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HARDNESS UNIFORMITY OF VICKERS HARDNESS BLOCKS FOR THE HIGH HARDNESS RANGE

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Abstract: Hardness uniformity is the most important property of hardness block and depends on the quality of the starting material, process of obtaining, heat and surface treatment. All parameters must be selected very carefully to achieve homogenous microstructure with no defects. Vickers hardness blocks for the high hardness range were produced by two different processes of powder metallurgy; conventional liquid phase sintering in hydrogen and sinter HIP. Nano powder of WC with a grain size of 150 nm with specific surface area of 2,5 m^2/g and addition of grain grow inhibitors was used as staring powder. Hardness uniformity of the produced blocks was investigated in this paper. Surface of the blocks was divided into radial and circumferential divisions forming different sections on the block surface. Hardness measurements were performed in each section with one level of the test force. Applied force was 9.807 N what corresponds to measuring method HV1. For reliable hardness measurement five indentations were measured in each section. Overall 40 indentations were performed based on which conclusions about the hardness uniformity of the Vickers hardness blocks were brought. Hardness uniformity was tested by analysis of variance, ANOVA, for single factor in to order to determine if significant hardness variations across the block surface are present. From carried research was concluded that hardness distribution on test surfaces of Vickers hardness blocks has a trend according to a process of obtaining.

Keywords: **Vickers hardness blocks, powder metallurgy process, hardness uniformity, surface division, ANOVA**

1. INTRODUCTION

Hardness blocks are an important and indispensable part of the measuring chain of traceability assurance for measurement quantity of hardness. Their quality directly affects the measurement uncertainty of hardness measurement. For this reason, the hardness blocks must cover the entire hardness range. Vickers hardness blocks are mainly produced for the hardness values up to 900 HV from steel heat treated depending of the desired hardness range. Development of materials with significantly improved mechanical properties requires a development of hardness blocks from that field. If the daily hardness tests are carried out in a certain hardness range it is advisable to check the adequacy of testing also in that range. Also, during calibration of hardness testing machine by indirect method is recommended to choose the hardness block corresponding to everyday hardness measurements [1].

The most important property of reference Vickers hardness blocks is a uniformity of hardness across the whole surface.[2] Material, method of obtaining and heat treatment must be selected carefully to achieve a homogeneous microstructure.[1] The place of measurement on the test surface should not affect the measured hardness values in case of optimal hardness blocks. However, so far has been found that the place of measurement can affect the hardness values. In order to reduce error from this factor during calibration of Vickers hardness blocks indentations on prescribed locations should be made. Five indentations covering the entire block surface for reliable measurement are required according to ISO 6507-3.[3] Location of indentations depends on a shape of a block and is also prescribed in ISO 6507-3.[4]

Uniformity of hardness, besides above mentioned factors, depends also on hardness machine on which the measurement is made. For that reason primary or reference hardness machines are used for calibration of Vickers hardness blocks.

For the purpose of traceability assurance for the hardness range around 2000 HV Vickers hardness blocks have been developed by two different processes of powder metallurgy; conventional liquid phase sintering in hydrogen and sinter HIP. Hardness uniformity of Vickers hardness blocks developed by different processes of obtaining is investigated in this paper.

2. EXPERIMENTAL

Vickers hardness blocks for the high hardness range were produced by two different processes of powder metallurgy; conventional liquid phase sintering in hydrogen and sinter HIP. Nano powder of WC with a grain size of 150 nm with specific surface area of 2,5 m^2/g and addition of grain grow inhibitors was used as staring powder. Preliminary samples of Vickers hardness blocks are presented in Figure 1. The dimensions of disk-shaped Vickers hardness blocks are 20 mm in diameter and 10 mm of thickness.

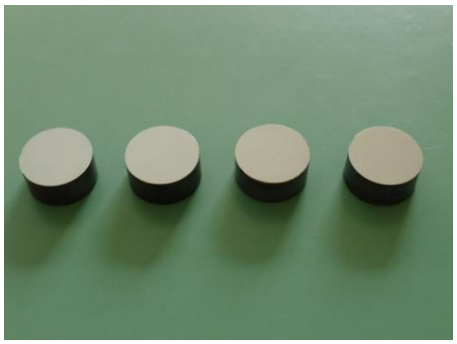


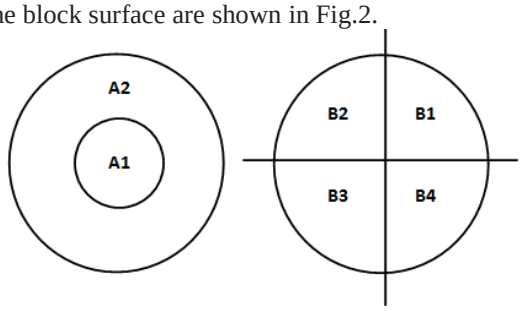
Figure 1: Preliminary Vickers hardness blocks

Two preliminary Vickers hardness blocks obtained by different powder metallurgy processes were chosen to test the uniformity of hardness across the whole surface. Characteristics of preliminary samples of Vickers hardness blocks are presented in table 1.

Table 1: Characteristics of Vickers hardness blocks

Vickers hardness block	**Process of obtaining**	**Grain grow inhibitors**	**Particle size, nm**	**Co content, %**
P28-1	sinter HIP	VC, Cr_2C_3	150	9
376-1	sintering in hydrogen	VC, Cr_2C_3	150	6

The test surface of the Vickers hardness blocks were divided into divisions for the purpose of hardness uniformity investigation. Two circumferential and four radial divisions of the block surface are shown in Fig.2.



Circumferential Division Radial division

Figure 2- Circumferential and radial divisions

Overall eight sections on the surface of Vickers hardness block have been formed (Fig. 3). For experiment such as that more than six up to 12 sections are recommended for hardness measurement[3]. The variation of hardness is rather stable for more than 12 sections[3].

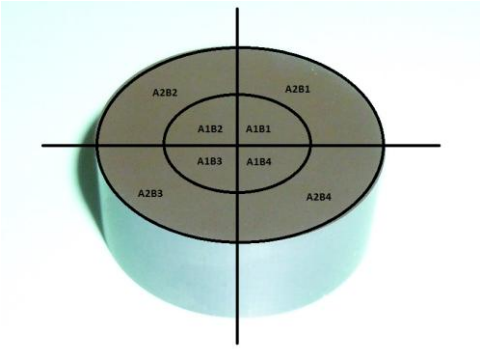


Figure 3 - Sections of Vickers hardness blocks

Hardness measurements were performed in each section with one level of the test force. Applied force was 9.807 N what corresponds to measuring method HV1. For reliable hardness measurement five indentations were measured in each section. Overall 40 indentations were performed based on which conclusions about the hardness uniformity of the Vickers hardness blocks were brought. Hardness measurements were carried out according to DIN EN ISO 6507-1:2005 on reference Vickers hardness machine installed in Laboratory for Testing Mechanical Properties at Faculty of Mechanical Engineering and Naval Architecture shown in Fig. 4.

Figure 4 - Reference Vickers hardness machine

In order to achieve more accurate and precise measurements of indentations, the diagonals were analyzed on opto-electric system installed in Laboratory for Precise Length Measurement at Faculty of Mechanical Engineering and Naval Architecture presented in Fig.5.

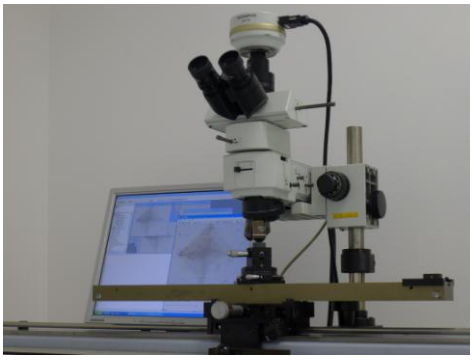


Figure 5 - Opto-electric system CCD-LFSB

Opto-electric system CCD-LFSB for calibration of precision measuring scales consists of uniaxial control system, an optical microscope with built-in high quality CCD camera with an increase of up to 200x and laser interferometer for measurement of a displacement achieved. The camera is connected to a computer software package Olympus DP-BSW manager that download and process images from the camera. The camera contains a CCD chip with a 145 million pixel progressive scan system.

For indentation analysis of Vickers hardness blocks software package was developed where the length of the diagonal of indentation is measured in pixels. The length of a single pixel, for a given increase, was calibrated with the use of 2D NPL standards and laser interferometer. Calibration was performed by measuring the dimensions of certain features (line thickness, width and spacing square mesh) at NPL 2D standard using a laser interferometer. The same measurement procedure (manual selection of start and end of features) later was used to measure the diagonals of indentations. On the captured images of Vickers indentations, using this software, the number of pixels in the features was determined.

Vickers indentation analyzed with opto-electric system CCD-LFSB is presented in Fig. 6.

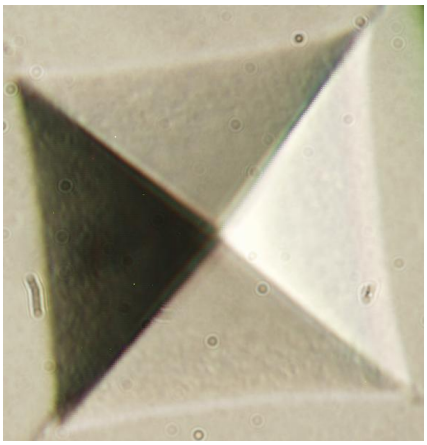


Figure 6 - Vickers indentation

On the basis of measured values of diagonals hardness values were calculated.

3. RESULTS

Results of calculated Vickers hardness for samples P28-1 and 376-1 are presented in tables 2 and 3. Hardness values were calculated according to equation: [5]

$$HV = 0{,}1891\frac{F}{d^2}$$

Where F is applied load in N, and d is average value of measured diagonals in mm.

Mean hardness values of each section are calculated according to equation:

$$\overline{X_j} = \frac{\sum_{i=1}^{n} X_{ij}}{n};\ j = 1,2.,3.k$$

Box plot of hardness values of each section with whiskers from minimum to maximum is shown in Fig. 7 for Vickers hardness block P28-1.

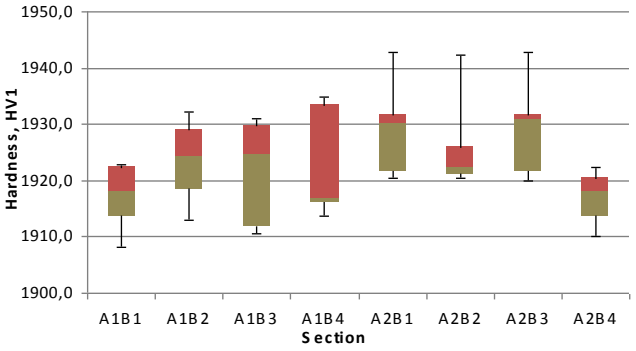


Figure 7- Box plot of hardness values of P28-1

Hardness variations in radial and circumferential division are not significant as can be seen from the box plot figure. Hardness values measured in each section are pretty much in the same range. Maximal mean hardness values amounts 1929,4 HV1 and was measured in section A2B1 and minimal amounts 1916,9 HV1 and was measured in section A2B4.

Box plot of hardness values of each section with whiskers from minimum to maximum for Vickers hardness block 376-1 is shown in Fig. 8.

Table 2- Measured hardness values of P28-1

Number of measurement	**SECTION**							
	A1B1	**A1B2**	**A1B3**	**A1B4**	**A2B1**	**A2B2**	**A2B3**	**A2B4**
1	1922,9	1932,3	1931,0	1916,1	1930,4	1942,3	1931,0	1922,3
2	1913,7	1924,2	1924,8	1916,8	1920,5	1921,1	1919,8	1913,7
3	1908,2	1918,6	1910,6	1934,8	1921,7	1926,0	1921,7	1910,0
4	1922,3	1913,1	1911,8	1913,7	1942,9	1920,5	1942,9	1920,5
5	1918,0	1929,1	1929,8	1933,5	1931,6	1922,3	1931,6	1918,0
$\overline{X_j}$	**1917,0**	**1923,4**	**1921,6**	**1923,0**	**1929,4**	**1926,4**	**1929,4**	**1916,9**

Table 3- Measured hardness values of 376-1

Number of measurement	SECTION							
	A1B1	**A1B2**	**A1B3**	**A1B4**	**A2B1**	**A2B2**	**A2B3**	**A2B4**
1	2131,0	2123,8	2128,8	2157,2	2167,6	2077,8	2118,8	2121,6
2	2122,4	2126,7	2133,9	2130,3	2133,2	2113,1	2136,1	2113,8
3	2175,8	2123,8	2145,5	2141,2	2126,0	2071,6	2117,3	2104,5
4	2136,8	2086,2	2129,6	2141,2	2137,5	2131,7	2141,9	2140,4
5	2176,5	2116,6	2136,1	2144,8	2131,0	2115,9	2114,5	2125,2
$\overline{X_j}$	**2148,5**	**2115,4**	**2134,8**	**2142,9**	**2139,0**	**2102,0**	**2125,7**	**2125,7**

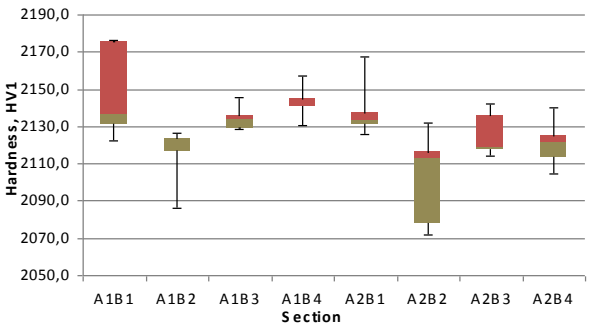


Figure 8- Box plot of hardness values of 376-1

Hardness variations in radial and circumferential division are significant as can be seen from the box plot figure. Maximal mean hardness value amounts 2148,5 HV1 and was measured in section A1B1 and minimal mean hardness value amounts 2102,0 HV1 and was measured in section A2B2. The difference between maximum and minimum value of measured hardness between sections is approximately 100 HV which is very significant hardness variation.

4. ANAYSIS OF RESULTS

Hardness uniformity of samples P28-1 and 376-1 was tested by analysis of variance, ANOVA, for single factor what is by far the most commonly-used technique for comparing means. Hypothesis H_0 states: arithmetic means of all eight sections are equal.

$$H_0: \mu_1 = \mu_2 = \mu_3 .... = \mu_8 = \mu$$

F-test was applied for testing the hypothesis H_0. The hypothesis is accepted if $F_{cal.} < F_0(\alpha=0{,}01)$. For specific degrees of freedom from the F-table the value F_0 is determined. Table value for 1% significance is 3.25 as can be seen from Fig. 9.

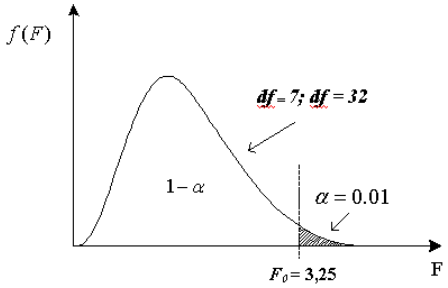


Figure 9 – F_0-value for significance 1%

Analysis of variance with specific calculated values for Vickers hardness block P28-1 is presented in table 4.

Table 4 – Analysis of variance for Vickers hardness block P28-1

Variance	Sum of squares	Degrees of freedom	Mean square	$F_{cal.}$	$F_0(\alpha=0{,}01)$
Between groups	840,8	7	120,1	1,67	3,25
Within groups	2300,0	32	71,9	-	-
Total	3140,8	40	-	-	-

Results from the table of variance show that the $F_{cal.}$ is smaller than the value of $F_0(\alpha=0{,}01)$ read from the table for F-test. F_{cal} is less than the theoretical value and the hypothesis H_0 is considered true. The premise that the arithmetic means of all eight sections are equal can be accepted. In other words from carried analysis of variance can be concluded that location of measurement on the surface of Vickers hardness block P28-1 doesn't influence on hardness values.

Analysis of variance with specific calculated values for sample 376-1 is presented in table 5.

Table 5 – Analysis of variance for Vickers hardness block 376-1

Variance	Sum of squares	Degrees of freedom	Mean square	$F_{cal.}$	$F_0(\alpha=0,01)$
Between groups	8459,8	7	1208,5	4,10	3,25
Within groups	9458,0	32	295,6	-	-
Total	17917,8	40	-	-	-

Results from the table of variance for Vickers hardness block 376-1 show that the $F_{cal.}$ is bigger than the value of $F_0(\alpha=0,01)$ read from the table for F-test. $F_{cal.}$ is bigger than the theoretical value and the hypothesis H_0 is not considered true. The arithmetic means of all eight sections are not equal. Location of measurement on the surface of Vickers block 376-1 influence on hardness values.

5. CONCLUSIONS

The purpose of the research was to investigate hardness uniformity of Vickers hardness blocks obtained by different metallurgy processes. For that purpose the surface of the blocks was divided into eight different sections in which five indentations were applied. From carried research is concluded that hardness distribution on test surfaces of Vickers hardness blocks has a trend according to process of obtaining. Each Vickers hardness block characterise different hardness uniformity in radial and circumferential divisions. Analysis of variance showed that process of obtaining influence on hardness uniformity across the block surface. The arithmetic means of hardness values measured on the Vickers hardness block produced by sinter HIP process are equal in all eight sections. The arithmetic means of hardness values measured on the Vickers hardness block produced by sintering in hydrogen atmosphere are not equal in all eight sections and the variation of hardness values are significant. Variations of hardness of Vickers hardness block developed by process of sintering in hydrogen are probably resulting from microstructure inhomogeneity and imperfections such as grain growth. From carried research can be concluded that for further development of Vickers hardness block sinter HIP process would be used. Sinter HIP eliminates the shortcomings of classical powder metallurgy process and achieves the superior properties of WC-Co hard metals compared to other procedure of consolidation. Further investigation of hardness uniformity on more samples of Vickers hardness blocks would be carried out.

5. REFERENCES

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[5] ISO 6507-3:2005 – Metallic materials - Vickers hardness test - Part 1: Test method