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Traceability for indentation measurements in Brinell-Vickers-Knoop hardness

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ABSTRACT

Hardness is an important material property describing a material's resistance to localized deformation from an indenter pressing or scraping against its surface. It is determined by measuring the indentation size realised on the tested material surface. This can be done by different hardness testing methods, like the Brinell, Vickers and Knoop scales. This project will investigate the indentation measurement phenomena for the above-mentioned scales to provide a better defined, more consistent, unified, and reliable measurement and traceability methodology to overcome the inconsistency between national metrology institutes (NMIs) and lower levels of indentation (hardness) measurements. The project outcomes will be used in the next generation of hardness definitions, instrumentation and standardisation to improve the accuracy of material testing in all engineering fields, including aerospace, automotive, health, industry and research and development.

1. Introduction

Knowledge of the critical mechanical properties of materials used in a vast array of industrial products, construction and infrastructure is essential to preventing health and safety hazards, controlling the costs of production and creating better-designed, healthier and more consumer-friendly products. This knowledge is obtained through the establishment of accurate, reliable and consistent reference standards for material properties at the NMI level and a link to end-user measurements for the lowest level test in a workshop through a continuous traceability chain. Brinell, Vickers and Knoop testing methods are three of the most important ones used for testing the hardness of metallic materials. Hardness is determined in these scales by measuring of the size of an indentation made with an indenter of known geometry and properties on the surface of the material with a known force. There are many types of indentation measurement systems and methodologies. The indentation size measurement is strongly dependent on the image magnification and processing instrument, the operator, and, if used, software. Due to the many influencing factors for hardness measurement as well as the variation of equipment used for measuring the indentations, there are significant inconsistencies among NMIs, as well as between NMIs and secondary - and testing laboratories. This is due to a lack of a well-defined methodology and specifications for the variety of indentation measurement instruments used. This was also clearly demonstrated in 2005 at the NMI level in the CIPM Brinell pilot study, CCM.H-P3 [1], where significant deviations in measurement results between the participants were found due to the variations in the numerical aperture (NAs) used in the imaging system. The inconsistency in measurements increase noticeable and is much more evident lower down the measurement chain. Traceable propagation of the reference value from NMIs to testing laboratories is quite difficult, due to the variety of simple devices used for testing without controlling all parameters.

In hardness block calibrations, measurement results that are frequently inconsistent with the nominal values of the blocks highlight the lack of standardised measurement methodology and instrumentation, although all instruments used are compliant with relevant ISO hardness standards from a length measurement point of view. The same situation occurs in testing machines; different types of testing machines may differ in their measurements of the same reference blocks, although they comply with the relevant hardness standards in terms of each component constituting the hardness scale such as force application, indenter used, indentation measurement system and measurement cycle. This can be attributed to a lack of a commonly accepted definition of the measurand (indentation) and uniformity in the methodology used, a lack of traceability between NMIs and from the NMI level to the end user as well as to the producers of instrumentation equipment and a critical dependence on the operator's subjective decision on the precise location of the indentation borders.

This project aims to overcome these critical problems by establishing the necessary methodologies and references for consistent, unified, reliable and traceable hardness indentation measurements from the NMI level to testing laboratories. This is done in 4 steps:

1) Diameter measurements of Brinell indentations in sink-in, pile-up, isotropic or non-isotropic materials give different results with different imaging and measurement systems because there is neither a physical definition for nor a common agreement on the location of the border of indentation. It is important to objectively define or at least agree on the boundaries of the indentation and its diameter as a starting point for the traceability that can be propagated from the highest level of measurement to the end-user. Consistent measurement demands that NMIs and DIs deliver a standardised measurand to the calibration and testing laboratories, which can then also be used by industry. It is also necessary to adequately define and agree upon the conditions under which measurements shall be performed to supplement unified and standardised Brinell indentations in the

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world of metrology. The same situation applies for Vickers indentations as well as Knoop ones, this time to define the corners of the indentation, which are the end points of the diagonal length that are measured for the determination of the hardness values of material.

2) Once a reference definition is constituted it needs to be propagated along the measurement chain. This makes it necessary to develop long-term stable and high-quality real or dummy reference indentations to be used as transfer standards for propagation of the indentation measurement methodology as well as assurance of the traceability and consistency of measurement results. The proposed set of reference indentations will ensure the traceability of real indentation measurements in three dimensions rather than two dimensions as is currently possible. The set of indentations will be produced for all hardness scales and levels used in industry, giving rise to the need to settle discrepancies between different hardness scales, which the project aims to address as well.
3) At the testing laboratory level, the problem of achieving consistent and traceable hardness measurements is much more obvious due to the variety of simple devices used for testing without controlling all parameters. Different magnification, imaging and measurement devices result in varying determinations of the border of indentations and thus indentation sizes, which ultimately results in inconsistent hardness measurements. This situation introduces the necessity of investigating the link between the industrial testing devices and the reference devices present at the NMIs. Determining the correlation between the reference systems and the testing systems is one of the most important parts of this project, as it can guide the construction and design of the next generation of hardness measuring machines. The list of recommendations that will be provided to hardness instrument producers will enable consistent and comparable measurements and will change the future development of these instruments.
4) Finally, after defining the measurand (indentation) and ensuring its propagation to the lower level of measurement stages, another problem confronted is not related to either the definition of the measurand or to the instrument itself, but to the operator, the person who performs the measurement. Another parameter that brings about deviations in measurements are the decisions taken by the operators about the location of the indentation borders. Automation of measurements is seen as the means to overcome this issue while also resulting in labour and cost efficiencies.

2. Development of definition and measurement methodology for hardness indentations of Brinell, Vickers and Knoop

Due to the testing process, the indentations produced in hardness testing have no sharp edges. Instead, the indentations gradually merge into the undeformed surface. Sometimes material is pushed out of the indentation (pile-up behaviour), whereas other times surrounding material is pulled down with the indent (sink-in behaviour) as in Fig. 1.

Both make the transition from the undeformed surface to the indentation even more complicated to define [3,4].

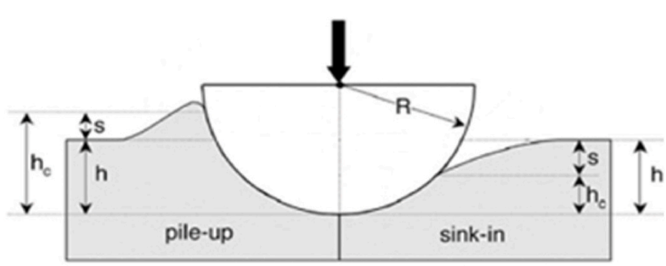

Fig. 1. Cross section of an indentation profile showing pile-up and sink-in behaviour.

Current hardness test methods determine the size of the indentations using optical systems like light microscopes as shown in Fig. 2r the indent, the size of the optical lens used and the illuminance have an influence on the back-reflection of the light off the hardness block back to the lens. Furthermore, the incidence of light back into the lens depends on the plane of focus. In addition to the effects introduced by the microscope itself certain parameters of the block, like its surface roughness [4,5], also influence the measurement results.

To address the different effects of measurement tools a precise definition of the borders of the indentation itself is required in order to minimize any uncertainty caused by the individual understanding of the indentation size by different operators. For this, a set of indentations is produced for all three hardness testing methods (Brinell, Vickers and Knoop), serving as a stable reference for all future developments within the scope of this project.

To gain a representative sample, a wide selection of different hardness reference blocks is used. The most important objective is a precise, unified definition of the borders of the indentations for both pile-up and sink-in behaviour of the materials tested, requiring indentations in both harder and softer materials. Indentations are performed on reference blocks made out of steel, aluminium, copper and brass. Furthermore, the effects of the surface roughness on the optical measurement systems are investigated by performing similar indentations on at least two blocks with at least three different surface roughness levels (polished to non-polished). A total of 76 indentations (36 for Brinell, 24 for Vickers and 16 for Knoop) were produced on a total of 28 hardness reference blocks. These indentations will serve as reference indentations in this project.

The reference indentations are measured and characterized by multiple consortium members utilizing both conventional optical methods as well as state-of-the art methods like scanning tactile and laser confocal measuring systems to measure the 3D surface areas of indentations exhibiting both pile-up and sink-in behaviour. Using these results, an analysis of the different measurement methods (scanning tactile and confocal laser versus standard optical measuring systems) will be performed. With the 3D surface data, the last point of contact between the indenter and the material will be determined and accounted for in the universal definition of indentation borders [7]. This allows for the development of universal definitions for Brinell, Vickers and Knoop's hardness indentation boundaries.

The current lack of specifications for a focal point makes it difficult to compare the indentation size measurements. Therefore, a definition for the correct focus must be established. With the inputs of the first step, the influence of the focal point on the measured indentation size is investigated in order to establish specifications for the focal point in the measurement of indentation sizes. Using the reference indentations, multiple measurements are performed at the focal point as well as above and below the focal point for all three hardness testing methods (BVK). Based on this analysis, specifications for the most appropriate focal points will be determined for indentation size measurements.

With the basic definitions set, further effects like the influence of illumination and related factors as well as different surface roughness levels on the measured indentation size can be investigated. For these, an additional 10 indents of different diameters are created on the three selected surfaces with differing roughness for Brinell (optional as well for Vickers and Knoop). The blocks are then investigated by different consortium members using the above-described unified methods to

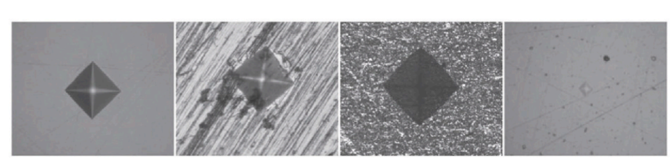

Fig. 2. Vickers indentation images, from left to right: a distinguished image, with surface damages, with poor optical conditions, with orientation and position flaws [6].

examine and establish a relationship between surface roughness levels and indentation size measurements. In addition, the influence of the illuminance and the numerical apertures of the lenses and their relationships on the indentation size for both bright-field and dark-field illumination conditions and for both pile-up and sink-in behaviour are investigated.

3. Development of transfer standards for Brinell, Vickers and Knoop indentations

Calibration of Indentation Measurement Systems (IMSs) used for Brinell, Vickers and Knoop hardness is performed along the *x*- and *y*-axes only using the standard scale and stage micrometer. However, due to the geometrical shapes of Brinell, Vickers and Knoop hardness indentations have 3-dimensional (*x*, *y*, and *z*) elements. This variance between the 2-dimensional reference used for the calibration of IMS and the 3-dimensional object to be measured results in systematic errors. A 3-dimensional indentation transfer standard is needed to enable more accurate and reliable calibrations.

For this reason, a new transfer standard to be used as a substitute for or concurrent with the current standard scale and stage micrometer methods used for calibrating manual, automatic and semi-automatic IMS of Brinell, Vickers and Knoop hardness scales is developed. In the first step, a literature review is performed to find suitable candidate materials to be used for the production of indentation transfer standards for Brinell, Vickers and Knoop hardness. Carbon steel, brass, aluminium, ceramic etc. will be reviewed as candidate materials for minimum and maximum nominal hardness for Brinell hardness (100–650) HBW, Vickers hardness (100–1000) HV and Knoop hardness (100–1000) HK. The selection is based on the material's long-term stability, desired surface properties and varying hardness levels. Once a material is chosen, optimum design features of the indentation transfer standards, such as optimum shapes (e.g., circle, square, triangle), thickness and minimum surface roughness, need to be defined by reviewing available data [8].

The transfer standard should cover all possible indentation sizes and shapes that influence measurement results due to their 3-dimensional properties and effects on optical measurement devices. The diameters and diagonal lengths of the indentations on the transfer standards as well as the number and interval of indentations for each sample needed for calibration of hardness indentation measurement systems will also be determined. The optimum hardness scale, force-diameter index and hardness level (diameter of indentation) for the transfer standards must be chosen so as to optimally meet requirements for the calibration of IMS and will directly influence the choice of material. A report will be drafted with the conclusions reached. The design and drawings of the indentation transfer standards, together with their mechanical properties for Brinell, Vickers and Knoop hardness scales, will be developed and the transfer standards themselves will be produced or bought.

A set of reference indentations for calibrating IMS of Brinell, Vickers and Knoop hardness are produced on the different blocks previously designed and acquired. The transfer standards will be evaluated for repeatability and reproducibility of the results of their measurements on the best national hardness standards available and a reference value (nominal diameter, diagonal length, measurement uncertainty, etc.) of the indentation transfer standards will be defined. A selected subset of the transfer standards is then circulated and used to validate the IMSs of the participating institutes.

4. Traceability chain from NMI to user level

The currently available traceability chain from NMIs to lower levels of secondary and testing laboratories covers only uniaxial static forces, applied perfectly through a known indenter at constant temperature, to test specimens that react in a purely axisymmetric manner. This is not how systems perform in practice, where there are additional effects due to short-term creep resulting from different force application times, indenter geometry, temperature, alignment, optical measurements on the indentation size and fabrication of the artefacts used as transfer standards [9–12]. For this reason, it is necessary to investigate all possible applications of Brinell, Vickers and Knoop hardness tests at user and industrial levels as well as the main instrumentation used to perform such tests to calibrate them according to international standards and, through the use of developed advanced uncertainty models, ensure a robust traceability chain for Brinell, Vickers and Knoop hardness tests.

A detailed review of commercially available Brinell, Vickers and Knoop hardness testing and calibration machines at the NMI, calibration laboratory and industrial levels across the countries of the participating institutes will be performed and information on measurement principles, force range, stiffness, indenter geometry, dynamic characteristics and optical bench will be collected. Associated instrumentation will also be considered during the review. International standards and documents such as ISO 6506 [13], ISO 6507 [14], ISO 4545 series [15], ASTM E92 [16], ASTM E384 [17], ASTM E10 [18], etc. and, where available, national standards and documents will be analysed. A wide selection of representative industrial applications, such as automotive, offshore, manufacturing, aerospace and nuclear, will be made and investigated. The industrial applications will be selected so as to cover a wide variety of materials and scales, chosen from the about 25 Brinell, 20 Vickers and 12 Knoop standardised scales covering, each, different levels of hardness: from very soft to very hard materials (aluminium, copper, brass, cast iron, steel, carbon steel, etc.).

In addition to the above-mentioned influencing factors due to the hardness block itself, there is a wide range of additional factors that need to be considered for BVK hardness tests. Some of them can be attributed to the machine (friction loss, elastic deflection, misalignment of the indenter holder, etc.), the measuring system (indicating error, poor resolution, numerical aperture of lens or illuminator, inhomogeneous illumination of the indentation, etc.), the force application system (deviation from nominal forces, deviation from time intervals of the testing cycle, test force dwell time, force introduction, overrun of test forces, indentation velocity, etc.) and the indenters (deviation from the ideal shape, indenter holder, damage, deformation under force, etc.) used. Environmental effects such as deviation and/or drift of ambient temperature, vibration and shocks also have an influence on the indentation produced. Certain influences have already been studied and their effects can be derived from available data. Information on the effects of short-term creep, the indenter's geometry, temperature and alignment of the applied load that is only partially known or not known will be studied and investigated.

The combination of the researched points from the machines and IMS used from NMI level downwards, the methods, procedures used and requirements of certain industries as well as the many different factors influencing an indentation and its measurement allows for the development of advanced uncertainty models describing the effects of the various application conditions in relation to the different types of hardness testing machines at NMI, calibration laboratories, and user levels as well as other associated effects on the measurement results of Brinell, Vickers and Knoop hardness tests.

With specific reference to the ISO 6506, ISO 6507 and ISO 4545 series, the main sources of uncertainty associated with the selected hardness tests from among representative scales and hardness values among all 25 Brinell, 20 Vickers and 12 Knoop scales will be determined and advanced measurement uncertainty models for hardness tests based on the major uncertainty components will be developed. These models will be verified by testing in a representative range of hardness testing machines, including both different hierarchy levels (NMI, calibration laboratories of hardness blocks and hardness machines and industrial testing machines) and different types of scales (Brinell, Vickers and Knoop). The validation report will be used as a contribution for revising the calibration guide EURAMET cg-16, Version 2.0 (03/2011): Guidelines on the Estimation of Uncertainty in Hardness Measurements [2], which currently covers Rockwell scales only.

The correct traceability of hardness should be based on a country having one NMI realising the hardness scales in a number of primary hardness standard machines used to calibrate primary hardness reference blocks and a number of calibration laboratories, generally accredited by their national accreditation body, using hardness calibration machines to calibrate hardness reference blocks. These blocks may then be used to calibrate industrial hardness testing machines. The hardness calibration machines will generally be traceable to the national hardness standard machines via comparisons using hardness blocks as transfer standards and the accredited calibration and measurement capability of the calibration laboratory will be based on the results of these comparisons. Calibration of hardness blocks in the hardness calibration machines will generally be carried out in accordance with a documented procedure, such as Part 3 of the related ISO standards, and the uncertainty of the calibration results will be dependent on the machine's calibration and measurement capabilities (CMCs), as well as on the performance of the instrument during the calibration. Similarly, the uncertainty of the calibration of the industrial hardness testing machines will be partly dependent on the uncertainty arising from the hardness reference blocks used for their calibration, and the uncertainty of any subsequent hardness measurement will depend in part on the uncertainty associated with the hardness testing machine. It can be seen that the uncertainty of the final hardness measurement is dependent on all of the previous traceability stages, and a clear and harmonised guidance on how to estimate all of these contributions is needed since at present it is partially, and sometimes with different approaches, given in annexes to some related national and international standards (i.e., ISO or ASTM standards) and in some international documents (i.e., EURAMET cg-16). The above traceability situation strictly covers only indirect calibration and verification, whereas direct traceability to the SI quantities involved in the hardness tests (force, length and time) should also be addressed.

Guidelines detailing improved calibration and test procedures will extend the present traceability chain for Brinell, Vickers and Knoop hardness tests from NMI to user level, ensuring that all influencing factors identified are properly considered by the calibration and test procedures will be drafted. These will give guidance on the steps necessary to assure traceability of hardness measurements and on the estimation of hardness measurement uncertainty for each of these steps from the NMIs to the end-user.

A set of recommendations for hardness instrument producers will be published, aimed at facilitating the development of the next generation of hardness testing machines for Brinell, Vickers and Knoop hardness measurements. Such recommendations will encourage instrument producers to update hardness machines to comply with updated documentary standards and the requirements for improved calibration and testing procedures. Recommendations will include technical specifications for the instrumentation so that they may be properly calibrated for assurance of traceable measurements.

5. Automation of indentation measurements

NMIs with Brinell, Vickers and Knoop CMCs perform the indentation measurements either manually, with the operator deciding the location of the border of the indentation by visual inspection or by using custom-made software to perform automatic and semi-automatic measurements.

In the current "automatic" measuring systems, an automatic measurement is taken in one direction only. The perpendicular length (diameter/diagonal length) measurement is made by rotating the indentation around its axis. Also, only one indent at a time can be measured this way, with no possibility to perform measurements in series on more than one indentation automatically, resulting in an at best semi-automatic measurement. In the scope of this project a truly automated IMS should be developed and realised at TÜBİTAK UME hardness laboratory shown in Fig. 3.

In a first step, currently available automatic indentation measurement systems for Brinell, Vickers and Knoop indentation measurements (either primary reference systems or calibration or testing machines) will be reviewed regarding their working principles, electro-mechanical structure and relevant software, including measurement algorithms and systems for auto-calibration of the system by making use of a certified line scale. The main parameters for the realisation of measurements made by the current systems will be determined and their performances will be evaluated in terms of accuracy and user-friendliness.

Findings will be used to develop and implement improvements to the present IMS at TÜBİTAK UME Hardness Laboratory [19] in order to endow it with the capability of marking the locations of indentations that are selected by the operator from the software of the IMS and later fully automatically measuring the produced indentations, creating a real automatic IMS. The tool used for marking the hardness reference blocks with the specified indentation diameters in accordance with the hardness scale and level, the targeted coordinates and the number of indentations will be produced and fitted to the IMS. Additionally, the modified IMS should have the capability to mark the correct position of the hardness block on the IMS plate for indentation measurements after realisation of indentations (to ensure identical positioning of the block for marking the location of indentations and for measurement after realisation of indentations).

A set of specifications for the improvements to the software of the IMS currently available at TÜBİTAK UME Hardness Laboratory to enable autofocus, edge detection by image processing for circular, square and equilateral-shaped indentations as in Fig. 4, automatic measurement of one indentation and serial automatic measurements of n number of Brinell indentations whose coordinates were already recorded by the software during marking for indentations suitable for automated measurements will be developed and the IMS accordingly adapted.

The fully upgraded system will then be tested and verified by comparing the measurement results within the same system between automatic and manual measurements as well as between different automatic IMS'.

Not all indentations are suitable for automatic measurement due to optical restrictions. The effective parameters are expected to be the lack of contrast, optical resolution, measurement conditions (lighting, etc.), shape and quality of indentation, etc. Using the upgraded TÜBİTAK UME IMS, the optical specifications of the material (hardness reference blocks), the optical devices, and the theoretical approach will be investigated to determine how the optical system, together with the software, recognises the surface during autofocus and the indentation border, with the aim of identifying optical limits for recognition. Also, different indentation shapes in relation to hardness levels and scales and the material of the hardness reference blocks will be investigated.

Measurements performed with different systems by the consortium members will be compared with automated measurements of the same indentations made at TÜBİTAK UME and a correlation between the indentation specifications and the accuracy of the automatic measurements will be determined. The purpose is to identify the characteristics

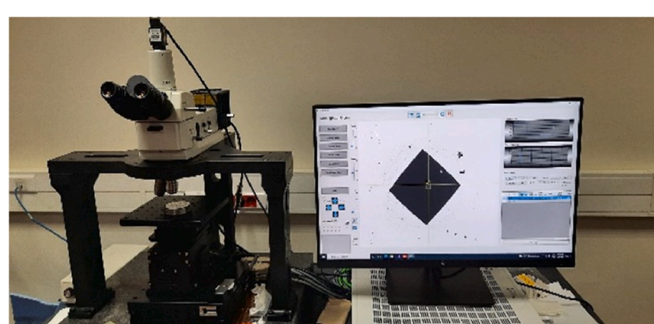

Fig. 3. Automatic IMS at TÜBİTAK UME hardness laboratory.

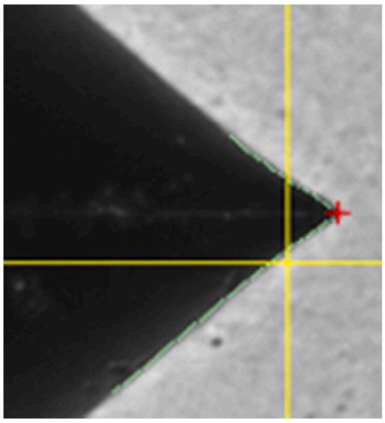

Fig. 4. Current edge-detection at TÜBİTAK UME IMS.

of indentations that are suitable for automated measurements.

6. Projected outcomes and impact on future hardness measurements

Hardness is not a physical quantity; it is a method-based quantity and thus the definition of the method and the measurands subject to consideration in hardness are very critical, as is the case in Brinell, Vickers and Knoop hardness indentation measurements. However, currently there is neither a standardised definition, testing methodology nor technical specification for the machines used in hardness testing. This is causing many difficulties in constituting a reference value and propagating it from the NMI level to the lower-level hardness measurement devices used in Europe. These problems are independent of the shape of the indentation and are equally prevalent for Brinell, Vickers and Knoop indents.

The project proposes a solution to the problem of inconsistent hardness measurements by defining and establishing a methodology for traceability from the NMI level to the end user by establishing universal definitions for Brinell, Vickers and Knoop indentations, their borders and a methodology for their measurement for the most widely used IMSs. Building on this, new 3D indentation transfer standards, uncertainty models for BVK hardness tests, a methodology to ensure traceable measurements from primary standards to the end-user, and automation of hardness measurements will all represent significant advances in hardness metrology. With the set definitions a set of recommendations for hardness testing instrument producers will be produced and disseminated. Uptake of the recommendations will enable a new generation of instrumentation for BVK hardness tests that produce results that are more consistent with the higher-level machines. On a wider scale, more accurate, reliable and consistent hardness measurements will benefit all industries in which such measurements are central to producing higher quality, durable and safer products. Developments and outcomes of the project will be shared with CIPM CCM Working Group for Hardness and EURAMET TC-M's newly established Working Group on Hardness, through which uptake of the knowledge and outputs by the metrology community will be facilitated.

The consortium will promote the results of the project within the standardisation community and will provide input into the standardisation process. The BVK indentation definitions, uncertainty models and methodology for establishment of traceability for BVK hardness measurements will be used as input to improve ISO 6506-1, ISO 6506-2 and ISO 6506-3 for Brinell, ISO 6507-1, ISO 6507-2 and ISO 6507-3 for Vickers, and ISO 4545 1, ISO 4545-2 and ISO 4545-3 for Knoop hardness tests, which currently have significant deficiencies. ASTM standards ASTM E92, ASTM E384 and ASTM E10 may also be improved as a consequence. Uptake of the knowledge, methods and other outputs generated by the project by the wider scientific and industrial communities will be assured through their incorporation into improved standards and guidance documents.

More consistent, reliable and traceable measurements of material properties will enable better, safer product design and manufacturing at a lower cost. Hardness tests are a widely used material testing method to obtain preliminary information about abrasion and strength properties of a material very easily and quickly. The durability of selected material is critical to the proper, predictable and safe functioning of manufactured products and components and the safety of the built environment.

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