Hardness Reference Blocks as Certified Reference Materials to be supplied by Inmetro to the Market

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Abstract. Reference materials for the Rockwell, Brinell and Vickers hardness scales are fundamental for the Brazilian industrial sector. Mechanical property hardness is the most common mechanical test used for quality control of raw materials in finished products as well as in research and development of new products and materials. A description of the main findings of two studies and the revalidation of the last on the demand of hardness reference blocks in Brazil is presented. This paper shows national demand of hardness reference blocks could be supplied on a large scale by Inmetro’s Force Laboratory.

Keywords: Certified Reference Material, Hardness Reference Block, Hardness Standard Block, Rockwell Hardness Scale, Brinell Hardness Scale, Vickers Hardness Scale

1. Introduction

Hardness is a mechanical property. Hardness can be described in a simplified way as a quantity associated with the mechanical resistance of all materials no matter if it is by quantitative methods (indentation ones, the most used with metals, and rebound tests, commonly used with plastics and rubber) or qualitative methods (Mohs, scratching or cutting tests). It is widely used for both quality control in production lines and checking desired or designed properties in industries´ receiving sector of materials and raw materials – not mentioning research and development of new products and materials – in steel, automotive, naval, aerospace, tubing, oil, machinery & equipment, kitchen & laundry appliances & products, and oil & gas industries among other industrial sectors [1-4].

Metrologically speaking Hardness is a quantity. Its measurements should have both metrological reliability and traceability [5]. These concerns are essential requisites that certified reference materials are to have in hardness field. A hardness reference block (or hardness standard block has three metrological roles being at the same time a materialized measure, a metrological standard, and a reference material (RM) that becomes a certified reference material (CRM) from the moment on it is calibrated by a recognized institution like a NMI (National Metrology Institute) [5,6] – Inmetro is the NMI in Brazil.

This paper aims to highlight the Brazilian demand for certified reference materials for the Rockwell, Vickers and Brinell hardness scales (HR, HV, and HB, respectively) [7-17]. In addition, it shows that one of the Inmetro (National Institute of Metrology, Quality and Technology)´s mission is...
to provide reliable certified reference materials to high technology and high added value industrial sectors [5].

Lafor (Inmetro’s Force Laboratory), that is the laboratory responsible for hardness metrology standardization in Brazil, has planned to help Brazilian industries more efficiently by supplying by itself CRMs for HR, HV, and HB for them. For the sake of information, nowadays all Brazilian industries acquire hardness reference blocks in the international market, paying high prices and still going through logistical problems for receiving such materials because all high-quality hardness blocks’ suppliers are located abroad. This is the only way that these companies have to maintain both traceability and reliability of the hardness measurements made in their premises [18,19].

This work will present the monitoring of the Brazilian demand for certified hardness reference materials within the last 20 years. This demand may be even greater if Mercosul (South American Single Market Agreement) countries were taken into account, which could be supplied on a large scale by Inmetro. It should be noted that Inmetro asked Brazilian Institute of Nuclear Quality (IBQN) for a Technical and Economic Feasibility Study on hardness reference blocks in Brazil and Mercosul in 1999 [6], in this paper called “1999 TEFS”. The most recent survey was carried out in 2016 by Inmetro’s Technological Innovation Division (part of Inmetro’s Department of Scientific Metrology and Technology “Dimci”) being focused in Brazil whose title was “National Demand Study on Certified Hardness Standard Block” [7], referred to herein as “2016 NDS”; this 2nd survey was revalidated by Ditec in 2019. Unlike 1999 TEFS, the most recent one focused on two scenarios: one of them dealt with demands related to large-size companies whereas the other included also the needs of medium-size industries.

2. Materials and methods

In this work a comparative methodology was used between 1999 TEFS and 2016 NDS. The future supply to the market of certified reference materials will require the same infrastructure that has been used in the last years for hardness metrology at Lafor. Some technical arrangements shall be necessary to be applied to the Primary Hardness Standardization System. An image of this entire system is shown in Figure 1, below.

![Figure 1 – Inmetro’s Primary Hardness Standardization Machine](image)

Figure 1 describes the primary system that provide metrological traceability to reference materials for HR, HV, and HB. This system is based on the application of force by dead weights, that is, the force produced by the direct action of acceleration of gravity on masses with traceable known values. In addition, the system is configured to meet all parameters involved in the calibration of reference materials in accordance with ISO and ASTM standards. Figure 2 shows some examples of hardness
reference blocks, also referred as hardness reference materials, which are from the lab collection of these RMs.

Figure 2 - Examples of hardness reference materials, also known as hardness reference blocks.

Figure 2 shows several hardness reference materials, where it is shown that they can be made from different materials, such as steel, brass or aluminium alloys. In addition, they may exhibit different geometries, thicknesses and different surface finishes depending on the hardness and manufacturer of the reference material.

For the metrological realization of the quantity hardness and consequently the certification of hardness reference materials it is necessary the use of hardness indenters. Indenters are dimensionally standardized metrological artifacts that perform the indentation into the surface of hardness reference blocks. Lafor has a collection of Rockwell and Vickers diamond indenters for certification of hardness reference materials. Figure 3 shows several types of images of a diamond Vickers indenter: in (3A), the tip of an indenter; in (3B), an indenter holder (the whole metal structure where the diamond tip is inserted) with its not visible tiny indenter at this tip to the right of the image, and, in (3C), an indenter apex (with a 20,007x magnification) which is at the intersection between the two lighter orthogonal diagonals in the image plane (like the intersection of the two laths of a cross). In (3C) it is possible a highlight of the topographic characteristics of this indenter tip e.g. a region nearby the indenter tip´s apex.

Figure 3 - Vickers diamond indenter images: (A) - indenter tip; (B) – indenter holder; and (C) - 20,007x magnification microscopy of the indenter tip.

In order to ensure the compliance of the diamond indenter to international standards, the geometric traceability of the measurements of relevant dimensional quantities for certification of reference materials is necessary. Figure 4 shows the Inmetro’s Primary Indenter Calibration System for the dimensional calibration of diamond Rockwell and Vickers hardness indenters.
Figure 4 - Inmetro’s Primary Indenter Calibration System for the dimensional calibration of diamond Rockwell and Vickers hardness indenters.

Measurements made in the Inmetro’s Primary Indenter Calibration System are in accordance with international standards and consist of measuring angles, lengths and radii related to indenter tips.

With a similar approach to 1st survey 1999 TEFS, that dealt with prospective studies and pertinent impact on hardness blocks market in Brazil and Mercosul, 2nd survey 2016 NDS focused only in Brazil. Since 2016 NDS study was conducted in 2016, it was revalidated in 2019 which concluded there were no significant variations in its findings of 2016 and 2019. Unlike 1999 TEFS, 2016 NDS took into account two scenarios. The first one considered only the demand of large companies of industrial sectors like metal-mechanical, machinery & equipment, automotive, car auto parts, electrical apparatus and materials, metallurgical, and transportation ones. And in the second scenario the demands of medium-sized companies were also inserted. This distinction was made because a reference material certified in hardness has a higher added value. E.g. big companies in the affected sectors have relatively more economic power and capital to invest in measures related to metrological reliability than the medium-size ones so their needs in terms of hardness reference blocks could be satisfied more easily than the minor companies. Measures related to metrological reliability means also that in some areas it can be necessary to satisfy mandatory requirements in terms of accreditation and evidence of metrological traceability of industrial goods of the related productive sectors. Such traceable measurements are performed by bodies accredited by the Inmetro’s Laboratories Accreditation Coordination (Cgcre). In last, big companies would have an easier access than minor ones to the international markets of countries that are signatories of the Mutual Recognition Agreement (MRA) of national accreditation and conformity assessment systems; as a consequence, Inmetro’s capability in providing hardness MRCs to the Brazilian demand means less costs to all impacted industries.

3. Results and Discussion
Table 1 [18-19] shows the amount of certified reference materials e.g. Rockwell, Vickers and Brinell hardness blocks determined by two studies: 1999 TEFS and 2016 NDS, the last being revalidated in 2019. For 2016 NDS two scenarios were considered in terms of number of hardness standard blocks involved: scenario 1 with only large companies and scenario 2 that included both large and medium enterprises; so this last one encompasses more blocks than the former.
Table 1 – Number of CRMs in hardness demanded in two surveys: 1999 TEFS and 2016 NDS [18,19]

| Survey            | Estimated Amounts |
|-------------------|-------------------|
|                   | Rockwell Hardness | Vickers Hardness | Brinell Hardness |
| 1999 TEFS         | 13,050            | 1,710            | 4,740            |
| 2016 NDS scenario 1 | 1,842            | 1,203            | 714              |
| 2016 NDS scenario 2 | 8,530            | 5,571            | 3,308            |

Comparative analysis of both surveys (1999 TEFS and 2016 NDS) shows different behaviours for each hardness scale in terms of the amounts of estimated blocks. Although 1999 TEFS included Brazil and/or South America and 2016 NDS only deals with Brazil it can be said there is an increase in the demand of hardness blocks between the time interval of both surveys. In 1999 TEFS there was more Rockwell blocks (13,050) than both Vickers and Brinell ones (1,710 + 4,740 = 6,450), the latter being 49% of the former. On the other hand in 2016 NDS there was an inversion in the demand of CRMs in hardness: the amount of Rockwell blocks (1,842 in scenario 1 and 8,530 in scenario 2) is less than the sum of Vickers and Brinell blocks (1,203 + 714 = 1,917 in scenario 1, and 5,571 + 3,308 = 8,879 in scenario 2). So in 2016 NDS the amount of both Vickers and Brinell blocks is about 104% of the Rockwell ones.

Figure 5 [18, 19] shows the analysis performed for certified reference materials for Rockwell hardness in 1999 and 2016 surveys.

Figure 5 – CRMs in hardness blocks demand for Rockwell scale in 1999 TEFS (in blue), in 2016 NDS scenario 1 (in red) with less 86% than Rockwell, and 2016 NDS scenario 2 (in green) with less 35% than Rockwell [18, 19].

Figure 5 shows that in relation to the 1999 TEFS (in blue) there was an 86% reduction for 2016 NDS scenario 1 (in red). This reduction is surely associated with the limitation of this study being only the demand estimated for large companies. Regarding 2016 NDS scenario 2 (in green), where, besides large-sized companies, medium-sized ones are also considered, a reduction of 35% was reported. In addition, the Rockwell hardness scale is the most used one because its hardness value is obtained directly in a hardness tester i.e. without the need to measure the dimensions (diagonals for Vickers and diameters for Brinell) of an indentation in a second metrological operation.

Figure 6 [18,19] shows that reference materials certified for Vickers hardness exhibit a distinct behaviour. In order to determine the Vickers hardness value it is necessary to analyze its indentation dimensions in a second operation being this hardness scale used a lot for the quality control of
products with high added value. It is associated, for example, to hardness of materials with surface finishes that need to have high wear resistance in sectors like orthopedic prostheses and industrial machinery and tools ones.

Figure 6 - CRMs in hardness blocks demand for Vickers scale in 1999 TEFS (in blue), in 2016 NDS scenario 1 (in red) with less 30% than Rockwell scale, and 2016 NDS scenario 2 (in green) with more 226% than Rockwell scale [18, 19].

Figure 6 shows that there is a substantial increase in block demand when scenario 1 (in red where only large companies are considered) is compared to scenario 2 (in green where large and medium-sized companies are jointly taken into account). Of course, one can expect that this growth can be even more significant if medium businesses are also considered. The behaviour of this growth of 226% in relation to the 1999 TEFS (in blue), may be related to the growth of a market that is dependent on the reliable measurement of the Vickers hardness, a market probably, or maybe a market maturity, not so present in the demand evaluated in 1999 when compared to 2016, and also 2019.

In Figure 7 [18,19] it can be observed the behaviour of the CRMs demand of the quantity Brinell hardness. There is a reduction of 85% in the number of Brinell blocks according to 1999 TEFS (in blue) when compared to scenario 1 of the 2016 NDS (in red). In relation to scenario 2 (in green), it is observed that the reduction is smaller, or around 30%. The Brinell hardness value, as well as the Vickers hardness one, depends on the indentation dimension’s measurement in a second metrological operation.

Figure 7 - CRMs in hardness blocks demand for Brinell scale in 1999 TEFS (in blue), in 2016 NDS scenario 1 (in red) with less 30% than Rockwell, and 2016 NDS scenario 2 (in green) with less 226% than Rockwell [18,19].
Figure 8, adapted from [18,19], shows the total of certified reference materials found for all three hardness scales Rockwell, Vickers, and Brinell in the surveys 1999 TEFS (in blue) and 2016 NDS scenarios 1 and 2.

![Diagram showing CRMs in hardness blocks total demand for all hardness scales Rockwell, Vickers, and Brinell in 1999 TEFS (in blue), in 2016 NDS scenario 1 (in red) with less 81% than Rockwell, and 2016 NDS scenario 2 (in green) with less 11% than Rockwell [18, 19].](image-url)

Figure 8 shows that, compared to 1999 TEFS, survey 2016 NDS scenario 1, that included large companies, showed less 81% HR, HV and HB blocks whereas survey 2016 NDS scenario 2, where medium and large companies are inserted, there is a decrease of 11% in the total amount of certified reference materials. So, 20 years later, after 1st survey 1999 TEFS, 2019 revalidation of 2nd survey 2016 NDS survey, the demand for certified reference materials still remains high in the Brazilian industrial sector. Nowadays this high-quality national market demand has been supplied by reference materials certified by other NMIs, in spite of a high cost, complex logistics and delivery time delays for the importation of these materials.

Lafor has been preparing to attend domestic market demand quickly and at fair prices. The laboratory is equipped with a Primary Hardness Standardization System (composed of a primary machine for indentations in hardness scales Rockwell, Vickers, and Brinell, and also a primary reference system for subsequent dimensional measurements of Vickers and Brinell indentations) that deals with HR, HV, and HB as of Figure 1. There is also a Primary System for Calibration of Diamond Indenters, Figure 4, for the calibration on diamond Rockwell and Vickers indenters. Faced with the availability of such equipment it will be possible to provide up to 50 reference materials to the market per week depending on the type of hardness CRM to be requested by clients. All these capabilities in hardness metrology has been running in accordance with ISO international standards’ series 6506, 6507, and 6508 that are related to hardness scales Brinell, Vickers, and Rockwell, respectively [7-17].

4. Conclusion
- This paper deals with analysis and comparison between two surveys: a 1999’s Technical and Economic Feasibility Study on production and certification of hardness standard blocks in Brazil and Mercosul, and a 2016’s National Demand Study on certification of hardness standard blocks only in Brazil, the latter been revalidated in 2019.
- On discussion of the findings of both 1999 and 2016 surveys Lafor has detected that there is a high demand from several national industrial sectors for certified reference materials for the Rockwell, Vickers and Brinell hardness standard blocks.
- With the ongoing maintenance and acquisitions, Inmetro can initially supply part of this demand. The production capacity of 100 to 200 blocks per month can still be increased in case of need motivated by the market. These certified reference materials may also be accompanied by a declaration of conformity to the relevant international standard, if desired by the customer.

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