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Finite Element Analysis Study on Effect of Indenter Tip Radius to Nanoindentation Behavior and Coatings Properties

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Abstract

This paper mainly focuses on the study of factors that affect the nanoindentation process and the determination of mechanical properties of coating film using computational finite element (FE) analysis. Attempts have been made in order to show the effect of indenter tip radius on the nanoindentation process of both hard and soft coating systems. It has been proved that larger load is required to reach a specific penetration depth when the indenter tip radius increases. Besides, the change in tip radius led to a more severe effect on hard coating system compared to soft coating system. Furthermore, pile- up and sink-in phenomena of the materials have also been proved. They affect the measurement of mechanical properties of coatings/films.

The developed nanoindentation FE models were able to simulate the indentation loading-unloading curves of the coating/substrate system. It also can be found that an extraction of intrinsic properties of the thin super-hard coating film, titanium diboride or TiB_2, was achieved. If the load-displacement curves of the simulation and experimental results can match with each other well, then the properties used in the simulation should be the actual properties of the coatings. The critical ratio of coating thickness to indentation depth for the property measurement was also presented. Hence, in order to get accurate measurements of properties of the film, it is necessary to know the limitation in which the penetration depth can be indented for the coating/substrate systems.

Key words : Finite element analysis, Nanoindentation, surface coating

Introduction

Since many engineering applications have shifted to smaller scales, the characterization of the materials' intrinsic mechanical properties including yield strength, elastic modulus and many others has become increasingly complex. Among the techniques used to measure the mechanical properties of nano-structured thin films, nanoindentation techniques have been widely utilized to measure thin film mechanical properties, particularly hardness, elastic modulus, scratch resistance, time or rate dependent properties and many others.(1-2)

In the late 19th century, the pioneer investigators played a key role in the analysis procedure of the elastic contact problem. Hertz(3) analyzed the problem of the elastic contact between two spherical surfaces with different radii and elastic constants. His solutions formed the basis of much experimental and theoretical work in the field of contact mechanics and provided a framework by which effects of non-rigid indenters can be included in the analysis. Boussinesq(4) developed the method based on potential theory for computing the stresses and displacements in an elastic body loaded by a rigid axisymmetric indenter. His method has subsequently been used to derive solutions for a number of crucial geometries such as cylindrical and conical indenters. Tabor(5) studied the indentation of a number of metals deformed by hardened spherical indenters. Sneddon(6) derived general relationships

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among the load displacement and contact area for any punch that can be described as a solid of revolution of a smooth function.

In the past two decades, it was realised that load and depth sensing indentation methods could be very useful in the measurement of mechanical properties of thin films and surface layers, and instruments for producing submicron indentations were developed. Elastic modulus and hardness are the two properties that are more frequently measured by the load and depth sensing indentation technique. Theoretical analysis of general indentation problems has received a great attention from many investigators. The investigators Oliver, Hutchings, Pethica, Doerner, Nix, and Joslin(7-9) then suggested a simple method based on measured indentation load-displacement curves and knowledge of the indenter area function or shape function that is the cross-sectional area of the indenter as a function of the distance from its tip. The most extensively used method to determine elastic modulus and hardness by nanoindentation called "Oliver and Pharr's method or O&P approach" was proposed by W. C. Oliver and G. M. Pharr(10) the slope of the unloading curve which is usually nonlinear was used to calculate the elastic modulus and provided a physically justifiable procedure for determining the depth which should be used in conjunction with the indenter shape function to establish the contact area at peak load.

Recently, great strides have been made in the development of nanoindentation equipments and nanoindentation techniques for investigating the mechanical properties of materials or thin film on sub-micron to nano scale.(11) Wang and Lu(12) used nanoindenter to quantify the elastic and plastic anisotropy in single crystals, and they found that the indenter orientation has no effect on the hardness and modulus measurement. Fujisawa, Swain, James, Tarrant, Woodard and McKenzie(13) have investigated mechanical properties of a range of tribological mitigating and biocompatible films deposited on a titanium alloy substrate using nanoindentation. Furthermore, the numerical simulation techniques have been developed and applied in many engineering applications which can be used in indentation problems as well.

Finite element (FE) technique is applied for studying the indentation process or improving the method used to extract the mechanical properties from the simulation data. Finite element modeling enables the measurement of mechanical properties of difficult samples where the conventional analytical treatments failed. However, it was found that changing some parameters during the indentation process may result in a tremendous change in the measured properties. Hence, in the past few decades, many investigators have brought to bear substantial efforts to find out what are the main factors that will affect the measurement of properties of the thin film.(10, 14-15) They have proved that the tip geometry of the indenter, instrument compliance, penetration depth, pile- up and sink- in of materials affect the measurement.

FE analyses on the critical ratio of coating thickness to indentation depth for the property measurement of hard coating on soft substrate have been performed by Sun, et al.(14) From these analyses, it can be concluded from the critical ratio of the coating thickness to indentation depth is proportional to the yield strength ratio and also the indenter tip radius. In the case of the soft coating on hard substrate, the hard substrate has a significant effect on the measurement of the mechanical properties of the soft coating. The plastic deformation is restricted within the soft coating during the indentation process, and material will pile up around the indenter and will lead to a larger contact area compared to the contact area measured by the analyses. Hence, the soft coating system has a very different indentation response compared to the hard coating on soft substrate.(15)

This study aims to elucidate the effect of indenter tip radius on hardness measurement of a coating/substare systems. Attempts have been made in the present work to simulate the nanoindentation process and study how the indenter geometries influence the plastic deformation behavior of various coating/substrate systems using the FE method. A conical indenter was chosen in the model in order to form the same projected area as the standard Berkovich indenter.

Finite Element Modeling (FEM)

In order to study how the indenter tips radius affects the finite element analysis of hard/soft coating on hard substrate, a two-dimensional (2D) axisymmetric model has been developed by using the capacities of the ABAQUS finite element code.(16) The hard/soft coating perfectly adhered to the substrate and was indented by a rigid conical indenter under condition of frictionless contact. The indenter has the same projected area-depth function as the standard Berkovich as it has a half angle of 70.3 °. The rigid indenter was simulated by using few tip radii, including perfectly sharp tips and round tips of 0.2, 0.5 and 1.0 µm. The schematic diagram of the geometry of a round tip indenter is shown in Figure 1, where r is the radius of the round tip indenter, d is approximate to 0.062 r and ξ is approximate to 0.0585 r.(14)

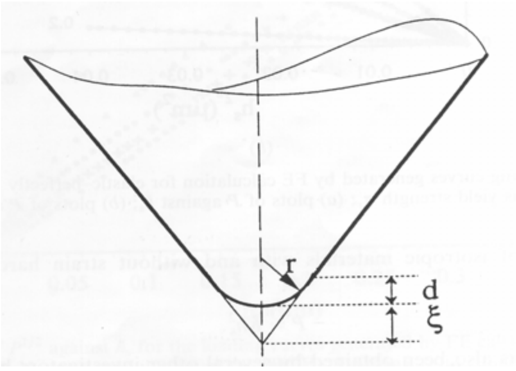


Figure 1. Schematic diagram of geometry of round tip indenter.(14)

Therefore, the indentation process can be modeled using the finite element mesh. The coating region and the adjacent substrate were finely meshed and continuously coarsened further away from the contact area (Figure 2).

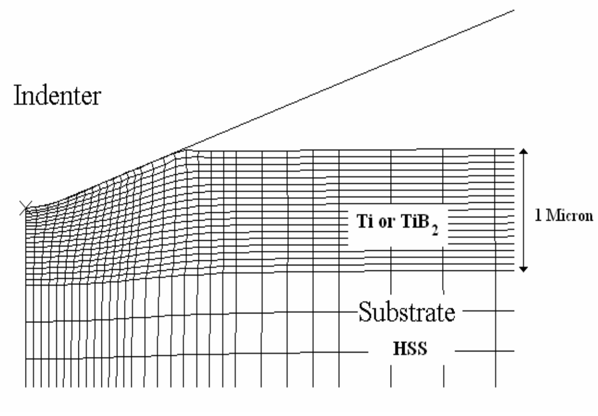


Figure 2. Mesh of the coated substrate with indenter

Both the coating and the substrate are considered to be isotropic which supposed to be linear elastic perfectly plastic material, as strain hardening is not considered in this case. In order to detect the contact between the coating and the indenter, the latter was set as master surface and the coating surface was defined as slave surface, where only the master surface can penetrate into the slave surfaces. The program can automatically detect the contact or separation of the master and slave surface. The indenter was set to penetrate through the coated substrate boundary conditions were applied to the centerline and bottom surface nodes, while the outermost side was assumed traction-free. Titanium was used as soft coating on the high-speed steel substrate, while the titanium diboride (TiB_2) was used as hard coating. Properties used in the model calculation of the coating and the substrate and thickness are shown in Table 1.

Table 1. Properties of coating and substrate used in the finite element analysis

Material	**Elastic Modulus, E (GPa)**	**Poisson Ratio, υ**	**Yield Strength , σ_y (Gpa)**	**Thickness (µm)**
High Speed Steel (HSS)	225	0.3	5	-
Soft-coating (Ti)	200	0.3	2	1
Hard-coating (TiB_2)	300	0.25	10	1

Results and Discussions

Nanoindentation Load-Displacement Curves

For layered material, the derived values of hardness, H, and elastic modulus, E, are due to the combined mechanical properties of the layer and substrate. The relative contribution of the properties of layer and substrate depends on the penetration depth. At shallow depth, the response is more characteristic of the mechanical properties of the coating alone. Hard-coated and soft-coated substrates have very different elastic deformation behaviors during loading process, due to the difference in Young modulus and Poisson's ratio values. Figure 3 and Figure 4 show the typical calculated loading/unloading curves of soft coating and hard coating, respectively, illustrating the influence of the indenter tip radius on the loading process of the coated substrate. It can be clearly seen that a slight increment in the indenter tip radii can significantly increase the load required to produce a specified indentation depth, in particular for the soft coating system. This is because the contact area between the indenter and the film increase as the tip radius increases. Hence, the load required is increasing, too.

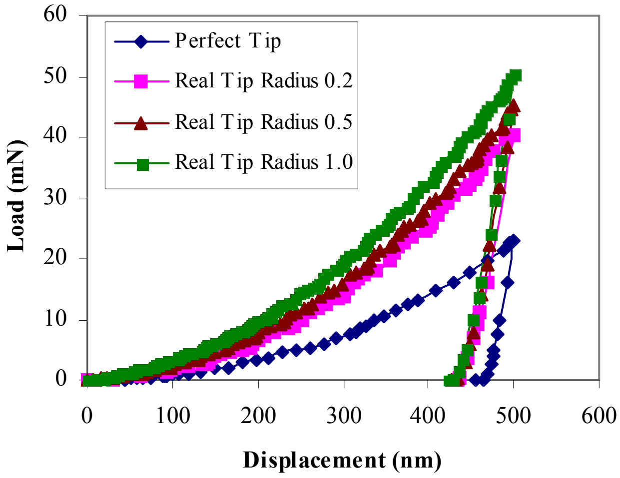


Figure 3. Loading curves of soft coating (Ti) on HSS substrate at 500 nm penetration depth.

From Figure 4 showing the load-displacement curves of hard coating on hard substrate it can be seen that increase in the tip radii led to the increment of the load required to reach a desired penetration depth, but the effect does not severe deviation compared to soft coated substrate (Figure 3). Nevertheless, it is very obvious that the hard coatings have better resistance to indentation process compared to soft coating system as they have a higher hardness. The load required for the perfectly sharp indenter to penetrate the hard coated substrate for 500 nm penetration depth is approximately 75 mN, while the load required for the soft coated substrate approximates only 20 mN.

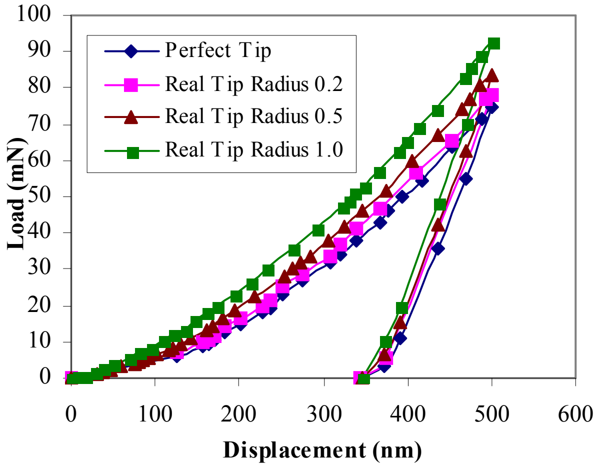


Figure 4. Loading curves of hard coating (TiB_2) on HSS substrate at 500 nm depth.

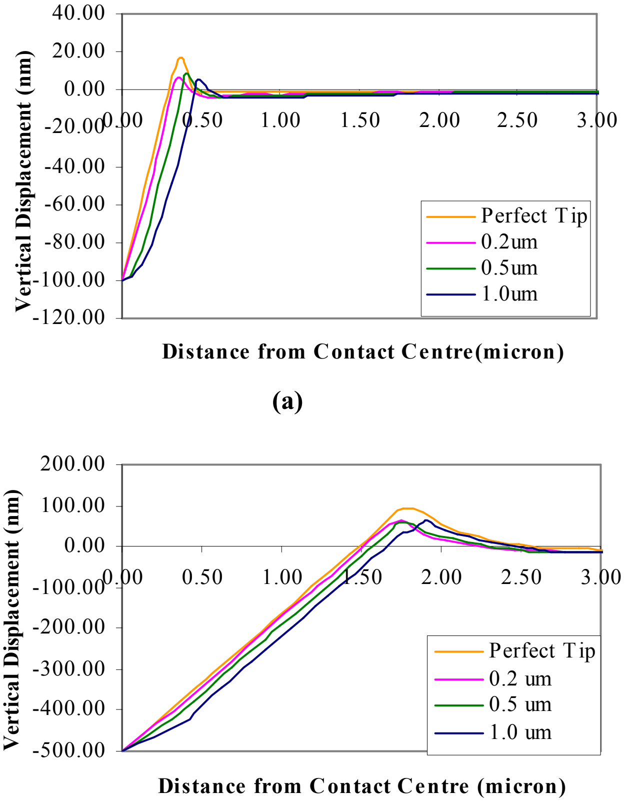
It can be concluded from the FE results that pile-up and sink-in of materials around the indenter affect load-displacement curves and properties interpretation. Actually, a well-annealed soft metal, which exhibits a high strain-hardening rate, will tend to show far field plasticity. Strain hardening near the indenter tip will cause plastic deformation to occur further and further away from the contact, causing the material to be displaced away from the indentation and result in sink-in behavior. By contrast, strain-hardened materials, which exhibit a low strain-hardening rate will deform locally, creating a pile- up of material around the indenter. Effect of sink- in and pile- up on the analysis will be discussed later.

Soft Coating (Ti) on HSS Substrate

As mentioned above, the harder substrate will restrict the plastic deformation process of the softer coating; hence the plastic deformation is constrained inside the coating and results in enhanced material pile-up around the indenter (Figure 5). The fundamental material properties that affect the pile-up effect are the ratio of the effective modulus to the yield stress, E_{eff}/σ_y, and the work hardening behavior. In general, pile-up is greatest in materials with large E_{eff}/σ_y, and little or no capacity for work hardening. The ability of work harden inhibits pile up because material at the surface adjacent to the indenter hardening of during deformation constrains the upward flow of material to the surface.

Another important parameter that affects the pile-up effect is the ratio of final indentation depth, h_f, to the depth of the indentation at peak load, h_{max}. The ratio of h_f / h_{max} can be extracted easily from the unloading curve in a nanoindentation experiment. The natural limits for this parameter are $0 \leq h_f / h_{max} \leq 1$. The lower limit corresponds to fully elastic deformation and the upper limit to rigid plastic behavior. The pile-up effect is large only when h_f / h_{max} is close to 1 and when the degree of work hardening is small; when $h_f / h_{max} \leq 0.7$, very little pile-up is observed even though the work hardening is very small.

Figures 5 (a) and (b) show the pile-up effect at 100 nm and 500 nm penetration depth, respectively. It is noted that the perfectly sharp indenter has a higher h_f than other round tip indenters, so the pile-up effect is more significant for this system. Indeed, pile- up of materials around the indenter will lead to a larger contact area than the predicted area. Errors in the contact area will lead to similar errors in the hardness calculation; the modulus will be in error by an area factor. More details and equations can be found in reference.(10)

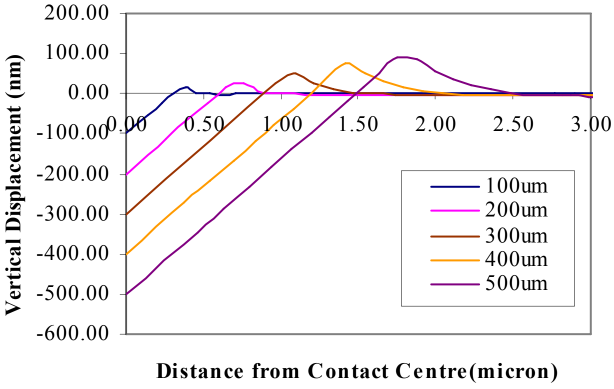


(a)

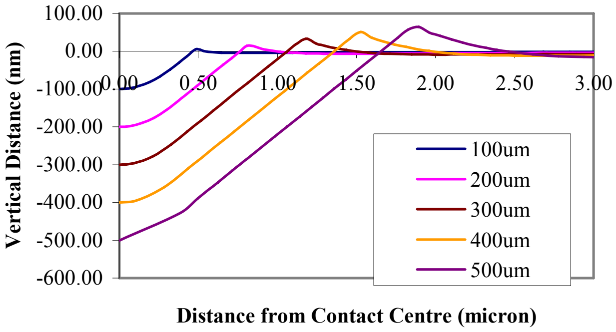
(b)

Figure 5. Amount of pile- up around the indenter at (a) 100 nm and (b) 500 nm penetration depth

Figure 6 illustrates the effect of penetration depth on the amount of pile- up. It can be seen that a larger penetration depth will lead to more pile-up of materials around the indenter for both perfect tip (Figure 6 (a)) and tip radius system (Figure 6 (b)).



(a)



(b)

Figure 6. Pile- up of material at different tip radius; (a) perfect tip and (b) 1.0 µm

Figure 7 shows the calculated hardness of the soft coating as a function of penetration depth at different tip radii. It can be seen that when the pile-up effect becomes larger (at higher depth), the contact area between the indenter and the sample also increases. Hence, the hardness at higher penetration depth should be less. However, Figure 7 shows an increasing trend of hardness at higher depth. This is due to the effect of hard substrate influencing the property of the soft coating. In addition, at different tip radii, the hardness of the film also varies. Figure 7 manifests that the perfect tip shows the lowest hardness because it leads to more pile-up height as shown in Figure 5.

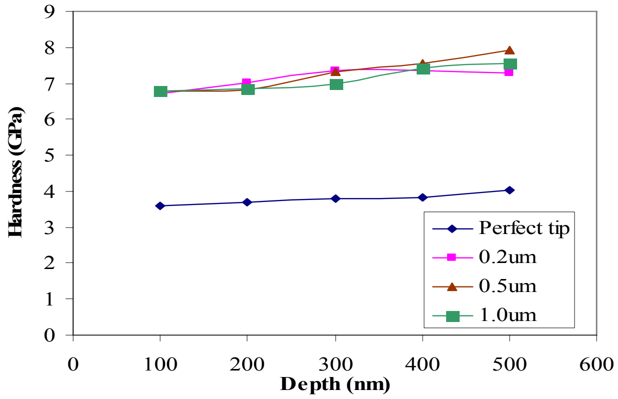


Figure 7. Hardness of the soft coating film at different tip radii.

Hard Coating (TiB_2) on HSS Substrate

Figure 8 indicates the amount of sink-in depth of materials around the indenter tip for different tip radii (Figure 8 (a) and Figure 8 (b)) and at various penetration depth. It can be seen that at higher penetration depth, the higher sink-in effect will take place, hence the contact area higher. As mentioned above, well-annealed metal that exhibits a high strain-hardening rate will result in sink-in behavior. As a result, the true contact area is always smaller than the predicted area when sink-in occurs. Therefore, the derivation of hardness and elastic modulus will be affected by the changes in the contact area.

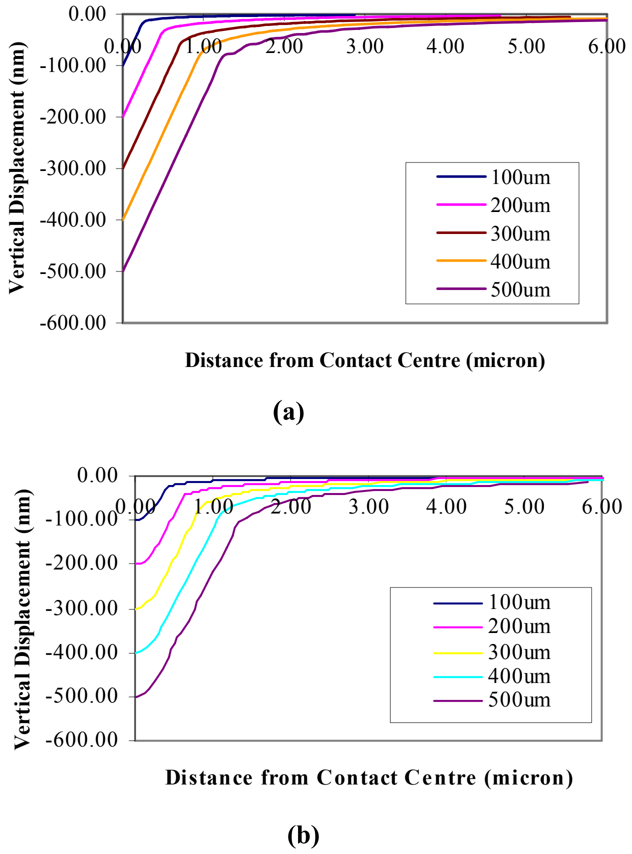


Figure 8. Amount of sink-in around the indenter (a) perfect tip and (b) 1.0 µm

Hardness is defined using the projected area of contact under the maximum load penetrated. It is noted that deeper a penetration leads to a higher sink-in effect, hence for a higher a contact area the hardness decreases as shown in Figure 9.

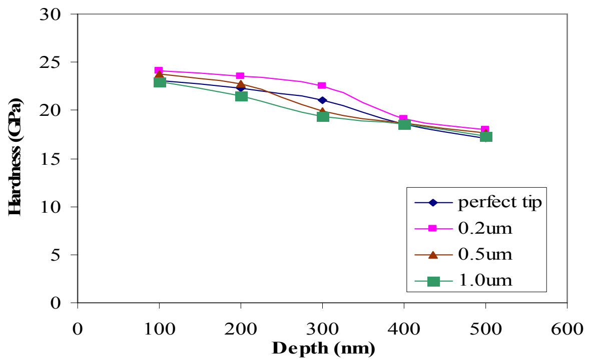


Figure 9. Hardness of the hard coating film at different penetration depth.

Conclusions

As to the finite element analysis of the hard and soft coatings on HSS substrate effecting indenter tip radii on the indentation response of the coated substrate, the main results are summarized below.

1. The load required to reach a desired depth is directly proportional to the indenter tip radii. However, soft coated substrate is more sensitive compared to hard coated substrate due to the pile-up effect.

2. For soft coated substrate, all the deformation is restricted within the coating unless the indentation reaches the coating and substrate interface in which the substrate effect will take place. The indenter with higher tip radii will have more material pile-up around the indenter, which is unfavorable. Furthermore, deeper penetration will also lead to more material pile-up around the indenter. Hence, a higher contact area affects the accuracy of the hardness and modulus of the film.

3. For hard coated substrate, the deformation is reduced due to the high hardness of the coating layer. Hence, higher load is required to penetrate the coated substrate. Higher tip radii and higher depth will cause more sink-in behaviors, and hence affect the accuracy of the definition of hardness and modulus.

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