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Provision of hardness standards - Final report

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Abstract

As part of a EUROMET agreement, the source of UK hardness standards were transferred to IMGC in Italy in 1988. Following pressure from UK industry, it was decided that a national centre of excellence in hardness measurement and calibration should be re-established in the UK.

This report describes the basis of hardness measurement and process of re-establishing national hardness standards at NPL. Two servo-controlled hardness machines have been designed and built by Instron Ltd and commissioned at NPL. Equipment was purchased, and modified where necessary, to measure the hardness indentations and hardness indenters. Problems encountered during commissioning are described together with additional work carried out to try to bring the equipment within the target specification. The main factors contributing to the uncertainty in measurements made with the UK hardness standard machines are considered and ongoing research aimed at reducing these uncertainties is summarised.

The launch of a UK calibration service is described and contact addresses are provided.

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Approved on behalf of the Managing Director, NPL,
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1 Introduction.

This report is submitted in completion of the contract MPU 8/45.4 'Provision of hardness standards' which was part of the DTI's National Measurement System (NMS) Programme for Mass, 1996-99. The first part of this report provides detail on the background to the work, the technical specification of the hardness standard machines, and ancillary equipment that has been purchased to establish a calibration service. The second part of the report considers the main factors contributing to the uncertainty in measurement made with the UK hardness standards machines. Finally, this report gives details on ongoing hardness research to improve the uncertainty budget of the Rockwell hardness machine.

1.1 Background.

The UK hardness standards held at NPL were transferred to IMGC in Italy in 1988. IMGC took over the responsibility of providing primary hardness calibrations to UK users. Through the British Measurement and Testing Association (BMTA), UK manufacturers of hardness equipment and users of hardness measurements requested that a national centre of excellence in hardness measurement be re-established in the UK. This should provide a local calibration service, a source of advice and consultancy to UK industry, and re-establish the UK's influence in the development of international hardness standards.

2 The UK hardness standards project.

Hardness measurements are cheap, quick, simple and relatively non-destructive and are therefore widely used in industry. Hardness is an unusual physical property; its measurement is essentially arbitrary and not susceptible to precise definition in terms of fundamental units of length, mass and time. In practice, hardness is measured in terms of the size of an impression made on a specimen by an indenter of a specified shape when a specified force is applied for a specified time. There are three principal standard methods for expressing the relationship between hardness and the size of the impression, these being Brinell, Rockwell, and Vickers. For each of these methods there are a range of scales, each a combination of applied load and indenter morphology, to cover the range of hardness encountered in practice.

Because of the arbitrary nature of hardness, procedural standards [1, 2, & 3] are critical in securing uniformity of measurements. The standards dictate performance requirements for reference hardness machines used to calibrate hardness test blocks. These standards require that hardness machines are directly calibrated - this means that the force, time, velocity, depth and indenter shape are all calibrated independently. The calibrated test blocks are then used to verify the performance of hardness testing machines.

In 1995 it was decided that UK national standards for hardness should be re-established to cover the highest priority Brinell, Rockwell, and Vickers hardness scales. Machines needed to be designed and built to make the necessary indentations. A single machine could be used to make the Vickers and Rockwell indentations because the required forces are similar. A separate machine was needed to make the Brinell indentations because the required forces are much greater. Equipment was also needed to measure the size of the indentations and to measure the morphology of indenters.

Before submitting a proposal, NPL reviewed the two most appropriate machine designs - deadweight and screw driven. Despite the metrological simplicity of deadweight machines, they lack adaptability and are expensive due to the manufacture of deadweight stacks. It was agreed that further advances in hardness metrology would best be made using servo-controlled screw drive

machines to apply test forces via high precision load cells. As a result of this decision, NPL submitted a joint proposal with Instron Ltd, a manufacturer of hardness testing machines.

The machines needed to have an uncertainty in the applied force of as low as $\pm 0.03\%$. This places requirements on both the force measurement and force control. High precision loadcells with linearity, hysteresis, and creep characteristics that exceed BS EN 10002-3 Class 00 [4] were purchased and calibrated using NPL deadweight force standard machines and specially written software. The servo-control mechanism must provide accurate control over the applied force and the indenter motion. This has the benefit of avoiding any force overshoot, a problem in some deadweight machines. A major advantage of this control system is the ability to accurately reproduce a wide range of loading cycles.

These machines will also be suitable for instrumented indentation hardness testing (universal hardness) for which ISO standards [5] are in the process of being drafted. Such hardness tests may eventually replace the Brinell, Rockwell and Vickers scales currently used as the tests provide additional information about the properties of the material.

The two national standard hardness machines were specified by NPL with the assistance of Instron Ltd, and then designed by Instron under sub-contract to NPL. On approval of the design by NPL, Instron Ltd were sub-contracted to manufacture and supply the hardness machines, and associated control software. By the end of 1998, NPL had taken delivery of both the hardness machines, the equipment for measuring indenter morphology, and an image analysis system for measuring Brinell and Vickers indentations. The assistance of IMGC was used in specifying the indenter and indentation measuring equipment. These were commercial developments of equipment built by IMGC. The image analysis microscope was obtained from another part of NPL. The laser interferometer used to measure the travel of the microscope stage was designed and manufactured at NPL.

2.1 Hardness scales.

The national standard hardness machines have been designed to cover the hardness scales most commonly used in UK industry. These scales are based on those requested by BMTA and were agreed with the United Kingdom Accreditation Service (UKAS) and are shown in Table 1. An additional requirement of the hardness machines was that they have the adaptability to provide additional hardness scales when required. The present scales are seen as the minimum requirement for the UK. The extension of hardness scales is part of the 1999-2002 NMS Programme for Mass.

Table 1. Hardness scales supported.

	Scales	Nominal Force (N)
Brinell	HB10/3 000	30 000
	HB10/1 000	10 000
	HB5/750	7 500
	HB2.5/187.5	1 900
Rockwell	HRB	100 to 900
	HRC	100 to 1 500
Superficial Rockwell	HR30N, HR30T	100 and 300
Vickers	HV10	100
	HV30	300

2.2 Hardness machines.

Both of the hardness machines supplied by Instron were modifications of commercial material testing machines. Machine 1 applies forces from 30 N to 1 500 N and Machine 2 applies forces from 1 500 N to 30 kN.

Machine 1 (Figure 1) is based on the Instron RT2000 hardness machine, and is able to cover the force and displacement demands of the Rockwell scales HRA to HRK, superficial Rockwell scales HR15N to HR45T, Vickers scales from HV5 to HV100, and Brinell scales HB2/4 to HB5/125. To eliminate torque from the screw drive, the machines are designed with twin reaction columns. The applied force is measured by high accuracy load cells, traceable to NPL force standards. Indenter depth measurement is by a Heidenhain sensor with a resolution of 1 nm. The Heidenhain depth measurement sensor is calibrated using laser interferometry traceable to the UK realisation of the metre at NPL. The calibration of the time control of the loading cycle is traceable to the UK time standard at NPL.



Figure 1. Machine 1, applies forces from 30 N to 1 500 N.

Machine 2 (Figure 2) is based on the Instron 5567 tensile/compression testing machine, and applies the forces required for the Brinell scales HB2.5/187.5 to HB10/3 000. This machine also has a Heidenhain depth measurement sensor for accurate monitoring of position during testing.

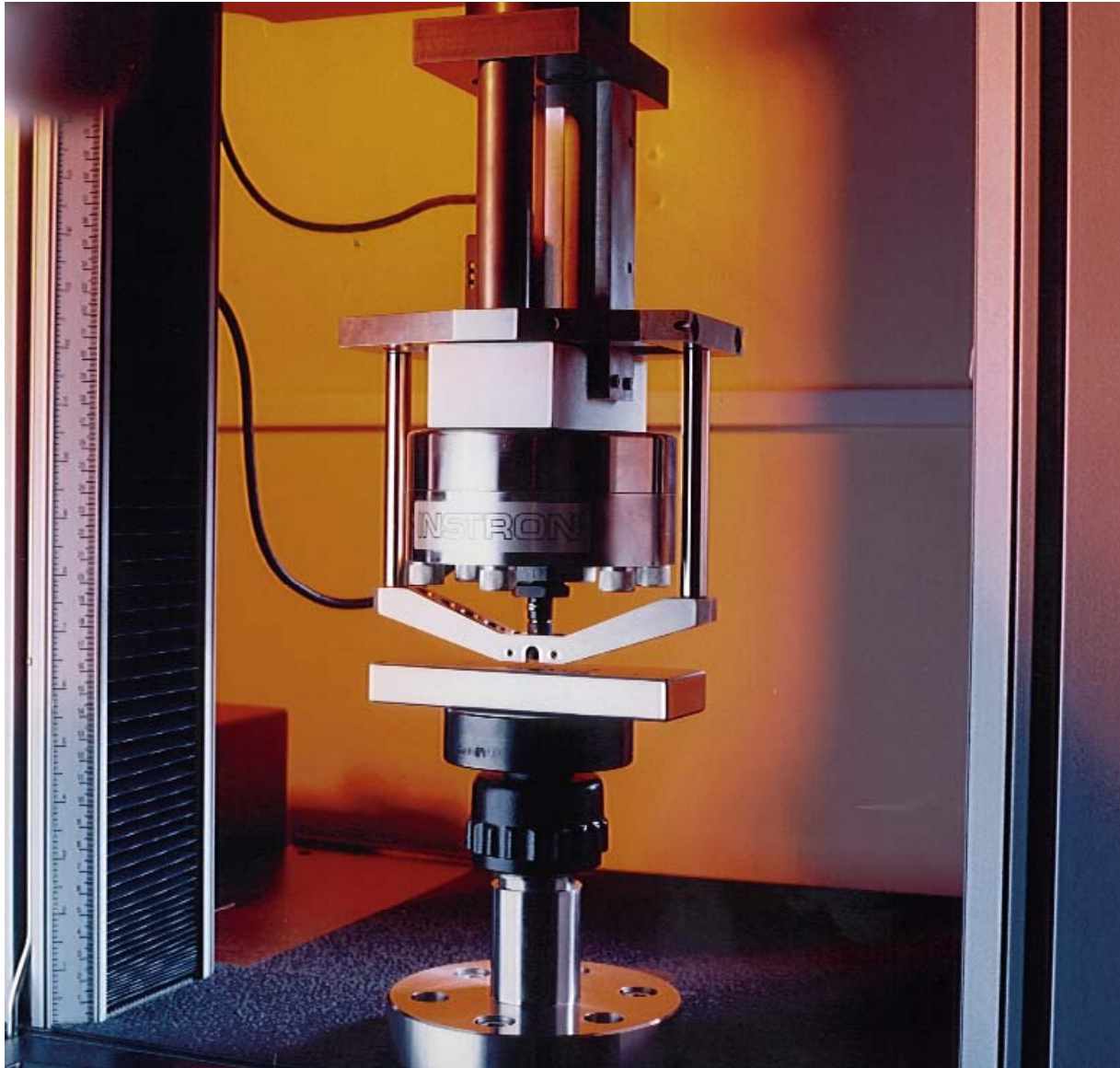


Figure 2. Machine 2, applies forces from 1 500 N to 30 kN.

Both machines are interfaced to their own PC driven control system based on Instron's Merlin and Wavemaker software products [6]. This software uses generalised waveforms for closed loop control.

2.3 Indentation measurement system.

Previous experience at NPL indicated that the best way to measure hardness indentation width, albeit primarily in micro-hardness [7], was using an optical microscope and stage fitted with a laser interferometer. The microscope is required to have high precision objectives of sufficient numerical aperture to distinguish the indentation from the bright surface of a hardness test block. The determination of the edge of the indentation depends on the illumination, magnification, and numerical aperture of the objective lens. Using an image analyser to remove operator dependence, the image display must be optimised for grey levels to ensure standardisation of contrast for imaging

the ‘true’ edge of an indentation. Since there is no ‘true’ edge, the system can only be validated by comparison with other national standard laboratories.

The indentation measurement system (Figure 3) consists of a microscope with CCD camera to image the indentations, an interferometer to measure the movement of the stage, and image analysis software that controls the measurement process. The system has been developed by Graftek Italia Srl for IMGC. It minimises the human manipulation of eyepiece cross-hairs used to identify the diagonal vertices of a Vickers indentation or the tangent to a Brinell indentation. The software identifies the edges of the indentations by comparison with templates. The measurement is accomplished by positioning one vertex of the indentation in roughly the centre of the image and measuring the distance the stage has to move to position the opposite vertex in the centre. The final value is the sum of the stage movement plus the distance of each vertex from the exact centre of the screen. The method corrects for any misalignment of Vickers indentations with the horizontal axis.

The stage movement is measured using a Jamin interferometer and mirrors attached to the side of the microscope stage.

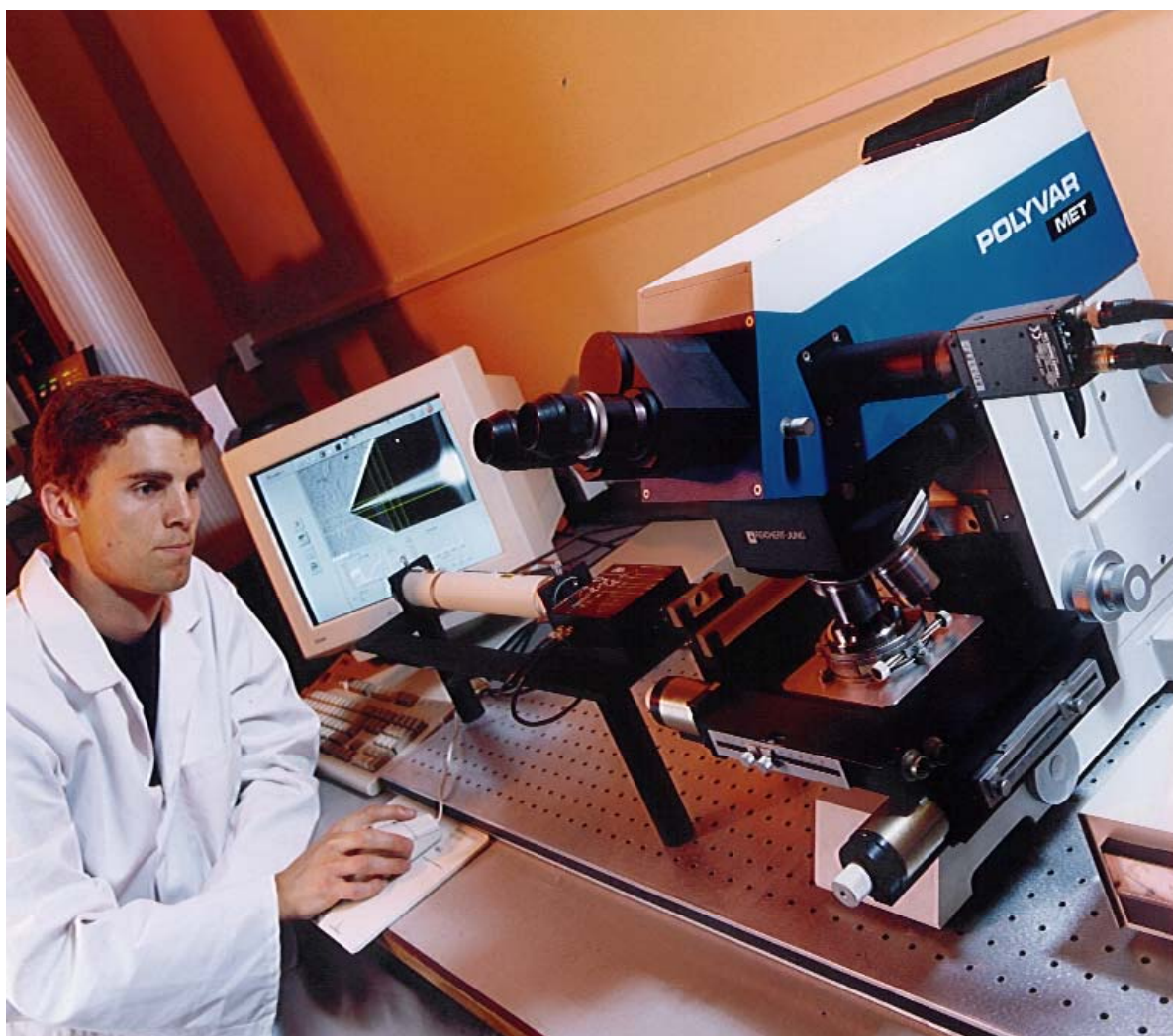


Figure 3. Indentation measurement equipment.

2.4 Indenter characterisation system.

The indenter characterisation system was developed by LTF Gal-Indent for IMGC. It covers the requirements of the standards for the measurement of Rockwell and Vickers indenters [2 & 3]. The

system (Figure 4) comprises an interferometer sine bar for the measurement of the angle of the cone of Rockwell indenters and the angle of the faces of Vickers indenters, and a rotary table for measuring the spherical tip of Rockwell indenters. Both systems are connected to a computer system that performs automatic data acquisition. The development of the indenter characterisation system is described elsewhere [8 & 9].



Figure 4. LTF Gal-Indent system.

The sine bar workstation is based on a Mirau interferometer mounted on a metallurgical microscope equipped with a x10 DI objective lens for splitting the light beam into two beams. One beam is reflected directly to the observer, whilst the second beam is directed to the observed field and then reflected to the observer. The beams recombine to generate an interference pattern. Each fringe corresponds to a height change equivalent to half the wavelength of the illuminating light source (a GIF yellow-green filter is inserted in the microscope illuminator to provide a λ value of $0.55 \mu\text{m}$). This illumination process enables both the geometry and topography of the indenter surface to be analysed.

Angular measurement of the indenter is made by tilting the indenter holder of the sine bar such that the conical end of a Rockwell indenter, or the pyramid face of a Vickers indenter, is aligned normally to the optical axis of the microscope. Normal alignment is confirmed by observation of the diffraction pattern. Examples of aligned Rockwell and Vickers indenters are shown in Figures 5 and 6 respectively.

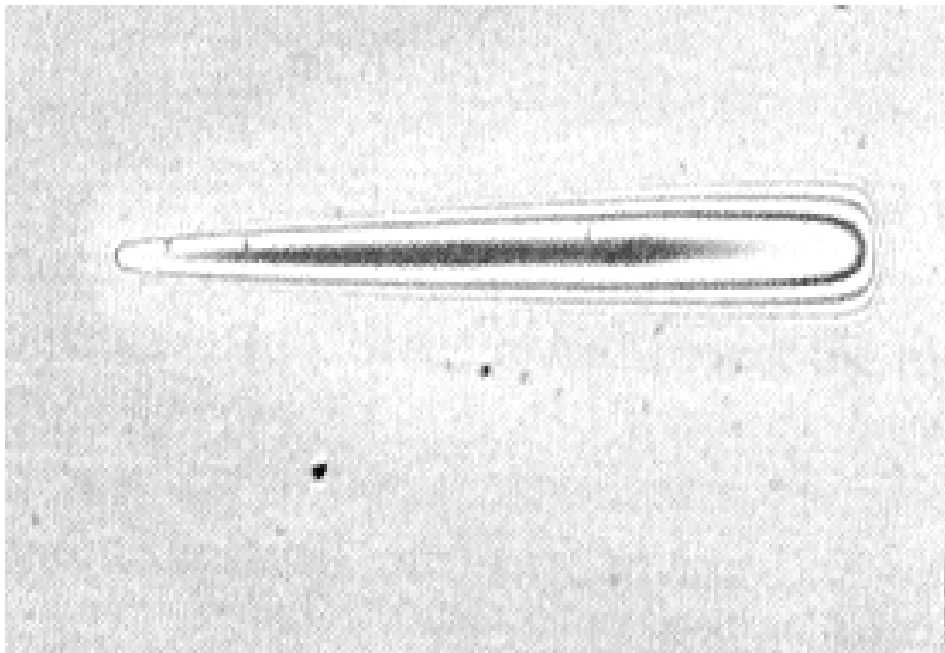


Figure 5. LTF Gal-Indent screen display of an aligned Rockwell indenter.

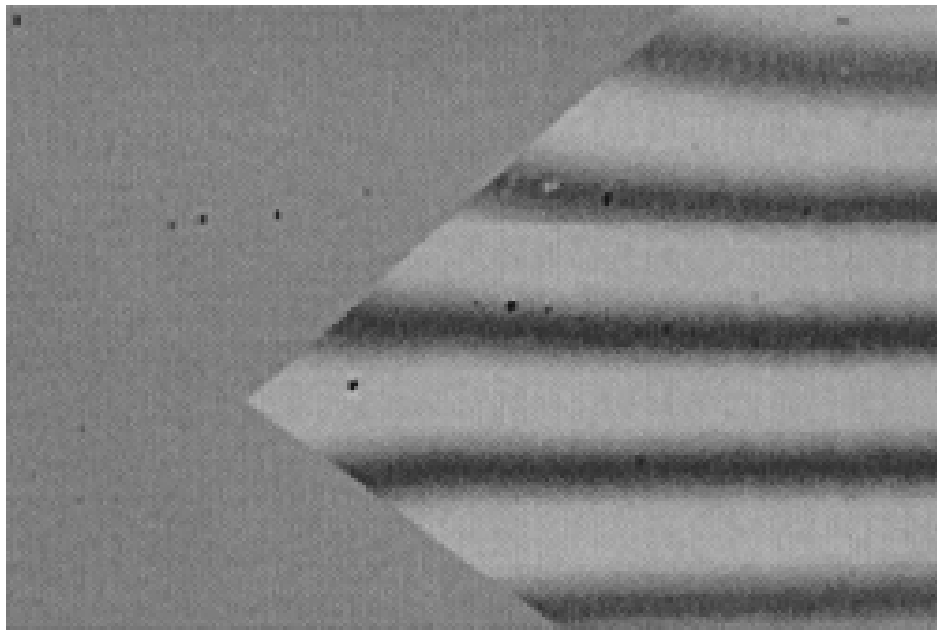


Figure 6. LTF Gal-Indent screen display of an aligned Vickers indenter.

For Rockwell indenters, the measurement fringes are observed on either two or four equi-spaced axial sections (i.e. four or eight generatrices). For Vickers indenters, all four faces of the pyramid are measured.

The geometric principle of the sine bar measurement is shown in Figure 7.

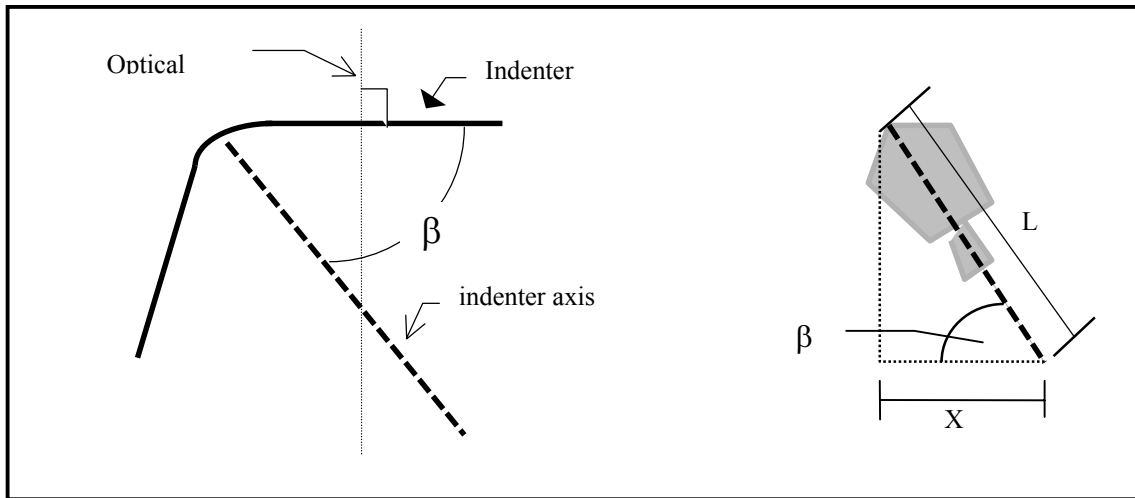


Figure 7. Geometric principle for measurement of indenter angle.

β , the angle between the axis of the indenter and the face of the indenter is calculated using equation 1.

$$\beta = \cos^{-1} (X/L) \quad (1)$$

The lateral displacement of the indenter holder (X) is measured using the Heidenhain gauge mounted on the sine bar. The hypotenuse (L) is a fixed length calibrated by NPL.

The Rockwell standard [2] specifies that the generatrix of the indenter conical angle adjacent to point where it blends with the spherical tip, should not deviate from straightness by more than 50 μm measured over a length of at least 400 μm . This measurement is made using the sine bar with the calibrated x10 DI objective lens. Figure 8 shows an example of the measurement of the straightness of the generatrix. The distance of 400 μm is indicated by the vertical lines.

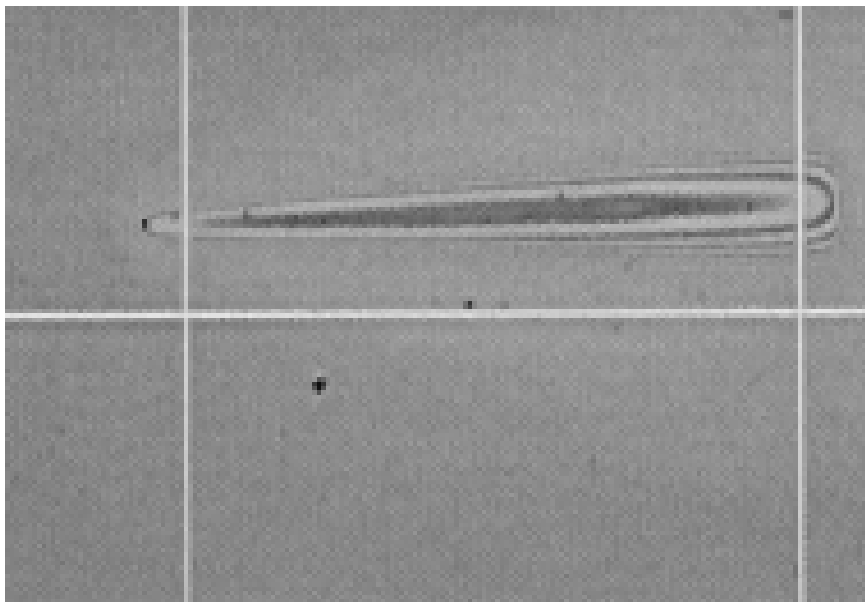


Figure 8. Measurement of the straightness of the generatrix of a Rockwell indenter.

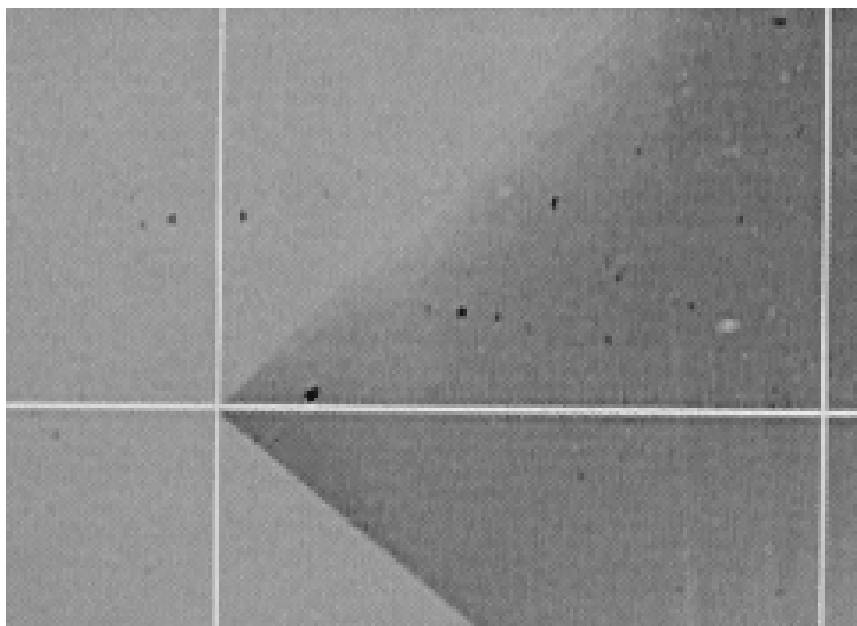


Figure 9. Measurement of the straightness of the generatrix of a Vickers indenter.

Unlike Rockwell indenters, the area over which the flatness of a Vickers indenter should be measured is not specified in the standards. Figure 9 shows an example of a flatness evaluation of a Vickers indenter, the fringe pattern being adjusted to give the minimum number of fringes on each face. For convenience, the same distance between the reference lines has been adopted as used for the Rockwell indenter.

The offset line of a Vickers indenter is determined from on-screen measurement of the chisel point using a x100 lens.

An additional requirement of the new ISO 6507-3 standard is the verification of the base squareness of a Vickers indenter. Unfortunately, at the time of purchase of the LTF Gal-Indent system this facility was not available. Subsequent development by LTF has provided a solution. The fringe

patterns on the four faces of the indenter are observed and the angle between the planes measured using an angle encoder. An assumption of this method is, that the pyramid axis and indenter holder axis coincide. Although, this is not necessarily true the error due to any misalignment is quoted as only a few tenths of a degree and is within the specified tolerance. This additional equipment is on order, and will be installed by LTF.

The tip radius of a Rockwell indenter is measured by mounting the indenter on the rotary table of the Gal-Indent system and using a LVDT that has been calibrated at NPL. The table rotates around the tip of the indenter to measure any deviation of the radius of the tip from the ideal value of $200\text{ }\mu\text{m}$. An example of a measurement is shown in Figure 10 and summary data provided in Table 2. In the example shown in Figure 10, the x-axis indicates the angular position around the circumference of the tip radius in degrees and the y-axis is a measure of the deviation of the indenter radius from the ideal value.

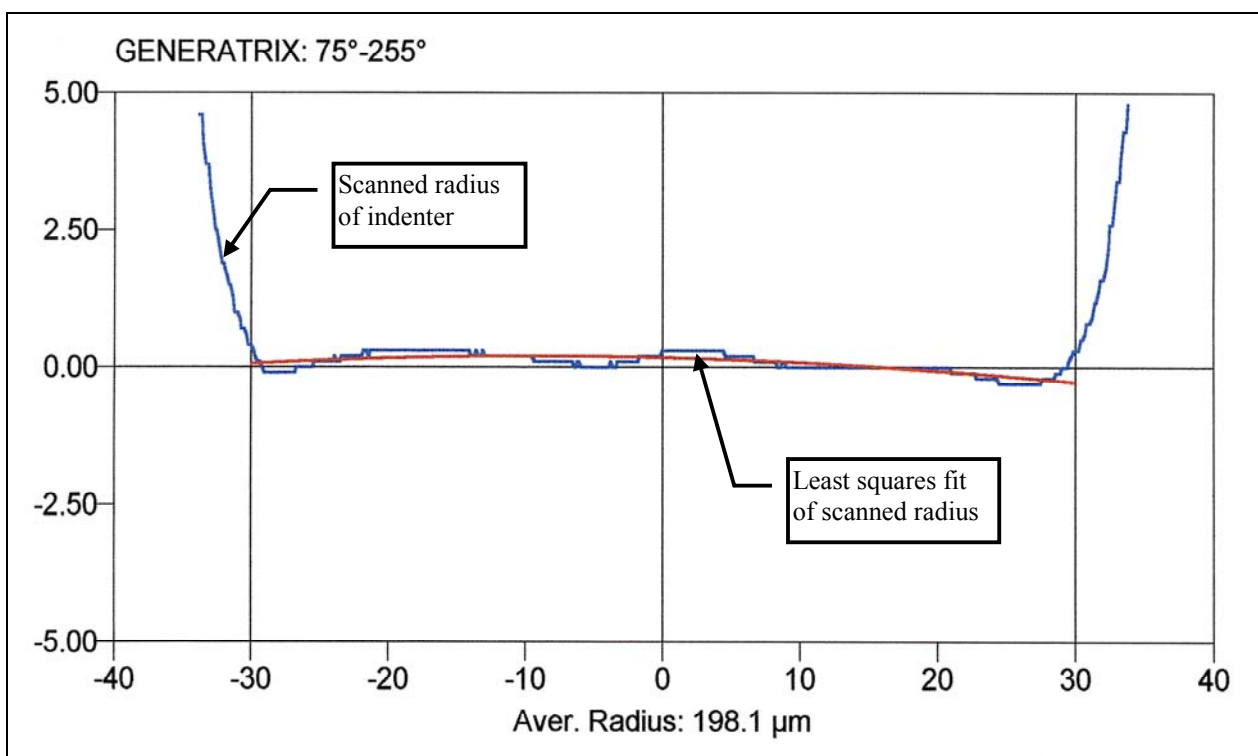


Figure 10. An example of a spherical tip radius measurement.

Table 2. Summary data of measurement of spherical radius of Rockwell indenter.

Section	Radius (μm)	Shape error (μm)	Deviation (μm)	Uncertainty (μm)
0° - 180°	200.0	0.1	0.0	0.3
15° - 195°	200.1	0.1	0.10	0.8
30° - 210°	199.8	0.1	-0.2	1.2
45° - 225°	200.2	0.1	0.2	1.5
60° - 240°	199.8	0.2	-0.2	1.6
75° - 225°	200.7	0.2	0.7	1.9
90° - 270°	200.9	0.2	0.9	2.4
105° - 285°	200.9	0.2	0.9	2.4
120° - 300°	199.7	0.1	-0.3	1.1
135° - 315°	199.5	0.1	-0.5	0.6
150° - 330°	200.0	0.1	0.0	0.6
165° - 345°	200.0	0.1	0.0	0.2
Average	200.13	0.20	0.13	1.48

3 Uncertainty in UK hardness measurement.

To establish a UK hardness calibration service of an acceptable uncertainty, the individual contributions to hardness uncertainties of the following must be assessed and minimised. There are contributions from the following:

- the standard hardness machines;
- the measurement of the indentation in both Brinell and Vickers measurement; and
- the characteristics of standard-grade indenters.

This section considers the above contributions in turn and highlights where further work is needed to reduce the contribution of uncertainty to an acceptable level.

3.1 Direct calibration of the hardness testing machines.

Calibration of the Instron hardness machines and their contribution to uncertainty of the machines' performance requires direct verification of the high precision load cell, the Heidenhain depth sensor, and the duration of the test cycle.

Indirect calibration of the hardness standard machines by intercomparison with other NMIs will provide further information on their contributions to the uncertainties.

3.1.1 Calibration of the load cell.

The application of the force by a servo- controlled screw drive can lead to two main sources of error - the measurement of the applied force and the ability of the screw drive to maintain the required force. Both of these factors were calibrated independently.

To estimate the uncertainty in the measured forces, two reference load cells were specially purchased and calibrated in NPL's 2.5 kN and 50 kN deadweight force standard machines. The load cells were placed in the hardness machines under a test block and a series of tests were carried out

in accordance with the recognised standard [10]. Figure 11 shows the calibration set up for machine 2. A similar set up was used for Machine 1.

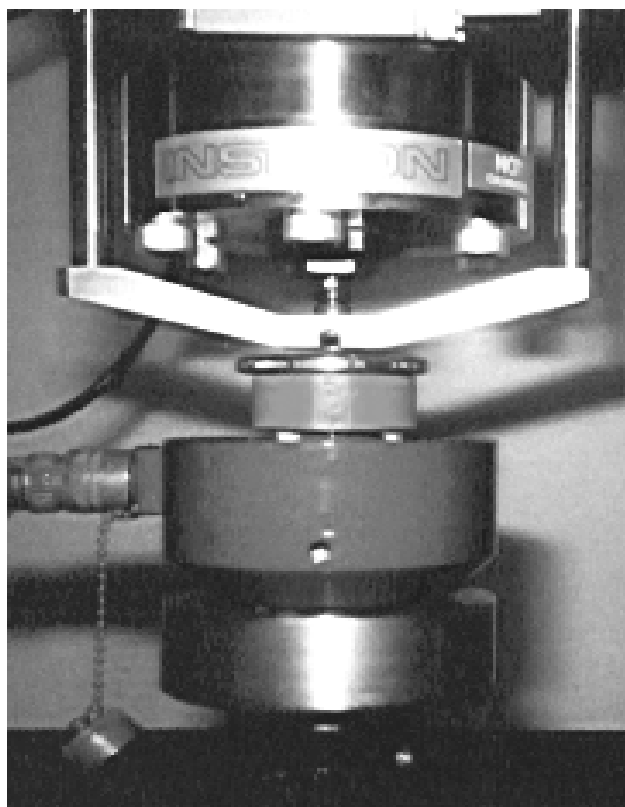


Figure 11. Machine 2, calibration of the 30 kN load cell.

The results of the initial calibration were used to make corrections to the force values of the hardness machines. The series of tests were then repeated using the corrected values. The results of these tests were used to assess the uncertainty in the measurement of the forces generated by the hardness machines. To ensure that there are no errors due to the long term drift of the load cells, the test forces are checked at monthly intervals. This is done by re-calibrating the reference load cells and then taking readings using the set up shown in Figure 11.

The uncertainty associated with the ability of the machine to maintain the correct force was assessed by analysing data recorded during a series of hardness tests. Data were recorded every 2 ms throughout the duration of a test. The uncertainty was calculated from the standard deviation of the data recorded during the application of a nominally constant test force. A sample of data taken during a test is shown in Figure 12

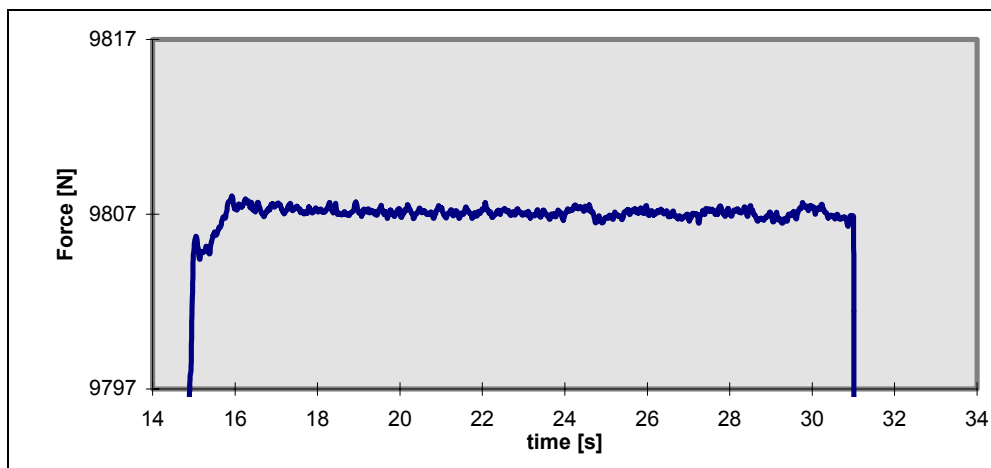


Figure 12. Data recorded during the test force of a HB 10/1 000 test.

The force contribution to the uncertainty is a combination of the ability to measure and maintain the force, the reference load cell, the national force standard machines, and the long term drift. The results for Machine 1 and 2 are shown in Tables 3 and 4, together with the force uncertainty requirements of the standards [1,2, & 3].

Table 3. Force uncertainty in Machine 1 for Rockwell and Vickers testing (95 % confidence level).

Force / kN	Maintain force / %	Measure force / %	Combined uncertainty / %	Standards requirement / %
1.5	0.016	0.010	0.030	0.100
1.0	0.020	0.010	0.034	0.100
0.3	0.024	0.015	0.059	0.100
0.1	0.024	0.028	0.082	0.100
0.03	0.120	0.075	0.280	0.200

Table 4. Force uncertainties in Machine 2 for Brinell testing (95 % confidence level).

Force / kN	Maintain force / %	Measure force / %	Combined uncertainty / %	Standards requirement / %
30	0.016	0.002	0.020	0.100
10	0.010	0.028	0.037	0.100
7.5	0.017	0.007	0.022	0.100
1.8	0.028	0.034	0.048	0.100

3.1.2 Calibration of the Heidenhain depth sensor.

Both Instron machines are fitted with Heidenhain depth sensors. On the 30 kN machine it is used to monitor the test cycle, whereas for the 1.5 kN machine it is required to measure the depth of the indentations during the Rockwell tests. For Rockwell test the Heidenhain is required to be accurate to within 0.2 μm , [2].

The positioning of the Heidenhain depth sensor on both machines is such that changes in the length of the load cell are measured as well as any changes in the position of the indenter. It was therefore necessary to evaluate the accuracy of the depth sensor, and the variation of the length of the load cell under load.

To measure the displacement of the indenter, laser light from a Jamin interferometer was reflected by a 45 degree mirror onto another mirror mounted around the indenter. This is illustrated in Figure 13. Two beams were used to ensure that the measurements were taken in line with the indenter thus avoiding the possibility of large sine errors.

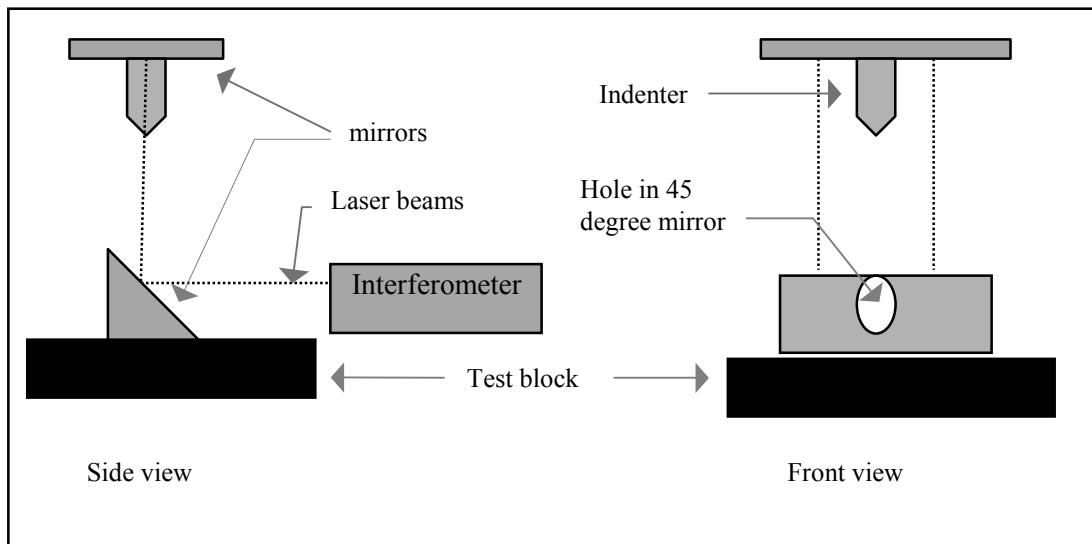


Figure 13. Simplified diagram of the set-up for calibrating the depth sensor.

The system was set up so that the laser beam exited the laser perpendicular to the motion of the indenter and, after reflecting off the 45 degree mirror, travelled parallel to the motion of the indenter. This minimised any cosine errors. A hole in the centre of the 45 degree mirror ensured that the indenter could make contact with the test block and apply a force. This enabled the evaluation of the deformation under load.

Initial results gave considerable concern over the performance of the depth sensor, and further tests only served to reinforce this concern. Although the Heidenhain gauge was functioning to the supplier specification, when mounted on the hardness machine its performance was out of specification. Figure 14 shows results of a calibration of the Heidenhain gauge against the interferometer.

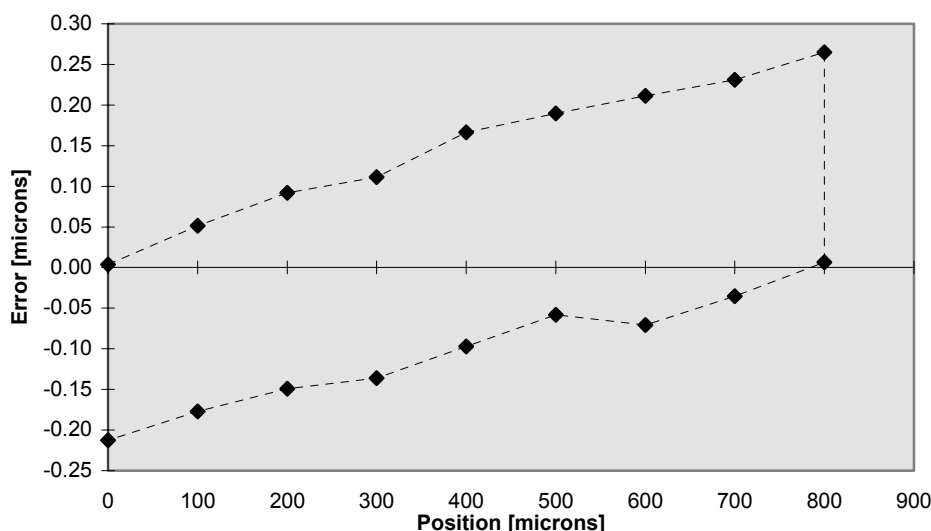


Figure 14. Calibration of the Heidenhain gauge showing hysteresis caused by the mounting of the gauge.

The two lines of data on the graph correspond to the indenter ascending and descending. The difference between the ascending and descending lines occurs when the indenter changes direction. The cause of this error was found to be the mechanical mounting of the Heidenhain gauge.

The Heidenhain was mounted on a reference cage that rested on the surface of the test block throughout the duration of a test. The reference cage had too much freedom to move, introducing errors due to the tilting of the reference cage. The friction between the bushes of the reference cage and the rods of the machine were of a level such as to cause sub-micron deformations of the cage.

The reference cage was redesigned with a view to providing lower friction, minimising tilt, and positive positioning of the Heidenhain gauge. The new design incorporated rolling element bearings which reduced the friction by a factor of two and the freedom to tilt by a factor of four. Despite this, and the reduction of Abbe errors achieved by the repositioning of the Heidenhain gauge, the uncertainties still did not meet the specifications. It was decided that, without significant improvements to the structural rigidity of the machine, it was not possible to meet the uncertainty requirements using a measurement system that was positioned at such a large distance from the actual measurement.

As part of the NMS Programme for Mass 1999-2002, an improved laser based measurement system to replace the Heidenhain gauge has been designed at NPL. The system is currently being manufactured. It measures the position of the indenter relative to the base of the test block by mounting corner cubes on either side of the indenter and measuring their position relative to a platform mounted on the anvil of the hardness machine.

3.1.3 Calibration of the duration of the test cycle.

The test cycle has an effect on the final measured hardness [11]. The greatest effects are produced by variations in dwell time and final approach velocity. The timing mechanism of both hardness machines was calibrated and the repeatability of each section of the timing cycle was measured.

To ensure that the force profiles were as close to the ideal cycle as possible, data from six tests for each of the hardness ranges were recorded. The results were analysed to determine the timing and

velocity of the different sections of the tests. The results are shown in Table 5, and are all well within the limits set in the standards [1, 2, & 3].

Table 5. Characteristics of the test cycle.

Test	Value	Uncertainty, 95 %
Brinell		
Application time	7.0s	0.2 s
Hold time	12.0 s	0.4 s
Rockwell C		
Initial preliminary load hold time	2.5 s	0.2 s
Velocity of application of total test force	0.04 mms ⁻¹	0.005 mms ⁻¹
Total test force hold time	4.0 s	0.3 s
Final preliminary load hold time	3.0 s	0.2 s
Vickers		
Application time	7.0 s	0.2 s
Hold time	14.0 s	0.3 s

3.2 Calibration of the indentation measurement system.

Brinell and Vickers indentations are evaluated using the indentation measurement system as described in Section 2.3. The main sources of uncertainty are due to the imaging of the indentation, the identification of the edges/vertices of the indentation, and the actual measurement of the distance between the two opposite edges/vertices.

3.2.1 Uncertainty in the measurement of the distance between edges/vertices.

The value for the diameter of a Brinell indentation or the diagonal of a Vickers indentation is a combination of two measurements. These are the movement of the microscope stage and the measurement of the distance between the centre of the screen and the two edges of a Brinell indentation or two corners of a Vickers indentation.

A Jamin interferometer measures the movement of the microscope stage. The frequency of the helium-neon laser used for the interferometer was calibrated against NPL's frequency standards and found to be stable to within 1 part in 10⁸. The readings from the interferometer were compared with a stage graticule and agreed within the uncertainty of the graticule (0.02 %). The movement of the stage was monitored to ensure that it was parallel to the laser beam.

Measurements made by the image analyser were calibrated using two methods. The first was to use a stage graticule and the second was to use the interferometer. The interferometer was a more accurate method but was only able to calibrate the x-axis. The image calibration was performed 5 times using each microscope objective. The repeatability was better than 0.5 %. When measuring an indentation, the contribution from the on screen measurement is typically less than 1 % of the indentation width, which gives a contribution to the overall uncertainty of the indentation diameter of less than 0.005 %.

There are no guidelines for imaging and identifying the edge of an indentation. The uncertainty was based on the repeatability of an indentation measurement rather than any deviation from a true value. A set of indentations were produced on a selection of materials using a range of forces. The same set of indentations were imaged by the same operator three times, to give an indication of the repeatability of the measurement. A different operator following the same procedure then measured the same set of indentations to assess any influence by the operator. The results of the measurement of the Brinell indentations revealed that the repeatability was less than 0.2 %.

3.3 Calibration of the indenter characterisation system.

Indenters are characterised by their angular geometry measured using the interferometer sine bar. Rockwell indenters are also characterised by their tip radius measured using the rotary table.

3.3.1 Calibration of the interferometer sine bar.

The angle is calculated from this horizontal displacement of the sine bar using a Heidenhain gauge. The Heidenhain gauge was calibrated by NPL. The resolution of the sine bar is 0.5 minutes of arc with a reproducibility of 1.5 minutes of arc, the hypotenuse (L) shown in Figure 7 has been calibrated by LTF and verified by comparison with IMGC [12]. Straightness of the generatrices of Vickers and Rockwell indenters are measured by analysis of the interference fringes using the calibrated x10 DI objective lens which was calibrated using a calibrated graticule. This measurement gives the flatness of the face/cone to within 0.5λ or $0.28 \mu\text{m}$.

Measurement of the line of conjunction between the faces of a Vickers indenter is limited by the resolution of the microscope. The measurement is made using the x100 objective lens which was calibrated using a calibrated graticule.

3.3.2 Calibration of the rotary table.

The tips of Rockwell indenters are measured by comparison with a calibrated ruby sphere [12] of $200 \mu\text{m}$ radius. The LVDT that measures the deviation was calibrated against NPL length standards.

3.4 Overall uncertainty in UK hardness measurement.

The uncertainty of the 1.5 kN hardness tester, used for Rockwell and Vickers hardness testing, will be re-assessed when the new depth sensor has been installed. Results from the work carried out in its current state combined with the expected uncertainty of the new depth sensor indicate that the uncertainty of Rockwell measurement will be less than 0.3 HR and the uncertainty in Vickers hardness associated with the production of Vickers indentations will be less than 0.02 %.

The 30 kN hardness machine can produce the required indentations for Brinell testing with a contribution to the uncertainty in measured hardness of less than 0.25 HB.

The uncertainty in the value of the measured hardness due to the measurement of the indentations is less than 0.2 %. The majority of this uncertainty is due to the repeatability of the measurement procedure.

For Brinell and Vickers it is the measurement of the indentation that is the source of the majority of the uncertainty in the hardness value. When this uncertainty is combined with the uncertainty in the production of the indentation the overall uncertainty due to the national standard machines is less than 2 HB for the four supported Brinell scales. For the supported Vickers scales the uncertainty in

the measurement of the hardness of soft materials (hardness in the region of 250 HV) is less than 3 HV and for hard materials (hardness in the region of 2000 HV) the uncertainty is less than 40 HV.

With the exception of the superficial Rockwell scales, these uncertainties and expected uncertainties for the three methods, are comparable with those of other NMI's [13 & 14] and are better than those required by the relevant standards [1,2, & 3].

The overall uncertainty in blocks calibrated at NPL will be a combination of the uncertainty due to the national standard hardness machines and the uncertainty due to the non-uniformity of the hardness block. The non-uniformity of the hardness block will be calculated from the spread of the five tests performed to calibrate the block.

4 Establishment of UK hardness calibration service.

The ability to make certifiable hardness calibrations for Brinell was established at the end of March 2000. However the results of intercomparisons with IMGC show disagreement greater than the combined uncertainties of the two organisations by a factor of 2 and further work is already underway as part of the NMS programme for Mass 1999-2002 to identify the cause. For this reason, the service has not yet been actively promoted. A calibration service for the measurement of indenters has also been established but further intercomparisons are needed before it is fully verified.

The scope of the measurement services is scheduled to be extended in May 2000. This service will provide Brinell and Vickers standards [1 & 3] hardness calibration, as listed in Table 1. The service will also provide Rockwell and Vickers indenter characterisation, as described in references [2 & 3]. The additional Vickers indenter requirement for the verification of the base squareness, described in the new ISO 6507-3 standard is scheduled for September 2000. Rockwell standards, as listed in Table 1 and of an acceptable uncertainty will be dependent on the performance of the improved laser based indentation depth measuring system. This is scheduled for delivery in May 2000. It is therefore scheduled that the supported Rockwell standards will be available June 2000.

Further information on the UK hardness calibration service please contact the authors of this report:

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6 References.

- [1] BS EN ISO 6506 - 1, 2 & 3 : 1999, Metallic materials -Brinell hardness test
- [2] BS EN ISO 6508 - 1, 2 & 3 : 1999, Metallic materials -Rockwell hardness test
- [3] BS EN ISO 6507 - 1, 2 & 3 : 1998, Metallic materials -Vickers hardness test
- [4] BS EN ISO 10002-3 Tensile testing of metallic materials- part 3 Calibration of force proving instruments used for the verification of uniaxial testing machines.
- [5] ISO/CD 14577 - 1, 2 & 3 : 1999, Metallic materials - Instrumented indentation test for hardness and other material properties, Draft document.
- [6] TestProfiler Option for Merlin II, Instron publication, 1997.
- [7] Leigh I C, The micro-indentation hardness test: The practical realisation of a standard Ph.D. Thesis, University of Surrey, 1983.
- [8] Turotti F, Ligorì A and Gori G, LTF Spa contribution to the standardization of hardness measurements, HARDMEKO '98, Proceedings of International Symposium on Advances in Hardness Measurement, Beijing, China 21-24 September 1998, p. 41-46.
- [9] Barbato G and Desogus S, Measurement of the spherical tip of Rockwell indenters, Journal of Testing and Evaluation, JTEVA, v. 16, No. 4, July 1988, p. 369-375.
- [10] ISO 376 : 1999(E) - Metallic materials - Calibration of force proving instruments used for the verification of uniaxial testing machines.
- [11] Barbato G, Desogus S and Germak A, Experimental analysis on the influence quantities in the Rockwell C hardness test. HARDMEKO '98, Proceedings of International Symposium on Advances in Hardness Measurement, Beijing, China 21-24 September 1998, p. 67-73.
- [12] LTF Technical Report No. V9801-1, 15 October 1998.
- [13] World wide Unified Scales Rockwell Hardness test with Conical indenter, Results of the round robin test, differences between the laboratories, T Polzin, D Schwenk, December 1999.
- [14] Guideline to the determination of the uncertainty of the Brinell and Vickers measuring method, PTB, August 1999.