A Method for Measuring the Hardness and Elastic Modulus of the Surface Layer on Hot Forging Dies Using a Nano Indentation

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Abstract

The properties and characteristics of the surface layer of forging dies are critical for understanding and controlling wear. However, the surface layer is very thin, and appropriate property measurements are difficult. The objective of the present study is to determine if nano hardness testing provides a reliable means of measuring the surface hardness in forging die steels. Two hot die steels (FX and H13) were used in the investigation. These steels were heat treated for various times to produce specimens with different values of hardness. The heat-treated specimens were tested using three different hardness instruments -- a Rockwell hardness tester for macro hardness, a Vickers hardness tester for micro hardness and a nano hardness tester for nano scale evaluation of hardness. The results of this study indicate that nano hardness values obtained using a Nano Indenter XP Machine with a Berkovich indenter reliably correlate with Rockwell C macro hardness values and with Vickers HV micro hardness values. Consequently, nano hardness testing can provide reliable results for analyzing the surface layer of hot forging dies.

1. Introduction

Die wear is a major cause of failure for hot forging dies. Failure is caused by a rapid increase in wear that causes the forging impression to grow beyond specified tolerances, which effectively terminates die life. Understanding die wear requires improved understanding of changes that take place in the surface layer of hot forging dies during use. The following conditions cause changes to the microstructure and properties of the die steel surface layer during forging: 1) time at high temperatures due to contact with the hot workpiece, 2) shear stresses associated with metal flow and friction, and 3) contact pressure, where contact pressure depends on the properties of the work material, part shape, and die design. Macro and micro hardness measurements are thought to be too coarse to effectively determine the strength of the layer on the surface of a hot forging die. Nano hardness testing, because of its small indentation, may provide a method to study the changes that occur to the surface layer.

Die wear depends on the strength of the surface layer of the die steel at forging temperatures. It is possible that the wear is the result of over-tempering of the surface layer of die steels. If the decrease in strength at temperature is related to over-tempering of the surface layer of a forging die, this effect can be observed at room temperature by either metallographic analysis or hardness testing. Detailed metallographic observation of tempered martensite is too expensive to be practical, but changes in hardness can be used to evaluate tempering.

As an initial step in evaluating wear in hot forging dies via hardness testing of the surface layer, the relationships between nano hardness, micro hardness, and macro hardness are compared using samples with uniform properties so that the different hardness tests evaluate the same microstructure. Samples from two die steels were tempered at different temperatures. This study shows that the values measured by nano hardness, micro hardness, and macro hardness tests provide comparable results. Consequently, nano hardness testing is a viable method for studying the changes in the mechanical properties of the surface layer of hot forging dies.

2. Experimental Procedures

2.1 Materials

An FX die steel (0.8 Ni, 1.15 Cr, 0.5 Mo) and an H13 die steel (1.0 V, 5.0 Cr, 1.4 Mo) were used in this study. The initial materials were received as hardened 12.7 mm (0.5 in) slices from approximately 50 mm (2 in) diameter bar stock. Samples were prepared by furnace tempering in an air atmosphere for one hour. After tempering each sample was sanded to 400-grit paper to remove scale from the sample surface. Figure 1 shows a test piece that was tempered. Table 1 lists the tempering conditions.

2.2 Macro hardness testing

Macro hardness testing was performed on a Wilson-Tukon Rockwell hardness machine. Tests were performed using the Rockwell C test, which uses a constant 150 kg load and a diamond indenter. The diamond indenter has a sphero-conical shape with a 120^o cone and a 200 micron tip radius. The hardness reading is based on the measured displacement of the indenter [1].

The macro hardness indentations were taken from left to right through the centerline of the cylindrical piece and from top to bottom through the centerline. These macro hardness tests were performed prior to the sectioning of the specimen into four sections as shown in Figure 1. These measurements not only provided the hardness values but allowed verification of the uniformity of the microstructure. The only variation that was observed was a slight soft spot in the center of the H13 steel samples. This area was avoided for all other tests. About thirty Rockwell C measurements were performed on each sample.

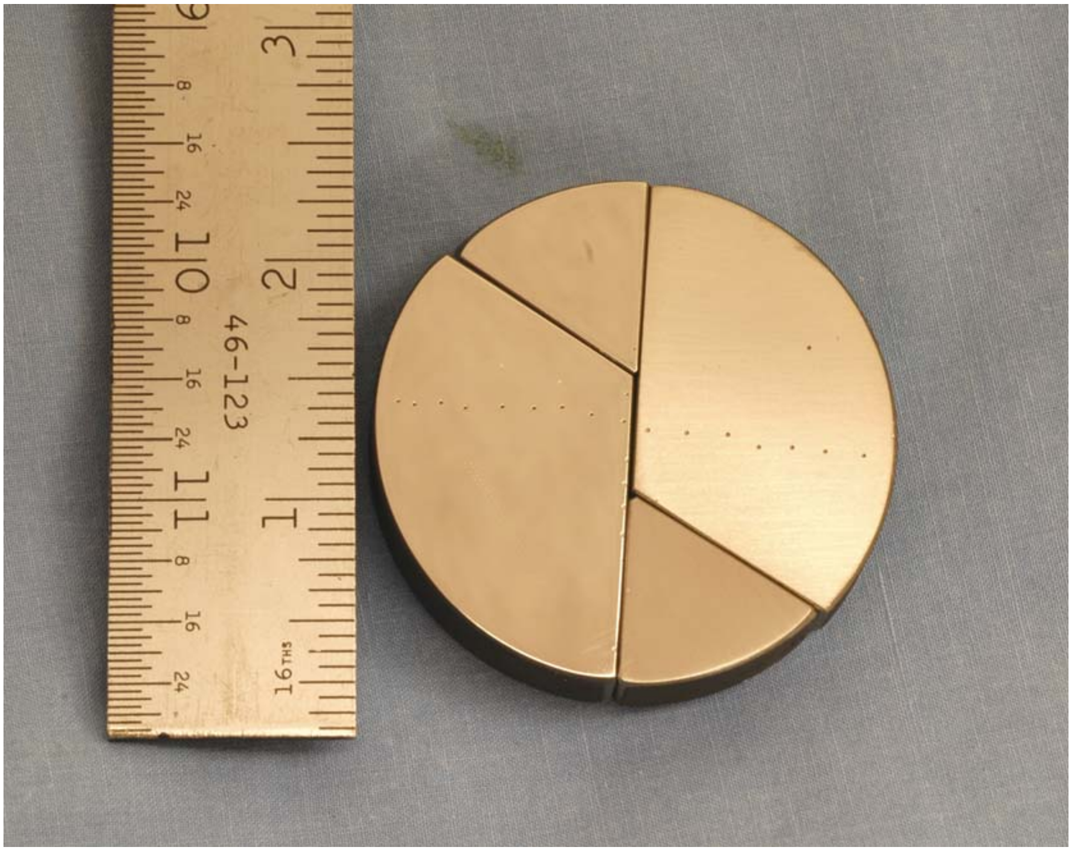


Figure 1 Specimen geometry. Note: the macro hardness tests were performed before the specimen was cut into sections.

Table 1 Tempering Temperatures

FX Steel	Temperature ^oF (^oC)	H-13 Steel	Temperature ^oF (^oC)
FX-00	As-Received	H13-00	As-Received
FX-01	400 (204)	H13-01	900 (482)
FX-02	500 (260)	H13-02	1050 (566)
FX-03	600 (316)	H13-03	1150 (621)
FX-04	700 (371)	H13-04	1250 (677)
FX-05	800 (427)		

2.3 Micro hardness testing

Micro hardness testing was performed using a Vickers indenter with a square pyramid shape. The micro hardness tests use a constant 500 g load with a hold time of 10 s. The indentation size is measured, and a look up table is used to determine the Vickers hardness value [1].

For convenience the samples were cut so that they would fit into the micro hardness tester. Each sample was cut into the geometry shown in Figure 1. One of the two small triangular pieces was used for micro hardness testing. The other small triangular piece was used for the nano hardness tests. The samples were cut and polished with a 1-micron diamond slurry prior to testing.

Micro hardness tests were performed 0.5 mm (0.02 in) apart with about sixty test measurements on each sample.

2.4 Nano hardness testing

Nano testing was done with the Nano Indenter XP machine. Figure 2 [2] shows schematic diagrams of the indentions produced by various indenters, which are used for hardness testing. A Berkovich indenter, which is a triangular pyramid, was used for nano testing. A high-resolution actuator was used to measure indentation, and a high-resolution sensor was used to measure the penetration. The area of contact is determined from indentation depth and the geometry of the indenter. The measured load is the force required to produce the indentation. The load and the area of contact are used to calculate a nano hardness value that has units of stress. The nano hardness value is a measure of the pressure required for indentation not the strength of the material being tested. In these tests, a small oscillation was superimposed on the primary load signal so that hardness and modulus can be obtained over a range of the load displacement curve. The elastic modulus is measured from the elastic recovery during the unloading portion of the oscillation. Figure 3 shows a load versus displacement curve from one of the tests on the FX-02 sample. Figure 4 shows a hardness versus displacement curve from one of the tests on FX-02.

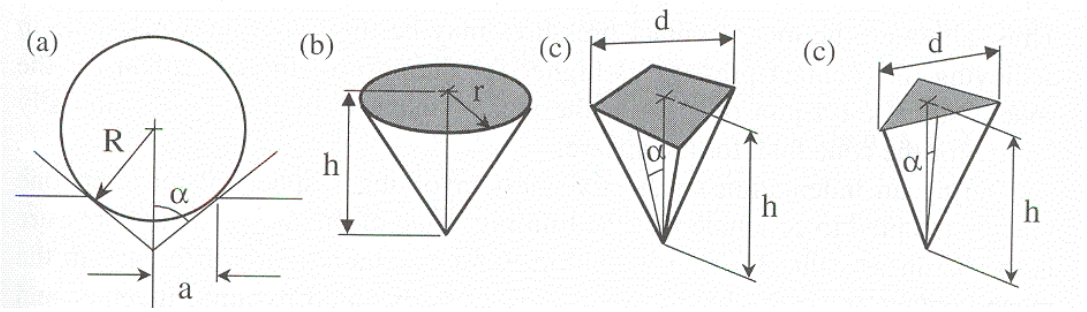


Figure 2 Schematics of various indentions with parameters. (a) spherical indenter (similar to the tip of a Rockwell indenter) with radius, R, angle, α, and indention radius, a, (b) conical indenter with radius, r, and depth, h, (c) pyramidal indenter (e.g. Vickers) with diagonal, d, depth, h, and angle, α, and (d) Berkovich indenter with length, d, depth, h and angle, α [2].

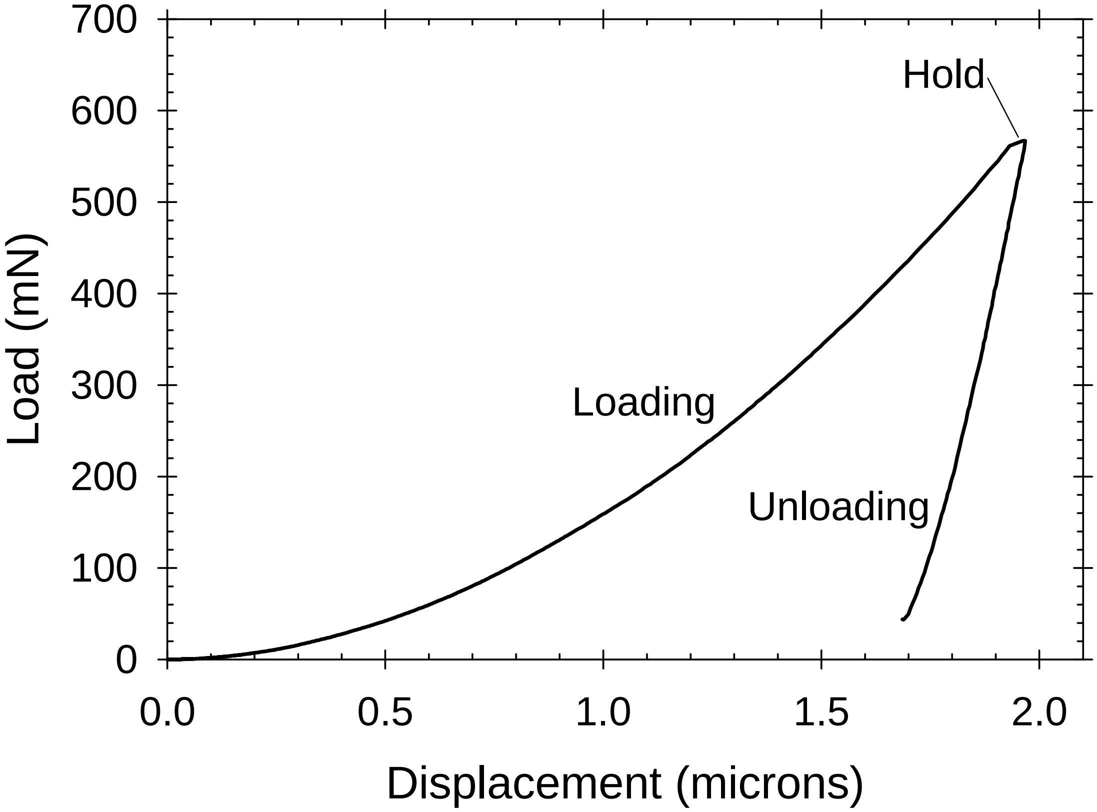


Figure 3 Load displacement curve for a nano hardness test.

The samples were cut to size (i.e. one of the small triangular pieces shown in Figure 1) and glued to a 25.4 mm (1 in) diameter aluminum cylindrical stud. Prior to mounting on the aluminum stud, each sample was polished with 1-micron diamond slurry.

In this study, the nano hardness tests were run to an indentation depth of about 2 microns where hardness and elastic modulus were determined over this penetration range. The load was increased until a depth of just over 1.9 microns was reached, then the load was held constant for about 10 s prior to unloading. A small load oscillation was imposed during loading so that hardness and modulus values could be obtained at each point during the indentation. The total test time was approximately 6 minutes. Indentations were spaced 50 microns apart in sets of ten indentations on three different areas of the sample for a total of thirty tests. The hardness values and elastic modulus were obtained by averaging the data between 0.6 to 1.9 microns on the loading curve as shown in Figure 4. The center of the reduced size sample was tested to avoid effects from any tempering that occurred on the edges during cutting.

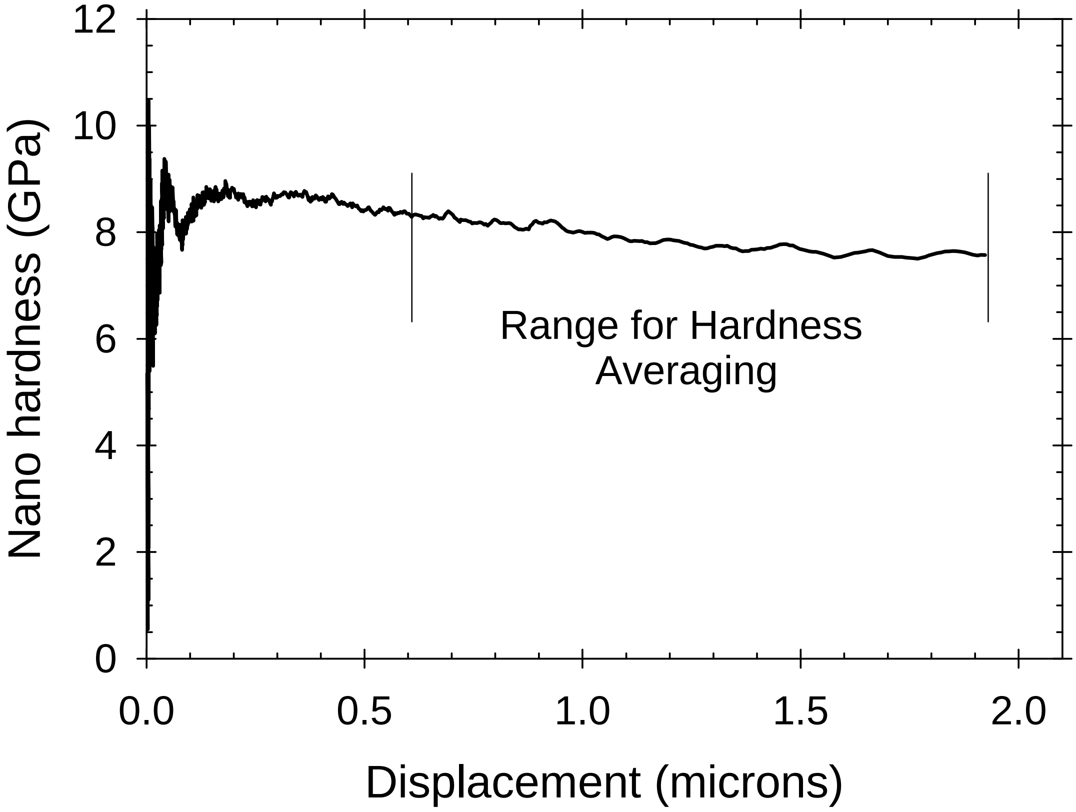
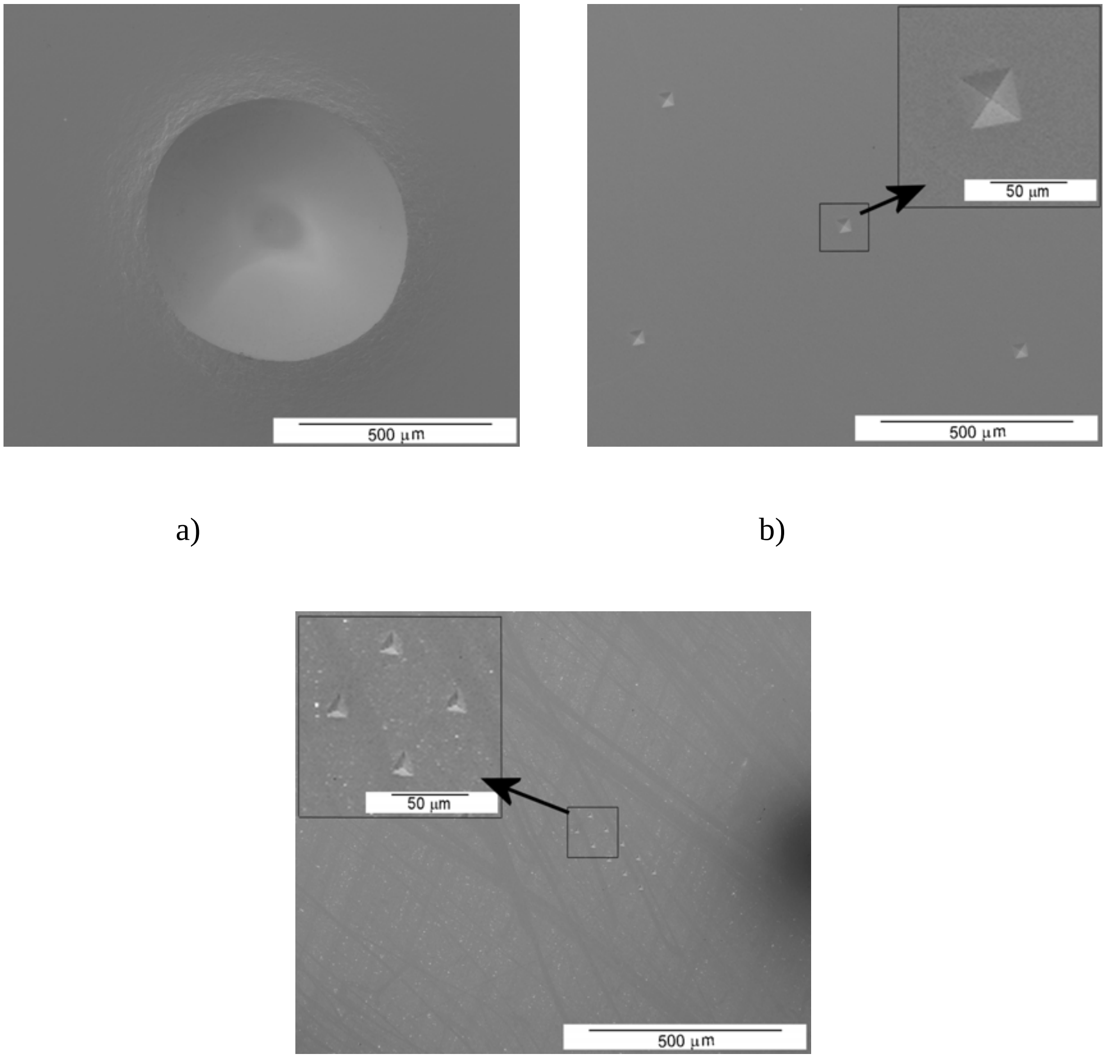


Figure 4 Hardness displacement curve for a nano hardness test.

3. Results

Figure 5 shows scanning electron microscope images of the indentations produced by the three types of hardness tests for the FX-02 sample. The three images in Figure 5 are at the same magnification, so a direct comparison of the indentation size and the volume of material tested by each method can be easily observed. The indentation size on the surface of the steel for the macro hardness test is about 650 microns (0.0260 in) in diameter. The indentation size on the surface of the steel for the micro hardness test is about 50 microns (0.0020 in) along the diagonal. The indentation size on the surface of the steel for the nano hardness test is about 10 microns (0.0004 in) along one of the triangular sides.

More importantly for the present study is the depth of the indentation. For the indentations shown in Figure 5, the depth for the macro hardness indentation is about 230 microns (0.09000 in). For the micro hardness the indentation depth is about 7 microns (0.00028 in). The depth for the nano hardness indentation is about 2 microns (0.00008 in). These differences in depth clearly show the advantage of nano hardness testing is evaluating the surface layer of forging dies.



a) b)

c)

Figure 5 Scanning electron images of hardness indentations all at same magnification. a) macro hardness, b) micro hardness with inset showing indention at a higher magnification and c) nano hardness with inset showing four indentations at a higher magnification.

Table 2 shows the results of the various hardness tests measurements. It can be seen from Table 2 that the stress values for nano hardness are unrealistically high for the strength of the steels. These stress values represent an indentation pressure rather than a material strength. Table 3 gives the elastic modulus measurements that were obtained from the nano hardness tests.

Table 2 Hardness Data

Sample ID	Macro (HRC)			Micro (HV)			Nano (GPa)		
	Average	Standard Deviation	Sample Size	Average	Standard Deviation	Sample Size	Average	Standard Deviation	Sample Size
FX-00	58.09	0.44	32	635.5	31.9	60	8.41	0.44	29
FX-01	57.44	0.36	29	608.9	26.3	60	8.01	0.48	30
FX-02	54.57	0.43	29	591.3	13.5	59	8.21	0.45	30
FX-03	52.46	0.41	30	527.9	15.8	60	7.66	0.22	28
FX-04	51.03	0.46	29	536.4	12.0	60	6.82	0.91	34
FX-05	49.34	0.18	29	507.6	11.0	59	6.93	0.74	29
H13-00	47.86	1.21	33	421.7	12.7	60	6.06	0.34	30
H13-01	47.75	1.59	29	420.0	13.8	60	6.13	0.18	29
H13-02	48.97	1.02	29	458.6	8.7	60	6.53	0.16	30
H13-03	45.87	0.41	29	440.0	6.3	60	6.36	0.20	29
H13-04	31.51	0.20	29	311.6	3.6	61	4.36	0.18	30

4. Discussion

4.1 Hardness results

Table 4 and Figure 6 show the relationship between macro hardness and nano hardness. The linear regression equation for this relationship is:

$$\mathrm{H}_{\mathrm{nano}} = 9.67 + 5.81 \cdot \mathrm{HRC} \tag{1}$$

where $\mathrm{H}_{\mathrm{nano}}$ is the nano hardness value in GPa and HRC is the macro hardness value.

Table 3 Elastic Modulus Data

Sample ID	Modulus (GPa)	Standard Deviation
FX-00	236.24	6.93
FX-01	225.34	8.10
FX-02	249.68	4.88
FX-03	242.22	5.29
FX-04	248.65	23.97
FX-05	229.56	14.55
FX Average	238.61	
H13-00	245.95	14.43
H13-01	253.62	5.60
H13-02	261.76	4.37
H13-03	272.32	4.73
H13-04	267.69	6.78
H13 Average	260.27	

Table 5 and Figure 7 show the relationship between micro hardness and nano hardness. The linear regression equation for this relationship is:

$$H_{nano} = 51.97 + 79.91 \cdot HV \tag{2}$$

where HV is the micro hardness value.

Table 4 Linear Regression and Analysis of Variance (ANOVA) for Nano Hardness as a Function of Macro Hardness

	Coefficients	P-value
Intercept	9.67	0.287
Macro Hardness (HRC)	5.81	4.77×10^{-6}

F-value for Regression	93.32
R^2-value for Regression	0.912

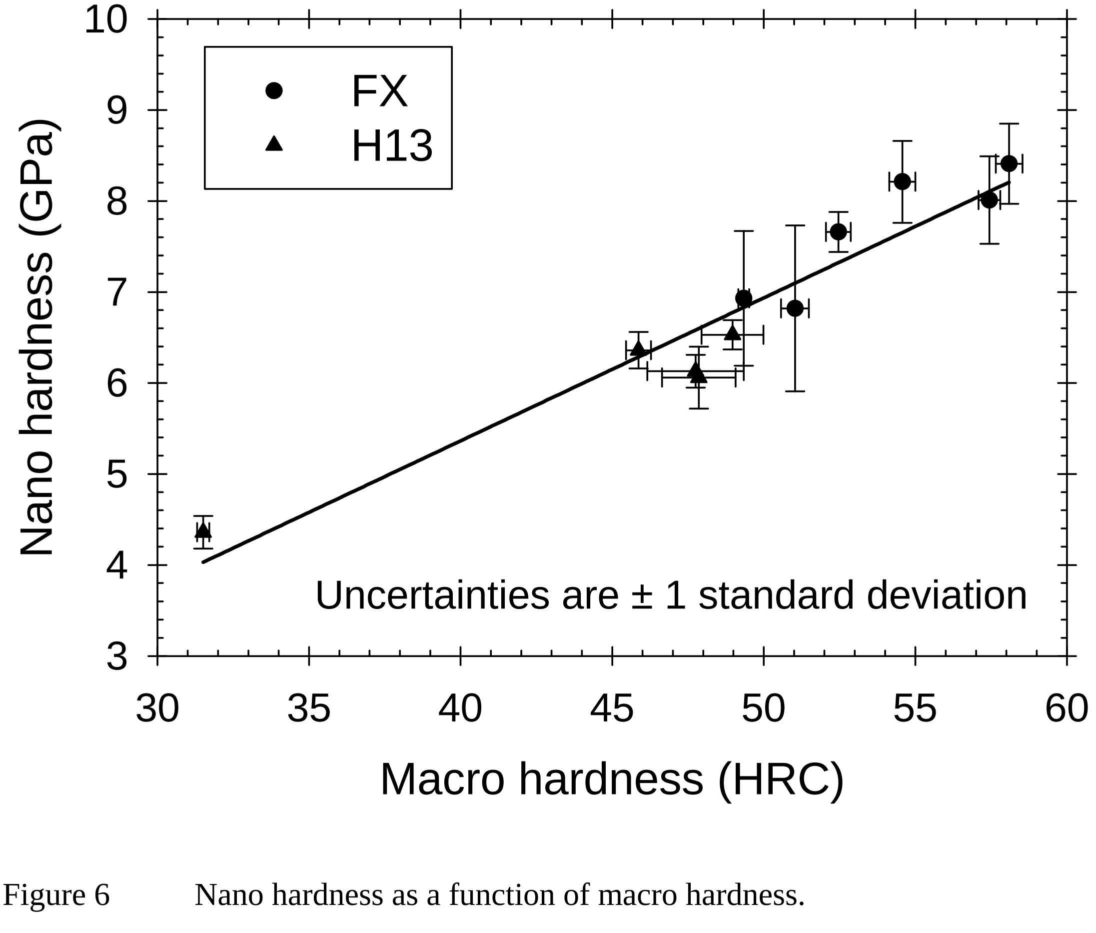


Figure 6 Nano hardness as a function of macro hardness.

Table 5 Linear Regression and Analysis of Variance (ANOVA) for Nano Hardness as a Function of Micro Hardness

	Coefficients	P-value
Intercept	51.97	0.068
Micro Hardness (HV)	79.91	0.424×10^{-6}

F-value for Regression	165.6
R^2-value for Regression	0.948

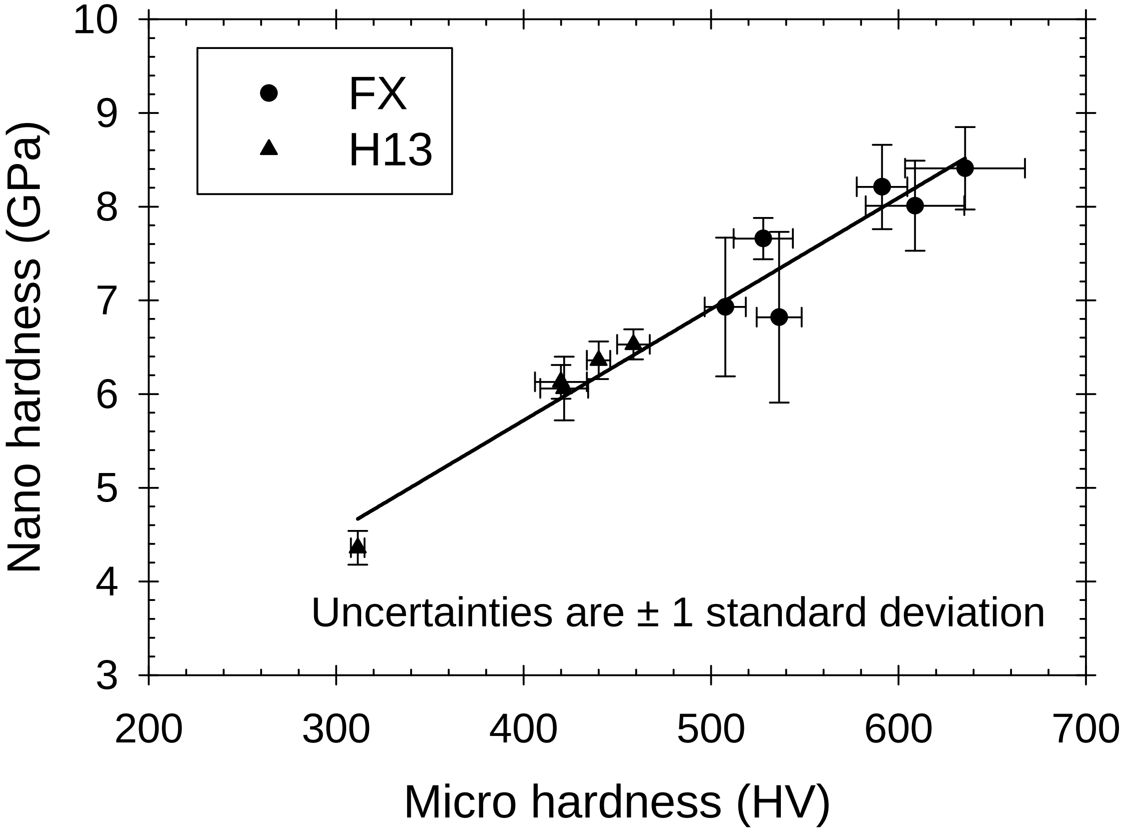


Figure 7 Nano hardness as a function of micro hardness.

Equations (1) and (2) are valid statistical relations. Figures 6 and 7 show that the data points are randomly distributed about each regression line. The regressions exhibit high F values, which indicate that there is a strong dependency between the independent and dependent variables. The P-values for the coefficients in Equations (1) and (2) are sufficiently small to indicate good reproducibility. The square of the correlation coefficient (R^2) for Equations (1) and (2) are 0.91 and 0.95, respectively. These R^2 values indicate that 91% and 95% of the total variation are explained by these linear regression equations.

In comparing Equations (1) and (2), it can be seen that Equation (2) is statistically better because the coefficients have lower P-values and the R^2 value is higher. As expected, it can be seen from Figure 5 that that the indentation size of the micro hardness impressions are much smaller than the macro hardness impressions. The indentation shape produced by the Berkovich indenter, which is used for the nano hardness tests in this study, is designed to be similar to the indentation created by the Vickers hardness indenter.

Even though Equation (2) is statistically more reliable than Equation (1), the experimental results show that nano hardness results from a Nano Indenter XP Machine with a Berkovich indenter can be reliably compared with both macro hardness (Rockwell C) values and micro hardness (Vickers) test results. Thus, the selection of an appropriate hardness test should be based on the problem being investigated. Since the forging industry uses Rockwell hardness and Vickers hardness values, the results of this investigation show that nano hardness test results from a Nano Indenter XP Machine with a Berkovich indenter can be used to reliably analyze superficial layers on the surface of hot forging dies.

4.2 Elastic modulus

The elastic modulus of the material is also obtained during a nano hardness test. Although the elastic modulus values are beyond the scope of the project, they were gathered and are presented in Table 3. The normally accepted value for the elastic modulus of steel is 200 GPa [3], but crystallographic orientation is known to cause variation in the elastic modulus of different steel grades. Since the elastic modulus was not measured by conventional means, it is not known if the discrepancies between the measured elastic moduli and the nominal value for steel are real or due to measurement issues. Nonetheless, it can be concluded that nano hardness testing provides a reasonable engineering approximation of elastic modulus.

It can be seen from Table 3 that for the FX steel, the magnitude of the standard deviations are such that the observed variation is random and related to measurement uncertainty. In contrast for steel H13, the elastic modulus exhibits a consistent increase with increasing tempering temperature. It is of interest to note that the range of tempering temperature for FX is 400 to 800 ^{o}F (204 to 427 ^{o}C) while the range of tempering temperature for H13 is 900 to 1250 ^{o}F (482 to 677 ^{o}C). At these higher tempering temperatures modest changes in crystallographic orientation might be possible.

5. Summary

Nano hardness values obtained using the Nano Indenter XP Machine with a Berkovich indenter reliably correlate with both Rockwell macro hardness values and with Vickers micro hardness values.

Nano hardness testing can provide reliable results for analyzing surface layer of hot forging dies.

Nano hardness testing can be used to determine a reasonable engineering estimate of the elastic modulus.

6. Acknowledgements

The work was performed with the support from a Finkl Challenge Grant administered by FIERF. The authors appreciate the Forging Industry Education and Research Foundation for awarding the grant. The authors also thank C. Ericksen of A. Finkl and Sons, Co. for supplying the steel for this study.

7. References

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