THE UNCERTAINTY EVALUATION OF NPL’S HARDNESS FACILITY

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Abstract: To understand the uncertainties in the calibration of hardness test blocks by NPL, it was necessary to evaluate the main factors contributing to the uncertainty in measurements made with the NPL hardness calibration machines. These factors were identified and then, where possible, directly verified. Factors that could not be directly verified were assessed using the best available information.

The verifications covered the force required to produce hardness indents, the measurement of the size of these indents, and the characterisation of the indenters.

As a result of the initial verification, modifications were made to the depth sensor on the Rockwell hardness tester.

Key Words: Calibration, Hardness testing, Uncertainties

1 INTRODUCTION
In 1995 it was decided that a UK national standard for Hardness should be re-established. By the end of 1998, NPL had purchased two hardness machines, a system for characterising indenters, and equipment for measuring Brinell and Vickers indentations [1]. This paper deals with the calibration of these four pieces of equipment.

For each piece of equipment it was necessary to identify the possible sources of error and then decide how to evaluate their influence. The calibrations of the hardness machines were designed, where possible, to provide traceability to the primary standards held at NPL, so that the uncertainty in the final measured hardness could be minimised.

2 MEASUREMENT SYSTEMS REQUIRING CALIBRATION
• Instron hardness testing machine capacity 1.5 kN for Vickers and Rockwell testing.
• Instron hardness testing machine capacity 30 kN for Brinell testing.
• Graftek indentation measurement system.
• LTF Gal indent Indenter characterising equipment.

3 CALIBRATION OF INSTRON HARDNESS TESTING MACHINES

3.1 Force Calibration
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.

3.1.1 Uncertainty due to the measurement of the force by the hardness machine
To estimate the uncertainty in the measured force, two reference load cells were used for the two machines (calibrated in NPL’s 2.5 kN and 50 kN national 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 [2]. The set up used is shown in Figure 1.

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 force by the hardness machines. The results are shown in Table 1.

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 in NPL’s force standard machines and then taking readings using the set-up shown in Figure 1.
3.1.2 Uncertainty due to application of force by hardness machine

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 during the test force. A sample of data taken during a test is shown in Figure 2.

3.1.3 Results of force calibrations

The results of the evaluations of 3.1.1 and 3.1.2 are shown in Tables 1 and 2. The total uncertainty is a combination of not only the ability to measure and maintain the force but contains contributions from the reference load cell, the national force standard machines, and the long term drift.

Table 1. Main uncertainties in the forces applied for Brinell testing (95 % confidence level).

<table>
<thead>
<tr>
<th>Force / kN</th>
<th>Maintain force / %</th>
<th>Measure force / %</th>
<th>Total uncertainty / %</th>
</tr>
</thead>
<tbody>
<tr>
<td>30</td>
<td>0.016</td>
<td>0.002</td>
<td>0.020</td>
</tr>
<tr>
<td>10</td>
<td>0.010</td>
<td>0.028</td>
<td>0.037</td>
</tr>
<tr>
<td>7.5</td>
<td>0.017</td>
<td>0.007</td>
<td>0.022</td>
</tr>
<tr>
<td>1.8</td>
<td>0.028</td>
<td>0.034</td>
<td>0.048</td>
</tr>
</tbody>
</table>
### Table 2. Main uncertainties in the forces applied for Rockwell and Vickers testing (95 % confidence level).

<table>
<thead>
<tr>
<th>Force / kN</th>
<th>Maintain force / %</th>
<th>Measure force / %</th>
<th>Total uncertainty / %</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.5</td>
<td>0.016</td>
<td>0.010</td>
<td>0.030</td>
</tr>
<tr>
<td>1.0</td>
<td>0.020</td>
<td>0.010</td>
<td>0.034</td>
</tr>
<tr>
<td>0.3</td>
<td>0.024</td>
<td>0.015</td>
<td>0.059</td>
</tr>
<tr>
<td>0.1</td>
<td>0.024</td>
<td>0.028</td>
<td>0.082</td>
</tr>
<tr>
<td>0.03</td>
<td>0.120</td>
<td>0.075</td>
<td>0.170</td>
</tr>
</tbody>
</table>

### 3.2 Depth sensor calibration

Both Instron machines are fitted with depth sensors. On the 30 kN machine it is used to monitor the test cycle. For the 1.5 kN machine it is required to measure the depth of the indents during the Rockwell tests.

#### 3.2.1 Calibration method

The positioning of the depth sensor on both machines is such that changes in the height of the load cell are measured as well as any changes in the position of the indenter. It was therefore necessary to evaluate two things: i) the accuracy of the depth sensor and ii) the influence of the variation of the height of the load cell during the test.

To measure the displacement of the indenter, laser light from a Jamin interferometer was deflected off a 45 degree mirror onto another mirror that was fixed round the indenter, (see Figure 3). Two beams were used to ensure measurements were taken 'in Abbe' thus avoiding large sine errors.

![Figure 3. Simplified diagram of the set-up for calibrating the depth sensor](image)

The system was set up so that the laser beam exited the laser perpendicular to the motion of the indenter and after hitting 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.

#### 3.2.2 Results of depth calibration

Initial results gave concern and further tests only served to reinforce this concern. The conclusion reached was that although the Heidenhain gauge was functioning satisfactorily, when mounted on the hardness machine it did not meet the requirements of the specification. Figure 4 shows results of a calibration of the Heidenhain gauge against the interferometer which pointed to the fact that the mounting was the problem.

The two lines of data on the graph correspond to the indenter ascending and descending. The difference between the ascending and descending occurs when the indenter changes direction. The most likely cause of this error is the mechanical mounting of the Heidenhain gauge.
The design of machine and the nature of the hardness test means that it is difficult to use the Heidenhain gauge in the optimum configuration (directly in-line with the indenter). It was decided to use an alternative measurement method that can be fitted in a more positive location.

The Heidenhain gauge has now been replaced by a laser based measurement system. The system is designed to measure the position of the indenter relative to the base of the test block. This is achieved by mounting corner cubes either side of the indenter and measuring their position relative to a platform mounted off the anvil of the hardness machine. The verification of the new system is under way.

### 3.3 Test cycle calibration

It is well known that a crucial factor in hardness measurement is the loading cycle. The timing mechanism of both hardness machines was calibrated and then the repeatability of each section of the timing cycle was measured.

To ensure 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 3.

**Table 3.** Characteristics of the test cycle.

<table>
<thead>
<tr>
<th></th>
<th>Value</th>
<th>Uncertainty, 95 %</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Brinell</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Application time</td>
<td>7.0 s</td>
<td>0.2 s</td>
</tr>
<tr>
<td>Hold time</td>
<td>12.0 s</td>
<td>0.4 s</td>
</tr>
<tr>
<td><strong>Vickers</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Application time</td>
<td>7.0 s</td>
<td>0.2 s</td>
</tr>
<tr>
<td>Hold time</td>
<td>14.0 s</td>
<td>0.3 s</td>
</tr>
<tr>
<td><strong>Rockwell C</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial preliminary load hold time</td>
<td>2.5 s</td>
<td>0.2 s</td>
</tr>
<tr>
<td>Velocity of application of total test force</td>
<td>0.04 mms⁻¹</td>
<td>0.005 mms⁻¹</td>
</tr>
<tr>
<td>Total test force hold time</td>
<td>4.0 s</td>
<td>0.3 s</td>
</tr>
<tr>
<td>Final preliminary load hold time</td>
<td>3.0 s</td>
<td>0.2 s</td>
</tr>
</tbody>
</table>

![Figure 4. Calibration of the Heidenhain gauge showing hysteresis caused by mounting of the gauge.](image)
4 INDENTATION MEASURING EQUIPMENT

Indentations produced during Vickers and Brinell tests are evaluated using the ‘indentation measuring equipment’. The main sources of uncertainty are due to the imaging of the indent, the identification of the edges/vertices of the indentation, and the actual measurement of the distance between the two opposite edges/vertices.

4.1 Uncertainty in measurement of the distance between edges/vertices

The value for the diameter/diagonal of the indentation is obtained by a combination of the measurement of the movement of the stage and the measurement of the positions of the two edges/corners on the screen.

The measurement of the movement of the stage is performed by a Jamin interferometer. 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 $10^8$. The overall uncertainty of the interferometer is less than 100 nm. The movement of the stage was monitored to check that there was no angular deviation with respect to the interferometer.

To calibrate the image seen on the screen two methods were used. 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 uncertainty of the calibration was found to be better than 0.5 %. Distances measured on the screen are less than 10 $\mu$m giving an uncertainty, due to the on screen component, of less than 50 nm.

4.2 Uncertainty in the imaging of the indentation

As there are no guide lines for imaging and identifying the edge of an indent, the uncertainty was based on the repeatability of the test 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 indents to assess any influence of the operator. (The results of these calibrations will be presented at the conference.)

Further information on the accuracy will be obtained by means of an intercomparison with other NMIs.

5 INDENTER CHARACTERISING EQUIPMENT

5.1 Measurement of geometry of indenters using the sine bar

The measurement of the angle of the cone of Rockwell indenters and the angle of the faces of Vickers indenters is achieved using the sine bar. The indenter is tilted until the face/edge is perpendicular to the lens of the microscope. A Heidenhain gauge measures the horizontal displacement of the base of the sine bar, (see Figure 5). The angle is then calculated from this horizontal displacement, $d$, and the length of the sine bar, $L$. Uncertainties were minimised by calibration of the Heidenhain gauge, accurate measurement of the length of the sine bar, and ensuring correct alignment of the indenter before taking a reading.

![Figure 5. Measurement of indenter angle](image)

The resolution of the system is 0.5’ of arc with a reproducibility of 1.5’ of arc [3].
Analysis of the interference fringes on the faces of Vickers indents gives the flatness of the face to within \( \frac{1}{2} \lambda \) or 0.28 \( \mu \)m. The offset line measurement is limited by the resolution of the microscope.

5.2 Measurement of the radius of the diamond tip of Rockwell indenters

The tips of Rockwell indenters are measured by comparison with a ruby sphere having a ‘perfect’ 200 \( \mu \)m radius. The LVDT that measures the deviation was calibrated against NPL length standards as was the Ruby sphere.

6 CONCLUSIONS

This paper has shown how the main sources of uncertainty in hardness measurement were identified and then calibrated. Details of the methods that were used to calibrate each of these parameters have been given. The results of the calibrations revealed that, with the exception of the depth sensor on the 1.5 kN hardness machine, the measurement systems had acceptable uncertainty levels for the calibration of test blocks. As a result of the calibrations a new depth sensor has been fitted to the 1.5 kN hardness machine.

Work is ongoing on how the above parameters influence the measurement of the hardness of test blocks. Further work is planned to expand the range of scales and to contribute to international hardness measurement.

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REFERENCES


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