"TRUE HARDNESS" OF SOME NON-FERROUS METALS

Peter BLAŠKO, Jozef PETRÍK, Branislav BUĽKO, Marek ŠOLC, Lenka GIRMANOVÁ, Martina HRUBOVČÁKOVÁ

TUKE - Technical University of Košice, Košice, Slovakia, EU, peter.blasko@tuke.sk

https://doi.org/10.37904/metal.2025.5158

Abstract

For finished products, it is sometimes necessary to measure hardness with minimal surface disturbance, where the indentations should not be visible to the naked eye. Applied loads that meet this requirement are below 0.9807 N for common metals. In this load range, the Vickers method is usually affected by the indentation size effect (ISE), which makes it difficult to interpret the measured values. If this effect is not taken into account, the measurement results can be misleading. For this reason, we recommend using the "true hardness", which is determined using the method presented. This article presents a method for calculating the "true hardness", which is not influenced by the test force when measuring the micro-hardness and essentially corresponds to the Vickers macro-hardness. Measurements were made using a Hanemann micro-hardness tester at loads in the range of 0.09807 N and 0.9807 N. The size and nature of the ISE and subsequently the "true hardness" were evaluated using Meyer's power law (index n), the Proportional Sample Resistance (PSR) and the Hays–Kendall method. The measurements were carried out on commonly used non-ferrous metals: Al, Mg, Cu, Ti, Ni, Co and W. A certain limitation of the method is that measurements must be taken on a metallographically polished surface, the quality of which influences the ISE parameters and consequently the "true hardness".

Keywords: Non-ferrous metals, load, micro-hardness, Indentation Size Effect (ISE), "true hardness"

1. INTRODUCTION

Sometimes it is necessary to determine the mechanical properties of the final product. Destructive testing is out of the question. A certain compromise is the hardness test, which can also provide secondary information also about other mechanical properties. The requirement is that the indentation should be as small as possible so as not to impair the aesthetic or functional aspects of the product. This requirement can be met by measuring the hardness using the Vickers method in the micro-hardness range (0.09807 N - 0.9807 N). The Vickers micro-hardness test is a widely used method for measuring the hardness of materials on a small scale.

The disadvantage of this method is the dependence of the measured micro-hardness value on various factors, such as the type of surface treatment leading to its roughness, the deformation strengthening, the quality of the indenter and, above all, the applied load. The indentation size effect (ISE) is a phenomenon in which the measured hardness value depends on the size of the indentation or the applied load while the size of the indentation is significantly influenced by the load. This can lead to uncertainties when determining the measured hardness of a material.

In terms of characteristics, ISE may not be manifest, it may be "normal" or "reverse". Studies have shown that ISE occurs because the relationship between the applied load and the indentation size does not always follow a simple law, such as Kick's law. ISE does not occur within the range of validity of Kick's law [1, 2].

"Normal" ISE is characterized by higher hardness values at lower loads. It is typical for brittle materials such as ceramics and glass, but also for heavily deformed metals. The most important factors that influencing "normal" ISE were identified in the work of Sangwal [3], Sangwal et al. [4], Gong et al. [5], Ren et al. [6], and Navrátil and Novotná [7]. These include the testing device (especially its part used to measure the diagonal

indentation and determine the applied load), the intrinsic properties of the tested sample (work hardening during indentation, the load to induce plastic deformation characterized by the parameter W, the elastic recovery of the identification and the elastic resistance of the materials), the technology of sample preparation (polishing). Other factors such as the friction between the indenter and the lubrication can also have a greater or lesser influence.

The "Reverse" ISE is characterized by lower hardness values at lower loads. It occurs in plastic materials, such as metals. Sangwal [3], who has studied this phenomenon, explains it as a result of a distorted zone near the interface between the effects of vibration and indenter bluntness, chipping of the tested material around the indenter, and the formation of cracks.

The concept of "true hardness" in micro-hardness testing refers to the hardness value that is independent of the Indentation Size Effect (ISE), mainly from the applied load. ISE is a phenomenon in which the measured hardness varies with the size of the indentation or applied load, often leading to discrepancies in determining the true hardness of a material. The models help isolate the "true hardness" value by accounting for the ISE. The Nix-Gao model, for example, is particularly effective in determining ISE-independent hardness by analyzing small-scale indentations [1, 8].

The relationship between the "true hardness" and the Vickers macro-hardness lies in their measurement scales and applications. The „true hardness" is an intrinsic property of a material, independent of factors such as the size of the indentation or the applied load. It represents the material's resistance to deformation at a fundamental level. Vickers macro-hardness, on the other hand, is a practical measurement that is carried out with a diamond-shaped indenter under a higher load (typically 1 kg – 100 kg). It provides a mass hardness value that reflects the overall mechanical properties of the material. While the Vickers macro-hardness can give an approximate indication of the "true hardness" for homogeneous materials, there may be variations due to factors such as grain size, surface roughness, or effects on the size of the indentation.

2. MATERIALS AND METHODS

The hardness of some metals as aluminium, magnesium, copper, titanium, cobalt, and tungsten with a purity of at least 99.5 wt% (technically pure) with the crystal system lattice FCC – face centred cubic (Al, Cu), BCC body centred cubic (W), and HCP – hexagonal close-packed (Mg, Ti, and Co) was analysed.

The samples were cut with a water-cooled diamond saw and sanded with water-cooled sandpapers in the order 80, 220, 240. … and 3000 ANSI/CAMI grit. The metallographic surface was mechanically polished with the water suspension of Al_2O_3 to a mirror finish and finally etched with a suitable etching agent (water solution 0.5 % HF for aluminium and titanium, 4 cm^3 HF + 100 cm^3 ethylalcohol for magnesium, 4 g $FeCl_2$ + 33 cm^3 HNO_3 + 1000 cm^3 H_2O for copper, 7.5 cm^3 HF + 2.5 cm^3 HNO_3 + 200 cm^3 H_2O for cobalt, and 33 cm^3 HF + 33 cm^3 HNO_3 + 34 cm^3 H_2O for tungsten and so on) to make hard intermetallic phases, grain boundaries, or discontinuities visible. Hardness was not measured in areas where these anomalies occurred.

The micro-hardness was measured by manual tester Hanemann, type Mod D32 fitted to microscope Neophot-32 with a magnification 480×. The discrimination of indentations measuring device is 0.000313 mm. Macro-hardness was measured on a HPO 250 tester. Before the measurement, both testers were calibrated using CRM (reference blocks), both meeting the requirements of the relevant standard ISO 6507-2 [9]. Calibrations as well as micro- and macro-hardness measurements (according to standard ISO 6507-1 [10]) were performed by one operator.

Applied loads P were 0.09807 N (10 g), 0.245175 N (25 g), 0.49035 N (50 g), and 0.9807 N (100 g) with five indentations at each load. The load duration (dwell) time was 15 seconds. The result was the “cluster” of 20 indentions for each sample. As for macro-hardness, a load of 98.07 N (10 kg) was used, at which five indentations were made. Measured values of micro-hardness are listened in **Table 1**.

Table 1 The average values of micro- and macrohardness and standard deviations (SD)

	HV0.01		HV0.025		HV0.05		HV0.1		HV10	
	average	SD	average	SD	average	SD	average	SD	average	SD
W	616	54.95	614	19.62	389	20.76	297	11.03	310	7.00
Ti	164	9.90	139	18.05	122	8.06	105	5.86	95	5.01
Co	427	44.50	371	11.78	340	6.55	293	6.07	223	5.62
Mg	47	2.27	45	1.94	52	4.33	45	1.52	42	4.11
Cu	83	2.52	74	1.80	68	1.35	65	1.00	62	4.45
Al	29	1.21	27	0.97	27	0.89	26	0.53	23	0.26

3. RESULTS

The methodology and procedures for determining the basic parameters that determine the character/type and size of the ISE calculation were based on work [8], also using works [3, 7, 11-13]. Meyer's power law, proportional sample resistance (PSR), and the Hays–Kendall approach were most commonly used to determine ISE characteristics.

As demonstrated by Ren et al. [6], Meyer's Power Law or Proportional Specimen Resistance model (PSR) describes ISE quantitatively.

Meyer's Law is easier way:

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

The parameters n and A are determined by exponential curve fitting to the indentation diagonal d (mm) the versus applied load P (N) or n and A_{ln} from the straight curve graph of ln (d) versus ln (P). The Meyer's index n, which is related to the "work-hardening index" is the slope, and the coefficient A_{ln} is the y-intercept of the line, as can be seen in **Figure 1** for cobalt. The same procedure is used for other metals analyzed. This relationship was derived for the spherical indenter but it has become common practice to use Tabor's interpretation of the strain-hardening by pyramidal indenter [13]. The index $n < 2$ for "normal" ISE, $n > 2$ for reverse ISE, and $n = 2$ for the micro-hardness given by Kick's Law. The values of n and A_{ln} are in **Table 2**.

Table 2 The values of ISE parameters: Meyer's index n, parameters related to elastic (a_1, c_1) and plastic properties (A, A_{ln}, a_2, c_2, A_1), parameter related to residual surface stress, parameter c_0 related to residual stress (a_1/a_2, c_1/c_2), and minimum load to initiate a visible indentation W

	n	A	A_{ln}	a_1	a_2	c_0	c_1	c_2	W	A_1	a_1/a_2	c_1/c_2
W	1.5352	283.0	5.6455	12.229	1118.1	-0.056	22.5200	763.7	0.086	1462.0	0.0109	0.0295
Ti	1.6807	204.2	5.3189	5.224	443.5	-0.054	10.4340	399.1	0.071	529.3	0.0118	0.0261
Co	1.7250	570.3	6.3462	7.135	1294.6	-0.057	16.7180	977.6	0.056	1481.4	0.0055	0.0171
Mg	1.9672	226.1	5.4211	1.038	226.6	-0.194	11.7020	97.6	0.040	229.5	0.0046	0.1199
Cu	1.8077	193.9	5.2671	2.016	307.1	-0.002	2.1997	304.2	0.030	335.2	0.0066	0.0072
Al	1.9280	228.6	5.4320	-0.006	310.2	0.087	-5.0850	379.0	0.007	310.7	0.0000	-0.0134

The PSR model of Li and Bradt (PSR) may be considered a modified form of the Hays/Kendall approach, described by equation (2) [5]:

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

Parameters a_1 (N.mm^{-1}) and a_2 (N.mm^{-2}) are related to the elastic and plastic properties of the material, respectively [4]. The parameter a_2 as state by Gong et al. [5] is related to load-independent "true hardness" (HPSR); it can be calculated by equation (3).

$$H_{PSRa2} = 0.1891 * a_2 \tag{3}$$

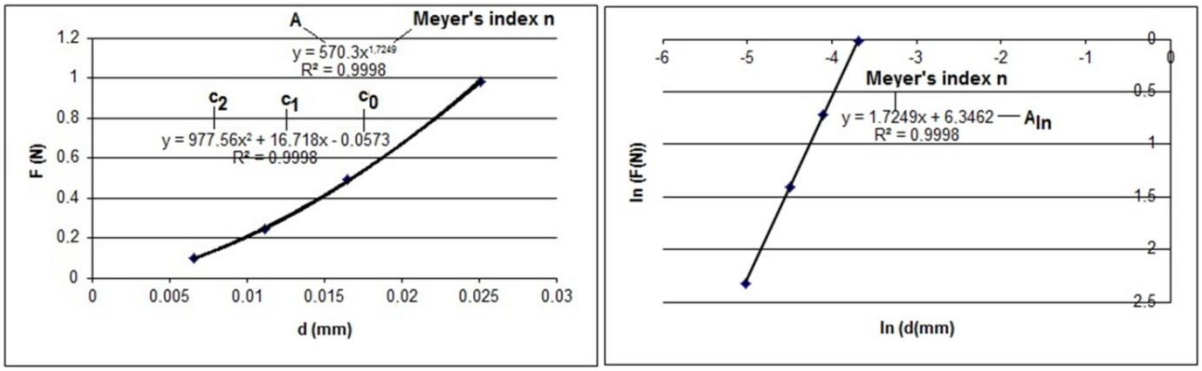

Figure 1 Determination of ISE parameters n, A, A_{ln}, c_0, c_1, and c_2, an example for cobalt

The parameters a_1 and a_2 of (2) may be obtained from the plots of P/d (N.mm^{-1}) against d (mm) as can be seen in **Figure 2**.

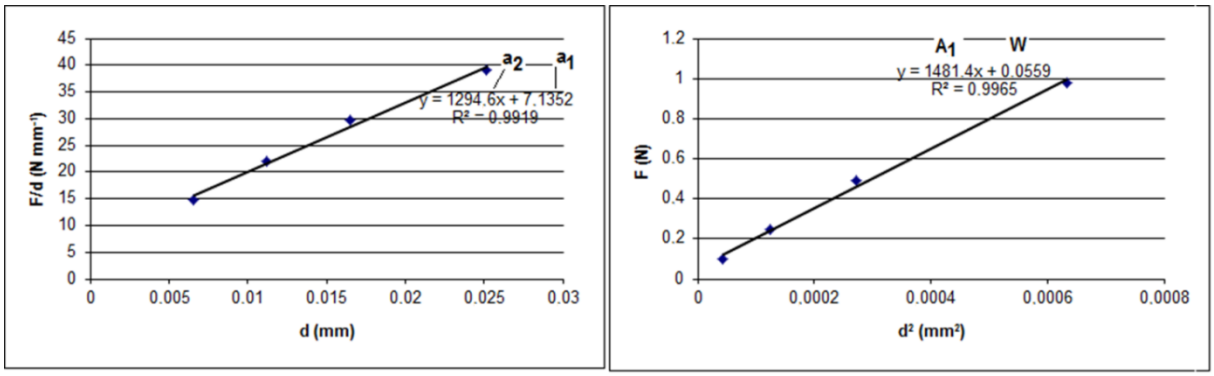

Figure 2 Determination of ISE parameters a_1, a_2, W, and A_1, an example for cobalt

Equation (4) can be regarded as a modified form of the PSR model.

$$P = c_0 + c_1 d + c_2 d^2 \tag{4}$$

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). The parameter c_0 is associated with residual surface stress 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. The ratio c_1/c_2 (or a_1/a_2) is a measure of the residual stress that arose during the manufacture of the [5].

Hays and Kendall proposed that W (N) is a minimum load to initiate a visible indentation. The relationship between applied load P and W is expressed by equation (5):

$$P = W + A_1 d^2 \tag{5}$$

where A_1 (N.mm^{-2}) is a parameter independent of load. As stated by Sangwal et al. [4] the values of W and A_1 may be obtained from the regressions of P (N) against d_2 (mm), **Figure 2**. The values of the parameters

obtained are in **Table 2**. The "true hardness" by analogy to a_2 can be calculated using A, c_2, and A_1 in equation (3). Calculated values of "true hardness" are in **Table 3**.

Table 3 Calculated values of "true hardness", comparison between of "true hardness" and micro-hardness HV0.05 and macro-hardness HV10 by paired t-test; p-values

	HPSR a_2	HPSR c_2	HPSR A_1	HPSR A	HV0.05	HV10
W	211	144	276	54	389	310
Ti	84	75	100	39	122	95
Co	245	185	280	108	340	223
Mg	43	18	43	43	52	42
Cu	58	58	63	37	68	62
Al	24	19	26	43	27	23
p	0.4278	0.1515	0.6638	0.1449	0.0877	-

Figure 3 shows a comparison of "true hardness" values calculated according to equation (3) using the listed parameters with a micro-hardness value of HV0.05 and a macro-hardness value of HV10.

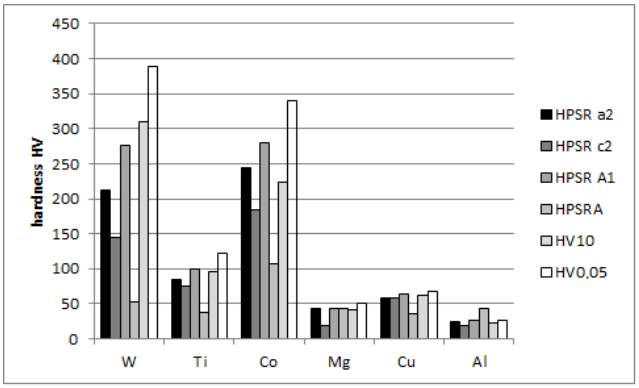

Figure 3 Comparison of "true hardness", micro-hardness HV0.05, and macro-hardness HV10

There is no clear trend in **Figure 3**. A certain exception is HPSR A, which is generally lower than HV10. On the other hand, the hardness HV0.05 is higher, which is due to the effect of "normal" ISE. At lower loads, this overestimation of micro-hardness is even more pronounced. Since the aim of the research was to replace the measurement of macro-hardness with micro-hardness in order to reduce the destruction of the sample as much as possible, was compared the values of true hardness and HV0.05 with the macro-hardness HV10 using a paired t-test (significance level $\alpha = 0.05$) [14], the p-values can be found in the last row of **Table 3**. According to conventional criteria, this difference between the "true hardness" calculated using the above methods is not considered statistically significant. The exception is the difference between the hardness HV0.05 and HV10, which we consider not quite statistically significant.

4. CONCLUSION

In the case to replace the measurement of macro-hardness with micro-hardness, it is appropriate to take the ISE into account. Its influence can be eliminated by calculating the "true hardness". This method requires a larger number of indentations and is therefore more time-consuming (5 standard indentations macro-hardness versus 20 standard indentations micro-hardness). A simplification is possible, theoretically one indentation for each selected load, while theoretically only 3 loads are possible, but with decreasing reliability. Determining the minimum number of micro-indentations and the minimum number of repetitions while maintaining

acceptable reliability could be the subject of further research. The paper is a pilot program; further research will be required to validate and verify the practicality of other metals as well as for the case of reverse ISE. In addition to the parameters that apply to the calculation of the “true hardness”, the equations presented also provide other parameters that, for example, provide information about the stresses in the samples.

ACKNOWLEDGEMENTS

This work was supported by the Slovak Grant Agency for Science VEGA 1/0199/24.

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