Evaluation of Steels Used for Hardness Standard Blocks Production

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Resumo: Os blocos-padrão de dureza são os padrões de referência responsáveis pela manutenção da rastreabilidade da grandeza dureza. Foram avaliadas não-uniformidade, composição química superficial e textura cristalográfica de três blocos de dureza Rockwell C, sendo esta a escala de dureza mais utilizada na indústria. Os resultados mostraram que os fabricantes podem utilizar materiais e rotas de fabricação diferentes para produzir blocos-padrão para mesma faixa e escala de dureza.

Palavras-chave: textura cristalográfica, dureza Rockwell C, Não-uniformidade, composição química superficial.

Abstract: The hardness standard blocks are the reference standards responsible for maintaining the traceability of the quantity hardness. Non-uniformity, surface chemical composition and crystallographic texture of three blocks of the most used hardness scale in industry e.g. Rockwell C were evaluated. The results showed that manufacturers can use different materials and manufacturing routes to produce standard blocks for the same range and hardness scale.

Keywords: crystallographic texture, Rockwell C hardness, non-uniformity, surface chemical composition.

1. INTRODUCTION

Hardness can be defined as the resistance to permanent plastic deformation, scratch or indentation of a softer material by a harder one. It is measured several times in the production line in order to assure the quality of several items produced, to perform sampling during the production processes and in materials receipt tests [1]. Brinell, Vickers and Rockwell are the most used indentation hardness scales for metallic materials. Rockwell hardness estimation is made as a function of the indentation depth [2]. Metrological reliability means all measurements shall be accurate and precise, have their uncertainties estimated, and have traceability to national or international standards. Certified reference materials shall be available to ensure the quality of hardness measurements established in last paragraph. These materials are transfer standards used to carry thru the traceability chain down the intrinsic metrological reliability from a primary system (reference system for a given quantity) to the productive sector. Thus, the hardness standard block of is this mentioned certified reference material being a key component to ensure valid macrohardness measurements [1].

The non-uniformity for a standard hardness block is the assessment of the homogeneity of hardness values on the surface of the standard block, according to ISO Guide 34 [3] and ISO 6508-3[4]. Since some of the properties of the crystals are strongly directionally dependent, the crystallographic orientation of the crystallites within the polycrystalline aggregate – the texture – plays an important role among these parameters [4a].

In this work, three hardness standard blocks with about 30 HRC (30 Rockwell C hardness) were selected from different manufacturers. Then, the influence of the variables crystallographic texture and chemical composition in the hardness non-uniformity was analyzed.

2. MATERIALS AND METHODS

Three standard hardness blocks were selected from different manufacturers. On cutting each block in three different regions a nine specimens were obtained. After that the specimens were grinded using SiC paper with different grades of roughness (from 220# to 1500#), thoroughly degreased with ethanol and rinsed with deionized water by ultrasonic vibration. Then the specimens were mechanically polished with diamond paste with 6, 3, 1 and 0.25 µm. Next, the specimens were also mechanically polished for crystallographic texture analysis on 0.06 µm colloidal silica. Finally, each specimen was cleaned with isopropyl alcohol PA by ultrasonic vibration and dried into nitrogen gas jetting.

For the crystallographic phases, grain sizes and surface chemical composition analyses, a FEI Company Nova Nanolab Dual-Beam Field Emission Gun Scanning Electron Microscope (SEM-FEG) was used. This equipment has also an EDAX-TSL system for crystallographic texture analysis using electron back-scattering diffraction (EBSD) technique. Energy dispersive spectroscopy (EDS) technique was used for the elementary micro-analysis of the specimens, which consists of an EDAX detector coupled to the SEM-FEG.

3. RESULTS AND DISCUSSION

3.1. Non-Uniformity Analysis

Relative non-uniformity (U_{rel}) is one of the most important criteria for the approval of reference material certified in hardness. So, for the hardness standard block to be considered a certified reference material on the HRC scale it must reach at most 1% U_{rel}. Table 1 shows the results of relative non-uniformity obtained for the three Rockwell C hardness blocks analyzed in this work and it can be seen they were approved according to U_{rel} criteria.

Table 1 - Relative non-uniformity of standard hardness blocks

Identification	Code	Hardness/HRC	U_{rel}/%
Standard block1	SB_1	30.0	0.4
Standard block2	SB_2	29.3	0.6
Standard block3	SB_3	31.2	0.4

3.2. Surface Chemical Composition Analysis

Figure 1 shows the elemental chemical analysis performed by the EDS technique. Although the hardness standard blocks have similar hardness values one can realize they are different in terms of chemical composition.

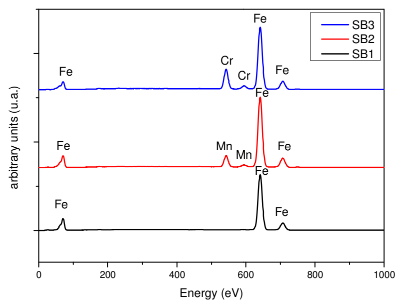

Figure 1 - EDS spectra shows the surface chemical composition of the standard blocks SB_1 (in black), SB_2 (in red), and SB_3 (in blue).

In Figure 1 it is possible to observe only the peaks of the higher amounts of elements present in the specimens. SB_3 has a significant amount of chromium (15.2% by mass) so it is expected that among the three standard hardness blocks studied in this work SB_3 presents the greatest surface chemical stability on its surface [5] (that means high resistance to the oxidation) and one can expect to some extent the behaviour of a stainless steel. SB_2 has a high Mn content whereas SB_1 has no presence of significant amount of alloy elements but carbon. Table 2 shows in more detail the chemical composition by weight (Wt%) of each element present on the surface of the three hardness standard blocks. Among the main elements on the surface not mentioning iron and carbon, SB_1 has only Mn and Si, SB_2 has a higher amount of Mn e some Si and Mn, whereas SB_3 has many alloy elements (and less Fe at the same time) including a lot of Cr and some Si, S, Mn and Ni.

Table 2 - SB_1, SB_2, and SB_3 surface chemical composition by weight analysis.

Elements	SB_1	SB_2	SB_3
	Wt%		
Si	0.5	0.5	0.7
S	0.0	0.0	0.5
Cr	0.0	0.3	15.2
Mn	0.6	2.4	0.6
Fe	92.3	91.5	77.0
Ni	0.0	0.0	0.5

3.3. Crystallographic analysis

Tables 3 and 4 show the crystallographic analysis of crystalline phases present in the specimens and the average grain size of the hardness standard blocks SB_1, SB_2, and SB_3.

Table 3 - Crystallographic analysis of standard hardness blocks SB_1.SB_2, and SB_3.

phases\specimens	SB_1	SB_2	SB_3
Ferrite	88.5%	84.4%	87.5%
Cementite (Fe_3C)	11.2%	15.0%	12.3%
$Mn_{23}C_6$	0.1%	0.3%	0.1%

Table 4 - Analysis of grain sizes of standard hardness blocks SB_1.SB_2, and SB_3.

Average grain size/ µm	Specimens		
	SB_1	SB_2	SB_3
	1.9	2.0	1.1

Analyzing Tables 3 and 4 it can be noted the ratios among the crystalline phases of the hardness standard blocks are very close and the presence of a slight amount of $Mn_{23}C_6$ probably as precipitates in grain boundaries. Taking into account both Table 1´s hardness values SB_2 < SB_1 < SB_3 and blocks have approximately 30 HRC, one can consider the average grain size is responsible for the different hardness values observed. All of the described phenomena means: SB_3 hardness standard block has both the smallest grain size and the higher hardness value; on the other side SB_2 has both the highest grain size and the smallest hardness value; and SB_1 has an intermediate behavior. So the smaller average grain size and chromium high concentration in the SB_3 gives it a slightly higher hardness value, as shown in Table 1.

Time, temperature, and transformation diagram in Figure 2, also known as TTT curve, shows the possible phase transformations for steels as a function of time and temperature [5]. Both Figure 2 and Table 3 show that crystallographic analysis has not detected the presence of the austenite phase e.g. retained austenite or uncompleted phase transformations and the unique microstructure present is the one that arised from the perlite transformation e.g. a ferrite and Fe_3C mixture.

Figure 3 shows the crystallographic mapping made by EBSD of the phases present in SB_1, SB_2, and SB_3. The ferrite matrix is in red, SB_1 has coarse perlite or coarse Fe_3C phase in green , and SB_1 and SB_2 show some finest ferrite phase of the same average grain size (Table 4).

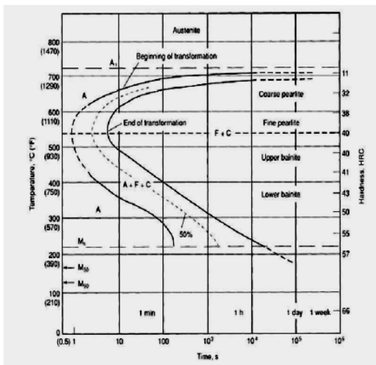

Figure 2 - TTT curve for steel [5].

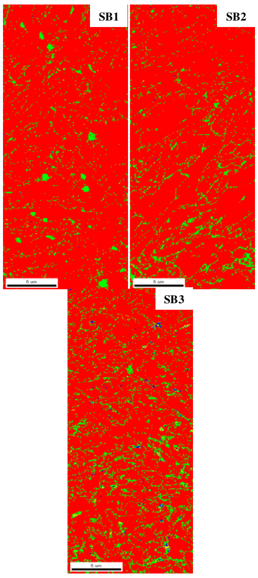

Figure 3 - Texture of crystallographic phases present in blocks SB_1, SB_2, and SB_3 where the areas in red correspond to the ferrite phase, in green to the Fe_3C phase, and in blue to the $Mn_{23}C_6$ phase.

SB_3 shows the distribution of a $Mn_{23}C_6$ network, a homogenous Fe_3C distribution and the lowest average grain size or highest hardness value.

Although the material properties depend primarily on chemical composition and previous thermo-mechanical operations treatments, results of this work could be seen as part of a recipe for producing 30 HRC standard blocks with a suitable microstructure. So a microstructure composed by ferrite matrix and the presence of perlite and a network of $M_{23}C_6$ phase seem to be a desirable microstructure since all three blocks from traditional suppliers have this character.

If an ideal manufacturer's selection was made to provide standard blocks for a national metrology institute, the most convenient choice would be the SB_3.For, in addition to having the proper non-uniformity, its surface stability against oxidation guarantees less variation of its hardness over time.

4. CONCLUSIONS

- EDS microanalysis showed that there are three different surface chemical compositions for the SB_1, SB_2 and SB_3 hardness standard blocks from different suppliers. Due to the high content of Cr the SB_3 standard block one can expect it has a somewhat oxidation resistance and chemical stability on its surface;

- The SB_1 and SB_2 hardness standards blocks have the same range of grain distribution and this fact can explain part of their hardness values closeness;

- Crystallographic texture of SB_1 and SB_2 are basically the same since both are composed of ferrite and Fe_3C;

- A microstructure composed by ferrite matrix and the presence of perlite and a network of $M_{23}C_6$ phase seems to be a must be character of a 30 HRC standard blocks with metrological reliability.

5. REFERENCES

[1] Oliveira, S.P. *et al* 2011 67º Congresso Anual da ABM.

[2] ISO Standard 6508-1:2005 "Metallic Materials - Rockwell hardness test - Part 1: Test Method (scales A, B, C, D, E, F, G, H, K, N, T)".

[3] ISO Standard 6508-3:2005 "Metallic Materials - Rockwell hardness test - Part 3: Calibration of reference blocks (scales A, B, C, D, E, F, G, H, K, N, T)".

[4] ISO Guide ISO Guide 34:2009 "General requirements for the competence of reference material producers".

[5] Metals Handbook, Prop.and Sel.: Irons, Steels, and High-Performance Alloys, 1990, **vol 1**, ASM International, Materials Park, USA.

6. ACKNOWLEDGMENT

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