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THE REMOTE LABORATORY AND MICROHARDNESS MEASUREMENT

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ABSTRACT: The measurement of micro-hardness with loads between 0.09807 N and 0.9807 N has been carried out in direct mode (appraiser measured the dimensions of indentations with a measuring device fitted to the micro-hardness tester, a part of microscope) and in modified remote modes. The reference block - certified reference material with defined specified hardness and its uncertainty was used as a specimen, particular measurement involved indirect calibration of tester. The influence of applied load on the measured value of micro-hardness was evaluated by Meyer's index n and PSR method. The difference between values obtained by particular modes is statistically significant.
KEYWORDS: Hardness test, micro-indentation, metal, CRM, remote laboratory

INTRODUCTION

Measurement of micro-hardness is frequently used for determination of hardness of small items or thin layers, and identification of individual phases in metallography. It can be carried out in a similar manner to the Vickers macro-indentation tests with diamond pyramid, except for considerably smaller loads. However, the most important and intractable problem associated with low loads (the deep of indentation is less than 10 μm deep as a rule [1]), is that concerned with change in indentation size [2]. The micro-hardness of solids depends on the applied load. The study of relationship between micro-hardness and load has been carried out not only for metallic materials, but also for semiconductors, glass, slag, ceramics and organic crystals [3-5].

"NORMAL" AND "REVERSE" ISE

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 [6]. The ISE may be caused by the testing equipment. The experimental errors 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 in this group [6-8]. Another source of ISE is intrinsic properties of the tested material (work hardening during indentation, load to initiate plastic deformation, indentation elastic recovery, elastic resistance of the materials) [7-9]. The effect of machining-induced residually stressed surface (grinding, polishing) of specimen and indenter/ specimen friction are also explanations of the ISE [6, 8-10]. In contrast to "normal" ISE, a reverse type of ISE (inverse ISE, RISE), 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 [9].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 [7].

A perfect measurement would obtain the true value of a quantity. True values are, by nature, indeterminable because a perfect measurement cannot be performed. Difference between the true value and the value obtained by a measurement is the error of measurement. The final result is an estimate of the true value. The measurement uncertainty is a parameter that characterizes the dispersion of the values that could reasonably be attributed to the result of measurement. The uncertainty is inversely proportional to the quality of measurement [11, 12].

The purpose of this paper is to evaluate the influence of load on the values of micro-hardness using Meyer's and PSR methods, to evaluate the influence of load on the uncertainty of measured micro-hardness and to compare the results obtained in direct and remote modes.

MODE OF LABORATORY

In engineering education, a key-activity to improve the learning process is hands-on experimentation, carried out by simulation tools or laboratory facilities [13].

Laboratory studies provide experience, try-and-error type of learning, building connections among other concepts and previously learned subjects. However, for many educational organizations it is not always *possible to provide such an experience for their learners, since the establishment and maintenance cost especially for the fields that need high level equipment in such laboratories. In such cases, several educational organizations are using some technologies for supporting their students remotely or virtually [14].*

In a traditional proximal laboratory, the user interacts directly with the equipment by performing physical actions (e.g. manipulating manually, pressing buttons, turning knobs) and receiving sensory feedback [15].

Online laboratories (Labs) became a very useful support for practical aspects of teaching methods since the time their technical basis got available world wide. A lot of online Labs are available via the internet. They support for example live experiments during lectures, distance learning courses (students work at home) or ed-to-ed scenarios (students work at an institute and access an online Lab at another institute to save costs for expensive equipment). As mentioned in many papers, online Labs can open possibilities for an experimental approach to a wide audience and are independent of opening hours. We can distinguish them by location and experimental equipment, as can be seen in tab. 1 [16].

Virtual Lab allows students around the world to log into a computer equipped with the suitable interface circuits, such as data acquisition systems connected to various sensors or communication modules, and perform real-time experiments [17].

Table 1. Concepts of Labs [16]

		Access to Laboratory	
		Local	Remote
Experimental equipment	Virtual	Local simulation	Virtual laboratory
	Real	Traditional (proximal) laboratory	Remote laboratory

Remote Lab (remote mode) can be defined as a laboratory (apparatus, rigs) that can be accessed and controlled via the internet (actual laboratory experiments that are run remotely via a web interface) [15]. Most of such Labs require complicated hardware and software for communication with human and specially to create a feeling of remote presence [18]. Users of remote Lab are able to interact with the apparatus, from any computer connected to the internet through interface applications that allow them to manipulate the equipment, monitor the process and receive the results of the experiment for analysis. In many cases, remote Labs can be made available 24/7 with minimal requirements for maintenance or staff intervention [19]. Remote Labs are intended to complement the hands-on laboratory experience by providing access to a greater number of laboratories, and more opportunity to run and re-run experiments at a time and from a location that is convenient for the student. Remote Lab conception requires technical, pedagogical and computer science competencies. Due to these requirements, it appears to be more complex than other e-learning contexts such as on-line courses, virtual classrooms, e-projects, role-playing, etc. however, this kind of training is essential for scientific and technical disciplines and fits a real need [13]. Motivations for remote laboratories development are:

- *sharing heavy, highly specialized and expensive instruments and equipments between institutions,*
- *anytime and anywhere Lab access, the students can login and carry out experiments from any place of the world,*
- *putting students in front of real situations and allowing them: to discover system behavior, to train by using instruments, to verify scientific theories, etc.,*
- *remote Labs give students the opportunity to work in the remote mode,which will eventually become important in engineering jobs,*
- *unlike simulations remote Labs provide real experience,*
- *remote Labs improve safety and security,*
- *complex experimental systems, including specific media addition such as cooling, inert gas maintained by specialist staff at a specific location, can be directly controlled,*
- *team members, working at different locations can take advantage of the same test-run results without extra travelling,*
- *long-term trials (reliability, failure performance) can be comfortably supervised from home, e.g. on weekends [13, 20].*

In broader sense, the process of remote measurement contains also objective explanation of remote measurement - conceptual solution to wider understanding of measurement. Conceptual solutions depend on measurement possibilities, monitoring and control in a real Lab (the equipment side), on knowledge, informational, technical, personal, organization but also financial situation of individual joined participants. Following situations may be counted in:

- *Concept A: Measurement systems and processes can be monitored and/or controlled locally – requires a physical presence in the laboratory.*
- *Concept B: Measurement systems and processes can be monitored and/or controlled remotely (virtually) – based on thin www client with connection to selected www server. In some situations, there will be a possibility of live stream from Lab - by webcams or virtual simulation [18].*

In accordance with aforementioned definition presented model of remote Lab is not a full-value (more or less corresponds with concept A) model of a remote Lab. The equipment (micro-hardness tester) is not operated from the user's side and the appraiser operating the tester is required on the equipment side.

The remote Lab allows dividing an appraiser hand-operating tester from evaluating process in the described experiment. The evaluation can be carried out by students during the lessons. The direct contact between the tester and the student (unless is required by his/her field of study) without a required competence and experience is excluded (the risk of the defect of expensive and sensitive equipment).

The appraiser carries out the indentations according to operating instructions (number of trials, load, identification of indentations) in remote mode. He/she controls the place of indentation, the distance between them, the load duration time and the loading rate. He/she is not distracted by measuring of the dimensions of indentations and by recording of measured values.

The indentations are photographed one at a time or in groups (cluster or its part according to size, the indentations could be scanned on-line and transferred into PC in a remote Lab, if appropriate. The appraiser scans the gauge (calibration line standard) for calculation of actual magnification, calibration of the dimensions measuring part of software and for calculation of the uncertainty of the gauge calibration.

The user (appraiser or student) measures the dimensions of diagonals of indentations comfortably in a remote Lab and calculates the values of micro-hardness. The quality of the user can be evaluated by type of error (bias, random) using Youden plot, Z-score, Mandela's statistics, analysis of uncertainty or measurement systems analysis (MSA) [21].

EXPERIMENTAL MATERIALS AND PROCEDURES

The tester Hanemann (Mod D32) fitted to microscope Neophot-32 and reference block – certified reference material (CRM) for indirect calibration with specified hardness H_c = 242 HV0.05 and standard uncertainty u_{CRM} = 5.4 HV0.05 were used. The applying loads P were between 0.09807 N (10 g) and 0.9807 N (100 g) with a 0.09807 N step size. An appraiser performed five indentations at each load in a row.

The result was a cluster of 50 indentations in 10 rows. The load duration time was 15 seconds, the loading rate was 0.042 $N.s^{-1}$ and the ambient temperature was 19.2°C.

Table 2. The normality (p - value), average micro-hardness value of all 50 indentation HV, micro-hardness HV0.05, "true hardness" H_{PSRc2} and relative expanded uncertainty of calibration U_{rel} (%) for direct and remote mode

Mode	Normality (p)	HV	HV0.05	H_{PSRc2}	U_{rel} HV0.05
Direct	0.2788	258	258	184	13.0
Remote 1	0.4875	248	272	150	7.3
Remote 2	0.3407	242	266	151	19.4
Remote 3	0.0626	292	306	96	36.2

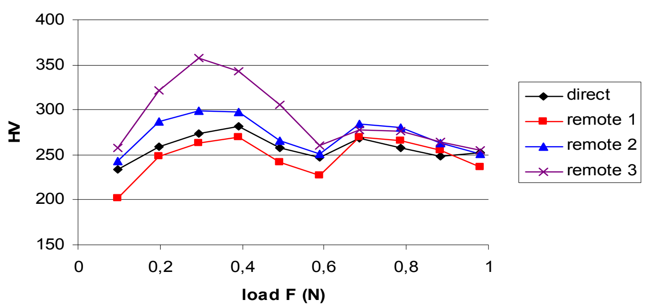

Figure 1. The values of micro-hardness

The length of two diagonals of the square-shaped Vickers indentation is measured by the appraiser immediately after each indentation with a calibrated micrometer attached to the eyepiece of microscope (magnification 480×) in direct mode. The cluster of indentations was thereafter photographed (scanned) for remote Lab (remote mode).

Computerized methods used software ImageJ in mode "remote 1" for measuring the indentation area and software TechDig. 1.1.b in mode "remote 2" for measuring of the diagonals. The diagonals on the hard copy of indentations were measured by slide caliper (scale division 0.01 mm) in mode "remote 3".

The normality (p – value), average micro-hardness value of all 50 indentation HV, micro-hardness HV0.05 and relative expanded uncertainty of calibration U_{rel} (in %, k = 2) for direct and remote mode are in tab. 1. The values of micro-hardness, measured at particular loads are in Fig 1 and the box-plots in Fig. 2. The results of calibration were used for calculation of relative expanded (k = 2) uncertainty U of the hardness values (in %), Fig. 3. The maximum value of U_{rel} is 10 % according to standard [22], only the mode "remote 1" match this requirement. The uncertainty depends on the mode and dencreases with increasing load. The ambiguity in the measurement of small indentation areas, particularly with pile-up or sink-in effects, results in over- or underestimation of the indentation area [23]. The remote mode simplifies the measurement of such indentations. The value of uncertainty obtained in remote mode is nevertheless higher in comparison with direct mode (except for "remote 1").

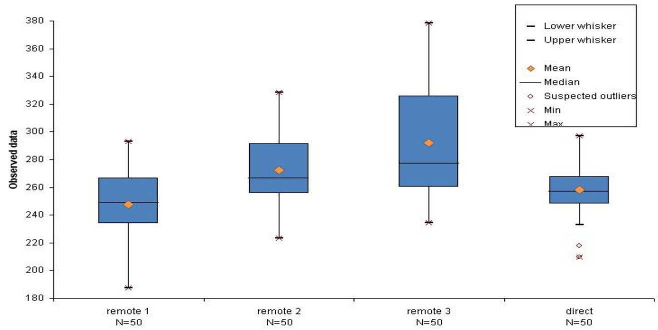

Figure 2. The box plot

It is necessary to remember that indirect calibration of micro-hardness testers is not routinely practiced process, unlike the (macro)hardness testers. Small dimensions of indentations diagonals and indentations with irregular shape are measured with difficulty. Small difference in reading of diagonals has a significant effect on the obtained (measured) value of micro-hardness and is a source of possible influence of appraiser's individuality and skill [24]. It is possible that high value of uncertainty of calibration is a result of low capability (high value of index %GRR, obtained by MSA analysis) [25].

Grubbs' test (significance level a = 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 particular clusters (50 indentations obtained in particular modes). As it can be seen in tab. 2, the normality was confirmed for all files. Absence of outliers suggests that the measurement process has avoided the gross errors.

According to the two factor ANOVA (Analysis of Variance) without replication the mode ($p = 2.79.10^{-6}$) and the load ($p = 1.06.10^{-5}$) have statistically *significant effect on the value of measured micro-hardness.*

The values of p of unpaired t-test, comparing the means of two groups (a = 95%) are in tab. 3. By conventional criteria, this difference is considered statistically significant between all modes. According to one sample t test the difference between the mean micro-hardness measured in particular modes (HV in tab. 1) and an expected value - standard hardness of CRM (H_c = 242 HV0.05) is statistically significant for all modes except for "remote 1".

Table 3. The values p of unpaired t-test.

	direct	remote 1	remote 2
remote 3	0.0001	0.0001	0.0032
remote 2	0.0009	0.0001	-
remote 1	0.0222	-	-

EVALUATION OF THE INFLUENCE OF THE LOAD ON THE MICRO- HARDNESS - Meyer's Power Law

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

$$P = Ad^n \quad (1)$$

The parameters n and A_{ln} are determined from a straight line graph of ln d (mm) versus ln P (N). Meyer's index n (work hardening coefficient) is the slope and A_{ln} is the y-intercept of the straight line, tab. 3.

When n=2, the micro-hardness is independent of the applied load and is given by Kick's Law. However, n<2 indicated "normal" ISE behavior, the measured micro-hardness decreases with applied load. When n>2, there is the reverse ISE behavior, measured micro-hardness increases with increasing of the load.

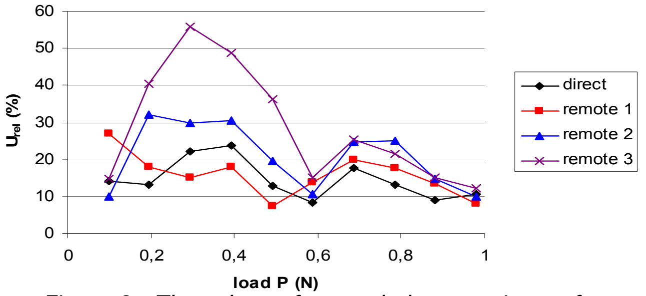

Figure 3. The values of expanded uncertainty u for particular loads and measurements

Table 3. The values of Meyer's index n and indices A_{ln}, c_0, c_1, c_2 for direct and remote mode.

mode	n	A_{ln}	c_0	c_1	c_2	c_1/c_2
direct	2.0209	7.3000	-0.9220	12.859	974.08	0.013
remote 1	2.1151	7.6304	-0.1620	19.435	792.50	0.025
remote 2	1.9846	7.2053	-0.1354	19.996	796.83	0.025
remote 3	1.8589	6.7591	-0.1645	28.576	506.83	0.056

The curves load vs. micro-hardness shows that the micro-hardness increases with load nonlinearly up tol about 0.5 N for the all modes and then remains practically constant (Fig. 1). The values of n, calculated in direct mode (n = 2.0209) and "remote 2" (n = 1.9846) practically confirm independence of the micro-hardness on load.

PSR (Proportional Specimen Resistance model) and modified PSR

Several authors [6, 8, 13] have proposed that ISE behavior may be described by the Eq. (2):

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

Gong et al. [2, 6] used an energy balance approach to examine the ISE and rearanged Eq. (2) into modified form of the PSR:

$$P = c_0 + c_1 d + c_2 d^2 \quad (3)$$

The values of constants c_0 (N), c_1 (N mm^{-1}) and c_2 (N mm^{-2}) of Eq. (3), obtained from the quadratic polynomial regressions of P/d (N mm^{-1}) against d (mm) are given in tab. 3. The parameter c_1 characterises the load dependence of micro-hardness (elastic properties). It consists of the elastic resistance of the test specimen and the friction resistance developed at the indenter facet/specimen interface [12, 13, 26].

The parameter c_2 may be a measure of the load-independent micro-hardness (plastic properties) sometimes referred to as "true hardness" H_{PSR} = 0.1891c_2. As can be seen in tab. 2, the "true hardness" is signifficantly less the measured hardness (HV 0.05 and HV) for all modes. The ratio c_1/c_2 may be treated approximately as a measure of the residual stresses due to machining and polishing [6, 27].

CONCLUSIONS

The differencies between direct and remote mode are statistically significant as far as measured values of micro-hardness but also uncertainty and indices, obtained by Meyer's and PSR methods. However, the differences are also beween particular remote modes. The influence of the load expressed by value of Meyer's index n was different for individual modes despite the identical specimen and equipment.

The relationship between differencies would be optimal, of course. It is possible, that observed difference is the result of raising uncertainty of measured values of the micro-hardness at low loads which partly obscures the occurrence and type of ISE.

Further research will be focused on preparation of factual and generalized knowledge about creative laboratory teaching at Higher Education Institution technical faculties especially in the field of Production Quality, especially: design, realization and work methods in Creative Laboratory Tuition at Technical Faculties (CRELABTE).

It is necessary to analyze the influence of repeatability, reproducibility, individual appraisers, methods of indentations diagonals measuring and other factors on the differences between modes at the same time. The results will be used for the improvement of analyzed system of Lab.

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