Microstructure and Pile-up effect on Nanoindentation measurements of FCC and BCC Metals

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Abstract
Nanoindentation is widely used for mechanical surface characterization. However, there is not a complete understanding about deformation mechanisms and integration of those in accurate presentation of mechanical properties measurements. This is mainly because of lack of precise measurements of pile-up and sink-in. Materials pile-up and sink-in changes during the indentation create errors in measured hardness and modulus of elasticity values. Efforts in past have been made to characterize pile-up and sink-in phenomena and different analytical approaches and experimental analysis have been reported. In this study, pile-up and sink-in behavior FCC and BCC metals are investigated and its relation to micro-structural characteristics and the grain orientations have been presented .Surface topographies for different indents are characterized by a state of the art technique with nanometer accuracy. Physical characteristics of pile-ups have been identified on the basis of knowing the crystal structure of materials and the geometry of three nanoindenter tips used for uniform and non-uniform stress distributions in the contact region: Berkovich, cubecorner, and conical. Extrinsically imposed stress conditions and their effect on material behavior in form of pile-up is studied here.

Introduction
When a material is indented, it generally forms either pile-up or sink-in with respect to the indented plane depending on various factors like strain hardening and the elastic modulus to yield strength ratio[1]. Nanoindentation calculations use the standard Oliver and Pharr method to calculate the hardness and elastic modulus[2].
Hardness is determined as:

$$H = \frac{P}{A} \qquad (1)$$

Where, A is defined as the projected contact area of the indentation at maximum load, P.
The contact area also appears in the expression for the reduced elastic modulus, E_r which is the combined moduli of the indenter and the sample as:

$$S = \frac{dP}{dh} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A} \qquad (2)$$

where S is the stiffness and A is the contact area. E_r is determined as:

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i{}^2)}{E_i} \qquad (3)$$

E, E_i ν, ν_i are the elastic modulus and poisons ratio of the specimen and the indenter respectively. Above relations do not consider the effect of pile-ups. These effects have been considered[3] and efforts in past have been made to account for these and people have come up with various ways to account for these with different materials like soda lime glass[4], but as Oliver and Pharr suggest in the most recent review, this still remains the holy grail in the area of instrumented indentation[5]. Orientation effects have been considered through simulation and a model has been proposed[6]. Area of pileup has been accounted as that of a semi ellipse in this study[7]. However, there is still a need for the exact measurement of pileups and a model for the prediction of these on the contact area in various metals. In this study, we have calculated the pile-up height of various indentations through Nano Vision™ using a special traceline method which can accurately measure the pileup heights and thus opens the door for the exact measurement of the pile-up area. The effect has been studied for copper, nickel and iron assuming different orientations and these results are compared with the experimental results to get a fair idea of the overestimation.

Experimental Method

Materials
The materials used in the study were chosen in such a way to span a majority of metals and thus indicate a general trend. Two types of FCC metals, nickel and copper were chosen because of the fact that copper has a lower elastic modulus and we expected to see better pileups compared to nickel. Also, both these would indicate the effect of other parameters as the stacking fault energy on other mechanical properties as hardness and elastic modulus. The results can thus be extrapolated to predict behavior in other FCC metals. Well defined slip systems in FCC metals also give us a better insight in the deformation mechanisms involved while indentation.

Iron is the most widely used BCC metal and was chosen as a representative for their behavior. The material used for calibration of the tips was fused silica which was chosen because of its isotropic properties and amorphous structure. The negligible time dependence on plastic properties of this material makes it ideal to be used as the standard calibration material. The elastic modulus of fused silica is approximately 72 GPa and the calibration of three different tips was based on the premise that this should not change with changing tips as the structure is amorphous.

Sample Preparation and Purity

Fused silica used for calibration was provided by the manufacturer and is intended for calibration purposes only. It was used as standard always. All three materials used in the study namely, copper, microcrystalline nickel and iron were 99.5% pure. Their purity was determined by the Electron Dispersive Spectroscopy technique.

All three metals were polished using the normal sequence of metallographic polishing papers to grade 1200 and then 1. 0.5 and 0.3 colloidal silica suspension solutions respectively. The specimen thus prepared has a mirror finish and can be used for indentation as well as examination under the Scanning Electron Microscope and Electron Back Scattered Diffraction Technique.

Indentation method

All experiments were done using MTS Nanoindenter XP at the Nanoindentation Laboratory in University of North Texas. The machine has a force and displacement resolution of 50 nN and .01 nm respectively. Indentations were done at room temperature. These experiments are sensitive to vibrations; therefore tests were generally done during the night. Since our purpose in this study is to find out the effect of various extrinsically imposed stresses on different crystal orientations in a poly crystalline material, we have used three different tips, namely Berkovich, cubecorner and conical. The first two are pyramidal in geometry and give us a very neat idea of pileup variation with changing angles of various planes in the pyramid and crystal orientation. The third has a perfectly symmetric stress distribution and can be used to see the effect on pileup by crystal orientations alone.

The indents were scanned by the indenter tip using a nominal load utilizing a unique feature in Nanoindenter XP called Nano Vision. The Nano Vision instrument extension works similar to the Atomic Force Microscope. Specialized test methods enable the indentation head to scan the specimen surface. As the sample is traversed under the diamond indenter tip, it causes deflection of the indenter tip which is the recorded and is used to create a detailed 3-D topological map of the specimen. It utilizes a special stage operated by piezoelectric and can be used to measure surface topography with nanometer precision because of low compliance of the system. The pileup height measured in this way was used for further analysis in the study. All indentations were performed using XP Indent and Scan Displacement Limit method and the residual depth was fixed at 1000 nm in all of the above tests in order to get an equivalent ground for the measured pile-ups. A nominal fraction of the peak load was used for scanning the indent. The allowable drift rate was fixed at .05 nm/s. All the tests were done at one strain rate of 0.05 nm/s

Each material was indented with all three indenters on 3×3 matrix. Each indentation was separated by approximately 50 microns. Pile-up heights were measured for all these indentations through traceline method in Nano Vision™.

Results and Discussion

Below shown are Nanovision profiles for copper, iron and nickel when indented with Berkovich, conical and cubecorner indenter respectively.

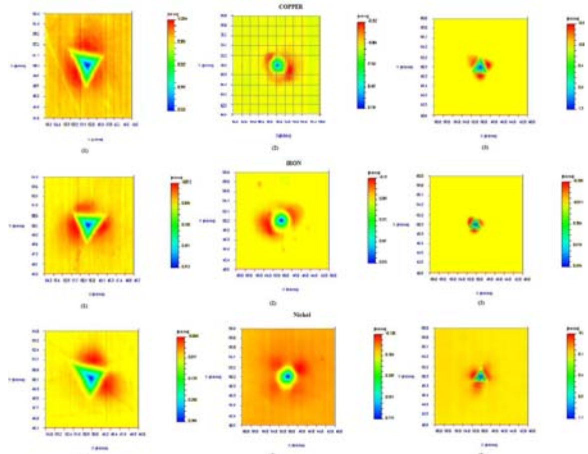


Fig 1. Top views of indented surface by Nano Vision for Cu, Fe, and Ni, top to bottom and by Berkovich, conical, and cubecorner tips from left to right, respectively.

The pileups shown in Fig. 1 were measured by the traceline method with nanometer precision. A special method was devised to number the lobes. Below shown is an area id schematic for lobe nomenclature. This was common for all the indents studied. For conical indentations, however, the lobes were numbered 1 onwards starting anticlockwise. A maximum of up to 4 lobes were seen on one indent.

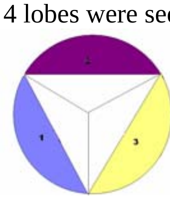
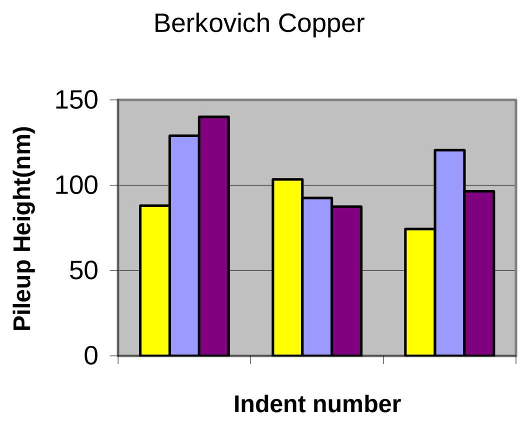
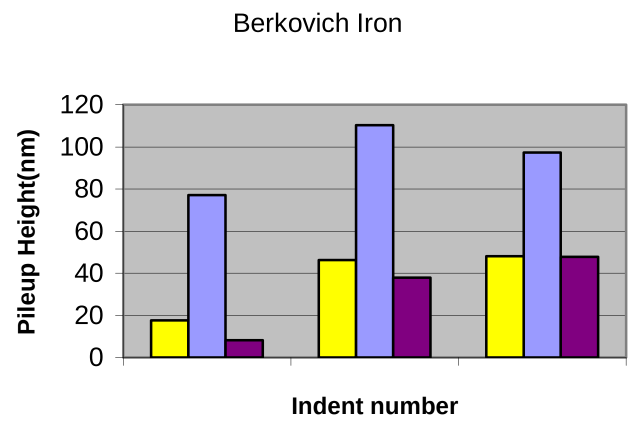


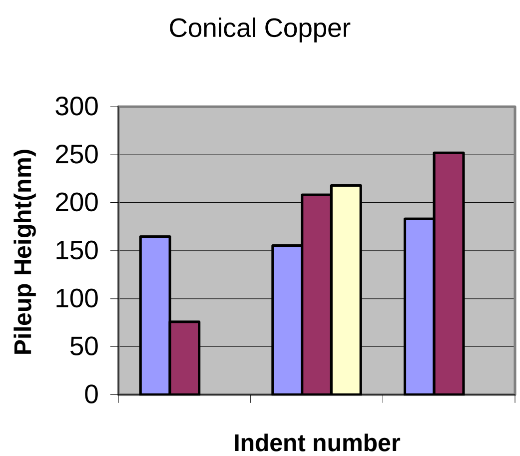
Fig 2. Area id schematic for lobe nomenclature



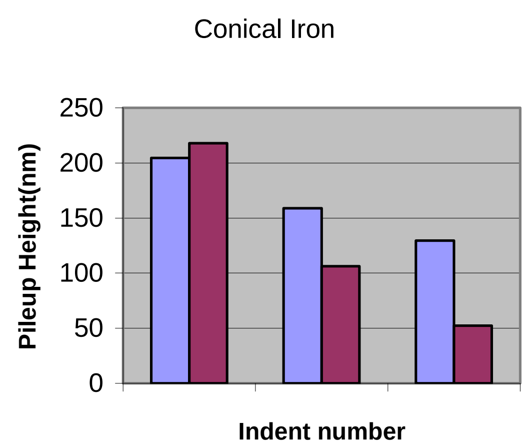
(a)



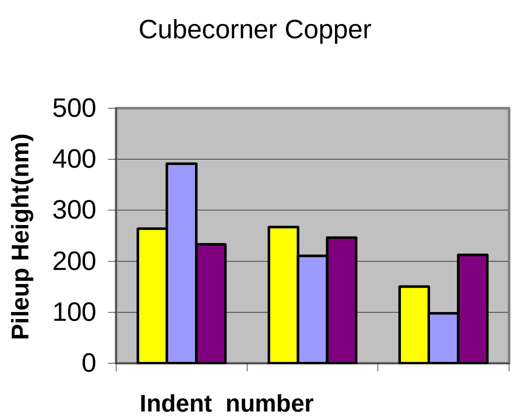
(a)



(b)

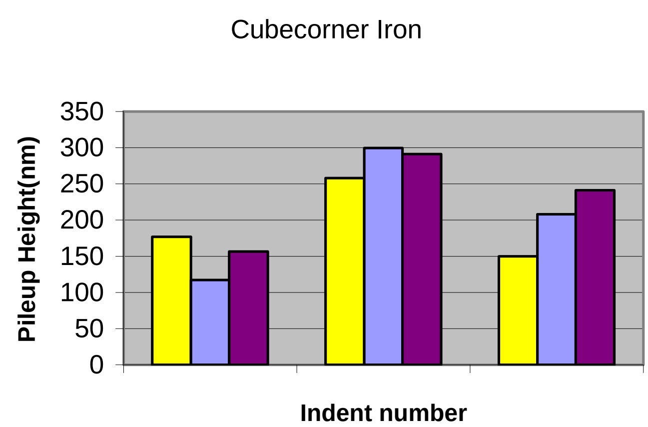


(b)



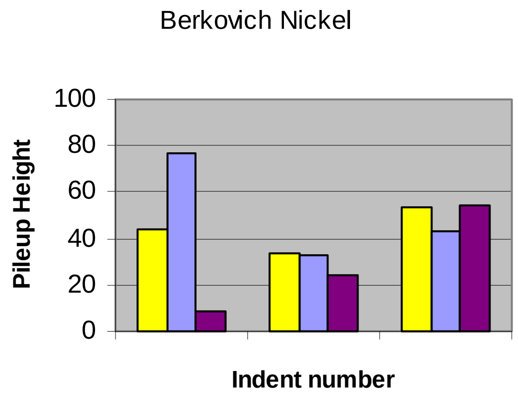
(c)

Fig 5. Pile-up Height versus Indent number for Copper. Different colors refer to pile-up heights in each lobe as specified in Fig 2 and text.

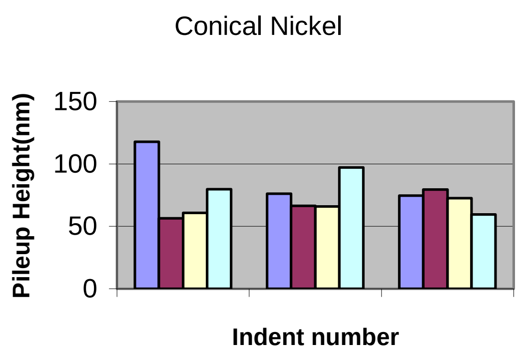


(c)

Fig 6. Pile-up Height versus Indent number for Iron. Different colors refer to pile-up heights in each lobe as specified in Fig 2 and text.



(a)



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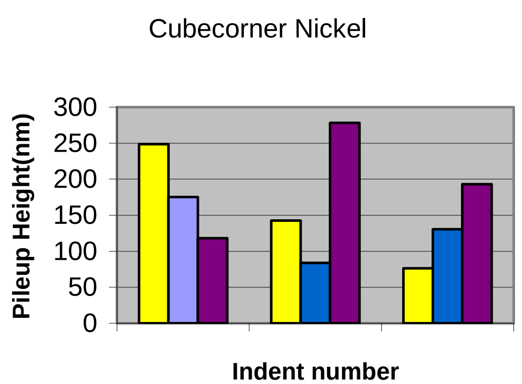

(c)

Fig 7. Pile-up Height versus Indent number for Nickel. Different colors refer to pile-up heights in each lobe as specified in Fig 2 and text.

As can be seen from the Nanovision profiles, there are certain regions around the indent with absolutely no pileups. This can be attributed to different grain orientations. Every grain orientation has different slip systems and depending on various slip directions, we see different pile-ups[1]. The regions with no pile-ups could be grain boundaries. Even certain regions where there are pile-ups, they are not uniform. This can be attributed to two conjugate grain orientations, behaving differently under the applied stress by the indenter. Very high pileups are seen with the cubecorner indenter; hence it can be said that the degree of overestimation is highest with cubecorner. There are various models proposed for calculation of pile-up area[1]. With this measurement of pile-up height, they can be verified and the degree of overestimation of hardness and elastic modulus can be calculated. Pile-up depends on the contact area of the indenter[3] which agrees with the experimental data referred to here which clearly shows that amount of pile-ups are different for different indenter geometries.

Conclusions

Based on above observations following conclusions can be drawn.

1. The pile-up heights are not uniform in all the three sides of indentation.
2. Pile-up heights are strongly related to the orientation of grains in the each material examined.
3. Indenter geometry plays an important role on pile-up height and hence the contact area.
4. With these measurements, it is possible to calculate contact area underestimation.

References

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