NPL REPORT CMAM 31

Proposed future research for hardness standards

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Abstract

This report draws on experience gained during the commissioning of NPL's new hardness standards facility, of discussions at a focus group meeting on hardness held in September 1998 and consultation with other National Measurement Institutes. It describes the hardness research planned as part of the DTI's National Measurement System Programme for Mass, 1999-2002.

The proposed work falls into three key areas: necessary reductions to the uncertainty of measurements made using the NPL hardness standard machines and a comprehensive study of hardness uncertainties, provision of standards for an expanded range of hardness scales, and validation of UK hardness standards through international intercomparisons.

The reduction in the uncertainties of the hardness standard machines will be achieved by modification and monitoring of the existing equipment, including improving the depth sensing system used in the small machine (force capacity of 2 kN).

A study will be conducted of the factors contributing to the uncertainty of hardness measurements. The study will include a review of the most up-to-date work on hardness measurement available, supplemented with original measurements. This report will be of use to UKAS laboratories and will also provide the UK with some of the fundamental information needed to participate more effectively at ISO and EA meetings.

To provide UK industry with the range of hardness metrology support they require, their requirements must be understood. A survey will be conducted and the findings regarding additional hardness scales implemented.

It is important to the competitiveness of UK industry that UK hardness measurements are respected throughout the world. NPL will validate its standards through participation in a CCM intercomparison and an intercomparison with NIST in the USA.

ISSN 1369-6785

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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 milestone 12 'Propose future research' of the contract MPU 8/45.4 'Provision of hardness standards' which is part of the DTI's National Measurement System Programme for Mass, 1996-99. The report reflects the work programme agreed for the National Measurement System Programme for Mass, 1999-02, at the meeting of the DTI's Measurement Advisory Committee Working Group on 22 April 1999.

Under the above contract, NPL has been re-establishing a national standard for hardness in the UK. A measurement service is scheduled for launch by the end of September 1999.

One of the key objectives in the special study on hardness conducted for the NMS in 1995 [1] is that a centre of excellence in hardness be established in the UK. This centre of excellence needs to be able to provide the following:

- Advice to UK hardness users
- Traceability in hardness measurements
- Representation on international committees on behalf of the BSI and the UK
- A contribution to World-wide knowledge of hardness metrology so that UK measurements earn international respect.

2. Enhanced depth sensor to minimise uncertainty in depth measurement

2.1 Background

Part 3 of the draft ISO standard [2] for calibration of standardised blocks to be used for Rockwell Hardness Testing, requires the depth to be measured with an expanded uncertainty of 0.2 μm. The machines that have been built as part of the current programme incorporate high precision Heidenhain gauges. Measurements made at NPL on identical gauges showed them to have an uncertainty of 0.025 μm. Using a Jamin laser interferometer, considerable effort has gone into trying to verify and improve the performance of the gauges in-situ. Unfortunately, the measured uncertainty in depth measurement has only been reduced to 0.15 μm. This meets the requirements of the draft ISO standard but is not good enough for a national standard machine.

A review of the sources of this uncertainty has been conducted. The position of the depth sensor makes it prone to errors due to the hysteresis in the compliance of the components between the sensor and the indenter. There is also a contribution from Abbe-sine errors. These are present because the measurement system is not exactly in-line with the direction of motion. Any tilting of the components results in an error equal to the product of the distance off axis and the sine of the angle of tilt.

Various possible solutions have been considered. Most leading NMIs use deadweight hardness testers with a laser interferometer to measure the position of the indenter. A retro-reflector is mounted on top of the indenter holder and the laser is directed onto it along the direction of travel. In this way, the Abbe-sine errors are eliminated. In the case of the NPL machines, an improved sensor must be retro-fitted. This sensor must be compact, free from Abbe-sine errors and able to get as close to the actual indentation measurement as possible. It should also allow rapid data collection so that the indenter velocity profile can be measured and the sensor can be used for instrumented indentation hardness testing (see section 3, 'Increased hardness ranges').

2.2 Proposal

The most commonly used method of accurate displacement measurement is laser interferometry. The right system can have high accuracy and resolution. It can easily be made traceable to national standards for length and when operated in an air-conditioned environment, the laser's sensitivity to temperature can be overcome.

A suitable laser system to measure the displacement of the indenter holder would require two sensors to overcome the Abbe-sine errors and complement the existing design of the machine. To minimise the deformation of components in the metrology loop, the position of the indenter holder needs to be measured relative to the surface of the test block. The space between the indenter holder (actually the bottom surface of the load cell) and the hardness block is very restricted. The interferometer therefore needs to be small. To protect the machine from the heating effects of a laser, they must be well separated. The use of fibre optics would avoid having laser beams traversing long distances across the laboratory.

A provisional design developed at NPL is shown in Figure 1.

The signals from the optical fibres are reflected off the surface of the test block and off cats eye reflectors mounted to the indenter holder. These signals recombine in the interference blocks, which are designed so that they produce three signals of different phases (0 degrees, 90 degrees and 180 degrees). By optimising the use of the information contained in these three signals, the position of the cats eye reflectors relative to the test block can be obtained. The system will be able to accommodate test blocks with widely differing reflectivity, and with a sampling rate of kHz, the direction and velocity of the indenter can be determined.

The design is expected to be relatively inexpensive but have a measurement uncertainty of less than 50 nm. This would be sufficient to meet the requirements of a national standard machine for making Rockwell measurements, and the sampling rate would be sufficient for instrumented indentation hardness testing.

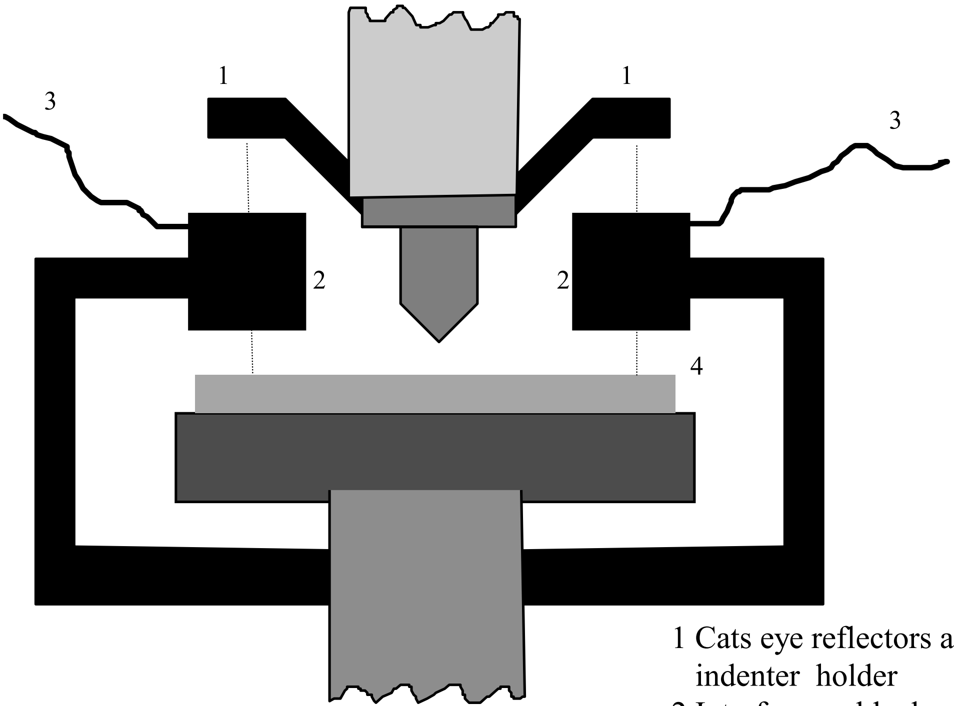


Fig.1 Preliminary design of depth sensor optics.

1 Cats eye reflectors attached to indenter holder
2 Interference blocks
3 Optical fibres
4 Test block

2.3 Stages of Development

1. Review alternative interferometer designs and find a commercially available solution
2. Carry out detailed design for implementation of proposed method and review
3. Manufacture or purchase optical and electronic components
4. Investigate and obtain digitising circuits to record data from the circuitry in parallel

3. Increased Hardness Ranges

The commonly used hardness scales are listed in tables 1 and 2. Those shown in bold are scheduled to be supported as part of the current NMS Mass Programme. These reflect the recommendations of the 1995 special study [1]. The recognised Brinell scales are shown by the combinations of force and indenter diameter used.

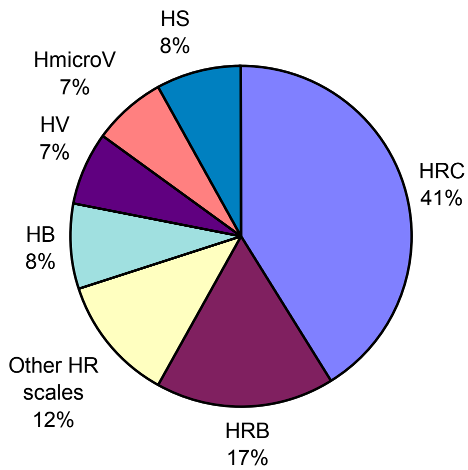
Type of scale								
Rockwell	HRA	HRB	HRC	HRD	HRE	HRF	HRH	HRK
Rockwell Superficial	HR15N	**HR30N**	HR45N	HR15T	**HR30T**	HR45T		
Vickers	HV5	**HV10**	HV20	**HV30**	HV50	HV100		

Table 1: Recognised Rockwell and Macro Vickers scales.

	Ball Diameter / mm				
Force/ kgf	10	5	2.5	2	1
3000	**X**				
1500	X				
1000	**X**				
750		**X**			
500	X				
250	X	X			
187.5		X	**X**		
125					
120				X	
100	X				
62.5		X	X		
40				X	
31.25			X		
30					X
25		X			
20				X	
15.6			X		
10				X	X
6.25			X		
5					X
4				X	
2.5					X
1					X

Table 2: Forces and ball diameters used for Brinell scales as recognised in the ISO standards [3,4,5].

At a recent meeting of the IMEKO hardness group, the president of Mitutoyo, one of the world's leading hardness test block suppliers, provided NPL with their sales figures for each hardness scale. These are reproduced below.



Key:

HRC- Rockwell C scale
HRB- Rockwell B scale
Other HR - other Rockwell
HB - Brinell scales
HV - Macro Vickers scale
HmicroV- Micro Vickers scale
HS - Shore hardness scale

Fig .2 Distribution of sales of standardised test blocks by Mitutoyo, grouped by test method.

It is essential that the appropriate scales are available to UK industry and that effort is not put into providing a measurement service for scales that are not used. To find out what additional scales are needed, UK industry will be consulted and a report written. This consultation should include UKAS, UKAS accredited laboratories, UK manufacturers of testing machines, hardness blocks and indenters, major users, IMGC (who are currently providing the UK traceability), and other NMIs.

Instrumented indentation ("Universal") hardness testing is one area that will be of great importance to the UK. Instrumented indentation hardness testing, is a recently developed method in which the penetration depth and force are both monitored during the indentation. It is therefore a more complicated test than conventional methods and it requires relatively expensive equipment. The force/depth profile can provide additional information such as Young's modulus [6]. The technique is fairly widely used at a micro level because it can give information about coating thickness. A DIN standard [7] has been written and an ISO standard [8,9,10] is in draft. There are as yet no test blocks available for these machines. There is a concern in the UK that overseas customers will begin to specify materials to instrumented indentation hardness standards and that they will be unable to make the measurements. The hardness determined using instrumented indentation hardness testing usually has a much greater uncertainty than that measured using conventional methods.

The versatility of the machines at NPL means that they will be able to deal with these extended ranges with little or no modification.

4. Study of the factors contributing to the uncertainty in measured hardness

Hardness measurement is an empirical test. Historically several companies developed a test based on the penetration of an indenter into the material. With a specified indenter and force, the smaller the indent, the harder the material. The three methods that have stood the test of time for hardness testing of metals are Brinell, Rockwell, and Vickers. The differences between the tests reflect the different problems they were designed to overcome. Rockwell is fast, Vickers can be used on small specimens and causes minimal damage, Brinell is relatively immune to surface finish and grain structure. The nature of these tests mean that the hardness of a material cannot be made traceable back to base units.

Each of the above methods has been standardised for many years. In an effort to produce World-wide harmonisation, ISO standards [2,3,4,5,11,12,13,14,15] have been written or are in the last stages of approval. The standards lay down acceptable limits for many of the measurement parameters such as indenter shape, force, indentation size measurement, and indentation velocity. Some of the requirements are arguably too stringent and place an unnecessary burden on industry. UKAS, along with similar organisations in other countries, are anxious to ensure that the requirements of the standards are met. They are also introducing requirements to estimate the uncertainty in the measured hardness and an EA document [16] has been drafted.

The issue of uncertainties is very important to UK industry. NPL organised a colloquium on the uncertainty of hardness measurement in 1999 and it was attended by a large number of companies. At the hardness focus group meeting in September 1998 organised during the formulation of the NMS Mass Programme 1999-2002, this topic was marked as a very high priority. At present there is little information available to UK industry and that which is available is not very accessible.

A literature search will be conducted, international experts will be contacted and, where necessary, fresh experimental work will be carried out. A report will be drafted pulling together the state of the art on hardness uncertainty measurement.

This work will be of use to laboratories that are being asked by UKAS to produce uncertainty budgets. It will also help establish the UK as a player in this field and enhance our representation on ISO and EA committees so that we are better able to eliminate some of the more unjustifiable requirements of standards and procedures.

There are many factors that contribute to the uncertainty of hardness measurement and these are summarised for Rockwell measurements in the EA document [16]. Measured Rockwell hardness is strongly influenced by the indenter. The uncertainties introduced by the allowable variations in the indenter are greater than those introduced by, for example, the force. The force is tightly specified in the standards because with the use of deadweights (with or without levers) it was thought to be an easy parameter to control. In practice, many lever machines are not meeting the criteria but it may be that this does not have a big impact on the measured hardness and the standards require modification. This example illustrates how a clearer picture of where the uncertainties come from will allow better standards to be written.

The standards specify the shape that indenters should have for use in a testing machine or a standardising machine. There are also discussions about indenters that should be used by NMIs in an effort to create world-wide agreed hardness scales. NPL has the ability to accurately characterise the shape of Rockwell and Vickers indenters. Previous work by IMGC [17] has shown that the geometry of the indenter is not the only variable that needs to be considered. Other possible causes for variation include, the mounting of the diamond tip, the orientation of the crystal structure, friction, and mechanical deformation. By using diamond tips with identical geometric characteristics but differing non-geometric characteristics the effect of these parameters could be better understood. Indenters could be obtained from several companies, with differing manufacturing methods, so that the influence of the processes can be assessed.

For Brinell and Vickers measurements the imaging of the indent has always been a problem even at national level. Investigation of the effects of different lenses on the measured hardness will be carried out.

Some of the work would have international importance due to the unique design of NPL’s national standard machines. Unlike other national standard machines NPL’s does not contain a deadweight but is controlled by a high accuracy load cell. The control of the force is such that different methods of loading can be investigated as well as accurate control of velocity and time. It will also be valuable to see how the results from a deadweight machine differ from that of a servo-controlled machine.

5. Intercomparison with NIST

UK companies who sell to aerospace companies have to measure hardness using ASTM standards. The most commonly used scale is Rockwell and the US measurements have differed significantly from European and Asia-Pacific measurements. September 1998's focus group meeting highlighted this point and was insistent that the UK intercompared hardness scales with NIST using Wilson test blocks. An intercomparison with NIST over several ranges would increase the ability of UK companies to trade with the US.

Agreement between NIST and IMGC has been poor in the past but this was believed to be due to the NIST indenters having a slightly smaller radius [18]. The world wide Rockwell intercomparison due to be completed in June 1999 [19] will give an indication of whether there is international agreement in the Rockwell scales.

6. CCM intercomparison

The exact nature of this intercomparison is still to be decided, but the UK needs to be involved if it wishes to be a major force on the international hardness scene. It is vital that the national standard at NPL agrees with those in the rest of the world.

NPL will be attending talks in Paris in May to discuss the ranges and format of the intercomparison.

Both the CCM and NIST intercomparisons need to be preceded by the improvement of the depth sensor.

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