Measurement 78 (2016) 358–365

Contents lists available at ScienceDirect

Measurement

journal homepage: www.elsevier.com/locate/measurement

Influential factors on hardness uniformity of Vickers hardness blocks for high hardness range

Tamara Aleksandrov Fabijanić *, Mladen Franz, Željko Alar

Laboratory for Testing Mechanical Properties, Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb, Croatia

ARTICLE INFO

Article history:
Received 4 November 2014
Received in revised form 28 June 2015
Accepted 13 July 2015
Available online 28 July 2015

Keywords:
Vickers hardness block
Sinter-HIP
Sintering in hydrogen
Hardness uniformity
Surface section
ANOVA

ABSTRACT

Reference Vickers hardness blocks for high hardness range were developed from WC 9–Co and WC 6–Co mixtures by two different processes of powder metallurgy, conventional liquid phase sintering in hydrogen and vacuum-sintering with subsequent hot isostatic pressing in one cycle, sinter-HIP, in order to analyse the appropriateness of the manufacturing process and the influence of starting characteristics of the mixtures on hardness uniformity and overall metrological characteristics. WC powder of 150 nm grain size and 2.5 m^2/g specific surface area with the addition of grain grow inhibitors was used as starting material. The emphasis of the research was placed on hardness uniformity of the test surface as it is the most important property placed on hardness blocks. For that purpose the 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 HV1 measuring method. A total of 40 indentations were performed, based on which the conclusions about hardness uniformity of Vickers hardness blocks were drawn. Hardness measurements and uniformity were tested by analysis of variance, ANOVA, for single factor in order to determine if significant hardness variations across the block surface were present. From the research conducted it was concluded that hardness distribution across test surfaces of Vickers hardness blocks had a trend according to a process of manufacturing and the amount of binder in the WC–Co mixture. The best material and metrological characteristics were obtained on the block manufactured by sinter-HIP with 9 wt.% Co.

1. Introduction

Hardness blocks are an indispensable part of the measuring traceability chain for the measurement quantity of hardness. Their quality directly affects measurement uncertainty of hardness measurement. For this reason, reference hardness blocks must cover the entire hardness range. Vickers hardness blocks are mainly produced for hardness values up to 900 HV from steel heat treated depending on the desired hardness range. Due to the development of new, advanced hard materials with significantly improved mechanical properties it is required to develop new reference hardness blocks with similar properties. If daily hardness tests are carried out in certain hardness ranges, it is advisable to check the adequacy of testing in that specific ranges as well [1,2]. Also, during calibration of a hardness testing machine by an indirect method it is recommended to choose a hardness block corresponding to everyday hardness measurements [2,3]. Reference Vickers hardness blocks for high hardness ranges are produced up to hardness values of approximately 1500–1600 HV. Hardness measurement in high hardness range poses many difficulties and problems. The most common ones are the cracking and chipping of material on the corners of indentation as a consequence of

* Corresponding author.
E-mail address: tamara.aleksandrov@fsb.hr (T. Aleksandrov Fabijanić).

http://dx.doi.org/10.1016/j.measurement.2015.07.030
0263-2241/

applied load [4,5]. For that reason, the requirement placed on blocks is that indentations should be free from cracks, which may influence hardness measurement and lead to higher accuracy errors and to the scattering of measured values. Furthermore, indentation size effect, impact or vibration and problems with length measurement due to very small indentation size may also occur. In hard materials obtained by powder metallurgy processes, like ceramics and hardmetals, indentations found after applying certain loads are mostly not the result of plastic deformation, but the result of material densification, displacement and fracture toughness [4]. Large WC grains can cause slight irregularities such as rounded tip, cusped tip, hairline tip or bent tip [4]. The combination of all these factors will result in indentation imperfections and problems during measurement.

The most important property of blocks is hardness uniformity across the whole test surface [6]. It depends mainly on the quality of the starting material, physical properties of the material used, microstructure and production process, as well as any post-heat and surface treatments. All influential factors must be selected very carefully in order to achieve a homogeneous microstructure [1]. Uniformity of hardness, together with the above-mentioned factors, also depends on measuring equipment and measuring conditions in which measurement is performed. For that reason, primary or reference hardness machines are used for the calibration of Vickers hardness blocks. The place of measurement can also affect measured hardness values. In order to reduce error from this factor during the calibration of Vickers hardness blocks indentations on prescribed locations should be made. Five indentations covering the entire block surface are required for reliable measurement according to ISO 6507-3 [6]. The location of indentations depends on the shape of the block and is also prescribed in ISO 6507-3 [7].

The development of reference Vickers hardness blocks was initiated in order to ensure measurement traceability for hardness values of approximately 2000 HV, as well as to enlarge the spectrum of materials and processes of manufacture. Different processes of powder metallurgy and different starting compositions were employed in order to investigate the appropriateness of the manufacturing process and the influence of starting characteristics of mixtures on hardness uniformity and overall metrological characteristics.

2. Materials and methods

2.1. Manufacturing processes and starting characteristics

Vickers hardness blocks were developed from WC 9–Co and WC 6–Co mixtures by two different processes of powder metallurgy; conventional liquid phase sintering in hydrogen and sintering in vacuum and hot isostatic pressing in one cycle, sinter-HIP. As starting material, a newly developed tungsten carbide nanopowder WC DN 2-5, produced by HC Stark (Germany), was used, with an average grain size of d_{BET} = 150 nm and a specific surface area (BET) of 2.5 m^2/g. The newly developed nanopowder was selected as starting material in order to develop a preliminary Vickers hardness block from fully dense, nanostructured WC–Co hardmetal with a very homogeneous and uniform microstructure and good fracture toughness whose microstructural characteristics would influence the reliability of measurements and improve metrological characteristics; measurement uncertainty of hardness measurement. As binder, HMP Co powder produced by Umicore (Canada) with a grain size d_{BET} of 210 nm was selected. Two different mixtures were prepared for the purpose of this research. Manufacturing processes consisted of several separate technological operations, such as milling, drying, waxing, granulating, dewaxing, compacting and sintering. Preliminary Vickers hardness blocks, approximately 25 mm in diameter and 10 mm in height, were consolidated. After consolidation by different powder metallurgy processes the blocks were finally finished to maintain the requirements according to EN ISO 6507-3:2005, concerning flatness, parallelism and surface roughness. Four preliminary Vickers hardness blocks selected for further analysis are presented in Table 1.

Two blocks manufactured by vacuum-sintering and hot isostatic pressing in one cycle, sinter-HIP, with different amounts of Co content in the starting mixture, WC 9–Co and WC 6–Co, were selected. In order to carry out a reliable comparison, two blocks produced by conventional sintering in hydrogen with equal starting mixture characteristics as sinter-HIP-ed blocks were selected. Preliminary Vickers hardness blocks after final finishing are presented in Fig. 1.

After sintering, the characterisation of porosity and microstructure was performed. The degree of porosity and uncombined carbon was determined by comparing the polished surface with photo micrographs from the standard (ISO 4505:1978). Microstructure analysis was extremely important. Homogeneous microstructure is the precondition for hardness uniformity throughout the test surface as it is the main feature of newly developed blocks. The analysis was performed using an optical microscope and a field emission scanning electron microscope, FESEM. The characterisation of the microstructure was based on various reactions of Murakami solution with different phases of the microstructure. The samples were etched in several steps in order to make the different microstructural constituents visible. The first etching of the polished surface was performed in Murakami solution for a period of 2–3 s for the detection of η-phase.

Table 1
Characteristics of Vickers hardness blocks.

Hardness block	Process of obtaining	Grain grow inhibitors	d_{BET}, nm	(BET), m^2/g	Co,% wt.
SH 1	Sinter-HIP	0.26%VC, 0.45% Cr_3C_2	150	2.57	9
SH 2		0.26%VC, 0.45% Cr_3C_2	150	2.57	6
SV 1	Sintering in hydrogen	0.26%VC, 0.45% Cr_3C_2	150	2.57	9
SV 2		0.26%VC, 0.45% Cr_3C_2	150	2.57	6