THE INFLUENCE OF SURFACE ROUGHNESS IN MICRO INDENTATION

CHUAH HUN GUAN

UNIVERSITI SAINS MALAYSIA

2013

THE INFLUENCE OF SURFACE ROUGHNESS IN MICRO INDENTATION

by

CHUAH HUN GUAN

Thesis submitted in fulfillment of the requirements
for the degree of
Doctor of Philosophy

November 2013

ACKNOWLEDGEMENT

In the journey of this research work, first of all I am indebted to my supervisor Professor Dr. Zaidi bin Mohd. Ripin. The accomplishment of this work would not have been possible without his guidance, patient and novel thoughts in providing valuable ideas for this research. In the strand of finance, his financial support gave me chance to continue the research. Furthermore, his unstinting encouragement, kindness and understanding always motivate me not only to complete the research but inspire me to think out of the box in my life.

I would like to indicate humbly my heartfelt thanks to my parents and Ms. Yap Sing Yoh for their warmest regards and support of my PhD study. The continuous difficulties and challenges through out this study were overcome with their encouragement and support.

I sincerely acknowledge Dr. N. M. Jennett for sharing with me the justifications and knowledge on the fundamental concept and technical issue of the loading and unloading process of the indentation technique. In addition, my appreciation should due to Prof. William D. Nix in clarifying the assumptions in the Nix-Gao model theory. In addition, I would like to extend my thanks to Dr. J. Alcala for his attention and rapid reply to my enquiry in the issue of friction in indentation size effect. I also like to express my gratitude to En. Ashammudin for his help in the laboratory.

In the journey of my PhD study, I did not traveled alone but accompanied with a large group of students and colleagues who are gifted in creating a caring and

fun environment. Therefore, huge thanks go to Ko Ying Hao, Yen Kin Sam, Lim Jiunn Shu, Ding Siew Hong, Ooi Ru Yen, Tan Wei Hong, Zhong Zong Leung, Yida, Maria, Hamimi, Teoh Yew Heng, Wong Wai Chi, Lo Jian Haur, Wong Chin Koon, Lim Kar Loon, Lim Phui Weng and Tan Yik Soon.

TABLE OF CONTENTS

LIFT OF TABLE

LIST OF FIGURE

LIST OF ABBREVIATIONS

KESAN KEKASARAN PERMUKAAN PADA LEKUKAN MICRO

ABSTRAK

Bagi lekukan mikro, ciri-ciri kebergantungan skala ditemui di mana saiz lekukan yang lebih kecil menghasilkan nilai kekerasan yang lebih besar, dikenali sebagai kesan saiz lekukan (KLS). Bagi lekukan skala kecil, saiz lekukan dan saiz puncak adalah kecil jika dibandingkan bagi ubah bentuk permukaan adalah “kasar”. Dengan itu, rintangan tersebut yang disebabkan oleh geseran permukaan adalah berkaitan dengan kekasaran permukaan yang akan mengubah tahap ubah bentuk yang menyumbang kepada saiz lekukan. Pengaruh kekasaran permukaan kepada kesan saiz lekukan dikaji menggunakan model rintangan spesimen berkadar (RSB). Dengan asas RSB, satu model baru kekerasan yang dinisbahkan $\frac{H}{H_o} = \frac{c_o + c_1 R_a}{a_2 d} + 1$ dibangunkan untuk menunjukkan perkaitan di antara KSL dan kekasaran permukaan. Kajian ini menunjukan indeks Meyer n meningkat dengan kekasaran permukaan. Daripada analysis menggunakan model RSB, permukaan yang lebih kasar memberikan rintangan spesimen berkadar yang lebih besar akibat daripada peningkatan a_1 iaitu pekali berkaitan dengan RSB. Model geseran yang bergantung kepada kekasaran permukaan $\mu_o = \sqrt{\frac{H_0^2\left[\sqrt{(1+\frac{h^*}{h_c - 2.46R_c})} + (\frac{0.39R_a}{h_c - 2.46R_a})\right]^2 - p^2}{\alpha p^2}}$ juga telah dibangunkan bagi meramal nilai pekali geseran dan model ini menunjukkan permukaan yang lebih kasar memberikan pekali geseran yang lebih tinggi di dalam lekukan mikro.

THE INFLUENCE OF SURFACE ROUGHNESS IN MICRO INDENTATION

ABSTRACT

In micro indentation, scale dependent behavior is encountered as the smaller indentation size produces higher hardness value which is known as indentation size effect (ISE). For small scale indentation, the indent size and the contact asperity size are relatively small if the deformed surface is “rough”. Therefore, the resistance attributed from the friction interface associated with surface roughness alters the deformation severity which contributes to the change of indent size. The effect of surface roughness on ISE in micro indentation is studied using the proportional specimen resistance (PSR) model. With the basis of PSR model, a new normalized hardness model $\frac{H}{H_o} = \frac{c_o + c_1 R_a}{a_2 d} + 1$ is developed to show the relationship between the ISE and the surface roughness. This study shows that the Meyer’s index n increases with rougher surface. From the analysis using the PSR model, rougher surface gives higher proportional specimen resistance due to the increase of coefficient related to proportional specimen resistance a_1. Subsequently, the normalized hardness value increases with roughness. In addition, a surface roughness dependent friction model $\mu_o = \sqrt{\frac{H_0^2\left[\sqrt{(1+\frac{h^*}{h_c - 2.46R_c})} + (\frac{0.39R_a}{h_c - 2.46R_a})\right]^2 - p^2}{\alpha p^2}}$ is developed to predict friction coefficient and the model shows that the rougher surface gives higher value of friction coefficient in micro indentation.

CHAPTER 1

INTRODUCTION

1.0 Overview

In this chapter, the research background and motivation are briefly described followed by the description of objectives and contributions of the research and end with thesis scope and outline.

1.1 Research background

Assessment of material hardness characteristic using indentation method measures the material's ability in resisting plastic deformation. The indentation method uses a solid indenter forced into a sample surface and followed by subsequent release of the force. The release of the force prompts the material elastic recovery together with resulting plastic deformation in the form of a permanent indent on the surface, the indentation size of which is taken as the measure of material's resistance against the applied load. The empirical hardness formulation of a material is based on the ratio of the applied force over the projected area of the permanent indent.

Conventional hardness test provides the hardening characteristic of material which in general gives a reasonably constant hardness value for macro scale indentation. Rockwell test is an example of the macro scale indentation technique using indentation peak load of 10 kgf (for most metal). In the early development of hardness test, the hardness value obtained with such large load was deemed as constant for all scale regimes. In the past few decades, enormous development in small scale mechanical structure in micro-electro-mechanical system (MEMS) yields strong interest in understanding the material mechanical performance in small scale regime (Cao, 2001, Garino et al., 2002).

The evolution from conventional hardness test to smaller scale indentation technique reveals a scale-dependent hardening behaviour of the tested material. This is observed when the specimen is subjected to smaller indentation load, The indentation size is reduced resulting in the increase of hardness value. This scale dependent hardness phenomenon is termed as indentation size effect (ISE) where the hardness increases with the decreasing indentation size (Pharr et al., 2010, Nix and Gao 1998, Ren et al., 2002). The ISE was first encountered in Vicker's micro hardness testing (Mukhopadhyay and Paufler, 2006). Figure 1.1 shows the ISE for Cu single crystals. In this figure, the material shows ISE which is attributed to the

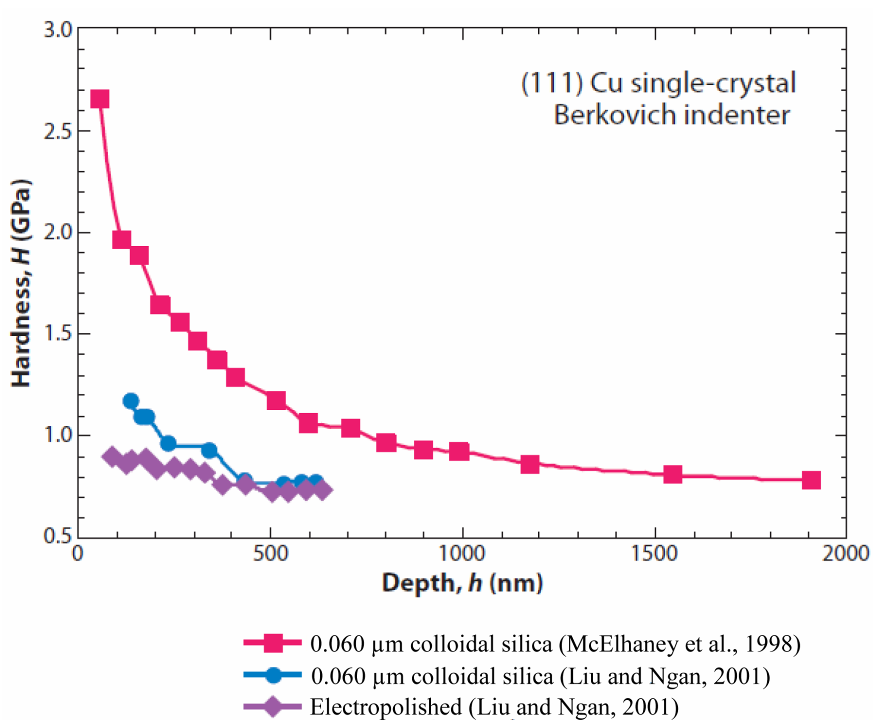

Figure 1.1 The ISE of Cu single-crystal attributed to the variation in indentation depth and different surface polishing process (Pharr et al., 2010).

variation of indentation depth and surface polishing process where the change of the indent size leads to the variation of hardness value.

Micro hardness tester with diamond pyramidal 4-faced indenter is usually used in small scale characterization as the sharp tip configuration easily induces plastic flow on the material surface at low indentation load. The indenter is known as Vickers indenter where the angle of two opposite faces is 136° to form a sharp configuration. According to ASTM E384, the load for micro hardness is within the range from 1gf-1000 gf. The European Standard EN ISO 14577-1:2002 specifies the tested load to be below 2 N for micro hardness testing. Compared to the conventional hardness test load, the load used in micro indentation is relative low and thus, it results significant ISE since hardness is scale dependent.

The ISE phenomenon remains a debatable research topic and many factors that contributed to ISE have been investigated. One of the most controversial factor that induces the ISE is the surface condition of the specimen. In general, most of the micro indentation studies prepared the specimen in well-polished condition without quantifying the surface roughness before indentation (Li et al., 1994, Ren et al., 2002, Chicot et al., 2007, Fares et al., 2009). Even in mathematic analytical studies, the material surface is assumed flat (Oliver and Pharr, 2004, Mata and Alcala, 2004, Guo et al., 2010). However, this assumption does not reflect the actual condition of the tested material surface. In reality, all surfaces have topographical configuration generally known as surface roughness. For small scale indentation such as in the micro hardness test, the size of the indenter tip is small compared to the asperity size for a very rough surface prepared for indentation. In such circumstance, the indenter tip deforms the asperity first rather than directly deforming the "surface" which is required to characterise the material in producing shallow indentation. Kim et al.

(2006) advocated that penetration process started with flattening of the rough surface (asperities) first followed by the deformation of the flattened surface. In agreement with this concept, Wai et al. (2004) suggested that penetration needs additional load to flatten the asperities in rough surface first where this is ignored in the flat surface assumption as shown in Figure 1.2. In this figure, load P_1 is applied to rough surface

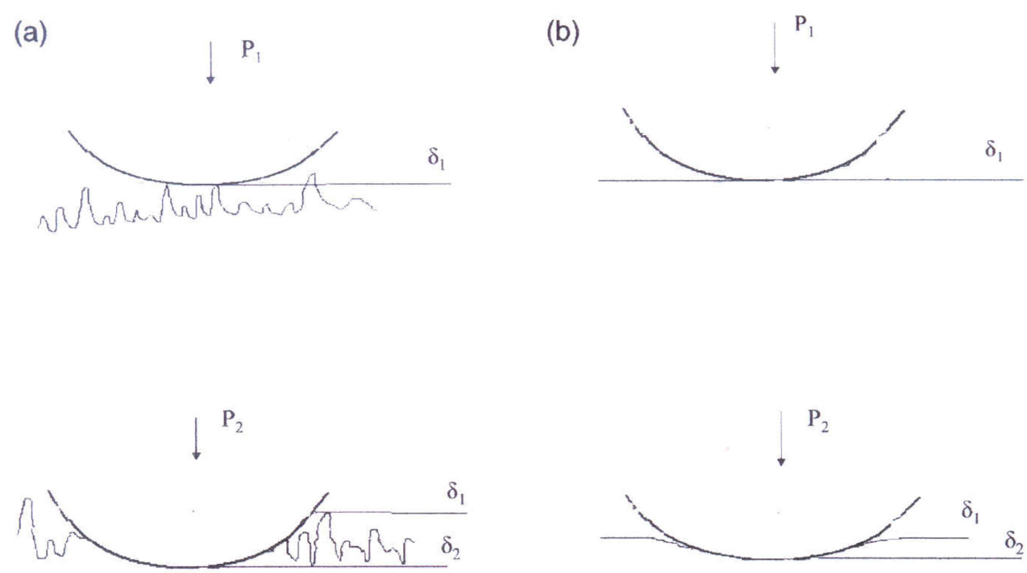

Figure 1.2 Indentation on (a) rough surface and (b) smooth surface (Wai et al., 2004).

and flattening of asperities occurs at the initial loading stage. The subsequent load P_2 required to make full contact between indenter and material while smaller load P_1 can make full contact on smooth surface. Since the amplitude of the load used varies with the different level of surface roughness, the resulting indent size is subjected to the ISE. To address this issue, BS EN ISO 14577-4:2007 limits the specimen surface roughness, R_a<0.05h_c where R_a is the surface roughness and h_c is the indentation contact depth. Nevertheless, this guide line does not guarantee that the ISE can be avoided with the prescribed surface roughness condition.

The phenomenon of ISE has been related to the friction effect of the contact interface between the indenter facet and material surface (Xu and Agren, 2004). Micro indentation commences when the indenter tip in initial contact with the topographical surface. Once the indenter tip is in contact with an asperity, friction is established at that contact interface. As the surface properties are inherent in the asperities of the specimen surface, interpretation of the surface roughness effect on the friction at the interface is imperative for finite element analysis. Although the all material are characterized with topographical surface condition, most studies modelled the material surface as flat configuration with frictional contact interface between indenter and material surface (Xu and Agren, 2004, Huang and Pelegri, 2007, Pelletier et al., 2009, Ponthot et al., 2009, Guo et al., 2010). In general, the friction resistance is represented by the friction coefficient μ. As an indication, the friction coefficient between the indenter and the SiO_2 material surface having μ values of 0, 0.1 and 1.0 require 1202.4 μN, 1210.1 μN and 1250.0 μN force respectively to deform the SiO_2 surface to the same indentation depth (Huang and Pelegri, 2007). In addition, the decrease in friction also creates a material accumulation around the indentation and induces the pile up effect which subsequently increases the indent size and results in the decrease of the hardness value (Xu and Agren, 2004, Sarris and Constantinides, 2013). Even though the "trial and error" approach with application of constant μ for the contact interface give results closer to the experimental result, the understanding and applicability of the friction coefficient μ induced by surface roughness remains unclear in justifying its role in ISE in micro indentation.

For a given series of load, ISE phenomenon can be observed when the hardness value increases with the decrease of the indent load. Several theories have been developed in order to explain the effect of ISE. Using Meyer's law, if the Meyer's index n is less than 2, the ISE is said to be present in the loading range. Although this theory is important in determining the presence of ISE but it lacks evidence in justifying the attribution of ISE. To overcome this, Li and Bradt et al. (1993) suggested that the additional load that yields ISE is attributed to the load used to overcome the proportional specimen resistance (PSR) which is proportional to the indentation size and related to the material elasticity and friction effect. Another well-established theory to describe the ISE is Nix-Gao model with the basis of material dislocation theory used to explain the material mobility during indentation (Nix and Gao, 1998). This model establishes that the normalized hardness H/H_o is inversely proportional to a given indentation contact depth. With the normalized hardness value, one can predict the ISE severity by taking the load independent hardness H_o as benchmark.

Although investigation on the effect of surface roughness on ISE has been done previously, there are two important issues remain unsolved in micro indentation.

a) Detail measure of ISE where the severity of change of indent size associated with surface roughness has not been addressed.

b) The actual friction coefficient arising from surface roughness effect which leads to ISE is still unknown.

Therefore, it is important to quantify the effect of surface roughness to ISE and determine the friction coefficient for the contact interface in micro indentation. The evaluation of PSR associated with different material surface roughness in a series of indentation load using PSR model has not been reported in the literature. This thesis establishes the relationship between the normalised hardness H/H_o with the coefficient related to proportional specimen resistance a_1 in order to evaluate the ISE attributed to the friction effect. In addition, the friction coefficient is determined in the micro indentation.

1.2 Objective

In order to address the unsolved issues on the measure of severity of ISE and friction coefficient associated to the surface roughness, the objectives of this study are:

1. To determine the influence of roughness on ISE severity in micro indentation using Meyer's law for a series of load and surface roughnesses for stainless steel, aluminium and copper material. These materials are chosen in order to examine the roughness effect of material with hard and soft nature where the variation of hardness of these materials renders different severity on the ISE.

2. To evaluate quantitatively the resistance attributed from the surface roughness in micro indentation using PSR model. With the basis of PSR model, a normalized hardness model is proposed which relates to a_1 in order to evaluate the PSR associated with roughness effect on the ISE where the normalized hardness is in the function of roughness.

3. To determine the friction coefficient μ for different surface roughness in micro indentation for stainless steel.

1.3 Contributions

The present research investigates the friction effect associated with variation of surface roughness in micro indentation. Proportional specimen resistance (PSR) model is a well established theory and used in many research works, but no detail study on the friction effect associated with surface roughness using PSR model has been done. The main contribution of this study is the establishment of the normalized hardness equation (H/H_o) using the PSR model to determine the severity of ISE due to the surface roughness. In addition to the determination of ISE severity, the normalized hardness equation can be taken as a correction factor to correct the ISE without roughness effect. With this correction factor, the materials can be characterized on a perfectly flat surface in micro indentation testing. The second contribution of this research work is to predict the roughness-dependent friction coefficient for indentation on three levels of surface roughnesses in micro indentation. This friction coefficient interpreted from the effect of surface roughness reflects the sliding contact interface response induced by the different topographical surface for micro indentation.

1.4 Thesis scope and outline

The scope of this thesis focuses on the evaluation of surface roughness effect and friction that contributes to ISE. First, the evaluation of ISE is done using Meyer's law, PSR model and a normalized hardness equation is proposed to measure the severity of ISE. Second, the friction level interpreted into friction coefficient is investigated for various surface roughnesses in micro indentation associated with the finite element analysis results.

With the objective of this study, the scope of this research focuses on the micro indentation study for metal material with hardness value range from 0.70 GPa to 4.03 GPa. The materials' surface roughness was confined from 6.22 nm to 132.79 nm for indentation with load range of 0.147 N to 1.962 N.

This thesis is presented in seven chapters. The first chapter briefly describes the background of the effect of surface roughness to ISE, models used to evaluate the ISE and contribution of present research. In addition, the objectives of present study were established in order to measure to measure the severity of ISE and determine the friction coefficient associated with effect of surface roughness and penetration load in micro indentation.

The second chapter reviews the concept of indentation method used for hardness test. An overview on micro indentation test is included in the following section. The contribution of surface roughness, the effect of friction and pile up to ISE, the theories employed to evaluate ISE and consideration in indentation procedure are presented in the end of this chapter.

In chapter three, the methodology of the research is presented. The experimental procedures which consist of polishing process, micro indentation and measurement method for three materials are described in detail. It is followed by the description on the development of finite element (FE) model for micro indentation.

In chapter four, the development of normalized hardness equation is established with the basis of PSR model. It predicts the ISE corresponding to the roughness effect for micro indentation. For the following section, the model of roughness dependent friction coefficient of micro hardness is developed and presented. For the model development, the surface roughness-Nix-Gao model associated with Tabor equation gives friction coefficient as a function of surface roughness.

In chapter five, the experimental result is presented and effect of surface roughness on micro hardness is evaluated using Meyer's law and PSR model. Detail discussions on the presence of ISE using Meyer's law and the analysis on the proportional specimen resistance under variation of surface roughness were elaborated in each section. This is followed by evaluation of ISE using the proposed normalized hardness equation.

In chapter six, the results of experiment and FE simulation are presented and evaluated accordingly based on the analytical model as described in chapter four. Discussions are drawn respectively based on the outcomes from the analysis.

This thesis ends with the conclusions and future works.

CHAPTER 2

LITERATURE REVIEW

2.0 Overview

In this chapter, literature review commences with the explanation on the technique of hardness followed by the introduction of micro-indentation test. The surface condition, friction factor and pile up in ISE are discussed in the subsequent section. Evaluation of ISE is presented with the existing theories. Lastly, this chapter ends with the important considerations in micro indentation test.

2.1 Micro indentation

Hardness characterization with indentation method is based on the resistance of a material to the plastic deformation. The indentation is achieved with the application of forced indenter loaded on the material surface and followed by the force removal from the material as shown in Figure 2.1. In this figure, the loading and unloading process induce the change in the deformed surface depth due to the

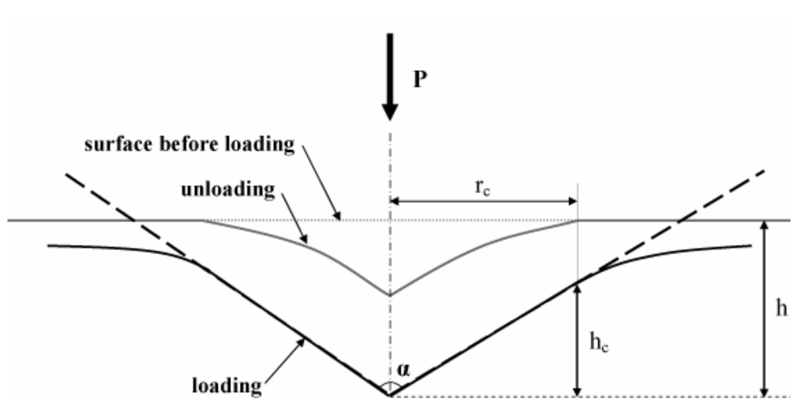

Figure 2.1 Indentation loading and unloading process (Oliver and Pharr, 2004).

elastic recovery where h is indentation depth, h_c is the indentation, r_c is the indent size from indentation center and α is the indenter angle. It should be noted that the contact indent size r_c during indentation is equal to the size of the residual indent at post indentation since the material at the contact region experiences the plastic deformation that leads to the formation of indent.

Conventional macro scale hardness characterization has been reported to produce almost constant hardness value (Voyiadjis and Peter, 2010). Nevertheless, the advent of small scale devices such as MEMS demands the understanding of material performance at small scale. This has lead to the application of micro-scale hardness characterization to evaluate the micro scale material behaviour where an indenter is driven into a well-polished material surface at a controlled load. According to ASTM specification E384, micro indentation involves the use of penetration load of less than 1kgf (ASTM, 2005). The European Standard EN ISO 14577-1:2002 specifies the load to be below 2N for micro hardness testing. Such small load on the examined material produces a very small residual indent on the surface. With the conventional optical microscope, the real contact surface or projected area is observed and the residual indent size is measured after the examined material experiences the elastic recovery.

2.2 Indentation size effect (ISE)

One interesting hardness characterisation phenomenon which can be observed is that the resistance against plastic deformation increases with the decrease in the indent size when the sample is loaded with smaller indentation load. This phenomenon was first encountered in Vickers testing in micro-scale indentation (Pharr et al., 2010). This size dependent behaviour exhibits “the smaller, the stronger" characteristic. This phenomenon has been investigated and attributed to scale dependent behaviour, surface condition, the friction interface between the indenter and material surface and pile up effect (Nix and Gao, 1998, Mata and Alcalá, 2004, Kim et al., 2007, Li et al., 2009).

2.2.1 Surface condition

Conventional large scale indentation for hardness testing in general does not require stringent surface preparation with fine polishing procedure, since the indenting depth is relatively large compared to the surface roughness, and as such, the surface roughness apparently does not influence the bulk hardness value.

In routine metallographic process, the polished surface provides a considerably flat surface for indentation. In reality, surfaces are characterized in topographical configurations with the surface roughness affecting the force required to deform the asperities (Greenwood and Williamson, 1966, Berke et al., 2010) and the size of the projected area of the residual indent. Tabor (1951) investigated the loads required to initiate the plastic deformation for soft copper with differing asperity radius when subjected to a hard flat surface. The asperity with radius of 0.0001 cm, 0.001 cm, 0.5 cm and 1 cm yielded at a load of 2.5×10^{-6} g, 0.025 g, 62 g and 250 g respectively and this shows that indentation load increases with the increase of asperity size. From the results obtained from the mathematical model, surface roughness is a factor that contributed to the load dependent plastic deformation (Tabor, 1951) where the asperities deformed elastically and followed by plastic flow until their area is sufficient to support the load applied by the hard flat surface.

Micro scale indentation requires the penetration using a small load, and since the size of the indentation is in the range of several micrometers, the surface roughness of the sample material becomes a crucial factor in affecting the resulting hardness value. Kim et al. (2006) suggested that the indentation process consists of initial flattening of the rough surface and followed by the deformation of the flattened surface. If the sample is prepared with very rough surface, the penetration size is comparatively small compared to the asperity size. Then, the indenter will contact and deform the asperities with the remaining energy used to deform the bulk. Wai (2004) found that additional load is required to deform the coarser asperities of the rougher surface of polystyrene sample. Therefore, the penetration on rougher surface generates smaller indent depth size which subsequently results in an exponential increase of hardness value below the penetrating depth of 0.2 μm (Bobji and Biswas, 1998).

Many studies have assumed that the material surfaces are flat, with the surfaces modeled as 2D flat platforms for indentation (Mata and Alcalá, 2004 and Xu and Agren, 2004, Ponthot et al., 2009, Wang and Fang, 2009, Guo et al., 2010, Strange and Varshneya, 2001). In order to evaluate the efficiency of 2D and 3D model, Shim et al. (2007) simulated the sharp indentation on 3D flat surface and both finite element (FE) models exhibited almost identical loading curves for increasing indentation depth. In addition, the indentation on 3D flat surface using Berkovich indenter gives the illustration of appearance of sinking which deviated from the ideal triangle geometry for the contact area as shown in Figure 2.2. In order to simulate the

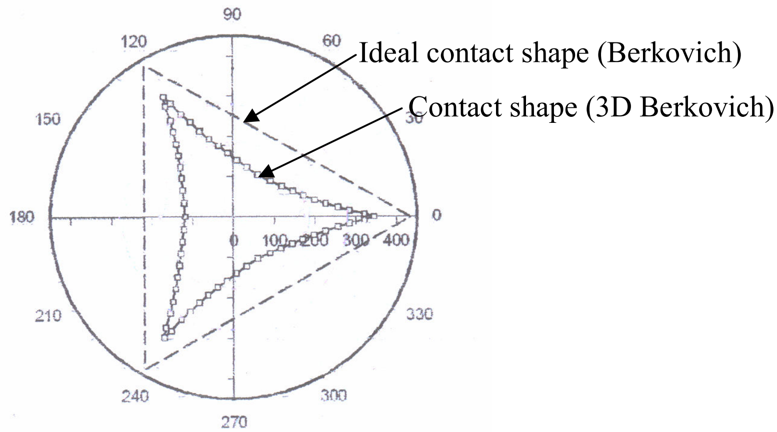

Figure 2.2 The ideal contact and sinking in contact geometry during maximum loading.

indentation closer to the actual surface condition of the sample material, Walter and Mitterer (2009) analyzed a material surface using the actual three-dimensional (3D) surface topography taken from atomic force microscopy data. The 3D indenting model for the nano indentation showed less scatter response in the load-displacement results than the 2D model and the 3D curve trend is similar to the result generated from the measured indentation. These results shows that the actual topographical surface should be taken into account in the FE model development but most of the recent indentation studies still assume the material surface is flat.

2.2.2 Friction effect

The solid surface represents the material boundaries and roughness is one of the parameter that can be used to describe the disordered texture of the natural state of a surface (Thomas, 1999) where it quantify the vertical deviations of the asperities height from it mean height level. Once the indenter touches and penetrates the rough surface, friction force is established for the asperities of rough surface to climb and interact with the surface of the indenter (Bhusan, 2005).

Since the roughness is random on the sample surface, the accountability of the effect of surface roughness in the friction at the contact interface is necessary in the micro indentation analysis. The effect of friction on the resistance against the plastic deformation can affect the indent size and this has been investigated using both experiment and computational analysis. Shi and Atkinson (1990) proposed that the friction governs the hardening behaviour that leads to the variation of hardness. This is supported by the indentation experimental set up under dry and lubrication for aluminium and copper material in load range of 15gf to 20kgf. The Vickers hardness values for both materials were found higher at dry unlubricated condition than the lubricated condition. Similar findings were also encountered by Wang et al. (2004) and Li et al. (2009) in the indentation of 304 stainless steel and $Pd_{40}Cu_{30}Ni_{10}P_{20}$ material under different lubrication condition of dry, application of degraded oil and anti-wear lubrication condition. By extending Shi and Atkinson (1990)'s work, Li et al. (1993) explained the ISE with the ratio of *S/V*, where *S* indicates the surface area and *V* indicates the displaced material volume during indentation. As the indentation size is small in low load or under lubricated condition, the interface area is larger compared to the displaced volume. The fact that friction increases at low load or small indent size indentation (Li and Bradt, 1993, Li et. al., 2009) reflects that the friction is not constant but it is load dependent. Nevertheless, determination of friction level is rather complex when indentation is done on the surface with random roughness.

During indentation, low friction condition induces higher material outflow and increases the projected area, which reduces the hardness value (Mata and Alcalá, 2004, Xu and Agren, 2004). Friction has also been reported to contribute to the appearent hardness value (Li et al., 1993, Shi and Atkinson, 1990) where friction at the contact interface strongly governs the hardening effect where the hardness value increases with friction resistance. Nevertheless, the friction effect associated with surface roughness that contributes to ISE remains unclear.

All the researches reviewed to date utilized a constant friction coefficient for contact interface between the indenter and material surface in the FE analysis (Guo et al., 2010, Pelletier et al., 2009, Xu and Agren, 2004, Mata and Alcalá, 2004, Ponthot et al., 2009, Huang and Pelegri, 2007, Berke, et al., 2010). Huang and Pelegri (2007) investigated the load required to deform the flat surface under different friction coefficient for contact interface. With three different friction coefficient values of 0, 0.1, 1.0, the loads of 1202.4 μN, 1210.1 μN and 1250.0 μN are required to deform the SiO_2/Si surface for the same maximum penetration depth. In addition, the material surface also experienced increase in contact area of 0.1151 μm^2, 0.11515 μm^2 and 0.11534 μm^2 respectively. Xu and Agren (2004) simulated the contact interface with increase of friction coefficient from 0 to 0.2 which resulted in the increase of contact area due to the extension of plastic core beyond the circle of contact. From the review made here, friction can be said as the factor that contributes to the change in the contact area which subsequently resulted in the variation of hardness. Despite the fact that friction is active on the interface and applied in these simulations, the constant friction coefficient used did not reflect the actual friction interface between the indenter facet and material surface.

2.2.2.1 Friction model of micro indentation

The influence of friction on the sharp indentation on perfect plastic solid was investigated by Johnson (1985) in which he proposes a friction model given by

$$\mu = 1 - H_o / H \tag{2.1}$$

where μ is the friction coefficient, H_o is hardness for frictionless condition and H is hardness when friction is present in the indentation process. Using equation (2.1), Xu and Agren (2004) found that the friction coefficients of the contact interface are below 0.2 for materials of different properties in the simulation of micro indentation. Nevertheless, the indentations were simulated on perfectly flat surface without consideration of the actual material surface topography.

For the sharp indentation, Mata and Alcalá (2004) investigated the relation of friction coefficient to indent size by taking into consideration the pile up effect. The study proposed a friction model written in the form of

$$H = \beta H_o (1 + \mu \cot \gamma) \tag{2.2}$$

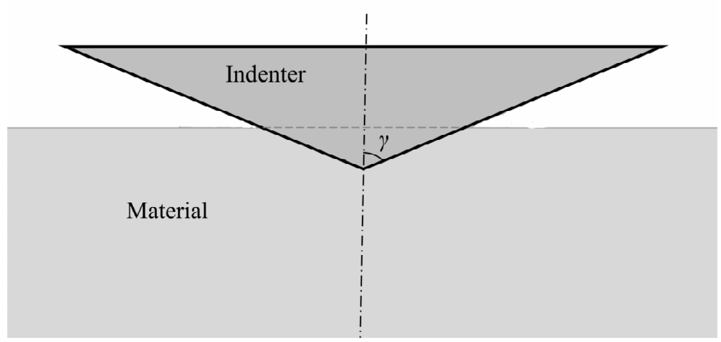

Figure 2.3 Indentation on material using sharp indenter with half apex angle γ.

γ is the half apex angle of conical indenter as shown in Figure 2.3. $\beta = \frac{\pi a_o^2}{\pi a^2} j$ is the pile up or sinking in parameter where a_o is projected area under friction condition, a projected area with friction contact and j is the normalized pressure. Nevertheless, the formula applicability is limited for the case of contact interface with constant friction coefficient value between 0-0.2.

Ponthot et al. (2009) suggested a friction model for indentation using defect conical indenter in the form of

$$\mu = \frac{H - H_o}{\lambda_o} \tag{2.3}$$

where λ_o is a constant related to pressure. By employing the parameter μ in the range of 0-0.15 for the contact interface in the numerical simulation, the H value increases with the increase of μ value for stainless steel, copper and aluminium. Even though the friction effect contributes to ISE, the relationship between friction level and surface roughness remains unclear that the friction coefficient still has not been determined for micro indentation.

2.2.3 Pile-up

Loading of indenter onto the surface not only pushes the particles deeper into the material but also creates plastic flow around the indentation. In some cases, the resulting plastic deformation removes and stakes the material near the indenter where the material accumulation will extrude higher than the normal surface level. The ensuing diameter can be twice the size determined at normal surface level (Tabor, 1951). This phenomenon is known as pile up effect. Figure 2.4 shows the pile up effect of the indentation by using pyramidal four face indenter. In this figure, the surface profile is higher than the surrounding undisturbed surface level near the residual indent (Zbib and Bahr, 2007).

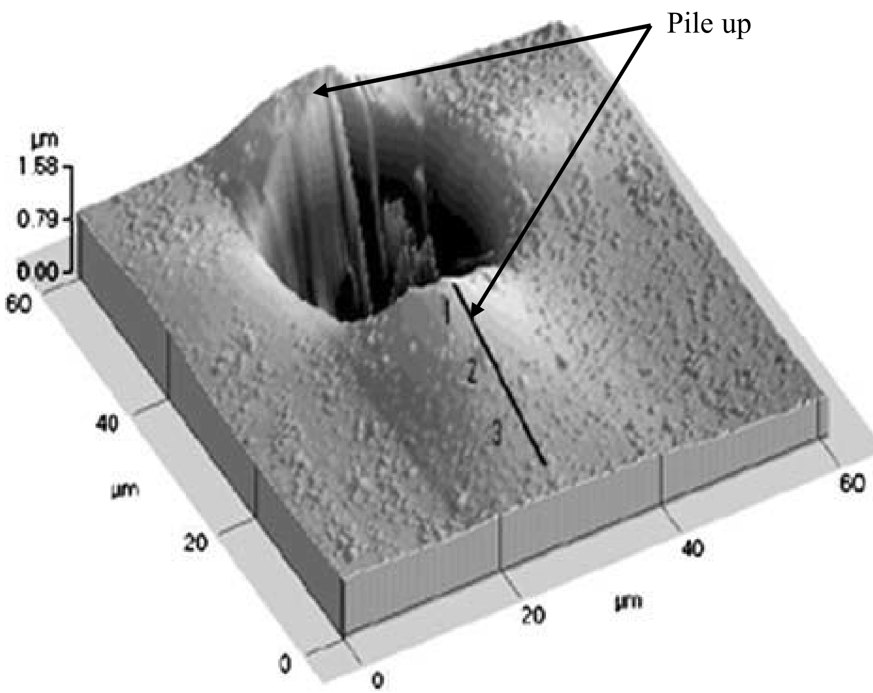

Figure 2.4 The indent with pile up effect in three dimensional view (Zbib and Bahr, 2007).

In the pile up effect, the plastic deformation severity can be determined by the size of the plastic zone of the material beneath the indenter. With low friction, the plastic core is smaller which causes the pile up effect (Ponthot et al., 2009). In further pile-up effect investigation, Guo et al. (2010) extended Ponthot's (2009) work and defined the pile up effect with parameter of β where $\beta = \frac{h_c - h}{h} \times 100\%$. With a decrease in friction coefficient, values β increase for all materials (Xu and Agren, 2004) where larger β value indicates more severe pile up effect thus increasing the projected contact area as shown in Figure 2.5. In this figure, the side view shows the pile up region indicated by the extrusion region while the additional projected area (grey colour area) resulting from pile up effect is shown in top view.

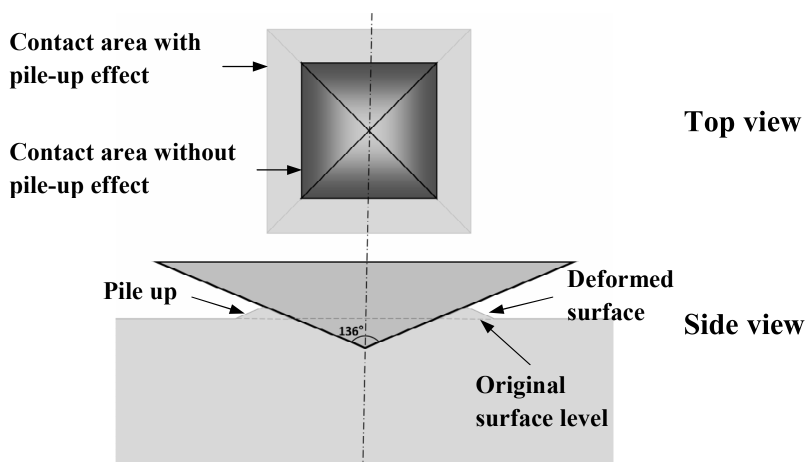

Figure 2.5 The widened area resulting from pile-up effect.

In order to enhance the accuracy of the hardness result, the widened projected area due to the pile up effect should be taken into account in the hardness calculation. In small scale indentation, small indentation with pile up effect generally produces poor observation using microscope and the projected area are estimated from the captured 2D images (Graca et al., 2007, Sangwal and Klos, 2005, Machaka et al., 2011, Soifer and Verdyan, 2005, Zhang et al., 2005, Zong, et al., 2006). Figure 2.6 (a) shows the 2D micro- and Figure 2.6 (b) the nano scale indent image of Nickel (Graca et al., 2007). From the figures, the deformed region (white in colour) can be seen at each side of both residual indents which represent the pile up region. Due to these unclear images, the wider border of the region due to the pile up effect can not be defined accurately resulting in error in the hardness calculation.

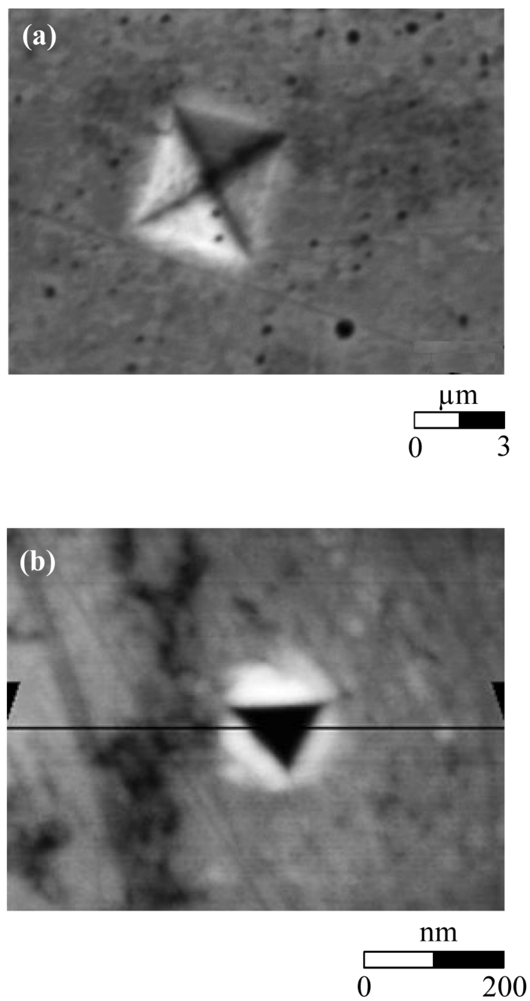

Figure 2.6 The 2D images of residual (a) micro- and (b) nano-indent on sample surface (Graca et al., 2007).

In order to include the extra widened area of the pile up effect of the projected area, Saha and Nix (2001) proposed that the extra area to be taken as semi-ellipse shape where it covers the area between the arc and indentation edge as shown in Figure 2.7. This method was used to predict the true contact area (Kese et al.,

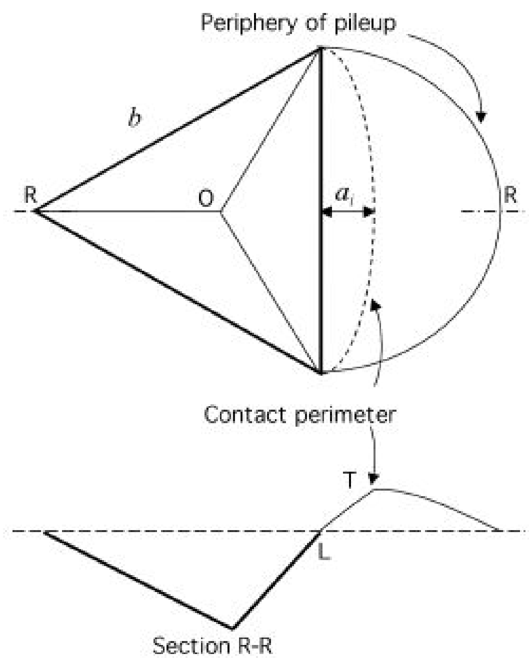

Figure 2.7 The pile up schematic diagram of a Berkovich indent and it's cross section. In top view, the semi eclipse arc is taken as the boundary of the projected area (Kese and et al. 2005).

2005, Kese and Li, 2006, Sun et al., 2008, Khan et al., 2009, Hussainova, 2011). Nevertheless, the arc shape is an assumption of the pile up area which it is different from the actual indentation.