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APPLICATION OF SCANNIG ELECTRON MYCROSCOPE FOR THE ANALYSIS OF REFERENCE HARDNESS BLOCK SURFACE QUALITY

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Abstract – One of the most significant problems in realising hardness traceability are reference hardness blocks failures. The work included testing on one standard block prepared in compliance with the requirements defined in EN ISO 6507-3 standard. It was calibrated on the reference hardness testing machine. For the analysis of the hardness block surface and the indentation, a scanning electron microscope TESCAN, type: VEGA TSS136LS with EDS sensor was applied. Failures caused by the reference hardness blocks finishing and failures due to the material inhomogeneity were noted. Microanalysis of the chemical composition on typical spots was carried out with EDS sensor.

Key words: Vickers hardness, scanning electron microscope, reference hardness block

1. SCOPE

The reference hardness blocks are applied for indirect verification of the hardness testing machines and their quality has direct influence on the measurement uncertainty of the calibrated device.

Material inhomogeneity problems and occurrence of impurities cause variations in the measurement results and are already known according to the published papers. Also, during the investigations, the reference hardness block surface may get contaminated by impurities, oxides etc. The mentioned phenomena affect the results of hardness measurements and for that purpose the following investigations have been carried out:

- surface inspection with the scanning electron microscope,
- microanalysis of the chemical elements on the investigated surface and in the indentation.

Surface inspection was carried out by the scanning electron microscope TESCAN, type: VEGA TSS136LS which is located in the Laboratory for Metallography at the Faculty of Mechanical Engineering and Naval Architecture, Figure 1. The device is high-quality computer-controlled electron microscope designated for operations with variable vacuum.

Figure 1 - Scanning electron microscope TESCAN

Chemical microanalysis was carried out on the EDS (Energy Dispersive X-Ray Spectroscopy) sensor, manufactured by OXFORD INSTRUMENTS in the detection range of 20 keV.

2. EXPERIMENTAL INVESTIGATIONS

Experimental investigations were carried out on the standard block with hardness being approximately 800 HV10 and the basic data presented in Table 1.

Table 1. Basic data about the standard hardness block

Code:	IN60115G HV10	
Manufacturer:	INDENTEC, Great Britain	
Shape:	Type c	
Dimensions:	ext. diameter: 60,4 mm thickness: 11,7 mm	

The standard block was tested regarding flatness, roughness, and parallelism, whose results should comply with the requirements of EN ISO 6507-3 standard.

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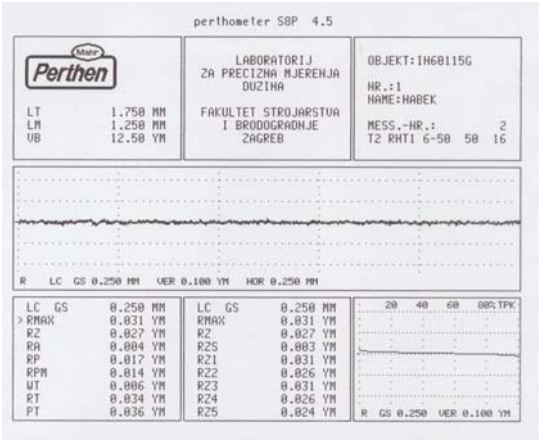
2.1 Testing the quality of block treatment

The deviation from flatness was measured by interferometer method using the interference microscope Carl Zeiss. Maximal deviation from flatness amounted to 0,15 μm, U = 0,06 μm (k=2, P=95 %) whereas the permitted deviation in compliance with the standard is 0,005 mm.

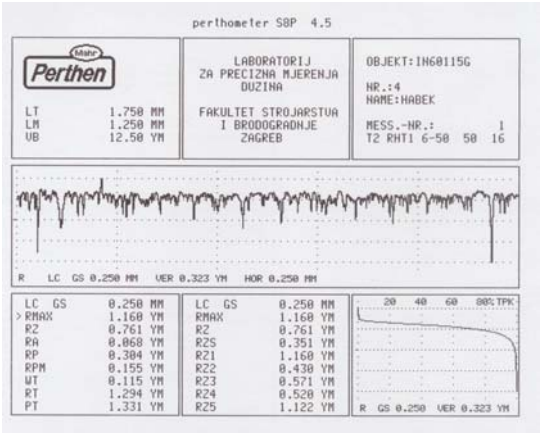
The permitted values of roughness on the measurement length of 0,80 mm are:

- test surface is $R_a \leq 0{,}05$ μm;
- contact surface is $R_a \leq 0{,}8$ μm.

Roughness tests were carried out on the electro-mechanical device for surface roughness testing designated: MU 2-127; manufacturer: Feinprüf; type: Perthometer S8P. Figure 2 shows diagrams of the results for one measuring point on the test and the contact surface.



a)



b)

Figure 2. Roughness testing
a) test surface,
b) contact surface of the block.

Maximal measured value of roughness amounted to:

- test surface: $R_a \leq 0{,}004$ μm,
- contact surface: $R_a \leq 0{,}103$ μm.

The permitted deviation from the parallelism of the contact and test surface should not exceed 0,010 mm per 50mm in compliance with EN ISO 6507-3 standard. The parallelism of standard block surfaces was tested on the comparison measurement device PGM; code: MU 41-363; manufacturer: MAHR; type: 826E; serial number: 08148. Maximal deviation from parallelism amounts to 0,59 μm; U = 0,4 μm (k=2, P=95 %).

The obtained results of testing flatness, roughness and parallelism for the standard block IN60115G may lead to the conclusion that the carried out surface treatment was of high quality and that the obtained parameters are far below the required limits.

2.2 Calibration

Calibration was carried out on the standard reference hardness testing machine 5030 TKV with measuring capability of ±1 % HV. The calibration was carried out in compliance with EN ISO 6507-3 standard, and the measurements plan is shown in Figure 3.

Figure 3. Measurement plan on the reference hardness block

Ambient conditions:

- temperature: 22 ºC ± 1 ºC,
- pressure: 1035 hPa.

Measurement conditions:

- load: 98,07 N,
- indentor drop speed: 1 mm/s,
- reading device resolution: 0,0001 mm,
- load duration: 8 s.

Table 2 presents the calibration results with the given expanded measurement uncertainty with k=2, P=95 %.

Table 2. Calibration results of reference hardness blocks IN60115G HV10

Ord.no. of measure ment	Measured value of diagonals			Calculated value of hardness, HV10
	d_1, mm	d_2, mm	d_{sr}, mm	
1	0.15151	0,15135	0,151430	808,70
2	0.15187	0,15139	0,151630	806,57
3	0.15142	0,15135	0,151385	809,18
4	0.15174	0,15152	0,151630	806,57
5	0.15131	0,15116	0,151235	810,79
		Mean value	0.151462	**808,36**
		Expanded measurement uncertainty		**8,29**

2.3 Testing by means of scanning electronic microscope

During surface investigation of the reference hardness block, serial number IN60115G, some failures have been noted, Fig. 4. Similar failures have been recorded on other reference hardness blocks [1]. Noted failures represent microcracks which are the result of production and finishing of the reference hardness blocks.

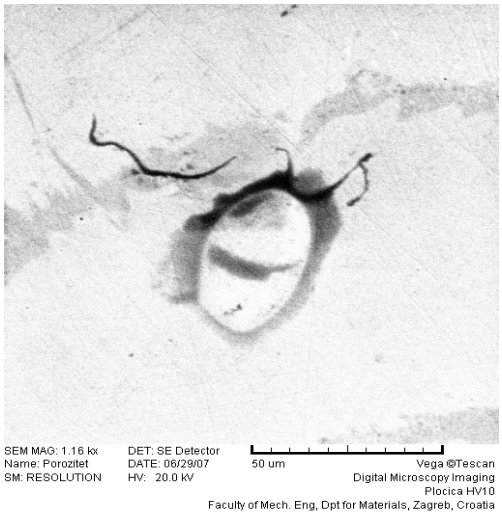


Figure 4. Failures on the investigated surface of the reference hardness block

Spots or areas on the reference hardness block surface which are visually different can be noted during scanning electron microscope analysis (Fig. 5).

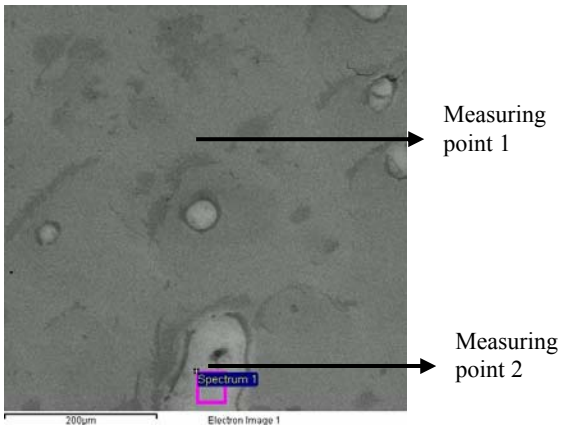


Figure 5. Visually different spots or areas on the reference hardness block

Different spots have not been noted on the entire block surface but only on some areas. The first assumption was that these spots are impurities as result of handling but after ultrasound cleaning in a bath the spots still remained. Thus, chemical microanalysis was carried out on one clean spot and on two inhomogeneity spots.

The comparison of the chemical elements spectrum inherented on spots with (Fig. 7) and without (Fig. 6) impurities showed some differences but without clear difference. This can have significant influence on the measuring results repeatability.

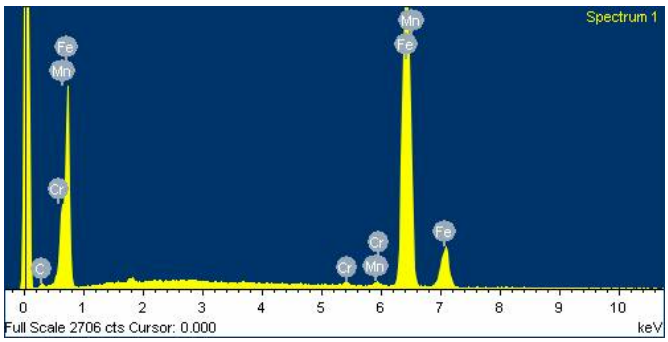


Figure 6. Chemical elements spectrum on the spot without inhomogeneity (measurement point 1)

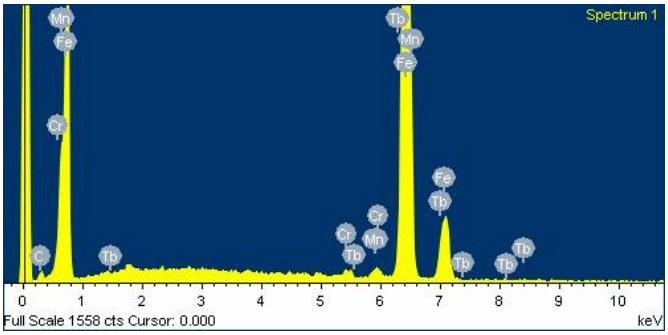


Figure 7. Chemical elements spectrum on the inhomogeneity spot (measuring point 2)

Besides, certain impurities have been noted (Fig. 8). The question is where these impurities come from.

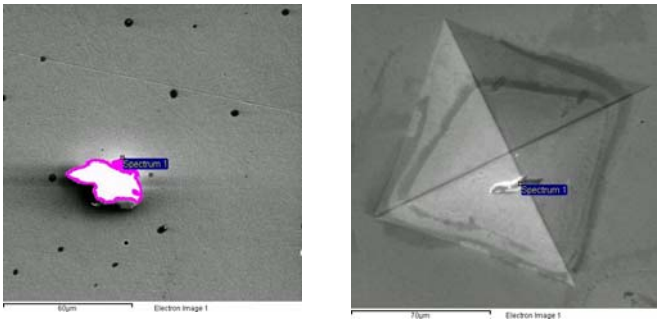


Figure 8. Impurities
a) on the reference hardness block surface and
b) in indentation

The impurities can be the result of indentation especially those in indentation itself, they can come from the environment or they are result from the indentor. If the impurities are from the environment they do not have significant influence on the measuring results [2] and [3] but for microhardness measurements they can be a problem. The impurities which are result of the material pasting on the indentor pages can have significant influence on the results

of the hardness measurements. The analysis of chemical composition of the impurities showed that they are the result of a reaction between the investigated surface and the environment (Fig. 9).

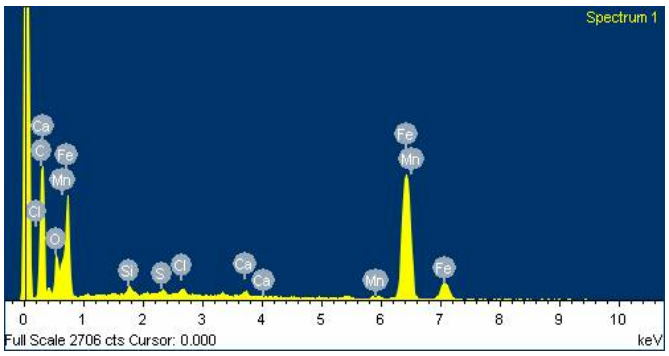


Figure 9: Chemical elements spectrum on impurity spot

3. CONCLUSION

The aim of these studies was to indicate the uncertainties that may result from the material and the manufacturing of the standard blocks as well as to present the possible application of the scanning electronic microscopy in studying such uncertainties. These preliminary research shows that the application of the scanning electronic microscopy may be of great assistance in the analysis of the cause of dispersion of certain hardness measurement results, i.e. it may tell us a lot about the quality of the standard hardness blocks. Therefore, the test of flatness, roughness and parallelism and some other tests defined by the standard are not sufficient in order to find out everything about the quality of the standard hardness block. The application of the scanning electronic microscopy enables the analysis of the quality of the entire test surface and the identification of defects resulting from the production and treatment, as well as failures caused by environmental impact. However, in order to analyse the condition of the entire test surface of the reference hardness block using this method, a longer time is required which may represent a certain problem.

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