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A Method for Measuring the Hardness of the Surface Layer on Hot Forging Dies Using a Nanoindenter

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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 to obtain. The objective of the present study is to determine if nanoindenter testing provides a reliable method, which could be used to measure the surface hardness in forging die steels. To test the reliability of nanoindenter testing, nanoindenter values for two quenched and tempered steels (FX and H13) are compared to microhardness and macrohardness values. 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 instruments—a Rockwell hardness tester for macrohardness, a Vickers hardness tester for microhardness, and a nanoindenter tester for fine scale evaluation of hardness. The results of this study indicate that nanoindenter values obtained using a Nanoindenter XP Machine with a Berkovich indenter reliably correlate with Rockwell C macrohardness values, and with Vickers HV microhardness values. Consequently, nanoindenter testing can provide reliable results for analyzing the surface layer of hot forging dies.

Keywords forging, mechanical testing, tool steels

1. Introduction

Die wear is a major cause of failure for hot forging dies. Failure is defined as the point where there is a rapid increase in wear that causes the forging impression to grow beyond specified dimensional tolerances. The strength of the surface layer of the die steel at forging temperatures determines the point of the rapid increase in die wear. The following conditions cause changes to the microstructure and the resulting strength 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. 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.

Macro- and microhardness measurements are thought to be too coarse to effectively determine the strength of the surface layer on a hot forging die. The small indentations associated with nanoindenter testing may provide a method to study the changes that occur in the surface layer. Since, experience in the forging industry is based on either micro- or macrohardness measurements, nanoindenter values should be related to micro- and macrohardness to provide credibility.

Hay and Pharr (Ref 1) have shown that nanohardness testing is suitable for isotropic materials that are not subject to creep at testing temperatures or that exhibit significant viscoelasticity. For most ceramics, hard metals and soft metals that strain harden, hardness and elastic modulus can be determined to an accuracy of ±10% (Ref 1). The conclusions of Hay and Pharr must be considered in conjunction results from work of Qian et al. (Ref 2) relating nanohardness to microhardness for copper, stainless steel, NiTi, fused silica, and silica with a (100) crystal orientation. They found that the applied load affects the comparison of nano- and microhardness, and for stainless steel, there is no load for which nano- and microhardness correspond (Ref 2).

Tempered die steels are not "hard materials," and they exhibit minimal strain hardening. So before using a nanoindenter to evaluate the surface layer of forging dies, there is a need to compare nanoindenter values to micro- and macrohardness for common forging die materials. The aim of the present investigation is to determine if relationships between nanoindenter values, microhardness, and macrohardness can be established for two commonly used die steels. The tested samples were tempered under different thermal conditions to develop a range of hardness. This study shows that the values measured by nanoindenter, microhardness, and macrohardness tests provide comparable results. Consequently, nanoindenter testing can be a viable method for studying the changes in the mechanical properties of the surface layer of hot forging dies that control wear.

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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) thick slices from approximately 50 mm (2 in) diameter bar stock. Samples were prepared by furnace tempering in an air atmosphere for 1 h. After tempering each sample was sanded with 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. The microstructure of the samples was either quenched or tempered martensite.

2.2 Macrohardness Testing

Macrohardness 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° cone and a 200 µm tip radius. The hardness reading is based on the measured displacement of the indenter (Ref 3).

The macrohardness indentations were taken from left to right through the centerline of the cylindrical piece and from top to bottom through the centerline. These macrohardness tests were performed prior to the sectioning of the specimen into four sections as shown in Fig. 1. The surfaces were ground with 400-grit paper prior to testing. These measurements provided the hardness values and 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 30 Rockwell C measurements were performed on each sample.

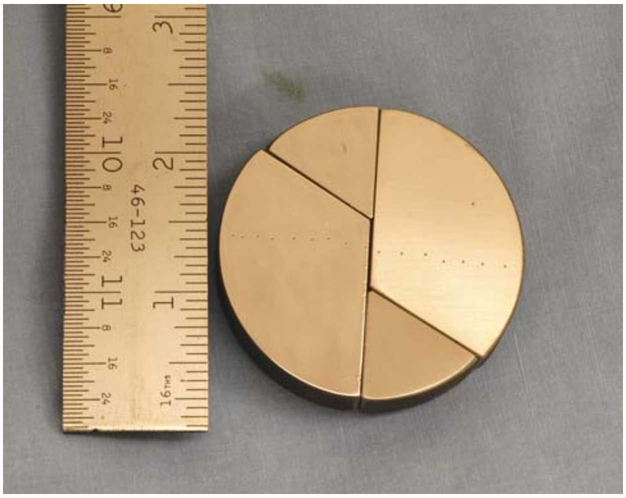

Fig. 1 Specimen geometry. Note: the macrohardness tests were performed before the specimen was cut into sections. The disk specimen was ground to 400-grit prior to testing. The two small sections were polished with 1-µm diamond paste before testing

Table 1 Tempering temperatures

FX steel	Temperature, °F (°C)	H-13 steel	Temperature, °F (°C)
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 Microhardness Testing

Microhardness testing was performed using a Vickers indenter with a square pyramid shape. The microhardness 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 (Ref 4).

For convenience, the samples were cut so that they would fit into the microhardness tester. Each sample was cut into the geometry shown in Fig. 1. One of the two small triangular pieces was used for microhardness testing. The other small triangular piece was used for the nanoindentation tests. These sectioned samples were cut and polished with a 1-µm diamond slurry prior to testing.

Microhardness tests were performed 0.5 mm (0.02 in) apart with about 60 test measurements on each sample.

2.4 Nanoindenter Testing

Nanoindenter testing of forging die steels causes both elastic and plastic deformation. Hardness is determined from the ratio of the maximum load to the projected area of contact. The projected area of contact is determined from an analysis of the indentation load-displacement curve that requires consideration of elastic recovery. This requires consideration of pile-up around an indentation and change of the indentation geometry due to elastic recovery. Also, for sharp indenters, such as a Berkovich indenter, at very shallow indentations, elastic contact can predominate because of tip rounding effects. When elastic contact predominates, the analysis of the effect of elastic recovery on the projected area of contact is more difficult to determine. Full details of the necessary procedures are provided elsewhere (Ref 1).

It should be noted that nanoindenter measurements have units of stress. The nanoindenter value is a measure of the pressure required for indentation not the strength of the material being tested.

Figure 2 shows schematic diagrams of the indentions produced by various indenters, which are used for hardness testing (Ref 5). In an analysis of the projected area of contact, it is found that similar relations exist for square cross sections like the Vickers pyramid and triangular cross sections like a Berkovich indenter (Ref 1).

The elastic modulus is taken from the slope of the unloading curve, and several methods are proposed. It is also indicated that the initial portion of the unloading curve should be used (Ref 1).

Nanoindenter testing was done with a Nanoindenter XP machine using a Berkovich indenter. A high-resolution actuator was used to measure indentation, and a high-resolution sensor was used to measure penetration. The apparent area of contact is determined from indentation depth and the geometry of the indenter. In these tests, a small oscillation was superimposed on

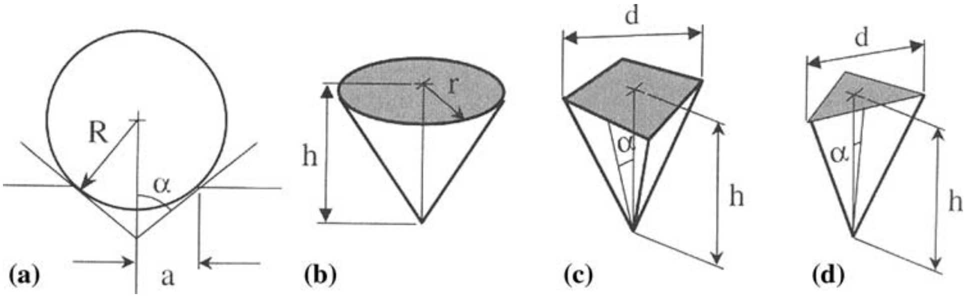

Fig. 2 Schematics of various indentions with parameters. (a) Spherical indenter (similar to the tip of a Rockwell indenter) with radius, *R*, angle, α, and indention radius, *a*, (b) conical indenter with radius, *r*, and depth, *h*, (c) pyramidal indenter (e.g., Vickers) with diagonal, *d*, depth, *h*, and angle, α, and (d) Berkovich indenter with length, *d*, depth, *h* and angle, α (Ref 5)

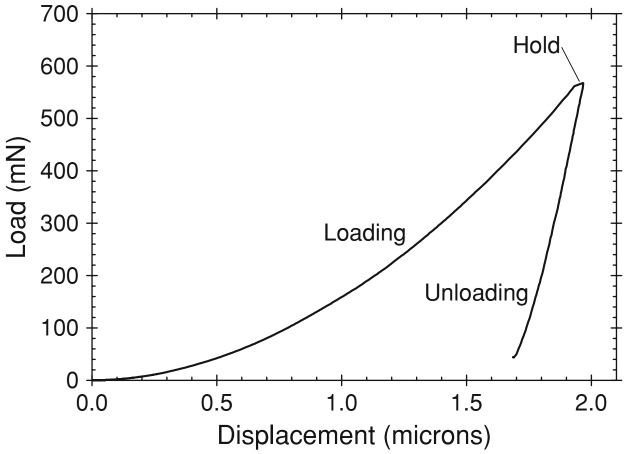

Fig. 3 Load-displacement curve for a nanoindenter test

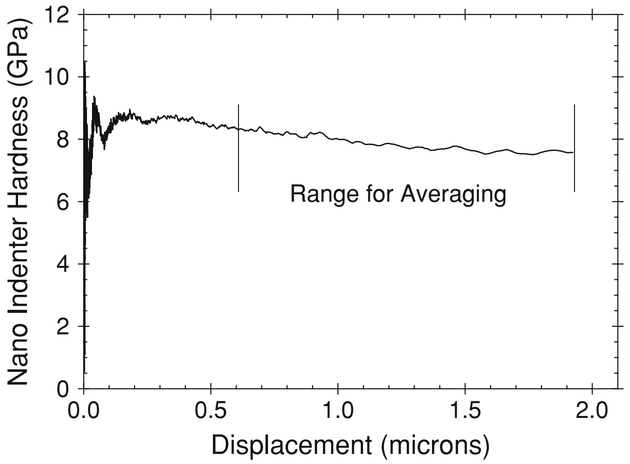

Fig. 4 Hardness as a function of displacement for a nanoindenter test

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.

The samples were cut to size (i.e., one of the small triangular pieces shown in Fig. 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-μm diamond slurry.

The nanoindenter tests were run to an indentation depth of 2 μm, and hardness and elastic modulus were determined over this penetration range. Although this depth is larger than the "nano-range" as defined in the ISO standard (Ref 6), a Nanoindenter XP machine was used. In effect, the test was a fine microindentation. The results in this study are called nanoindenter test results because of the testing instrument used and to clearly distinguish them from the other two sets of test data. Since the objective of the work was to compare the results for these three test methods, the deeper penetration was used to insure that the measurement was of material that was comparable to the material tested in the macrohardness and microhardness tests. A 2-μm indentation depth is adequate to evaluate the thin surface layer of a forging die, so measurements at shallower penetration depths are not needed.

The load during the nanoindenter test was increased until a depth of just over 1.9 μm was reached, then the load was held constant for 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 min. Indentations were spaced 50 μm apart in sets of 10 indentations on three different areas of the sample for a total of 30 tests. The hardness values and elastic modulus were obtained by averaging the data between 0.6 and 1.9 μm on the loading curve as shown in Fig. 4. The center of the reduced size sample was tested to avoid effects from any tempering that occurred on the edges during cutting.

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 Fig. 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 macrohardness test (Rockwell C) is about 650 μm (0.0260 in) in diameter. The indentation size on the surface of the steel for the microhardness test (Vickers) is about 50 μm (0.0020 in) along the diagonal. The indentation size on the surface of the steel for the nanoindenter test is about 10 μm (0.0004 in) along one of the triangular sides.

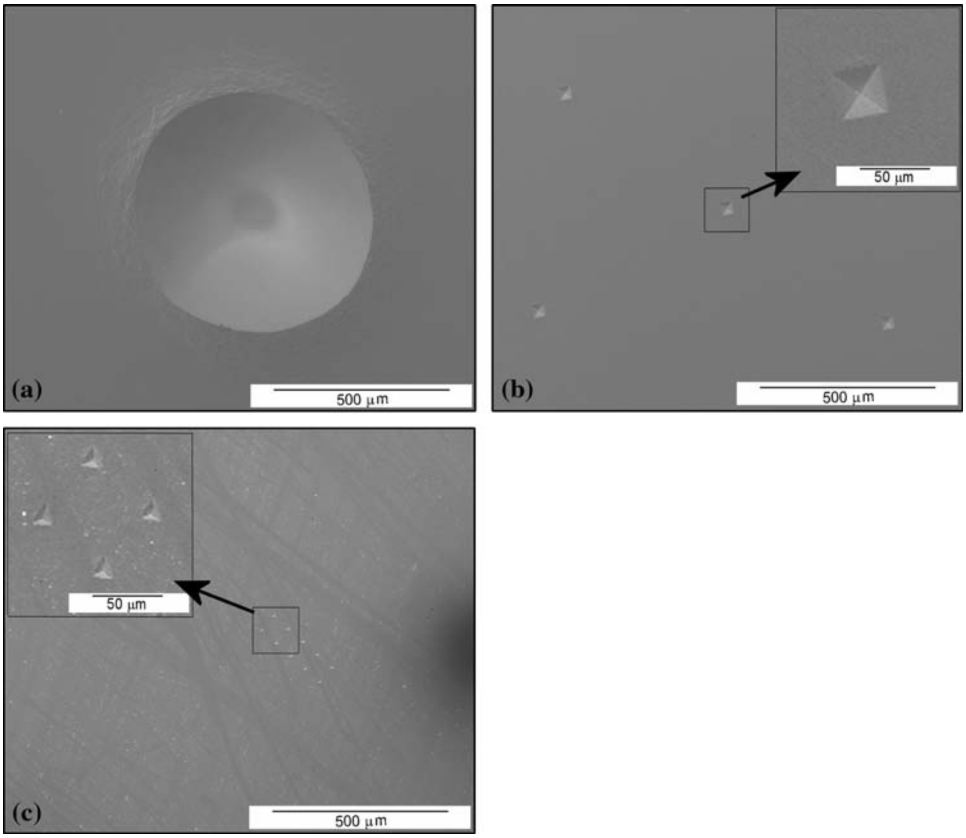

Fig. 5 Scanning electron images of hardness indentations all at same magnification. (a) macrohardness, (b) microhardness with inset showing indention at a higher magnification, and (c) nanoindenter indentation with inset showing four indentations at a higher magnification

Table 2 Experimentally measured data

	Macrohardness, HRC			Microhardness, HV			Nanoindenter hardness, GPa		
Sample ID	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

For a study of a forging die, the depth of the indentation is more important. For the indentations shown in Fig. 5, the depth for the macrohardness indentation is about 230 µm (0.09000 in). For the microhardness, the indentation depth is about 7 µm (0.00028 in). The depth for the nanoindentation is about 2 µm (0.00008 in). These differences in depth clearly show the advantage of nanoindenter testing for the evaluation of the surface layer of forging dies.

Table 2 shows the results of the various hardness tests measurements. It can be seen from Table 2 that the stress values for nanoindenter 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 nanoindenter tests.

4. Discussion

4.1 Hardness Results

Table 4 and Fig. 6 show the relationship between macrohardness and nanoindenter test results. The linear regression equation for this relationship is:

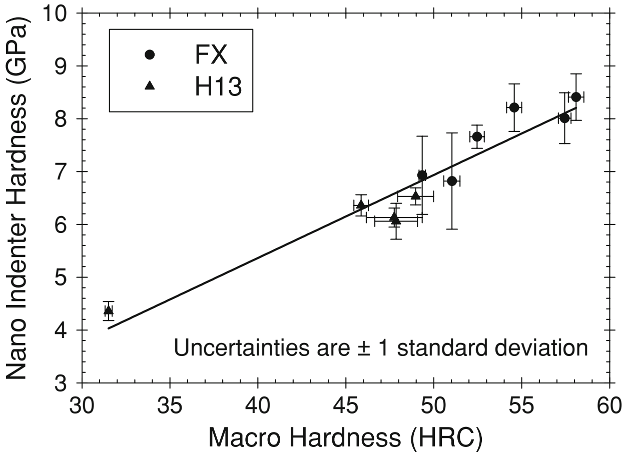

Fig. 6 Nanoindenter hardness as a function of macrohardness

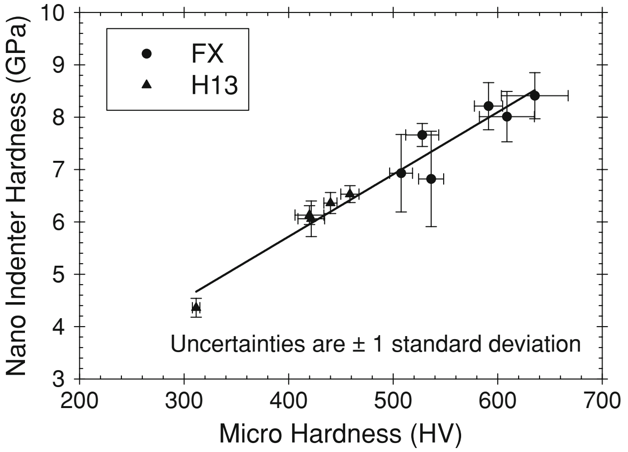

Fig. 7 Nanoindenter hardness as a function of microhardness

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 4 Linear regression and analysis of variance (ANOVA) for nanoindenter hardness as a function of macrohardness

	Coefficients	*P*-value
Intercept	9.67	0.287
Macrohardness (HRC)	5.81	4.77×10^{-6}
F-value for regression	93.32	
R^2-value for regression	0.912	

$$H_{\text{nano}} = 9.67 + 5.81 \cdot \text{HRC} \qquad \text{(Eq 1)}$$

where H_{nano} is the nanohardness value in GPa and HRC is the macrohardness value.

Table 5 and Fig. 7 show the relationship between microhardness and nanoindenter test results. The linear regression equation for this relationship is:

$$H_{\text{nano}} = 51.97 + 79.91 \cdot \text{HV} \qquad \text{(Eq 2)}$$

where HV is the microhardness value.

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 Eqs 1 and 2 are sufficiently small to indicate good reproducibility. The square of the correlation coefficient R^2 for Eqs 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.

Table 5 Linear regression and analysis of variance (ANOVA) for nanoindenter hardness as a function of microhardness

	Coefficients	*P*-value
Intercept	51.97	0.068
Microhardness, HV	79.91	0.424×10^{-6}
F-value for regression	165.6	
R^2-value for regression	0.948	

In comparing Eqs 1 and 2, it can be seen that Eq 2 is statistically better because the coefficients have lower *P*-values and the R^2 value is higher. The Vickers and Berkovich indenters have similar geometric effects on indentation geometry (Ref 1), so it is reasonable that Vickers and nanoindenter test values should exhibit a better statistical relationship.

Even though Eq 2 is statistically more reliable than Eq 1, the experimental results show that nanoindenter results from a Nanoindenter XP Machine with a Berkovich indenter can be reliably compared with both macrohardness (Rockwell C) values and microhardness (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 nanoindenter test results from a Nanoindenter 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

Although it is outside the scope of this study, the elastic modulus was determined and is reported as additional information. The normally accepted value for the elastic modulus of steel is about 207 GPa. It can be seen from Table 3 that the reported elastic moduli are significantly larger than 207 GPa. Since the elastic modulus of the steels in this study 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.

It can also 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 probably 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 204-427 °C (400-800 °F) while the range of tempering temperature for H13 is 482-677 °C (900-1250 °F). At these higher tempering temperatures, modest changes in crystallographic orientation that could affect the elastic modulus might be possible.

In general, it is reported that when there are discrepancies in nanotesting, both hardness and elastic modulus can be in error (Ref 1). If there is pile-up, measured hardness values can be as much as 60% higher than actual values and measured elastic modulus values can be as much as 30% higher than actual values (Ref 1). In the present study, the correlation between nanoindenter test values, microhardness, and macrohardness indicate that the nanoindentater measurements are consistent. In contrast, the elastic modulus measurements are consistent with a 20-40% increase in elastic modulus.

In other work on quenched AISI 52100 steel, it has been shown that both nanohardness and elastic modulus agree with expected values (Ref 7). The only difference between AISI 52100 and tempered FX and H13 die steels is that AISI 52100 has a much higher volume of carbide and the martensite is not tempered. From available evidence, it cannot be determined if the accurate determination of elastic modulus for AISI 52100 is due to the difference in microstructure or differences in the measurement technique that was used.

5. Summary

Nanoindenter test results obtained using the Nanoindenter XP Machine with a Berkovich indenter reliably correlate with both Rockwell macrohardness values and with Vickers microhardness values.

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

Determining accurate values of elastic modulus is more difficult than determining nanoindenter hardness values.

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