Computerized Image Analysis Software for Measuring Indents by AFM

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BIOGRAPHY

David J. Shuman received his MS in materials science and engineering in 2001 from Michigan Technological University of Houghton, Michigan. He has created patents and software related to SPM. He is the author/coauthor of three scientific papers and has received several citations. His research focuses on nanotechnology, AFM mechanical testing and computerized image analysis. Recently he has begun investigating methods to improve depth-sensing indentation measurements of polycrystalline materials.

ABSTRACT

Indentation testing measures the mechanical properties of materials from the nanoscale to microscale. An atomic force microscope can be used to image the residual indent. However, it is difficult to measure the indentation from a complex three-dimensional image, therefore automated software was developed to measure the depth, projected area and effective surface area. This software digitally reconstructs the fully loaded indentation shape from the residual indent image. The contact depth, contact area, indenter angle and tip radius are measured from the reconstructed image.

KEYWORDS

atomic force microscopy, nanoindentation, hardness, pile-up, image analysis, reconstruction

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INTRODUCTION

The objective of this research was to develop reliable image-analysis software to measure the complex shape of indents that have been imaged with an atomic force microscope (AFM) (Fig. 1) and provide more detailed and reliable results than traditional techniques. A common technique for measuring hardness is based on the Vickers microhardness tester, which requires a person to visually measure the diagonal of a residual indent impression with an optical microscope. How the optical microscope is focused and where the operator chooses to measure the diagonal may cause variable results. One method to measure the hardness without having to image the surface is by using a technique called depth-sensing indentation (DSI) [1]. A DSI tester records the displacement and force during the indentation process. Its load-unload curve is used to measure the indentation depth and calculate the sample hardness and modulus.

However, current technology for nanoindentation testing is only suitable for monolithic materials such as thin films. A common measurement artifact from nanoindentation is called the indent size effect (ISE), which is when the hardness appears to increase as the indentation depth decreases. However, when the contact area was measured correctly for single-crystal silicon the hardness remained constant into nanoscale depths [2]. Nazarov et al. [3] stated that with the nanoindentation technique "it is not necessary to make the difficult and unreliable measurements of the geometrical dimensions of contact area because information about the penetration depth of the indenter into the solid during the loading cycle allows us to exclude the influence of elastic recovery on the hardness value." This paper describes a computerized image analysis software technique that can measure the contact area plus several other dimensions from a residual indent including the elastic recovery. Measuring the indentations shape using computerized image analysis software reduces the human error and provides reproducible results.

The state-of-the-art of image analysis for indent image measurement depends on using a technique called threshold to measure the residual indent. A threshold is done by converting a grayscale image into a binary image such as the height grayscale for AFM images. After doing the threshold the highlighted binary pixels are summed to equal the total projected area. In order to avoid the surrounding surface noise the threshold height is placed slightly below the sample surface height, which causes an error in the projected area measurement. The outline around the indent is more complex than the threshold flat plane assumption because of surface roughness, sink-in and pile-up. Sink-in happens when the edge of the indent collapses below the surface height after indentation. Also, the threshold technique does not measure the surface area, contact depth or contact area.

MATERIALS AND METHODS

Software was used that automatically measures the indent depth, projected area, surface area, volume, pile-up dimensions, contact depth and contact area. Research by Stillwell and Tabor [4] found that a residual indent covered the same projected area as the fully loaded indentation. A residual indent has a smaller depth than the fully loaded indentation. This software digitally reconstructs the fully loaded indentation shape from a residual indent image [5]. From the reconstructed image the contact area and contact depth are measured and used to calculate the contact hardness, or projected area function. The projected area function could be used to calibrate a DSI. The pile-up dimensions relate to the

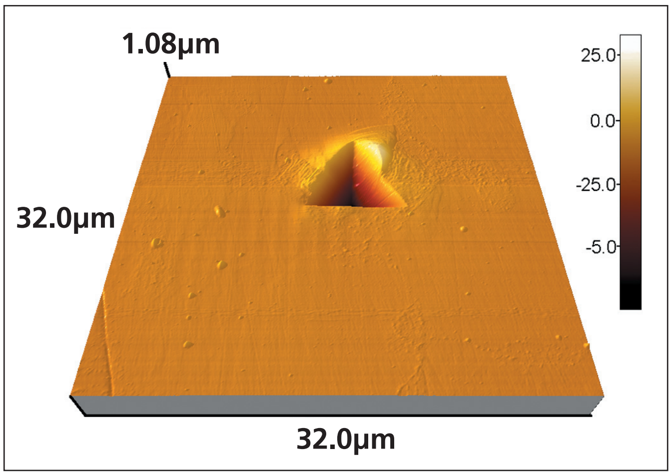

Figure 1: An atomic force microscope image of a Berkovich indentation made in AISI 1020 steel using 84.7 mN force (z-scale 4:1).

material's crystal structure [6]. Hardness test results depend on the indenter shape. If the indenter is too rounded then the hardness will have a significant error. The conical indenter angle and tip radius could also be determined using this software.

The computerized image analysis software called NanoMc was developed by the author. It was written using Microsoft Visual C++ as a dynamic link library (DLL) plug-in for Nanoscope III 5.12 or 5.13 from Veeco Instruments and also for SPIP software from Image Metrology. The Nanoscope and SPIP software handle the complicated graphical user interface. The advantage to the SPIP program is that it imports a wide variety of commercial scanning probe microscope images. It can be downloaded and evaluated for free by visiting the Nanomc.com website.

This paper used the same indentation image of an AISI 1020 steel sample for all examples. The sample rod was cut and polished using standard metallographic procedures. The rough surface was removed using SiC wet sandpaper, suspended 1 µm diamond paste and then the final polishing step was done using Struer OP-S. The indent was made using a Shimadzu ultra-microhardness depth sensing indentation tester with a Berkovich indenter at 84.7 mN force. A Veeco Instruments D3100 AFM was used to image the residual indent in tapping mode with a scan size of 32 µm, scan rate of 1 Hz, image size 512 × 512 pixels and a 220-µm long TESP. To improve the image quality the Nanoscope 'Amplitude Setpoint' was decreased until the trace-retrace cross-section lines coincided in the lowest part of the indent.

Indent dimensions

The indentation was divided into two parts above and below the sample surface height. The part below the surface height is the residual indent and the part above is the pile-up (Fig. 2). From the residual indent the NanoMc software measures the depth, projected area, surface area and volume. The NanoMc starts by creating an outline around the indentation using the 'Tangent Method' [5]. The tangent method is better for measuring indents than the threshold method because it accounts for surface roughness, sink-in and pile-up. Once the outline is made the indent measurements are straightforward.

The indent projected area is the flat plane at the sample surface height. The pixel width is equal to the scan size divided by the number of samples per line. Figure 1 shows an AFM indent image with a scan size of 32 µm at 512 × 512 pixels. Each pixel therefore has a width of 62.5 nm and an area of 3906.25 nm^2. The summation of all the projected area elements inside the NanoMc outline equals the total projected area. The projected area (Ap) and force (F) are used to calculate the indentation hardness $H_{(IH)}$ similar to that discussed in the ISO 14577-1 for nanoindentation testing [7]:

$$H_{(IH)} = F/Ap$$

The surface area covers the topography of the indent. A surface area element is quadrilateral in shape because the four sides have different lengths. The total surface area is the summation of all the surface area elements inside the NanoMc outline. The volume of each pixel is the projected area multiplied by the height from the projected plane to the indent surface. A summation of all the volume elements inside the NanoMc outline equals the indent volume.

Indent Measurements	NanoMc[1]	Nanoscope III[2]	SPIP[3]
Depth (surface to minimum), nm	751.7	750.37	751.7
Projected area, µm^2	25.14	30.057	25.6
Surface area, µm^2	26.67	—	—
Volume, µm^3	7.003	6.815	7.04
Projected area (with pile-up), µm^2	30.75	—	—
Surface area (with pile-up), µm^2	32.61	—	—
Volume (with pile-up), µm^3	7.003	6.815	9.94
Pile-up projected area, µm^2	52.10 *	49.764	53.9
Pile-up surface area, µm^2	52.64 *	—	—
Pile-up volume, µm^3	4.440 *	2.743	2.9
Pile-up center-of-volume, µm	2.670 *	—	—
Contact depth, nm	887.0 **	—	—
Contact area, µm^2	27.24 **	—	—
Indenter angle (conical), °	70.39	70.84 **	72
Indenter tip radius, µm	1.014	—	1.6
Indentation hardness, MPa	3369.13	—	—
Hardness MPa	3109.4	—	—

Table 1:
Various measurement results for the NanoMc and Nanoscope image analysis software.
*([1]Tangent Method. [2]Threshold Method. [3]Histogram Analysis or Pore Analysis. *Using 3.0 multiplied by the roughness standard deviation to identify the pile-up pixels. **Using an elastic constant of 0.18 for the NanoMc elastic reconstruction.)*

Pile-up dimensions

The pile-up area contains material forced above the sample surface height by the indentation process. A computerized method was created to measure the pile-up projected area, surface area, volume and center-of-volume. First the pile-up element had to be identified using a flood and fill-in technique. The flood and fill-in is done by identifying all the pixels in an area with a new color that is in contact with the seed color starting point. Any pixels near the indent pile-up outline greater than three sigma times the surface roughness were considered as pile-up. The seed points are started at each of the outline pile-up points. Once all the pile-up pixels are identified the pile-up dimensions are measured like that discussed above for the indent dimensions. The moment-of-inertia method from mechanical physics is used to find the pile-up center-of-volume and magnitude offset in relation to the indent minimum depth point.

Elastic reconstruction

For comparison the AFM indentation contact area could be used to calculate a hardness value that is comparable to Vickers hardness or Oliver-Pharr hardness scales depending on the type of indenter used. The residual indent AFM image depth cannot be used because it only has the resulting plastic deformation. The software digitally adds the elastic recovery back into the indent image. Figure 3 shows an indent cross-section before and after reconstruction. The depth of every pixel element inside the outline is lowered based on a value called the 'elastic constant' [5]. The elastic constant is adjusted by the user until the indent image has the same shape as the actual inden-

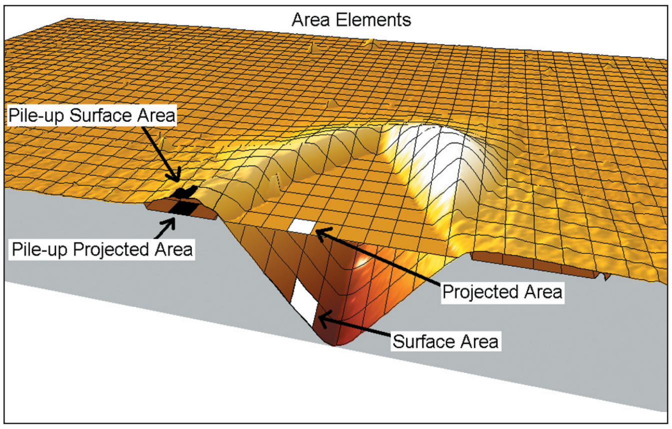

Figure 2:
3D cross-section reconstruction of the indent. The indent projected area and surface area elements are shown in white and the pile-up projected area and pile-up surface area elements are shown in black (z-scale 4:1).

ter. The reconstructed indentation image is used to calculate the contact depth and contact area. The force (F) and contact area (A_C), are used to calculate the hardness (H_C):

$$H_C = F/A_C$$

Indenter angle and tip radius

After the NanoMc elastic image reconstruction the modified indent shape can be used to determine the average indenter angle and tip radius. An indenter projected area function is built by measuring the projected area (Ap) at various heights (h) from the indentation minimum. When a second-degree polynomial curve is fitted to the projected area function (Ap) versus the height (h), the first term (a_0) can be used to calculate the average (conical) indenter angle (α) and the second term (a_1) the tip radius (R) according to the following equations [5]:

$$Ap = a_o h^2 + a_1 h$$
$$\alpha = \tan^{-1}((a_o/\pi)^{1/2})$$
$$R = a_1/2\pi$$

RESULTS

An indentation was made in AISI 1020 steel, imaged with an AFM, and measured using the new computerized image analysis technique. Figure 4 shows the NanoMc 'results image' with a pile-up outline. This results image was used to visually inspect the outline quality. The white outline was automatically added digitally to the main image by the NanoMc software. By visual inspection it was clear that the outline followed the parameter including the pile-up indenter contact area. The NanoMc 'Tangent Height' was set to 95%.

Table 1 shows the measurement results of the NanoMc 'Tangent Method' compared to the Nanoscope 'Threshold Method' and SPIP. In order to measure the indent dimensions with the Nanoscope the image height pixels were inverted. The threshold was adjusted until the indent area appeared completely red and the surrounding area showed the normal surface image. To measure the pile-up dimensions with the Nanoscope a box was placed around the pile-up area which eliminated other tall surface particles. The surface roughness was 3.2 nm (one sigma) measured in a 10 $\times$ 10 µm scan area. The NanoMc used three times the roughness for the flood and fill-in to identify the pile-up elements. The difference between the pile-up and indent volume indicates that 2.56 µm^3 of atoms were injected into the material's crystal structure.

The elastic reconstruction was used to measure the contact depth, contact area, indenter angle and tip radius. An elastic constant of 0.18 was found by adjustment until the indent face angle in the AFM image equaled the known indenter face angle. The depth measurements from the Nanoscope and SPIP were in agreement to within 0.17 % of the NanoMc. The other measurements had a large discrepancy attributed to the threshold method errors discussed earlier. Table 1 shows that the Nanoscope and SPIP pile-up volumes were 36% less than the NanoMc because the threshold was placed high above the surface height to reduce noise. The NanoMc accounts for the volume of the pile-up from the pile-up element height down to the sample surface projected area. Figure 5 shows the indenter angle and tip radius for a cross-section of the indent through the indentation minimum point.

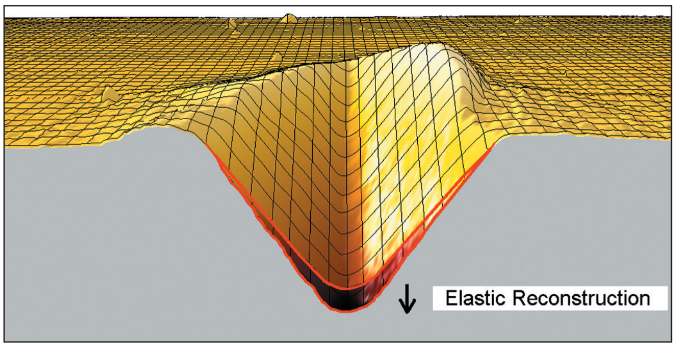

Figure 3:
A cross-sectional image of the elastic reconstruction process for a residual indent into the fully loaded indentation shape (z-scale 4:1).

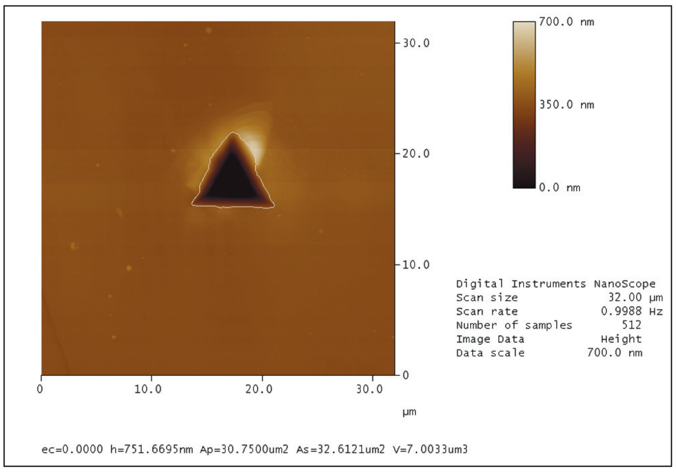

Figure 4.
A NanoMc measurement results image including the pile-up contact area shown by the white outline.

CONCLUSIONS

This research presented reliable AFM image analysis software to measure indents. The NanoMc software automatically measures the indent depth, projected area and surface area with or without the pile-up area. The form of the white outline in the NanoMc results image is used to inspect the measurements. An elastic reconstruction function converts the residual indent into the fully loaded indentation shape. The elastic reconstructed indent image was used to measure the contact depth and contact area. A second-degree polynomial curve fit to the height versus projected area was used to determine the average indenter angle and tip radius. The indentation hardness and contact hardness were calculated using the automated software measurements.

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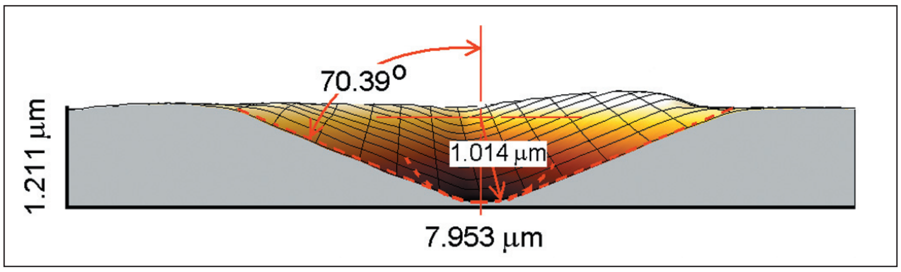

Figure 5:
The indenter tip radius and average angle measured after the NanoMc indentation reconstruction.