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USING NEW ATOMIC FORCE MICROSCOPE SOFTWARE TO MEASURE THE HARDNESS OF GRAINS AND MICROCONSTITUENTS

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Abstract - It is desirable to measure the hardness of individual grains and microconstituents to have control over the mechanical properties of materials. An ultra-micro or nanoindenter is required to make indents small enough to fit inside a single grain or phases that is smaller than 10 mm diameter. Because the indents are too small for an optical microscope an atomic force microscope was used to view the location and measure the contact area. Measuring the contact area of indents from an atomic force microscope image is unreliable because it is difficult to manually locate the indent edge. To solve this problem computerized image analysis software called NanoMc was used to measure the residual indent contact area. This software digitally reconstructed the residual indent back into the fully loaded indentation shape and then measures the contact area and depth. This method avoids the complicated tip rounding and load-frame compliance problems. As an example this method was used to measure the hardness of pearlite and ferrite microconstituents in SAE 1020 steel.

Keywords : hardness, AFM, software

1. INTRODUCTION

Materials engineer could have a better understanding of materials by measuring the fundamental properties at the nanoscale. One method to measure the strength of materials at a small size is by using indentation hardness testing. Hardness, *H*, is equal to the force, *P*, divided by the contact area, A_c, when the indenter is at full load (1).

$$H = \frac{P}{A_c} \tag{1}$$

Forcing a hard diamond indenter into a material makes an indentation. Nanoindentations that are around 200 nm deep, are too small to be measured optically. Researchers have devised techniques to measure the contact area of the indentation without optical imaging by using a depth-sensing indentation (DSI) hardness tester [1, 2]. Their methods use the depth of the indentation along with its known geometry to calculate the contact area and then the hardness. Unfortunately the DSI technique works best with monolithic materials but not very well for polycrystalline materials.

An example application would be to measure the hardness of individual grains of polycrystalline or multiphase materials. The indentations must be small enough to fit inside the area of a single grain typically less than 10 mm. Indents of this size would be too small to be measured optically. An atomic force microscope (AFM) is an appropriate tool to image and measure the contact area of this size of an indentation. However measuring the indentation shape from the AFM image is unreliable. Therefore, the objective of this research was to measure indentation contact area using software.

Typical AFM's create a 3-dimensional digital image of the residual indent that can range from a few nanometers up to a micrometer deep. However, it is impossible to measure the indentation contact area from a residual indent image because of elastic recovery. Elastic recovery causes the residual indent image to be smaller than the fully loaded indentation.

Commercially available computerized image analysis software called NanoMc was used to measure the contact area [3, 4]. The NanoMc software can digitally reconstruct the residual indent images back into the fully loaded indentation shape. The elastic reconstruction is based on the known indenter geometry and the elastic properties of the sample material. After reconstruction the software automatically outlines the indentation and measures the contact area. The hardness was calculated using the force from the hardness tester along with the NanoMc indentation contact area. This method eliminated the tip rounding and load-frame compliance errors associated with the DSI technique. The mathematical and image analysis behind this software method are explained in previous works [3].

2. EXPERIMENTAL DESCRIPTION

2.1. Sample Preparation

One sample of SAE 1020 steel was selected and prepared for these experiments. This material was chosen because its properties are well known and it has two microconstituents of different hardness: ferrite and pearlite. The hardness of ferrite and pearlite grains will be compared to the bulk hardness of ferritic and pearlitic steel.

Sample preparation was done using standard metallographic procedures. The sample came in the form of a 2,54 cm diameter rod. It was cut into disk samples. The

samples were hand polished using a series of SiC wet sandpaper, and then diamond 1 mm for 5 minutes. The final polishing step was done using suspended colloidal silica for 5 minutes using a product from Struers™. A chemical agent in the polishing solution helped to decorate the sample by etching a few nanometers deep into the cementite. An AFM was used to image the surface of the polished sample to measure its roughness. The root-mean-square (RMS) roughness of the image scan area was 2,5 nm (one sigma).

2.2. Indentation Procedure

A Shimadzu™ ultra-micro hardness tester (DUH-W201S) was used to make the indents. A standard 115° Berkovich indenter was used. This type of hardness tester applies the force by sending an electrical current through a magnetic coil. The electron flow causes a magnetic force to be applied to a permanent magnet and then onto the indenter. This machine has a force range from 0,0196 to 1960,0 mN with an accuracy of ±19,3 mN.

A scratch was intentionally made in the polished sample from the center to the edge. The center point of this scratch marks where the indentation pattern will start. This mark would later be used to assist in finding the tiny indents with the optical microscope attached to the AFM. An indent around 300 nm deep and 2 mm across looks like a speck of debris with a typical optical microscope. The indents are separated equally by using stage control screws. A pattern of equally spaced indents is easy to distinguish from the surface debris. For the grains that are visable with an optically the indenter was placed directly in the center of the desired grain.

The proper force to create indents around the desired depth was found by making a series of indents over a range of forces. The depth should be less than 10% of the half of the diameter of the grain. This 10% value is the same rule-of-thumb used to avoid substrate effects when testing thin-films. In this case it was used to avoid the deformation of the surrounding microstructures.

Over 26 indents were made in the SAE 1020 steel sample. The first 6 indents were used to find the force required to make an indent less than 10% of half of the grain diameter. By random selection the grain diameter was about 6 mm so therefore the depth should be less than 300 nm. A force of 5,0 mN made indents about 250 nm deep. Then twenty indents were made using 5,0 mN force, 30 s approach rate, and 30 s hold time. The laboratory conditions during the hardness tests were 19°C and 61% humidity.

2.3. Imaging

A Veeco Instruments Dimension 3100 scanning probe microscope (SPM) was used to image each of the residual indents. This microscope was setup to operate as an AFM, in tapping mode, using a 220 mm length TESP cantilever. Before imaging the indents the AFM was calibrated using the built-in software calibration procedure. This calibration procedure involved scanning a 1 mm sample grid at various scan rates, scan sizes and orientations. Imaging an 800 nm step height was used for the z-calibration. Other calibration grids were imaged to calculate the accuracy. It was found that the error was less than 5% in the x, y and z directions after the scanner had relaxed.

The optical microscope of the AFM was used to locate the indents near scratch. Because the indents were equally spaced apart it was easy to distinguish them from the sample debris. The indents were imaged in the same order they were made by the hardness tester. Images were made with the same scan rate of 1 Hz. The scan size was either 10x10 mm or 15x15 mm. Before capturing the image the scanner was allowed to relax. The scanner was assumed to be sufficiently relaxed when the polish scratch lines appeared straight.

2.4. Hardness Measurement

The SPM image analysis software NanoMc was used to measure the indentation contact area. This software automatically locates the indent in the AFM image, measures its contact area and calculates the hardness. The software asks the user to enter the force used to make the indentation and allows for adjustment to the outline location quality. The software takes about 6 seconds to measure the indent using a 450 MHz computer processor. It displays an image of the indent with a white outline and then a dialog box pops-up showing the hardness value. The white outline is used to visually verify that the contact area measurement was done correctly.

The hardness was measured for each indent. The hardness tester force was assumed to be accurate based on the manufacturer calibration. The displacement from this depth-sensing indentation tester was not used. Instead the AFM with the image analysis software NanoMc was used to measure the contact area. The indent was digitally reconstructed in order to measure the contact area. As a test of the elastic reconstruction the average of the three face angles of the reconstructed indent image can be compared to the face angles of the Berkovich indenter.

The NanoMc software can measure the pile-up contact area but it was ignored for now because its effect on hardness has not yet been sufficiently researched therefore, this analysis only considered the contact area below the surface height.

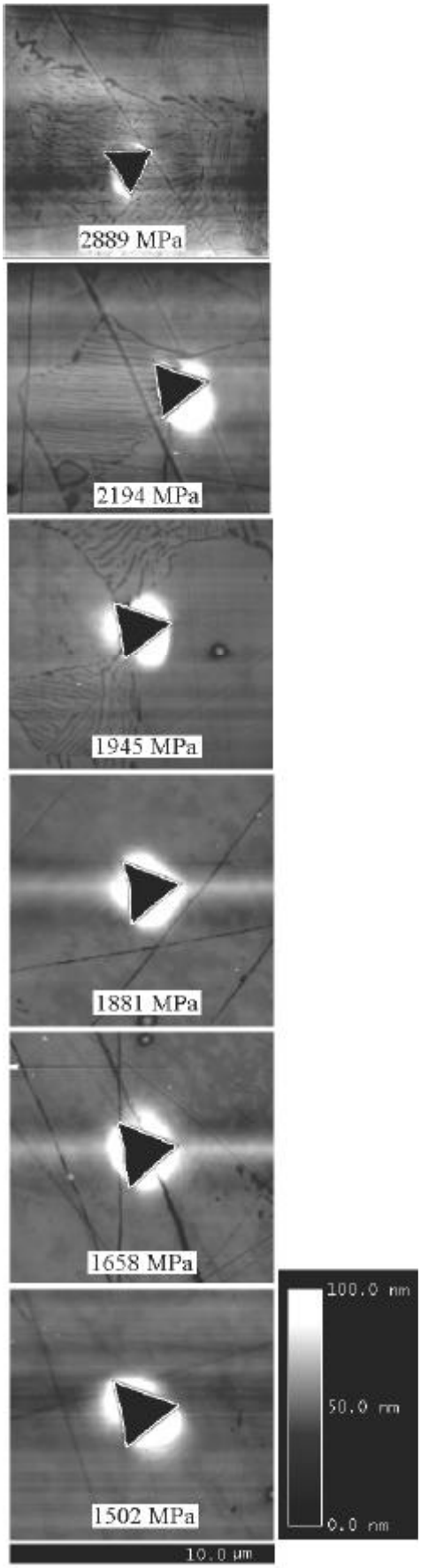

Figure 1. The hardness of SAE 1020 steel made using 5,0 mN force, 30 s approach rate, 30 s dwell time, and a Berkovich indenter. The average contact depth was 282 nm.

3. RESULTS AND DISCUSSION

3.1. Results

Figure 1 shows a series of indents made in SAE 1020 steel. The number shown at the bottom of each indent image is the hardness. Twenty indents were imaged however only the six best indent images in ferrite and pearlite were used. Indents that were contaminated by debris or clearly defective were not used. A white colored bulge can be seen in the 1881 and 1945 MPa indentations. By image manipulation it was possible to improve the aesthetic appearance however, that would alter the indent contact area measurement. Therefore, image correction was not performed on the measured indent.

Hardness equaled the force divided by the NanoMc contact area. The white outline on the indents shown in Figure 1 was digitally added by the NanoMc software to indicate where it made the measurement. Each of the indents was elastically reconstructed prior to measuring the contact area. For this steel sample the “elastic constant” was found to be 0,183 in both the ferrite and pearlite regions. This “elastic constant” is the amount of elastic recovery upon unloading under the indenter. The elastic reconstruction method was tested by taking an average of the sidewall angles of the reconstructed indent image. The average was approximately 65^{o} for all three faces which nearly equals the face angle of the Berkovich indenter. Only in the tip rounded area was it different.

3.2. Discussion

Figure 1 gives the hardness for various locations in SAE 1020 steel each made using 5,0 mN force. The ferrite region had less hardness than the pearlite region. As the amount of pearlite under the indenter increased the hardness increased as shown by the sequence of images. The hardness ranged from 1502 MPa for ferrite to 2889 MPa for pearlite. The hardness consistently corresponded with the microstructure. The 1502 MPa ferrite indentation had a contact depth of 344 nm and for the 2889 MPa pearlite it had contact depth of 220 nm as measured by the NanoMc software after elastic reconstruction.

These hardness results compared well to those published by Rodríguez and Gutierrez [5]. The results were comparable because both used a Berkovich indenter. Their hardness tester went up to 650 mN force. The hardness at infinite depth of ferritic steel ranged from 1,6 to 2,5 GPa because of increasing alloy content. These results showed that the hardness for ferrite grains ranged from 1,5 to 1,9 GPa. For pearlitic steel at infinite depth the hardness was 2.9 GPa. This reasearch measured a hardness of 2,9 GPa directly in the center of a pearlite. Both the ferrite and pearlite hardness measurements corresponded well with the bulk materials. At the ferrite-pearlite boundary the hardness was proportional to a mixture of the two microconstituents.

Contrary to the results of Rodríguez and Gutierrez the indent size effect (ISE) was not observed [5]. According to their results the hardness at our experimental depths of 200 to 350 nm should be 2,3 GPa for ferrite and 4,5 GPa for

pearlite. The reason why their results had the ISE was probably caused by the tip rounding error. This AFM/NanoMc method of measuring the contact area avoids the tip rounding measurement error.

Ferrite hardness had a variation of about 400 MPa. Other researchers have found that the crystallographic orientation will influence the hardness for iron and aluminum [6, 7]. The crystallographic orientation influences the shape of the pile-up. By studying the pile-up shape in Figure 1 a difference was observed for 1502, 1658 and 1881 MPa. The hardness of the indent with 1502 MPa had a pile-up distribution that was different than the 1658 and 1881 MPa indents.

4. CONCLUSION

To improve hardness testing accuracy an AFM was used to measure the contact area in order to avoid the tip rounding and frame-compliance. Software called NanoMc was used to measure the AFM images of residual indents. It digitally reconstructed the indent into the fully loaded indentation shape and then automatically measured its contact area. This method was used to measure the hardness of ferrite and pearlite of SAE 1020 steel. The hardness results compared with bulk ferritic and pearlitic steels. Ferrite showed a hardness variation that was possible due to a difference in crystallography orientation.

This method appears to be a viable technique to measure microconstituents and crystallographic influences on hardness.

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OTHER REMARKS:

- The NanoMc software can be obtained by visiting the Internet website: www.nanomc.com