

ESTABLISHMENT OF A MARTENS HARDNESS CALIBRATION SERVICE

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Abstract – As part of the UK's National Measurement System Mass Programme, and as a result of industrial demand, it was decided to establish a Martens hardness calibration service.

This report describes the basis of Martens hardness measurement, work done to determine the ability of NPL's 1,5 kN hardness machine to measure Martens hardness, the process of establishing the calibration service, and the setting up of instrumented indentation hardness tests.

Keywords: Hardness, Martens

1. INTRODUCTION

Conventionally the hardness of a material is a measure of its resistance to permanent deformation caused by a force applied to its surface through an indenter, with the measurement made after the removal of the test force, such that elastic deformation is ignored.

Instrumented indentation hardness provides the ability to measure the indenter penetration h under the applied force F through the testing cycle and is therefore capable of measuring both plastic and elastic deformation of the material under test.

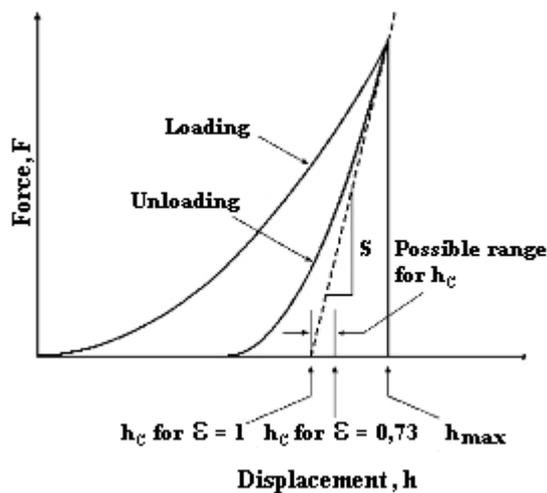


Fig. 1. Typical force/displacement curve

The Martens hardness value is obtained by dividing the maximum test force F by the surface area of the indenter

penetrating beyond the original surface of the test piece $A_s(h)$ [1].

To set up a Martens hardness calibration service, several areas first need to be investigated. These included the frame compliance of the hardness machine, thermal drift effects within the system/depth measuring device, the best way to analyse the data files, the area function of the indenter, and the timing and profile best suited to running a Martens hardness test [2].

This paper describes the work done to set up a Martens hardness calibration service, the problems found in the process, and their resolution.

2. EQUIPMENT

The work was performed in NPL's 1,5 kN hardness standard machine (Fig. 2). A high accuracy load cell, traceable to NPL force standards, measures the applied force. Indenter depth measurement is by a laser interferometer system, traceable to the UK realisation of the metre at NPL [3].

The machine is PC-controlled and uses generalised waveforms, under closed loop control, to run standard indentation profiles.

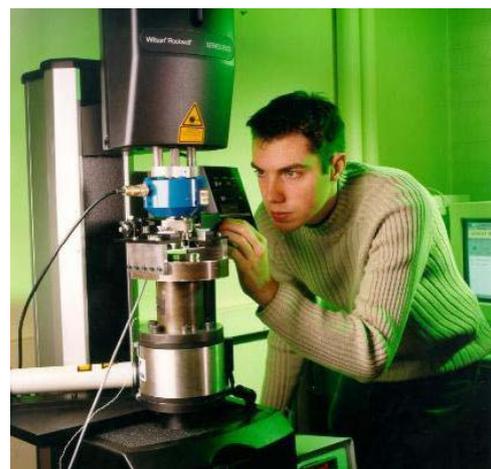


Fig. 2. NPL 1,5 kN hardness machine

The Instron Merlin™ software used to control the machine streams data from all input channels - depth, force,

time etc. - at a sample rate selected by the user throughout the hardness tests. This data is stored as a text based file.

3. WORK

3.1 Frame compliance:

The applied test force not only acts on the test piece surface, it also acts on parts of the testing machine and these are elastically deformed.

This elastic deformation causes an increase in the measured indentation depth, which is not experienced at the indentation contact, but occurs between the reference planes in the testing machine.

This additional compliance must be taken into account as it acts directly to increase the measured value of h . With instrumented indentation tests it must be taken into account at all forces as it can decrease the gradient of the tangent to the removal curve of the test force. The increase in measured h is especially significant at high forces.

Due to the construction of the 1,5 kN hardness machine, the machine compliance should be very low, as the indentation depth is measured via a laser interferometer, at a point as close to the indenter tip as possible.

An estimate for the frame compliance of the 1,5 kN hardness machine was obtained by experimentation.

The experiment used a hardness block of known Young's modulus and a 5 mm diameter hardmetal (cemented carbide) ball indenter. A series of tests were made at different forces spanning the range of the machine.

Within the elastic region of indentation of the test block, the frame compliance was calculated by taking the difference between the measured depth at the maximum force, and the theoretical depth (y) that the indenter should reach, due to the elastic properties of the block and the ball indenter, based on the Young's modulus and Poisson's ratio of the block and indenter, the applied force, and the diameter of the indenter.

The following equation [4] was used:

$$y = 1,040 \times \sqrt[3]{\frac{P^2 C_E^2}{D}} \quad (1)$$

$$\text{where } C_E = \frac{1 - \nu_1^2}{E_1} + \frac{1 - \nu_2^2}{E_2}$$

Subscripts refer to the two bodies (1 = the block, 2 = the indenter), ν = Poisson's ratio, E = modulus of elasticity, P = force, and D = diameter of indenter. The elastic parameter values used were: $E_1 = 207$ GPa, $E_2 = 540$ GPa, $\nu_1 = 0,3$, and $\nu_2 = 0,26$.

For the forces in the plastic region, the indentations made were measured optically using a laser interferometer and image analysis system and this measurement, along with the calculated elastic recovery of the block, was included in the adjustment of the test depth measurement.

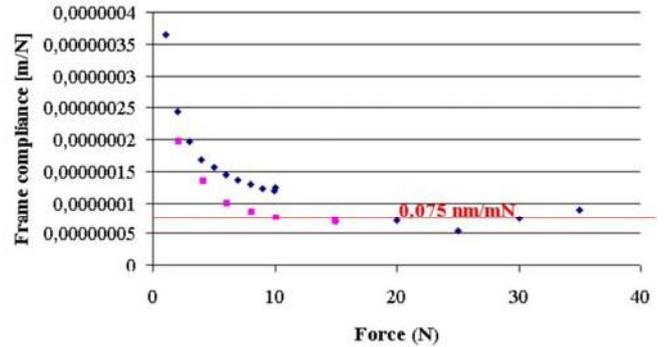


Fig. 3. Frame compliance results

The frame compliance value of $0,075 \text{ nm} \cdot \text{mN}^{-1}$ shown in Fig. 3 was initially used. However, when this value was used to correct the indentation data for an indentation into steel, the force removal curve slope became negative indicating that the frame compliance was too large. A second estimate was made to gain a match with the modulus for the steel block. This second test estimate was $0,008 \text{ nm} \cdot \text{mN}^{-1}$, an order of magnitude lower than the first estimate, and was the value finally used. The reason for the difference was unknown, although it could have been partly due to the difference in compliance between the Vickers indenter used in this test and the ball indenter used for the frame compliance tests.

3.2 Signal conditioning:

During these series of tests, examination of the force/displacement curves showed some strange recovery effects on removal of the forces. This problem was found to be due to a difference in bandwidth between the load and depth channels of the hardness machine. The bandwidth of the force collected by the Merlin™ software is 10 Hz while the depth data has a bandwidth of 1,75 kHz. This causes the force to lag behind the depth; the faster the speed, the greater the lag. To adjust for this problem, a second order 10 Hz filter was used to adjust the data output from the Merlin™ software. As can be seen (Fig. 4 & Fig. 5), this resulted in a much improved force/displacement curve.

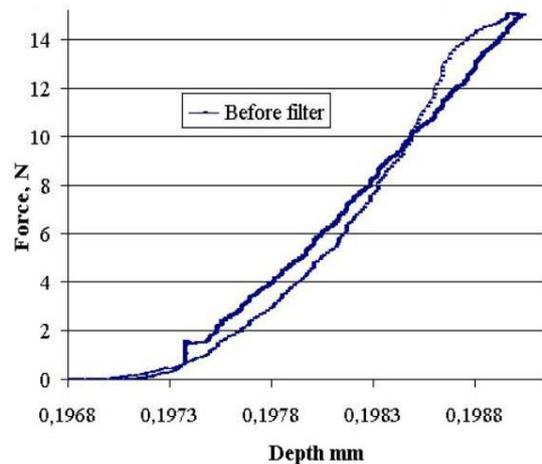


Fig. 4. Force/displacement curve, before filtering

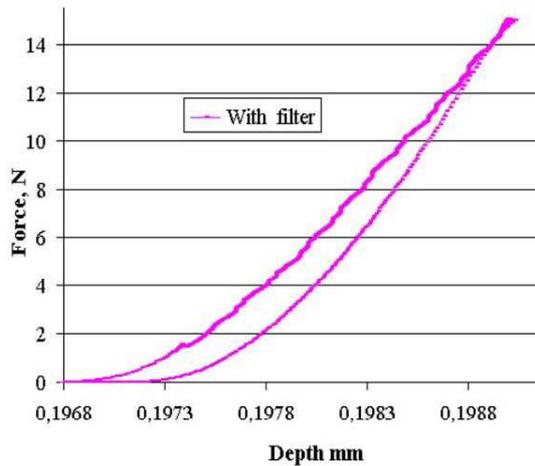


Fig. 5. Force/displacement curve, after filtering

3.3 Indenter area function:

The Martens hardness value as well as data from the instrumented indentation test is based on the contact area of the indenter, but only the indentation depth is measured. Crucial differences can be found when comparing the actual contact area with the area calculated assuming an ideal indenter geometry.

To maintain reproducibility of the results it is necessary to determine the actual contact area and use it in the calculation of the materials parameters.

The area function of the Vickers indenter used was calculated from the certified geometric calibration of the indenter but assumed no rounding of corners or the tip.

Base squareness angles were 89,90°, 90,00°, 90,03°, and 90,07°. The angles between opposite faces of the indenter were 136,07° and 136,08° giving an average facet angle of 68,04°. The length of line of conjunction between opposite faces was 370 nm. Angular uncertainties, at a 95 % confidence level, were ±0,065°.

Within the stated measurement uncertainty, the base is square and the indenter is mounted with no tilt from the vertical indentation axis. Inputting these geometries into a MathWorks MATLAB® programme, designed for this use, the area function was calculated to be:

$$A(h) = 4 h^2 \tan^2 68,04^\circ + 370 h \tan 68,04^\circ \quad (2)$$

3.4 Thermal drift:

Time dependent drift in the readings of the depth sensor will cause discrepancies in the measurements. Hold periods at a low force before and after the main force application were used to take readings to monitor this and adjust the data accordingly.

Due to a difficulty in monitoring the thermal drift, it was necessary to have a 60 s hold for the pre-load and final load. This problem in the thermal drift is thought to be due to a resolution limit within the system, creating steps within the depth measurement. The steps are large enough that only two or three can be resolved over the drift experienced in the

longer 60 s hold period. The thermal drift was therefore corrected by averaging over the 60 s pre-load hold.

The depth measurement resolution is due to the laser using a channel originally specified for a Heidenhain depth sensor. A software upgrade is currently being performed to increase the available resolution and improve both the monitoring of thermal drift and also the standard depth data.

3.5 Load cell creep effect:

Any creep effect within the load cell also needed to be assessed, as this would not be visible within the data. Isolating this ensures a better analysis of the thermal drift of the depth sensor. To do this, a reference load cell was used to monitor the force. This reference load cell was initially calibrated within NPL's 2,5 kN deadweight force standard machine and the creep response was recorded. A smaller load cell also had its creep response recorded using calibrated hand weights. Using both these load cells within the 1,5 kN hardness machine, it was possible to identify any error caused by creep response within the machine's load cell.



Fig. 6. Machine set up for creep analysis work

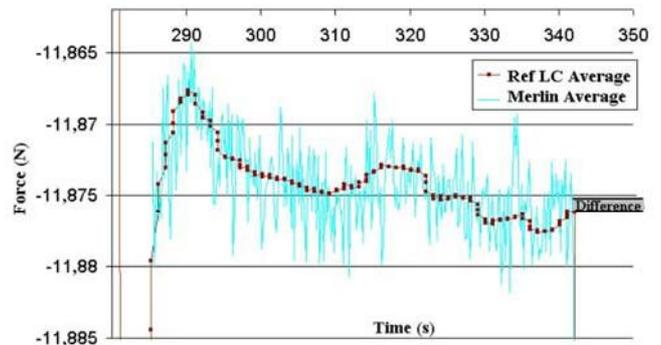


Fig. 7. Creep analysis results

Fig. 7 demonstrates that any error caused by the creep response of the load cell appears to be insignificant compared to the resolution of the load control.

3.6 Zero point assignation:

For the actual test, the Merlin™ software was set to record data at a rate of 20 Hz and, the approach velocity for contact was set to 0,005 mm·sec⁻¹. This combination sample rate and contact velocity enabled a reasonable approximation of the zero point to be made, from analysis of the graph.

4. RESULTS

A MATLAB® programme was written and used to extract the relevant data from the text file output of the Merlin™ software. This MATLAB® programme is also used to apply the second order 10 Hz filter to adjust for the force/depth data lag, as well as to convert the data into a format which allows a further MATLAB® programme, used for data analysis to gain the required results from the data. The second MATLAB® programme adjusts for frame compliance and calculates the results using the input area function (2). This has also enabled us to carry out further instrumented indentation work, and to be involved in the EC DESIRED instrumented indentation project.

Using the final set up, a series of tests was made on a steel test block with increasing forces, the results being shown in Fig. 8. Reasonably repeatable results are shown as demonstrated by the similar track of the force application curves and the resulting Martens hardness values given in Fig. 9.

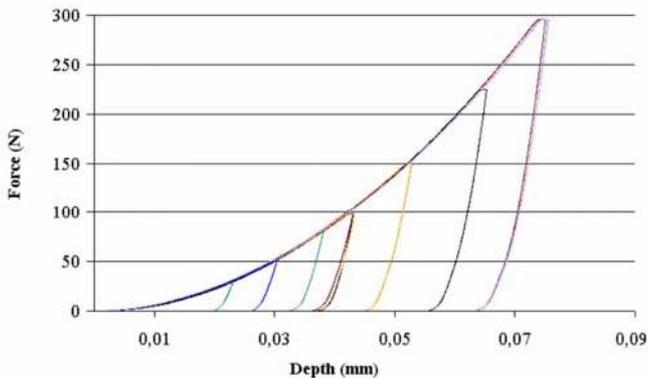


Fig. 8. Force/depth curves for Martens hardness tests

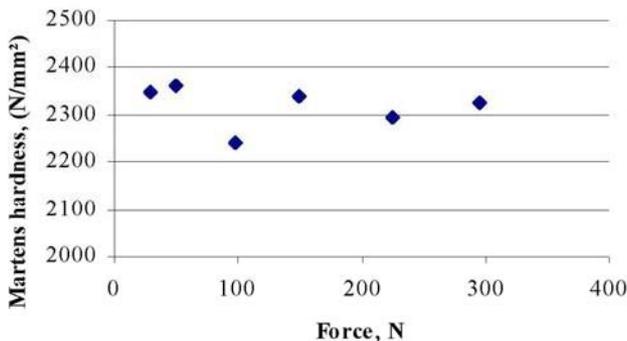


Fig. 9. Martens hardness values for a steel block

5. CONCLUSION

Some of the areas required to set up a Martens hardness service have been evaluated within this work. This has enabled initial Martens hardness test work to begin, as well as instrumented indentation test work and NPL's involvement in the EC DESIRED instrumented indentation project. Further work though is now required to evaluate the uncertainties associated with this service, and final procedures for test work and data analysis, before a final Martens hardness calibration service can be launched.

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