

Measurement Note

Mapping Indentation Hardness

Summary

Local mechanical property information can be rapidly obtained from a measurement of microindentation hardness. A new scanning microhardness indentation test machine (SIMM) has been developed for this purpose at NPL. The system uses the principle of depth sensing hardness, because the measurement process can be fully automated. A diamond indenter is applied to the surface of the testpiece and hardness is calculated from the load and the indentation depth. A raster of indents can be positioned by computer control to map the variation in hardness.

Advances in computer aided measurement electronics and sensitive displacement measurement transducers enable hardness systems to measure the depth of an indent during testing rather than determining the indent area post test [1,2]. A major advantage of this method is that dynamic measurements can be performed, i.e. a load versus displacement curve can be measured. Also, the subjectivity of the operator is removed. The traditional hardness values can in principle be obtained without the need to measure the indentation optically. More significantly, properties of the material such as the indentation elastic modulus and elasto-plastic hardness and dynamic properties (such as creep and relaxation) can be determined. The NPL SIMM has been used recently for a variety of purposes, such as mapping welds and surface coatings.

This Measurement Note highlights a microhardness mapping case study for characterising heterogeneity in hot deformed compression testpieces in a range of representative engineering metals. NPL, in partnership with the Universities of Sheffield and Wales (Swansea) is working towards good testing practice in dynamic high temperature compression techniques; recently to produce guidelines for microstructural examination and to define local deformation conditions to compare computed and measured maps of microstructural evolution to provide information for the validation of appropriate models that include a consideration of strain history. Measurements of the spatial variation in mechanical properties are being used at NPL to complement measurements of microstructural variation.

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NPL 
National Physical Laboratory

[Mapping (MN)]

Instrument Characteristics

The NPL instrument has been constructed with a relatively high speed facility to map large areas quickly. Welds provide classic examples of features that benefit in their interpretation from a knowledge of spatial variation in mechanical properties. An example of a weld microhardness map is shown in Fig 1. The map was formed from an X by Y array of about 17000 indentations. The indents were placed on a square array, each indent separated by about 100 μm , using a load of 0.2 N. The indent diameters were typically 20 μm . The map was generated automatically by leaving the machine to run continuously over a period of 37 hours. Clear definition of the different zones of structure can be seen and it is possible to zoom and investigate local behaviour or study line maps in X or Y directions.

In the SIMM force and displacement are monitored continuously during a complete indentation cycle, which can typically be completed in 15s. The machine currently operates

in the micro to mesoscale range, 0.02-10 N, (approximately 2 gf-1 kgf), with a load resolution of 2 mN (0.2 gf) and a depth sensing resolution of 20 nm, using a specially designed capacitance displacement sensor. It is currently being uprated to be able to apply a wider range of loads, 0.01-50 N, (approximately 1 gf – 5 kgf), without loss in load or displacement resolution over a spatial area of 25 $\times$ 25 mm (compared with 10 $\times$ 15 mm currently).

Hardness maps can be obtained using the high speed indenter up to 60 times faster than nano-scale machines working at the micro-scale. For instance a matrix of 30 $\times$ 30 indents takes 2.5 hours as opposed to 6.25 days under typical operating constraints defined in the Draft ISO/CD 14577-1 (1999). This means that the mapping of a significant surface area is not only possible, but also realistic and it may be used both in a quality control context as well as for research and development. Hardness is determined using the principles outlined in the draft ISO standard for Instrumented Hardness Tests, ISO DIS 14577.

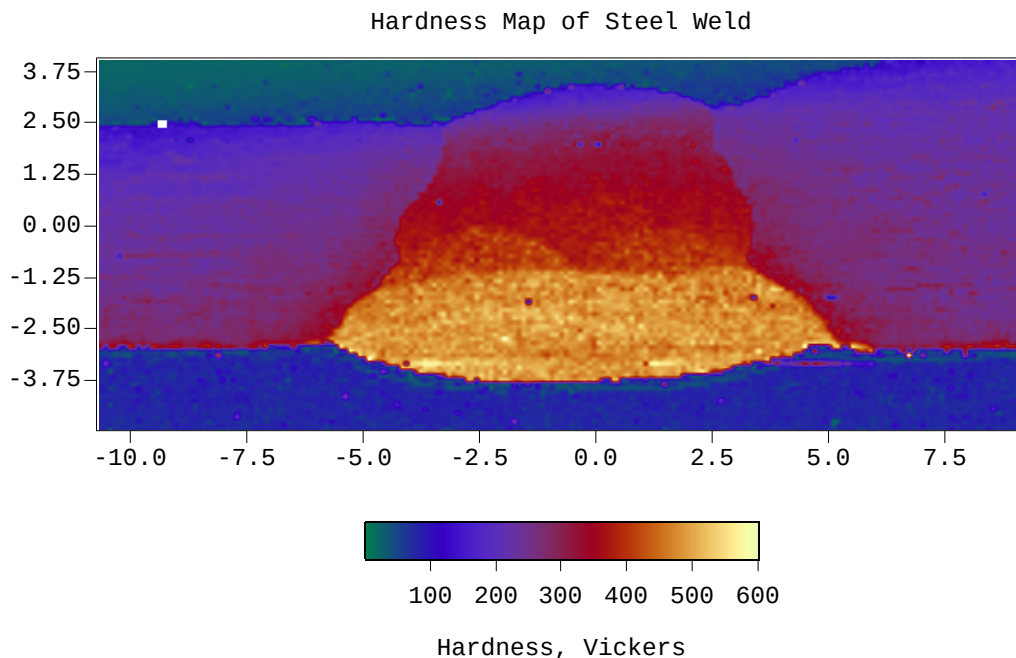


Fig 1 Hardness map of a weld in a ferrous material.

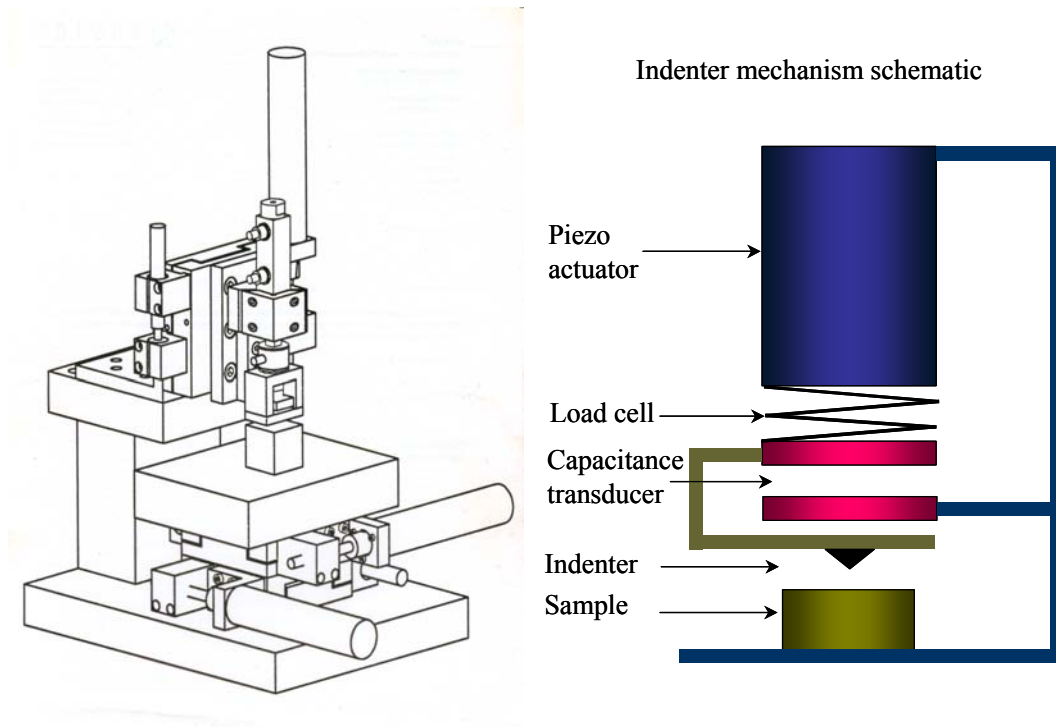


Fig 2 Schematic diagram of mapping hardness system.

Three linear slides provide the X, Y and Z movement, (Fig 2). Each slide is driven by a 1 mm pitch screw thread, powered by a servo controlled DC motor. A microprocessor controller determines the position of each slide with one micrometre resolution. Control feedback is via a 500 line rotary encoder attached to the shaft of each motor. The load is measured with a conventional strain gauged load cell, and the Vickers indenter was taken from a conventional microhardness system. The loading mechanism uses a piezoelectric actuator. A piezoelectric system has the potential to be quick, with a high resolution. However, the main drawback is a limited range of movement of around 60 μm . The complete assembly is mounted on a motorised Z stage. The motor is driven to within a few micrometres of the surface, and the piezoelectric mechanism is used to perform the indentation. From this position the Z motor is only used to accommodate any roughness or misalignment of the sample surface.

To operate the system the piezoelectric actuator is expanded by the application of a high voltage, and this causes the indenter to move towards the sample. The motion of the indenter is measured

by the capacitance transducer and is a measurement relative to a static capacitance plate which is in turn mechanically connected to the sample surface. When the indenter touches the sample surface a load is registered on the load cell. Loading is continued until the desired load is reached. The design of the loading mechanism is such that although the strain gauge load cell is effectively a soft spring, the compression of the load cell does not register on the displacement measurement system as the load cell is above the capacitance transducers, (Fig 2). This means that there is no need to correct for the load cell stiffness in the final load against displacement curves.

Typical design range and resolution parameters for the system are given in Table 1. The hardness values were calculated using the standard Vickers formula

$$HV = 2P \sin \theta / d^2 \quad (1)$$

$$HV = 1.8544P/d^2 \quad (2)$$

where P is the indent load in kgf and d is the corner to corner diameter of the indentation in

mm and θ is the angle between the indenter face and the vertical ($136^\circ/2 = 68^\circ$). The depth of the indentation, h , is equal to $d/7$ ($d/2\sqrt{2}\tan\theta$). Therefore, expressing HV hardness as a function of indentation depth.

$$HV = P/26.42 h^2 \quad (3)$$

Vickers hardness is defined as the ratio of applied load to contact area between indenter and sample under load.

Table 1 Typical Range for 0.5 kgf (approximately 5 N) and 0.005 kgf (approximately 50 mN) indents

Hardness, HV0.5	200	500	1000
Indent Diameter, μm	68	43	30
Depth, μm	9.7	6.1	4.3
Hardness, HV0.005	200	500	1000
Indent diameter, μm	6.8	4.3	3.0
Depth, μm	1.0	0.6	0.4

Current system resolution: Load = 2 mN;
Displacement = 0.02 μm .

The control software for performing indents, displaying and storing load displacement curves, and calculating the SIMM hardness values, was written in Labview[®] using modular routines. The principles of depth sensing hardness at the microscopic level (i.e. application loads of typically 5-20 N) have been extensively demonstrated by Thurn et al [3] on a range of

ceramic materials. The depth sensing hardness HV (SIMM) is defined by P/A , where A is the projected area of contact, and this is calculated from

$$HV (SIMM) = F/(24.5 \cdot h_c^2) \quad (4)$$

where h_c is the indentation contact depth under load (Fig 3).

The hardness at each point is determined by using the following method on the load/displacement curves (Fig 3). Firstly the surface is found by fitting a polynomial to the initial 5% of the loading curve. This is followed by finding $h_{\max}$, the depth at maximum load. A linear fit to the top 20% of the unloading curve is then extrapolated down to zero load, to give the unloading zero, h_r . The indentation contact depth under load, h_c , is then determined using

$$h_c = h_{\max} - 0.75 \cdot (h_{\max} - h_r) \quad (5)$$

The major difference between the method adopted for the NPL machine and the ISO guidelines is that the data from the NPL machine was not corrected for the machine compliance, since a satisfactory method for this correction has yet to be determined: typical load/displacement curves for tests on Al alloy are shown in Fig 4.

Setting the approach rate of the piezo-actuator controls the loading rate of the system. Therefore, the system is displacement controlled and the loading rate depends on the material hardness and indentation depth. Most experiments are set up so that it takes several seconds to ramp the load up to the maximum, and so loading rates vary from around 0.01 to 1 N/s.

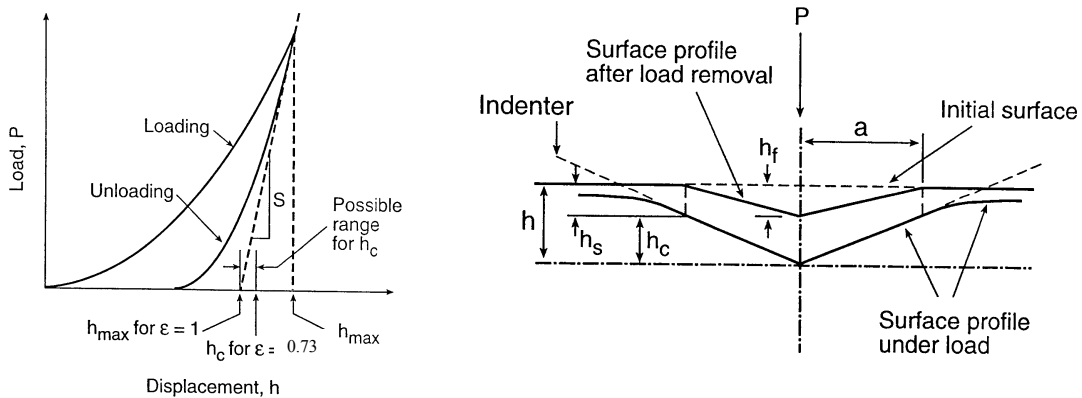


Fig 3 Schematic diagram of typical load/displacement curve and depth parameters [4].

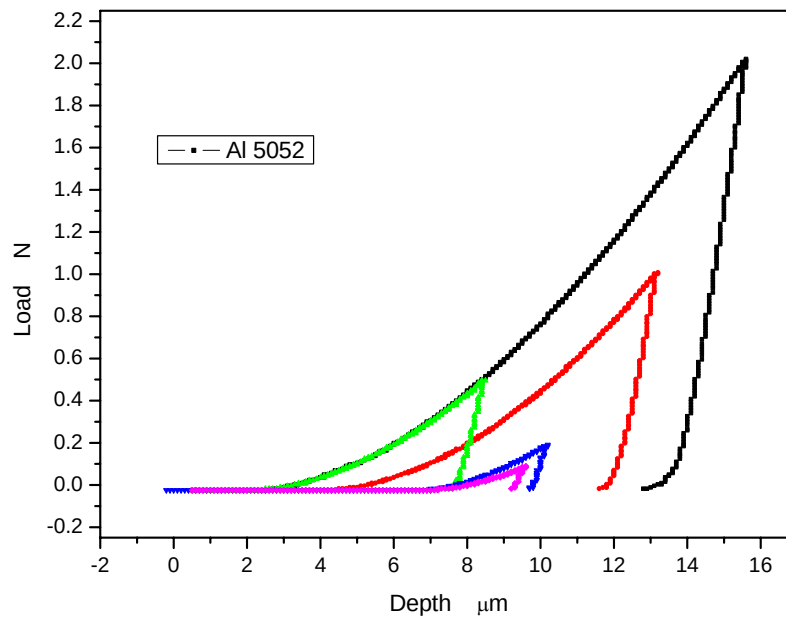


Fig 4 Typical load/displacement curves for depth sensing indents at different loads on the AA5052 aluminium alloy.

Instrument Performance

Microhardness values depend, not only on the applied load, but also on the scale of the underlying microstructure of the material being investigated. In order to evaluate the capabilities of the current system four representative engineering materials were chosen for this study:

- 316 stainless steel
- Waspaloy (Ni superalloy)
- AA5052 (Al alloy)
- Ti-6Al-4V (Titanium alloy).

An example 40×40 array is shown in Fig 5 for indents at 0.2 N in the Waspaloy Ni superalloy.

Suitable surfaces for indentation were prepared by careful metallographic standard grinding and polishing and the following experiments were performed to investigate the metrology associated with making microhardness measurements in each of these materials: Place single indents in each material (*To study the shape of the load/displacement curves*); Place a 2×5 array at different loads (0.05 N – 2 N) with indents spaced by $50 \mu\text{m}$ (*To study the load dependence of microhardness values (calculated using depth*

sensing principles or by optical measurements of indent diameter); Place a series of arrays of different spacing and at two applied loads, nominally a high and a low load – 1 N and 50 mN (*To study the effects of indent spacing on microhardness values*); Place large arrays; up to 200×150 indents (*To investigate the time dependence of the mapping facility*).

An example of an indent array with respect to the scale of the underlying structure for Waspaloy and Ti6Al4V is shown in Fig 6.

For each material an array of 2×5 indents was applied at each of six loads (0.05, 0.1, 0.2, 0.5, 1 and 2 N). These initial exploratory tests provide an indication of scatter in SIMM hardness values and their dependence on load. The coefficients of variation increased significantly with decreasing load below about 0.5 N. The variation was also higher in the two phase material Ti-6Al-4V, as might be expected, dependent on the position of the indentation with respect to each phase (α , β or mixture). A plot of indent diameter as a function of applied load is shown in Fig 7 for the four materials, where indent diameter was measured using either a microscope (d_m) or indirectly for the SIMM universal hardness value.

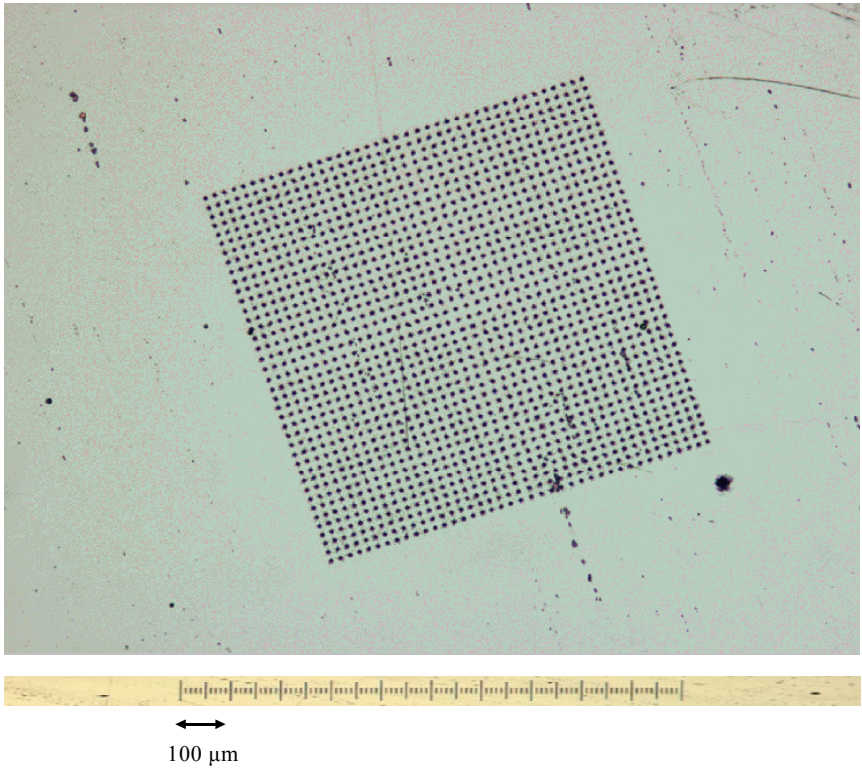
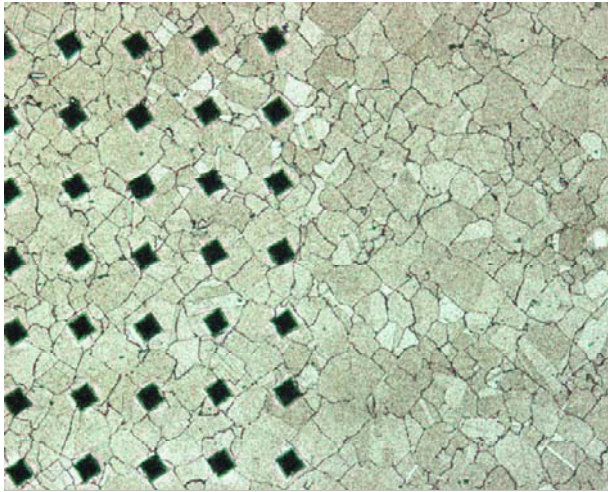
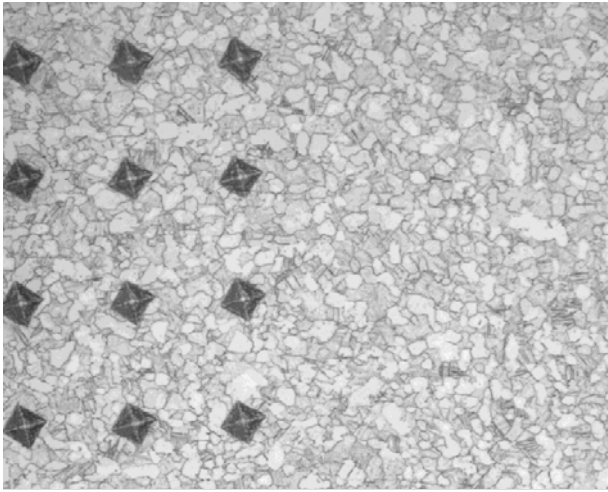


Fig 5 Example 40 × 40 array at 0.2 N in Waspaloy.



Waspaloy



Ti 6Al 4V

Fig 6 Indent arrays at 2N (100 micrometre spacing) in Waspaloy and 2N (80 micrometre spacing) in Ti6Al4V, subsequently etched.

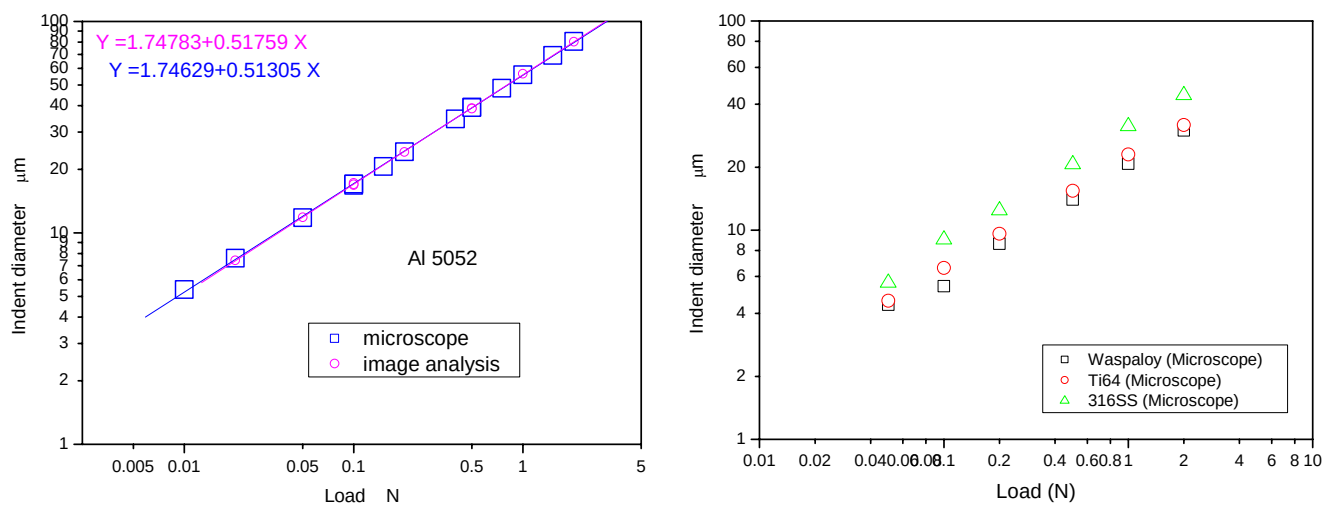


Fig 7 Dependence of indent diameter on applied load.

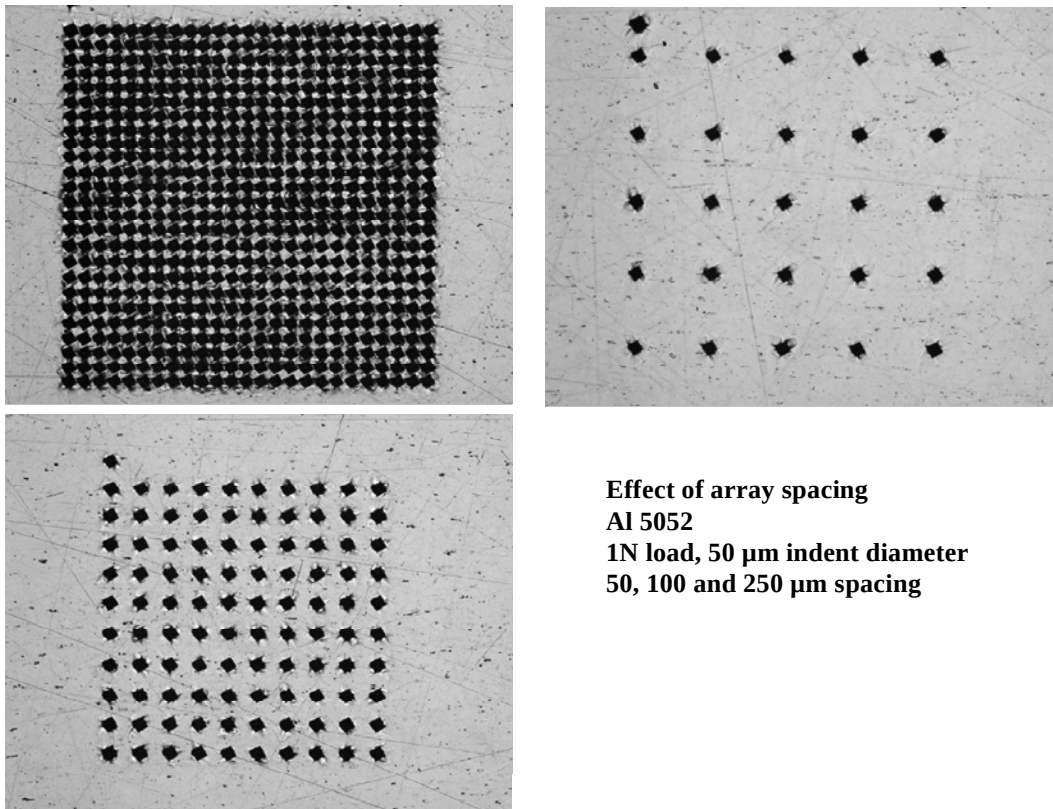


Fig 8 Indent arrays in Al 5052 alloy.

The effects of array spacing were studied for the Ti-6Al-4V and Al 5052 alloys at two applied loads, 0.05 and 1 N. For the Ti alloy indent diameters of about 4 and 20 μm were developed for the low and high loads respectively. For the Al alloy the equivalent diameters were about 12 and 50 μm respectively. For the Ti alloy the indents were placed in arrays spaced by 4, 10 and 20 μm at the low load and 20, 50 and 100 μm for the high load. For the Al alloy the indents were placed in arrays spaced by 20, 50 and 100 μm at the low load and 50, 100 and 250 μm at the high load (Fig 8). The arrays were set to cover about the same area, about 1 mm $\times$ 1 mm resulting in arrays of 625, 100 and 25 indents respectively.

The results of the study of indent array spacing are analysed in two ways:

- as a plot of indent array number (i.e. in chronological sequence). The first indentation is placed at the top left hand corner of each image, moves right (+X direction) to the end of a row, drops down one indent spacing (+Y direction) and then moves left along the row (-X direction), drops down one indent (+Y direction) and so on.
- as a cumulative plot of SIMM hardness, ranked from low to high. Rank value (R) is calculated from the formula $R_i = (n_i - 0.5/N)$ where N is the total number and n_i is the SIMM hardness value of the i^{th} indent (ranked from low to high).

For the Ti alloy no obvious trend of hardness with indent number was observed for any of the applied conditions when plotted in sequence. The same behaviour was observed in the Al alloy for all conditions except the closely spaced high load (1 N) results. In the latter case there was clearly an effect of indent spacing on measured SIMM hardness values from the second row onwards. The first row (with indents following one after another) did not show an effect. Whereas for the second and subsequent rows where indents were affected by the deformation field of indents above as well as behind, the apparent hardness was higher. However, when the results are plotted as ranked values an effect of indent spacing can be seen for the high load arrays in the Ti alloy. For the Al alloy the effect was only noticeable for the 25 $\times$ 25 array (50 μm spacing 1 N load). In both

Ti and Al alloys there was no effect for the low load arrays.

Case Study

Representative SIMM measurements were made on compressively deformed axisymmetric cylindrical samples of Waspaloy, Ti6Al4V, Al5052 and 316 stainless steel. The specimens were deformed by the University of Wales (Swansea) as shown in the matrix in Table 2. The degree of strain homogeneity in specimens was governed by frictional constraints; with low friction corresponding to uniform strain and high friction equivalent to heterogeneous strain. Temperature heterogeneity was increased by high strain rates and changing rates did not significantly affect patterns in strain.

Table 2 Compression testing conditions

Specimen Number*	Temperature $^{\circ}\text{C}$	Strain Rate s^{-1}	Friction Condition	Strain
N1	P	0.01	lub	0.5
N2	P	0.01	dry	0.5
N3	P	1	lub	0.5
N4	P	1	dry	0.5

N = T (Ti6Al4V), A (Al5052), W (Waspaloy) or S (316 stainless steel).

P = 950 $^{\circ}\text{C}$ (Ti6Al4V), 400 $^{\circ}\text{C}$ (Al5052), 1040 $^{\circ}\text{C}$ (Waspaloy) or 950 $^{\circ}\text{C}$ (316 stainless steel).

The axisymmetric compression specimens had an initial height of 12 mm and an initial diameter of 9 mm. They were heated to testing temperature within 10 minutes and water quenched within 3 seconds at the end of the compression test. Tests were carried at true constant strain rate. High friction conditions were attained by using dry specimens and low friction by lubricating with a glass mixture. Following deformation, specimens were sectioned across a diameter and in a plane parallel to the cylinder axis. All were prepared by grinding and polishing using coarse and fine diamond wheels.

SIMM maps from the four Waspaloy cylinders are shown in Fig 9. The maps were obtained in a 90 $\times$ 100 array with 2 N indents spaced by 30 μm in diameter. The high friction tests show a harder zone close to the top and bottom surfaces corresponding to a region that has not recrystallised during deformation. SIMM maps

for the 316 stainless steel cylinders are shown in Fig 10. The array parameters were similar to those used for the Waspaloy microhardness maps. The high friction tests again show strong heterogeneity in structure and the SIMM maps are currently being compared with metallographic measurements to establish the source of the heterogeneity. SIMM maps for the two high rate tests on the Ti6Al4V samples are shown in Fig 11. In this case the differences in

microhardness are likely to be due to temperature variations in the deformed cylinders that result in changes in the volume fraction of the α and β constituent phases. Typical variations in strain (Waspaloy) and temperature (Ti6Al4V) obtained by FE models of the axisymmetric deformation process are shown in Fig 12. The similarity in strain distribution and hardness maps for the high rate, high friction test on the Waspaloy sample is particularly noticeable.

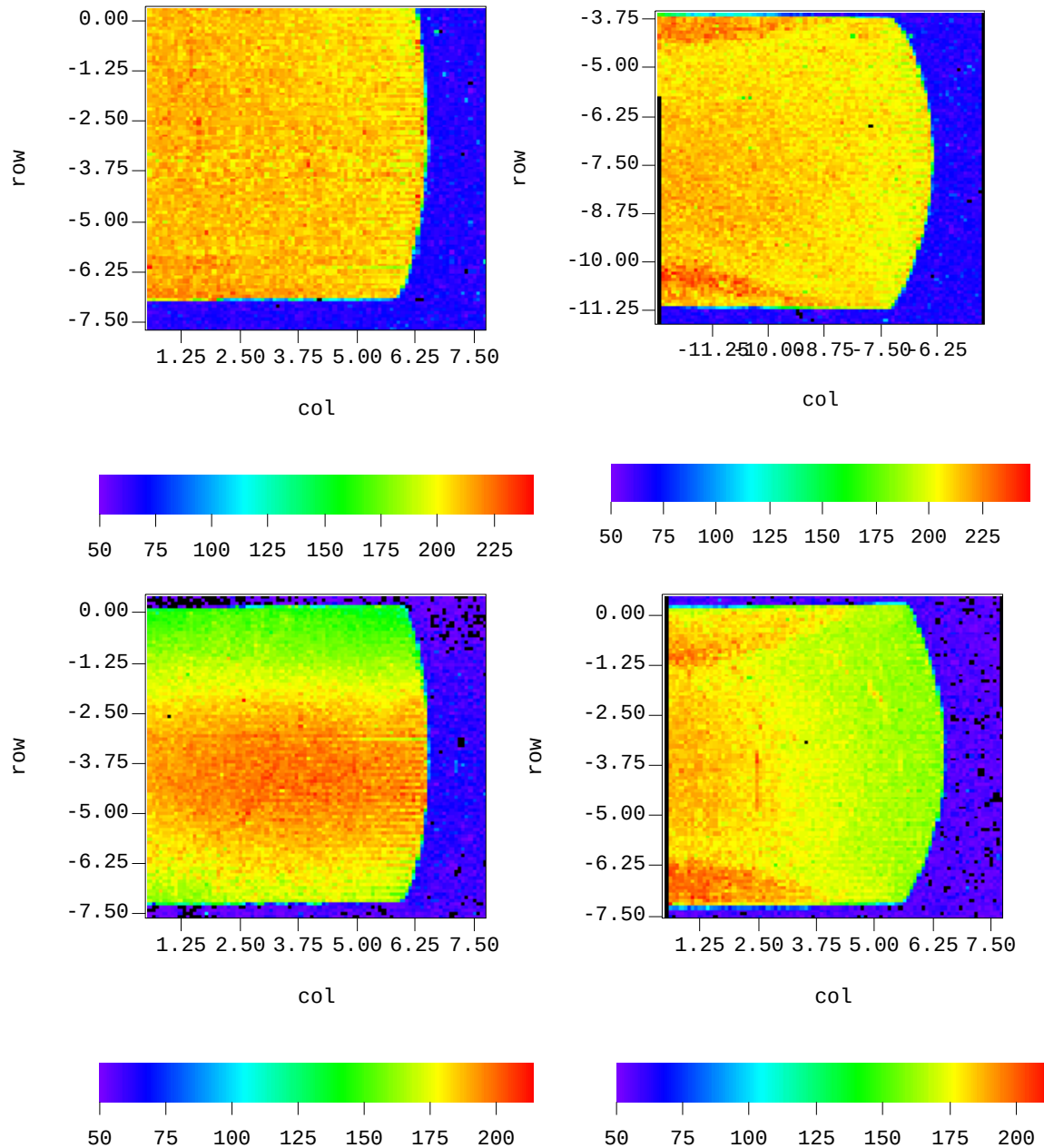


Fig 9 SIMM maps of Waspaloy alloys for the different test conditions (Coloured Hardness scale; high strain rate tests on the bottom row, high friction tests in the right column).

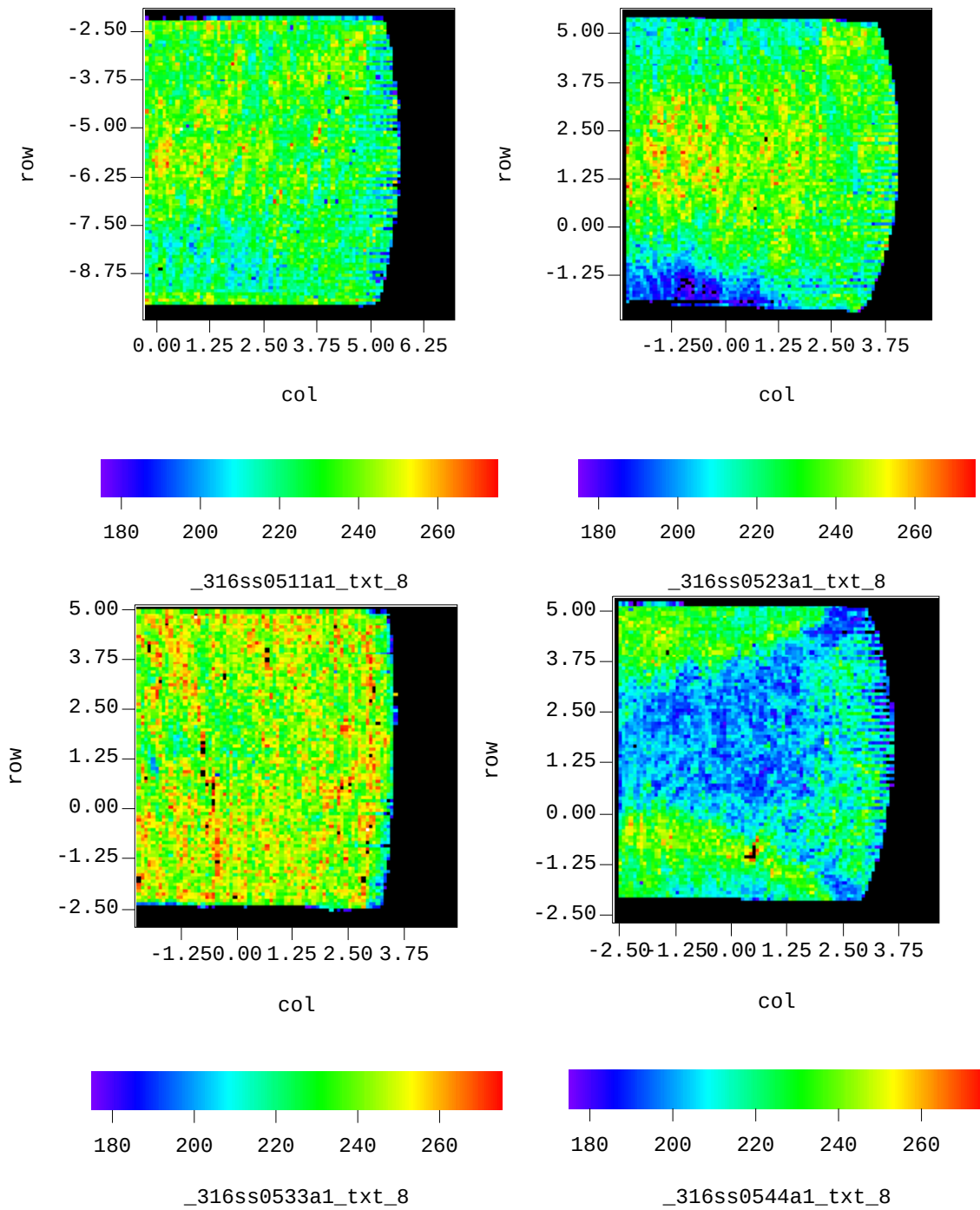


Fig 10 SIMM maps of 316 stainless steel alloys for different test conditions (Coloured Hardness scale; high strain rate tests on the bottom row, high friction tests in the right column).

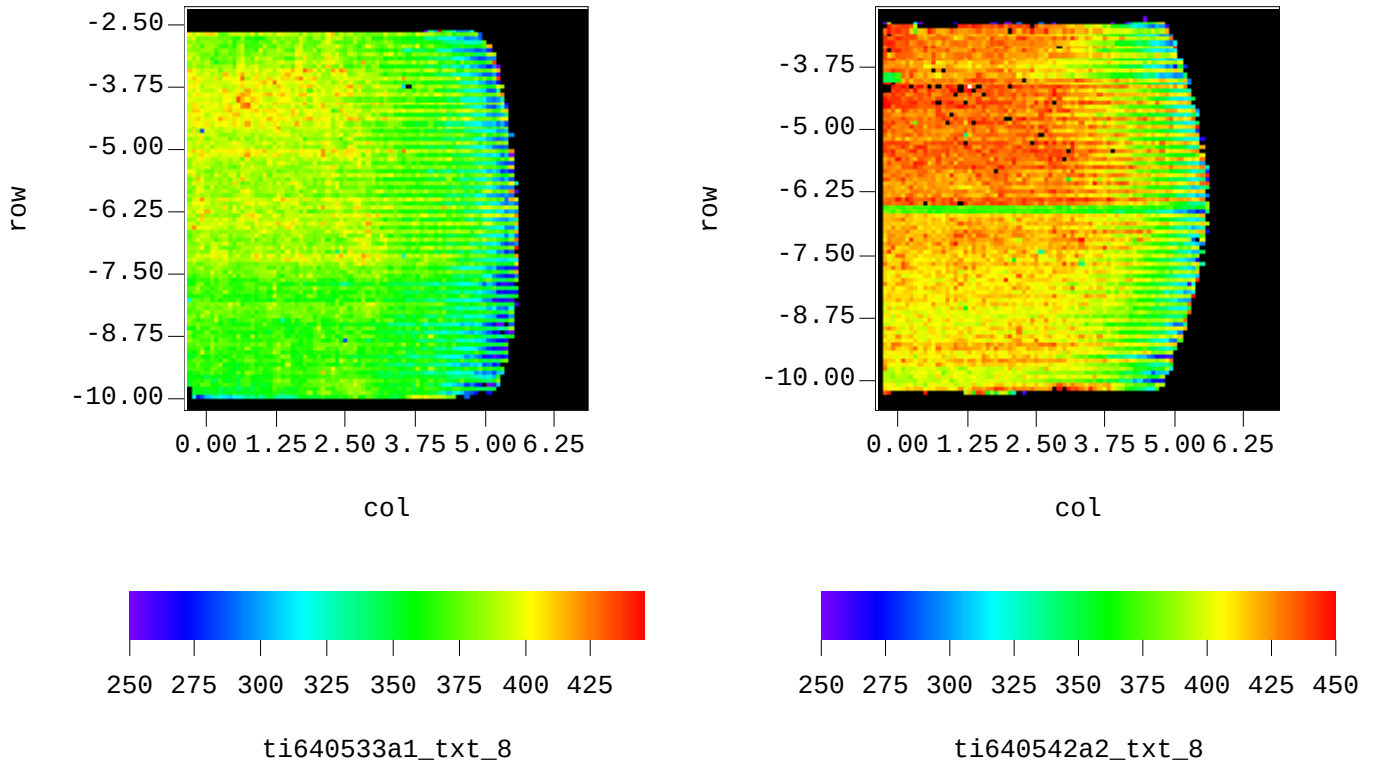


Fig 11 SIMM maps of high strain rate tests on Ti6Al4V samples (Coloured Hardness scale; high friction test on the right side).

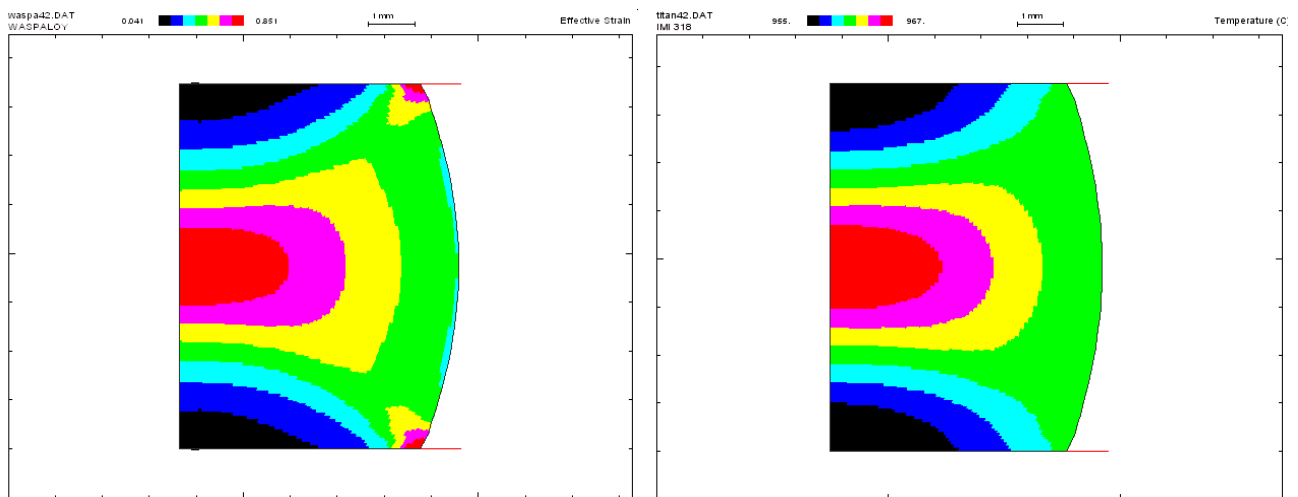


Fig 12 FE model predictions of strain in Waspaloy and temperature rise in Ti6Al4V (high friction, high strain rate (courtesy Prof R Evans, University of Wales, Swansea).

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