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DEVELOPEMENT OF REFERENCE VICKERS HARDNESS BLOCKS BY THE POWDER METALLURGY PROCESS

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Abstract: Development of reference hardness blocks for the high hardness range by the powder metallurgy process will be presented in this paper. Development would start with the selection of powder, matrix and grain growth inhibitors as well as parameters related to the technological process of obtaining which are related to milling, compacting and sintering. Mentioned would be realized in order to ensure the measurement traceability of the Vickers hardness for the Republic of Croatia for the high hardness range and to contribute to the spectrum of materials and processes of manufacturing reference blocks for the Vickers hardness on the global level.

Keywords: *reference hardness blocks, Vickers hardness, powder metallurgy, nano-powders*

1. INTRODUCTION

Reference hardness blocks are used for the indirect method of calibrations of hardness testers and to verify the adequacy of hardness test. The role of reference hardness blocks as reference materials is getting more and more important due to traceability assurance of calibration and testing laboratories. [1] There are three basic requirements placed on the reference hardness blocks; homogeneous microstructure that means uniform hardness over the entire test surface, stability of hardness over the time and reliability of hardness standard values. [2]

Development of new materials with significantly improved mechanical properties; higher hardness, higher compressive and flexural strength, higher modulus of elasticity, better wear resistance, improved chemical resistance, etc. requires the development of reference hardness blocks from this area. If the daily tests on the hardness tester are performed in a specific hardness range than is advisable to check the adequacy of testing and to calibrate hardness tester with the reference hardness blocks in that hardness range.[3] Conventional Knoop or Vickers hardness test methods are most commonly used to quantify hardness of ceramics and hard metals.[4] Reference hardness blocks for the high hardness range so far been developed by powder metallurgy process, more precisely, by the process of hot isostatic pressing that achieves theoretical density and eliminates porosity as one of the critical sizes of the conventional methods of powder metallurgy. On that way is achieved homogeneous microstructure as a basic requirement that is placed on the reference hardness blocks.

The process of development of reference Vickers hardness block for the high hardness range by the conventional sintering process from newly developed nano powders is described in the paper. The development of the reference hardness blocks is still in the process and is planned to be realised within the year.

2. STARTING POWDERS AND TECHNOLOGICAL PROCESS OF OBTAINING

Mechanical properties and microstructure of the sintered material depend on a variety of factors that could be categorized into two groups: those that relate to the characteristics of the applied powders (origin, method of obtaining, purity and appropriateness of carbide and binder, chemical composition, geometry and grain size) and those related to the technological process of obtaining (the characteristics of milling, compacting and sintering). Consequently the selection of starting powders, carbide and binder phase, is the first and very important step in the process of developing reference Vickers hardness blocks. After a detailed analysis of all the demands that are placed on the same for starting materials were selected newly developed nano-powders of tungsten carbide with a grain size in the range 95-150 nm and specific surface area of 2.5 to 4.0 m^2/g while for the binder material is selected half micron cobalt powder. One of the biggest potential problems of the powder metallurgy method is retaining small grain size of milled powders in the sintered product [5] and therefore are selected powders with the addition of grain growth inhibitors. Characteristics of starting powders are presented in the table 1.

Table 1: Characteristics of starting powders

Powder	Grain size, nm	Grain grow inhibibors %
WC DN 2-5	150	0,2-0,3 % VC 0,4-0,5 % Cr_2C_3
WC DN 2-5	150	0,2-0,3% VC
WC DN 4-0	95	0,4-0,5 %VC, 0,8 % Cr_2C_3

Characteristics of applied nano powders are improved mechanical properties such as hardness and toughness as well as homogenous microstructure which is first and the most important requirement placed on the reference hardness blocks. Starting powders analysed on the field emission scanning electron microscope are presented in figures 1, 2 and 3.

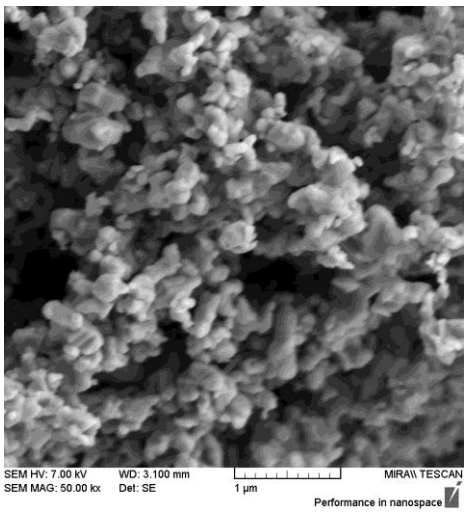

Figure 1 –WC D-N 2-5 with addition of VC and Cr_3C_2

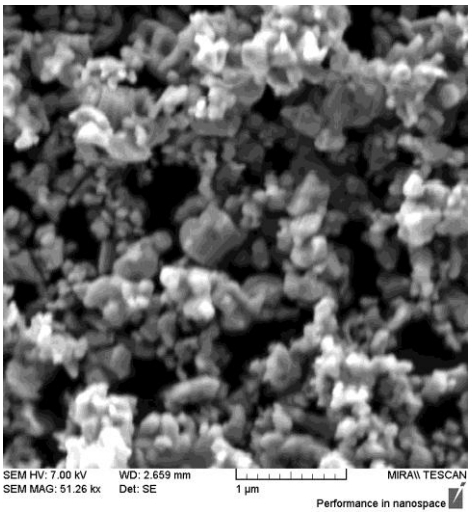

Figure 2 –WC D-N 2-5 with addition of VC

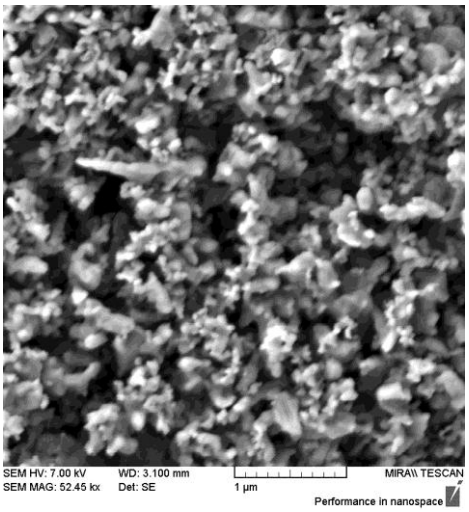

Figure 3 –WC D-N 4-0 with addition of VC and Cr_3C_2

From the starting powders different batches have been made varying the cobalt content. From every batch samples of materials from which can potentially be developed reference Vickers hardness blocks were made. Batches characteristics are presented in table 2.

Table 2: Batches characteristics

Batch	Starting powder	Grain grow inhibitors	Grain size, nm	Co content, %
DN 2-5 CRV-9/366	WC-DN-2-5	VC, Cr_2C_3	150	9
DN 2-5 V-9/369	WC-DN-2-5	VC	150	9
DN 2-5 CRV-6/376	WC-DN-2-5	VC, Cr_2C_3	150	6
DN 4-0 CRV-4/377	WC-DN-4-0	VC, Cr_2C_3	95	6

Formation of the batches is a result of the first operation in technological process of obtaining; milling. Parameters of milling such as milling speed and milling time are very important variables that should be taken into account when determining the first phase of the technological process of obtaining and impact on the microstructure of the final product. Milling time will depend on the type of mill, milling intensity, relative size of the ball and powders and temperature of the system. [6]

Technological process of obtaining reference Vickers hardness blocks, more precisely powder metallurgy process, consists of number of unbreakable operation as presented in figure 4. Each operation is very complex, dependent of many factors that can affect on the satisfaction of basic requirements; uniform hardness, hardness stability and reliability of hardness.

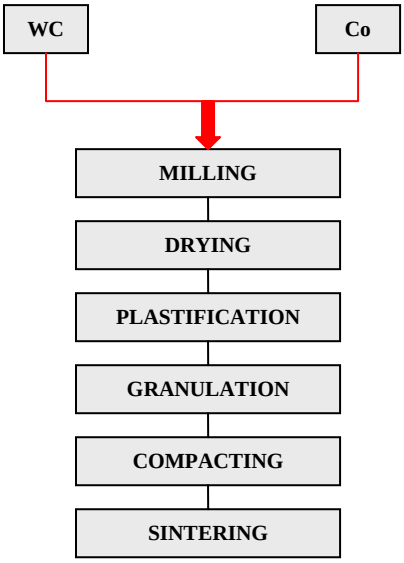

Figure 4 –Technological process of obtaining

3. DEVELOPED SAMPLES OF MATERIALS

Samples of nanostructured hard metals were developed as described previously. Dimensions of samples are 5x6x20 and are presented in figure 5.

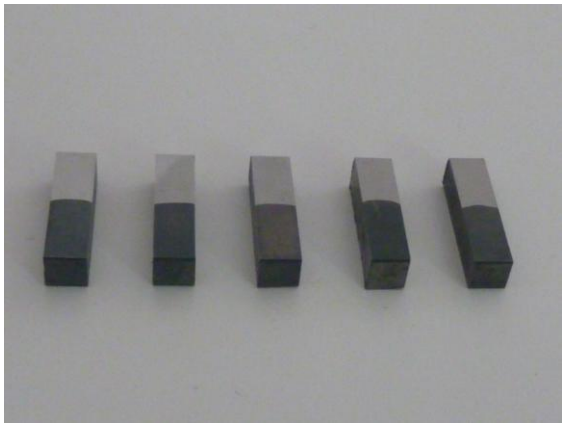

Figure 5: Samples of developed material

Preliminary samples were made in order to analyse microstructure, porosity and uncombined carbon. Porosity and uncombined carbon have been tested on the polished surface of the developed sample according to EN ISO 4505. Porosity and uncombined carbon have not been detected on the polished surface of samples. According to photomicrographs the level of porosity can be defined as A00 and uncombined carbon as C00. Microstructure of developed samples obtained by sintering on different temperatures was analyzed on the optical and scanning electron microscope according to EN ISO 4499-2. Some of the microstructures analysed after etching procedure in the freshly prepared Murakami reagent on the scanning electron microscope are presented in figures 6 and 7.

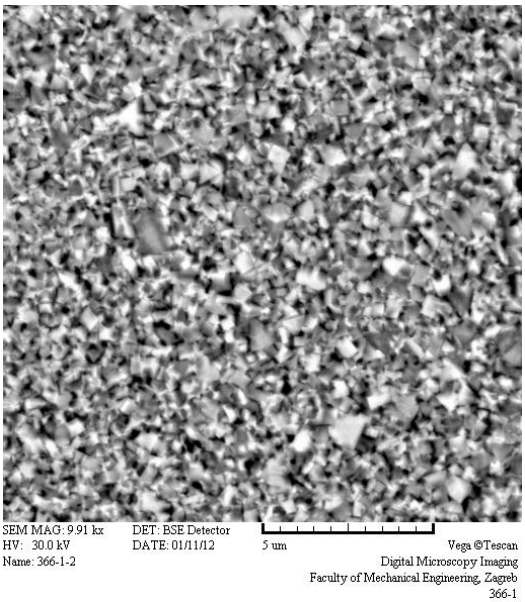

Figure 6 **- Microstructure of the sample 366-1, sintering temperature 1400°C**

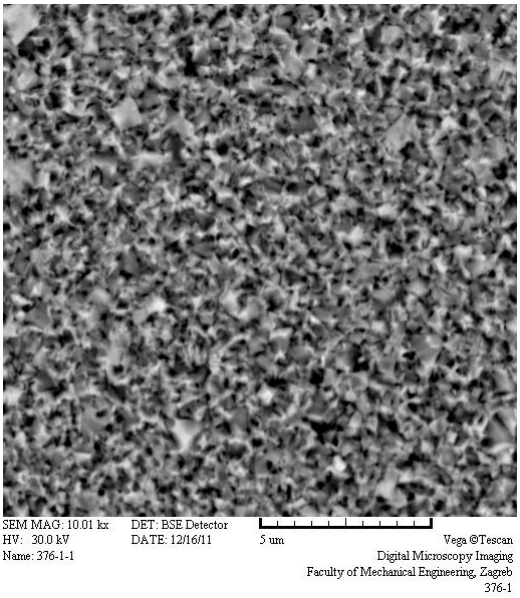

Figure 7 **- Microstructure of the sample 376-1, sintering temperature 1420°C**

Image analysis is used for the microstructure evaluation. Only tungsten carbide and cobalt binder were detected. Microstructure is homogenous over the whole surface of samples without abnormal grain growth that implies that the parametars of technological process of obtaining such as milling parametars, sintering temperature, etc. are properly selected.

4. HARDNESS MEASUREMENTS

Preliminary hardness measurements on the developed samples were also carried out in order to test hardness uniformity. On every sample five indentations on different positions over the entire surface have been made. Measurements of hardness were carried out in the Laboratory for Testing Mechanical Properties at the Faculty of Mechanical Engineering and Naval Architecture on the reference hardness tester which fulfils the general requirements specified in ISO 6507-3 and is presented in the figure 8.

Figure 8 - Reference hardness tester

Reliability of hardness values is assured by fulfilment of requirements according to EN ISO 6507-1 related to the applied load, loading velocity and duration of load application during the measurement procedure. The results of hardness measurement are presented in the table 3.

Table 3: Measured values of hardness

Sample	Measured diagonal	Average value of diagonal, $\overline{d}$, mm	Measured hardness value, HV 20	Non - uniformity U_{rel}, %
366 - 1	0,1759	**0,17608**	1797,9	**0,34**
	0,1761		1793,9	
	0,1764		1787,7	
	0,1758		1800,0	
	0,1762		1791,8	
369 - 1	0,1791	**0,17878**	1757,7	**0,84**
	0,1779		1734,3	
	0,1782		1751,0	
	0,1794		1728,5	
	0,1793		1730,4	
376 - 1	0,1674	**0,16774**	1985,2	**0,42**
	0,168		1971	
	0,1681		1968,7	
	0,1675		1982,8	
	0,1677		1978,1	
377 - 1	0,1657	**0,16542**	2026,1	**0,42**
	0,1650		2043,3	
	0,1652		2038,4	
	0,1655		2031,0	
	0,1657		2026,1	

Maximum permissible value of non-uniformity U_{rel} for the hardness values bigger than 225 HV and for the applied load in the range from HV 5 to HV 100 is 1 %.[7] As can be seen from the table the uniformity of hardness of developed samples satisfies the requirements according to EN ISO 6507-3. Non uniformity of hardness is the biggest for the sample 369-1 and amounts 0,84% what is still less than required value. Non-uniformity of hardness of other samples is smaller than 0,5% what indicates that the microstructure is homogenous.

5. CONCLUSION

The development process of the reference Vickers hardness blocks provided good first results. From the analysis of microstructure and preliminary hardness measurements can be concluded that the developed samples of materials satisfy some of the requirements placed on the reference hardness blocks. With the application of newly developed nano-powders with addition of grain grow inhibitors and described technological process of obtaining it is possible to achieve homogenous microstructure and to retain small grain size of the powder in the sintered product.
On that way two goals can be achieved:

- Development of the material used for the first time for the production of the reference Vickers hardness blocks whose metrological characteristics shall be equivalent to the

existing one. On that way contribute to a range of materials and procedure of obtaining on a global level

- Improvement of the traceability for the high hardness range

6. ACKNOWLEDGEMENTS

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7. REFERENCES

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