Indentation mechanics of layered solids

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INDENTATION MECHANICS OF LAYERED SOLIDS

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SCHOOL OF MECHANICAL & AEROSPACE ENGINEERING

2006

INDENTATION MECHANICS OF LAYERED SOLIDS

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Abstract

Micro or nano probe instruments, such as nanoindenter and surface force apparatus (SFA), are used for the measurement of surface energy and other material properties of thin film or microelectro mechanical systems. Continuum mechanics models of contact load - deformation is necessary to interpret the measured load - displacement data. At micro or nanoindentation test of thin films, the adhesive surface forces operating between the indenter and the thin-layer will contribute to the deformation. An appropriate mechanics model is required to explain the experimental measurements. In this thesis, the Johnson-Kendal-Roberts (JKR) adhesion theory for bulk elastic solids is extended to the adhesion analysis of layered thin film system. A numerical approach to study the indentation of layered solids in the presence of adhesion is presented. The finite element (FE) calculation effectively takes into account both adhesionless and adhesion indentations.

In the elastic three-layer geometry, the numerical version of JKR theory is used to carry out a specific case study for the SFA experimental measurements. In the SFA measurements, contact radius-load curves with and without adhesion are obtained by chemically treating the surfaces. The experimentally measured geometrical and mechanical data of this SFA are used as input parameters in the FE calculation. The contact load of FE analysis is separated into adhesionless and adhesion parts, which are connected by the adhesion parameter. The effective moduli measured from SFA experiment are in good agreement with that computed by FE. The SFA measured contact radius-load curves in the presence of adhesion are in reasonable agreement with extended theory. The pull-off force provides the most direct measure of the surface energy, and is found to be a strong function of the adhesion parameter. For the experimental SFA geometry, the FE analysis shows that pull-off force $P_c = 1.75\pi wR$, which is intermediate to the JKR limit ($P_c = 1.5\pi wR$) and the DMT limit ($P_c = 2\pi wR$).

For the elastic two-layer geometry, adhesion between a spherical indenter and thin compliant elastic film applied on a substrate has been investigated. During the modeling and calculation, the net load and displacement of indentation are decomposed into non-dimensional adhesionless and adhesion components. The variations of contact size versus load, load versus displacement for a series values of adhesion parameter are computed. By analyzing the numerically calculated load and compliance data across the entire range of contact radius, empirical relations for the compliance and hence for the net load as a function of contact radius are obtained. The minimum value of load for a given adhesion parameter represents the pull-off force at which the surfaces separate abruptly. The pull-off force is a function of adhesion parameter, thin film Poisson's ratio and the stiffness ratio. The maximum non-dimensional pull-off force can be 0.95, which is very different from the JKR limit 0.5. The effect of friction between the indenter and thin film surface on the deformation behavior of the layered system is analyzed. The friction effect on the load and displacement functions is very small except for the transition region. The experimental verifications demonstrate that the extended numerical version of JKR theory is applicable for the adhesion analysis of layered materials.

The adhesionless and adhesion contact between the spherical indenter and linear incompressible viscoelastic thin film applied on a rigid substrate is analyzed by extending the empirical results of elastic thin film. For the adhesionless analysis of viscoelastic thin film, the contact cycle is separated into two phases, i.e. closing contact and opening contact. The corresponding principle is used in the closing contact phase. The controlled linear and sinusoidal loading/unloading cycles are considered. The variation of contact radius versus time through such a ramp cycle is calculated and compared with the results of bulk viscoelastic material. Johnson's analysis for the adhesive contact of bulk viscoelastic spheres is extended to the adhesion analysis of viscoelastic thin film. The load controlled adhesion experiment has been modeled by applying the fracture mechanics principles of a moving crack. The variation of non-dimensional contact radius versus load is calculated for a series of values of loading rate parameter. The pull-off forces needed to separate the

contact surfaces are enhanced based on different loading rate parameter.

Protective stiff and strong thin films are routinely deposited on a soft ductile substrate to tailor many surface properties in a controlled fashion. When this layered system subjected to a normal load, the substrate may undergo plastic deformation before triggering any other failure mechanism. Finite element method is used to perform an accurate numerical study of the normal indentation of an elastic harder film on a Ramberg-Osgood hardening elastic-plastic substrate by a rigid sphere. The solution is compared with the similarity solution of the bulk elastic-plastic solids and purely layered elastic solids. The effects of elasticity, yield strain and strain hardening of the substrate on the indentation mechanism are explored. The indentation force and depth as a function of contact size in non-dimensional form for a series of substrate yield strains and strain-hardening exponents are calculated. The deformation mechanism of layered solids from elastic deformation to finite plastic deformation is shown as an indentation map. The stress distribution is not continuous in the interface between thin film and substrate, which may lead to the layer-substrate interface failure or interfacial delamination.

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List of Abbreviations and Symbols

AFM	Atomic Force Microscope
BEM	Boundary Element Method
DMT	Derjaguin-Muller-Toporov
FEM	Finite Element Method
JKR	Johnson-Kendall-Roberts
MEMS	Micro Electro Mechanical Systems
M-D	Maugis-Dugdale
SEM	Scanning Electron Microscope
SFA	Surface Force Apparatus
E	Young's modulus
E_i	Young's modulus of i th material
E^*	Combined elastic modulus, $E^* = [(1 - \nu_1^2)/E_1 + (1 - \nu_2^2)/E_2]^{-1}$
E'	Plain-strain Young's modulus, $E' = E/(1 - \nu^2)$
E'_e	Effective modulus
E'_i	Plain-strain Young's modulus of i th material, $E'_i = E_i/(1 - \nu_i^2)$
G	Energy release rate
G_c	Critical energy release rate
J_2	Second invariant of stress tensor
K_I	Stress intensity factor of mode I
K_{II}	Stress intensity factor of mode II
P	Net load
P_0	Adhesionless load
P_a	Adhesion load
P_c	Pull off force
P_m	Maximum load

List of Abbreviations and Symbols

$\bar{P}$	Non-dimensional load
$\bar{P}_c$	Non-dimensional pull off force
R	Spherical indenter radius
a	Contact radius
a_0	Contact radius at zero load
a_c	Contact radius at pull off force
a_m	Maximum contact radius
$\bar{a}$	Non-dimensional contact radius
$\bar{a}_0$	Non-dimensional contact radius at zero load
h	Thin film thickness
k	Stiffness ratio, $k = E'_2/E'_1$
n	Strain hardening exponent of plastic material
p	Pressure
p_0	Maximum pressure
p_m	Mean pressure
t	Time
u_z	Normal displacement
w	Work of adhesion
z_0	Equilibrium distance between atoms
$\Phi(t)$	Creep function
$\Psi(t)$	Relaxation function
α	Non-dimensional adhesion parameter
β	Apparent surface energy factor
γ	Surface energy
δ	Indentation depth
δ_m	Maximum indentation depth
$\bar{\delta}$	Non-dimensional indentation depth
ϵ	Strain
ϵ_0	Yield strain
ϵ_e	Effective strain

List of Abbreviations and Symbols

η	Viscosity constant
θ	Phase angle, $\theta = \tan^{-1}(K_{II}/K_I)$
ν	Poisson's ratio
ν_i	Poisson's ratio of i th material
σ	Stress
σ_0	Yield stress in uniaxial compression or tension
σ_0^s	Yield stress in pure shear
$\sigma_1, \sigma_2, \sigma_3$	Principal stresses
$\sigma_r, \sigma_\theta, \sigma_z$	Principal stresses in axisymmetry state
σ_e	Effective stress
$\sigma_{\hat{r}\hat{z}}$	Tangential stresses in local Cartesian coordinate system ($\hat{r}, \hat{z}$)
$\sigma_{\hat{z}\hat{z}}$	Normal stresses in local Cartesian coordinate system ($\hat{r}, \hat{z}$)

Chapter 1

Introduction

1.1 Background and motivation

Thin films are used in many applications where properties such as resistance to abrasion, corrosion, permeation, and oxidation, or special magnetic and dielectric properties, are needed to meet specialized functional requirements [1]. In these applications, the essential point is that these films are made of special substances, which are different from the bulk material and can therefore be used to tailor many surface properties of the workpiece in a controlled fashion.

Typical example for the application of thin film structures is protective coating layers, which offer improved performance in many areas including injection molding, pharmaceutical and biomedical applications, military uses, and even sports and food service equipment. New coatings are being developed to withstand extreme temperature for the purpose of providing non-stick surfaces in molds and dies, gears and bearings and military weapons. Thin film coatings of steel medical instruments and baking equipments as well as skis, are further examples of applications of protective coatings used in non-ideal conditions.

In addition, the past decade has observed the rapid growth of Micro Electro Mechanical Systems (MEMS) as a very important area of engineering application of thin film technology [2]. In MEMS devices, the mechanical and electrical components are integrated together by using the microfabrication technique based on related thin film technologies. The efficiencies of high volume production and low unit cost achieved by the microelectronics industry over the past years have been successfully translated to the MEMS industry. A host of commercially promising and important devices with unique capabilities at

very small scales such as sensors, actuators, power producing devices, chemical reactors, and biomedical devices have been produced using the microfabrication techniques developed during the past decade. These devices have found wide applications and increasing markets in telecommunication, automobile, chemical, biomedical and many other industries.

With recent advances in engineering, thin film technology is playing an increasingly important role in spearheading technological advancements for future society. Thin film technology is also closely tied to nanotechnology, which is one of the key technologies of the near future. Nanocomposite, nanophase or nanostructured bulk materials and coatings will become tomorrow's workhorse in new-generation manufacturing and precision engineering industries. Thin film coating is already an important part of precision engineering, and it will play an ever-increasing role in nanotechnology.

It has been recognized now that a good understanding of the mechanical behavior is essential for improving the reliability and lifetime of thin films and thus devices based on thin film technologies. The mechanical properties of geometrically constrained materials, such as thin films, often differ significantly from those of their bulk counterparts, which is usually ascribed as the size effect [3]. The mechanical properties of thin coating or layers are commonly measured by a 'contact test' in which a micro or nano probe with a spherical tip is pressed into contact with the surface of the test material and then withdrawn (unloading) until the surfaces snap apart [4]. The contact force and relative displacement (or contact radius) are measured throughout the test. To extract the mechanical properties, such as film hardness, yield strength, modulus etc, from the experimental measurements, it is indispensable to apply a suitable theory.

The contact mechanics of two elastic bulk-solids in the absence of surface forces is well established by Hertz [5]. However, at small deformation scales, the Hertz theory is not suitable, because the adhesion force between the contacting solids is comparable with the applied load, and can not be neglected. The force between individual atoms and molecules, often described through a surface energy, is referred to as the adhesive

force [6]. The influence of adhesion between solid surfaces on their mechanical performance becomes increasingly important with smoother surfaces, smaller length scales, and lighter loads. Due to the effect of adhesion force between two contacting solids, the contact area exceeds that predicted by the Hertz theory, a contact area of finite size is seen at zero load and a tensile force is required to pull the surfaces apart. To model the contact problem with adhesion, Johnson-Kendall-Roberts (JKR) [7] have modified the Hertz theory by accounting for adhesion within the flattened contact region. Later, Derjaguin-Muller-Toporov (DMT) [8] provided an alternative method to include adhesion forces, which act in an annular zone around the contact area without any change in the deformation profile of the contacting solids. These two theories give different expressions for applied load. At first it was thought that these theories were incompatible, until Tabor [9] suggested that each applied to opposite ends of the spectrum of a non-dimensional parameter which expressed the ratio of the magnitude of the elastic deformation to the range of surface forces. Comparing the two theories, Tabor has shown that the JKR theory describes the adhesion contact problem well when the surface forces are short range in comparison to the elastic deformations they produce (i.e. compliant materials, high surface energies, large radii of curvature); while, DMT theory applies well in the case of long-range surface forces with a Hertzian geometry (i.e. stiff material, lower surface energies, small radii of curvature). Following previous researches, Maugis [10] recognized the analogy between adhesion and fracture and showed both experimentally and theoretically, how a wide variety of geometries in adhesive contact could be brought together and treated in a unified and consistent way by the principles of linear elastic fracture mechanics. By applying a Dugdale crack-closure model where the adhesion forces are assumed to be constant on an annulus around the contact area, Greenwood [11] also provided a numerical connection between the JKR and DMT theories. For those contact problems involving compliant elastomers, the experimental results demonstrate that the JKR theory is more effective [12–14].

Nowadays, nano-probe instruments, such as Surface Force Apparatus (SFA) [15–17]

or Atomic Force Microscope (AFM) [18–20] are routinely used to extract the mechanical properties of thin films by subjecting them to indentation process. At these micro or nano indentation loads, the adhesive surface forces operating between the indenter and the thin-layer will contribute to the deformation. To analyze adhesion contact problem of thin films, the standard JKR theory is not applicable for its semi-infinite elastic half-space hypothesis. A novel method based on linear elastic fracture mechanics principles coupled with non-dimensional analysis and finite element (FE) method was developed by Johnson and coworkers [21, 22] to extract the surface energy of the contacting layered system. However, this numerical version of JKR theory is only an initial model. Its validity and application need to be further explored, especially when the incompressibility and viscoelasticity of thin film are considered.

1.2 Objectives and scopes

The basic aim of the proposed research is to develop effective adhesion mechanics models for different types of thin film systems. The scope of this project can be summarized as follows:

- A detailed literature review on the contact mechanics of indentation with and without adhesion is carried out.
- The numerical version of JKR theory for three-layer geometry is validated by simulating a specific surface force apparatus experiment.
- The adhesion mechanics between a spherical probe and a thin elastic (both compressible and incompressible) film on a rigid and elastic substrate is investigated using a finite element method. Empirical relations of the contact load and compliance versus contact radius for flat punch and spherical probe geometries based on parametric studies conducted for the layered systems are developed. The effect of friction between the contacting surfaces on load-displacement is explored.

- Most compliant polymeric coatings are likely to have viscoelastic material behaviors. The contact mechanics of linear viscoelastic thin films with and without adhesion is investigated by extending the empirical results of elastic thin film.
- When a stiffer elastic thin film applied on a ductile elastic-plastic substrate, subjected to a normal load, the substrate may undergo plastic deformation before any other failure triggers. A good understanding of the plastic behavior is essential for improving the reliability and lifetime of thin film systems.

1.3 Organization of report

The report starts with a brief introduction in Chapter 1, which provides the background and the motivation for the proposed research, and then brings out the objectives and scopes of this project.

Chapter 2 reviews the pertinent literature on contact mechanics. It gives a comprehensive review of works on adhesionless and adhesion indentations of elastic, plastic and viscoelastic materials, covering approaches such as analytical methods, experimental measurements and numerical analysis.

Chapter 3 discusses the adhesion mechanics of a three-layer geometry. The numerical version of JKR theory is verified by simulating one specific SFA experiment. In Chapter 4, the numerical version of JKR theory is further extended to two-layer elastic half-space problem. A detailed analysis of adhesion mechanics between a compliant elastic coating and a spherical probe is presented. The effect of friction between the contacting surfaces to the adhesion is explored. The empirical relations for the contact load and contact compliance as a function of contact radius are obtained from the numerical data for practical layer-substrate material systems. In Chapter 5, the contact behavior of a linear viscoelastic thin film is investigated by extending the empirical results of elastic thin film on a rigid substrate. The load controlled loading/unloading adhesion experiment has been modelled by applying the results of fracture mechanics to the motion of the edge of circular contact

Chapter 1. Introduction

region. In Chapter 6, the indentation of a hard thin film applied on elastic-plastic substrate is simulated. The effect of yield strain, strain-hardening exponent, film/substrate elastic mismatch parameter on the onset of plasticity are explored. Competing deformation regimes are presented in the form of an indentation map.

The report ends with conclusions and possible extensions for future works of this proposed project in chapter 7.

Chapter 2

Mechanics of Contacting Solids

2.1 Introduction

‘Contact test’, such as indentation, has been the most commonly used technique to measure the mechanical properties of materials because of the ease and speed with which it can be carried out [23–26]. In a typical contact test, a spherical tip indenter is brought into contact with a flat surface under a controlled load or controlled displacement, then unloaded until the surfaces separate. During this test process, the contact load, displacement or contact size are recorded automatically using appropriate instrumentation on the test system. From the load-displacement data, many mechanical properties such as hardness, elastic modulus and surface energy can be determined without imaging the indentations. During the past two decades, the scope of contact test has been extended down to the micro or nano meter range. In recent years, the study of mechanical properties of materials on the nanoscale has received considerable attention. These studies have been motivated partly by the development of nanocomposites and the application of micro or nanometer films for miniaturization of engineering and electronic components, and partly by newly available methods for probing mechanical properties in small volumes. In many situations, adhesion forces are extremely small compared with the force needed to deform the contacting materials, allowing its effects to be neglected. However, if the contacting materials are compliant and possess high surface energies, or the measurements are in the micro or nano scale, the adhesion force operating between the contact surfaces will contribute to the deformation and cannot be neglected.

In contact mechanics, most research works were devoted to mechanical analyses of indentation testing with the aim of gaining a thorough understanding of the relation between

constitutive properties and global indentation parameters. The methods developed and the results obtained have also been used in order to analyze other related contact problems, such as powder consolidation, flattening of surface asperities and inelastic impact.

Indentation problems can be analyzed by analytical modeling, numerical formulations and via experimental measurements. Advanced experimental tools have been designed and numerical solution techniques have been developed making it possible to determine contact parameters with high accuracy. Empirical and semi-empirical relations frequently used for material characterizations have been given a solid theoretical foundation and also improved [27].

This chapter provides a broad review of various aspects of the contact mechanics of elastic, plastic and viscoelastic solids with and without adhesion.

2.2 Adhesionless contact

2.2.1 Elastic solids

The stresses and deflections arising from the contact between two elastic solids have practical application in hardness testing, wear and impact damage of engineering ceramics, the design of dental prostheses, gear teeth, and ball and roller bearings. Most of these problems can be reduced to the contact between a rigid indenter and a flat half-space.

The contact problem of an frictionless elastic half-space indented by an elastic indenter, which includes both stresses and displacement boundary conditions, is sketched in Figure 2.1. The boundary conditions required for the solution of equilibrium equations of elasticity can be described as

$$u_z(r/a, 0) = \delta - f(r/a), \quad 0 \leq r/a \leq 1, \quad (2.1)$$

$$\sigma_z(r/a, 0) = 0, \quad r/a > 1, \quad (2.2)$$

$$\tau_{rz}(r/a, 0) = 0, \quad (2.3)$$

Figure 2.1: Indentation of an elastic half-space by a rigid axisymmetric frictionless punch.

where δ is the penetration depth of the punch, a is the contact radius, $f(r/a)$ is its shape function (with $f(0) = 0$), which must conform with the surface of the half-space inside the contact area. Furthermore, all stresses must approach zero at infinity. For a given shape of punch, the function $f(r/a)$ depends on a .

The first solution of such a problem for the contact of two elastic spheres is due to Hertz [5] in 1882. Hertz assumed a hemispherical pressure distribution in the contact area, and obtained a complete solution for the problem.

Hertz theory

When two non-conforming solids are brought into contact, they touch initially at a single point (axisymmetry) or along a line (plain-strain). Under the action of the slightest load, they deform in the vicinity of their point of first contact so that they touch over an area, which is finite small compared with the dimensions of the two bodies. The problem of a spherical punch pressing against an elastic half-space or of the contact of two elastic spheres was solved by Hertz [5], who gave the stress distribution in the area of contact, the penetration of the punch (or the approach of the two spheres) and the radius of contact as a function of the load.

As shown in Figure 2.2, two elastic spheres with radii R_1 and R_2 are brought into contact. If no pressure is applied between the two spheres, the contact occurs only at point O (in the absence of surface energy). If one sphere is pressed on the other with force P , the centers of the two spheres approach by a quantity δ , and the contact is achieved on a

Figure 2.2: Hertz contact of two elastic spheres.

circle of radius a . The Hertz contact theory makes use of the following assumptions:

1. The ratio a/R is small;
2. No tensile stress exists in area of contact;
3. The surfaces are frictionless.

The normal displacement within the contact area is given by

$$u_z(r) = \delta - \frac{r^2}{2R}, \quad r \leq a, \quad (2.4)$$

where $R = [1/R_1 + 1/R_2]^{-1}$, is the reduced radius of curvature. A distribution of pressure which gives rise to displacement satisfying equation (2.4) was proposed by Hertz as

$$p = p_0 \sqrt{1 - \left(\frac{r}{a}\right)^2}, \quad (2.5)$$

where p_0 is the maximum pressure and equal to 1.5 times the mean pressure given by $p_m = P/\pi a^2$. The pressure gives rise to normal displacement

$$u_z(r) = \frac{1 - \nu^2}{E} \frac{\pi p_0}{4a} (2a^2 - r^2), \quad r \leq a. \quad (2.6)$$

Using equations (2.4) – (2.6), other contact parameters can be expressed as

$$a = \left(\frac{3PR}{4E^*} \right)^{1/3}, \quad (2.7)$$

$$\delta = \frac{a^2}{R} = \left(\frac{9P^2}{16RE^{*2}} \right)^{1/3}, \quad (2.8)$$

$$p_0 = \frac{3P}{2\pi a^2} = \left(\frac{6PE^{*2}}{\pi^3 R^2} \right)^{1/3}, \quad (2.9)$$

where $E^* = [(1 - \nu_1^2)/E_1 + (1 - \nu_2^2)/E_2]^{-1}$, is the reduced (or combined) elastic modulus.

The stress distribution in the contact material is useful for the analysis of the material fracture and yielding process. Hertz calculated the stress distribution on the surface and along the axis of symmetry. The variations of principal stresses σ_r , σ_θ and σ_z on the horizontal surface is shown in Figure 2.3. The hoop stress σ_θ is always compressive. The radial stress σ_r is compressive in most of the area of contact, while it is tensile outside the area of contact and reaches a maximum value at the edge of the contact circle. For brittle materials, a mode I crack initiated on the edge of the circle then penetrate below the surface. This phenomenon is known as *Hertzian fracture*.

The principle stresses as a function of the normalized depth z/a along the z-axis are shown in Figure 2.4. The hoop stress σ_θ is equal to the radial stress σ_r at the axisymmetric

Figure 2.3: Stress distributions on the surface for Hertz contact.

Figure 2.4: Stress distributions on the z -axis for Hertz contact.

axis. All the principle stresses have the maximum values on the surface and decrease rapidly with depth. The stress σ_z has higher value among the three. The square root of second invariant, i.e. $\sqrt{J_2}$, has a maximum value at $a/z \approx 0.5$, which is referred as *Hertz point*. In an elastic-plastic material, the plastic deformation begins when $\sqrt{J_2}$ reaches the yield stress in simple shear or $1/\sqrt{3}$ of the yield stress in simple compression.

Circular flat punch indentation

The problem of the rigid and circular flat punch of radius a pressing on the surface of an elastic half-space under a load P was solved by Sneddon [28] with the shape function of the punch being given by

$$f(r/a) = 0. \quad (2.10)$$

The solution was found to be

$$\delta = \frac{1 - \nu^2}{2E} \frac{P}{a}, \quad (2.11)$$

$$\sigma_z(r) = -\frac{P}{2\pi a^2} \frac{1}{\sqrt{1 - (r/a)^2}}, \quad r < a, \quad (2.12)$$

$$u_z(r) = \frac{1 - \nu^2}{\pi E} \frac{P}{a} \sin^{-1} \frac{a}{r}, \quad r \geq a. \quad (2.13)$$

The profile of the elastic half-space near the edge of the contact is shown in Figure 2.5.

Figure 2.5: The displacement profile of an elastic half-space indented by an axisymmetric flat punch.

2.2.2 Plastic solids

When a solid is subjected to an indentation load, it can undergo elastic, plastic or creep deformation, depending on the magnitude of the applied load, material constitutive behavior and operating temperature. At a small value of the applied load, the solid deforms elastically. Under sustained and large loadings, however, most of them exhibit some degree of plasticity or viscoelasticity, which are two kinds of inelastic behaviors. As the load is increased further, the plastic zone grows until the entire region surrounding the indenter undergoes a plastic deformation, which is referred to as fully plastic indentation. Under viscoelastic conditions, a material has a time-dependent stress-strain relation, whereas in plasticity the material is considered time-independent in its stress-strain behavior.

During the normal indentation of an elastic-plastic material, when the yield point of the ductile material is exceeded, the onset of plastic deformation commences. Initially, the plastic region underneath the indenter is small and completely contained within the surrounding elastic material. Hence, the plastic strains are of the same order of magnitude as the surrounding elastic strains. The plastically displaced material is fully accommodated by elastic expansion of the surrounding solid. This is referred to as the ‘constrained deformation’ regime because the flowing or plastically deforming volume is fully constrained by the surrounding elastic medium. As the applied strain is increased, i.e. as the

load is increasing or as the indenter is made sharper, a greater pressure beneath the tip is required to produce the necessary expansion. Eventually, sufficient pressure is achieved where the plastic region reaches the free surface, allowing the displaced material to escape via ‘uncontained’ plastic flow along the sides of the indenter.

Onset of plastic yield

The onset of plastic yield, or constrained deformation, is assessed by applying an appropriate yield criterion [29]. The two most commonly applied criteria are the Tresca’s maximum shear stress criterion and the von Mises maximum shear strain-energy criterion. According to Tresca criterion, yielding occurs when the difference between the maximum and minimum principle stresses reaches the yield stress in simple compression (or tension) σ_0 , i.e.

$$\max(|\sigma_1 - \sigma_2|, |\sigma_2 - \sigma_3|, |\sigma_3 - \sigma_1|) = \sigma_0, \quad (2.14)$$

where σ_1 , σ_2 and σ_3 are the principal stresses in the state of complex stress. In the von Mises criterion, yielding occurs once the deformation energy equals the deformation energy at yield in simple compression or pure shear. Therefore, by the von Mises criterion, yielding occurs when the square root of the second invariant, $\sqrt{J_2}$, of the stress deviator tensor reaches $1/\sqrt{3}$ of the yield stress in simple compression, i.e.

$$\sqrt{J_2} = \left\{ \frac{1}{6} [(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_3 - \sigma_1)^2] \right\}^{1/2} = \frac{\sigma_0}{\sqrt{3}}. \quad (2.15)$$

Relating the stresses from the yield criteria to the mean contact pressure under the indenter, one obtains

$$p_m = C\sigma_0, \quad (2.16)$$

where C depends on the indenter geometry and the coefficient of friction at the contact interface. The onset of unconstrained plastic flow, i.e. the point when the plastic yield zone reaches the free surface, is expected to occur when the contact pressure reaches the

yield stress given by rigid-plasticity theory. Based on a number of numerical analyses and indentation measurements with rigid spheres in elastic-plastic half-spaces, Johnson [30] found that $C \approx 2.7$.

The models of plastic material

Plastic deformation during indentation occurs in the neighborhood of the indenter, with the bulk of the material remaining elastic. The elastic region imposes a constraint on the plastic flow. The analysis of such an elastic-plastic behavior is very complex. Simple material behavior is often assumed in order to simplify the problem mathematically. Common descriptions of rate independent plasticity are summarized in Figure 2.6. They are (a) rigid-perfectly plastic behavior; (b) rigid-strain hardening plastic behavior; (c) elastic-perfectly plastic behavior; (d) general elastic-plastic behavior [31]. Some indentation problem solutions for these materials are also summarized below.

Figure 2.6: Various plastic behaviors: (a) rigid-perfectly plastic, (b) rigid-strain hardening plastic, (c) elastic-perfectly plastic, (d) general elastic-plastic.

(a) *The rigid-perfectly plastic material*

When the plastic deformation is severe so that the plastic strains are large compared with elastic strains, the elastic deformation may be neglected. Provided the material does not strain-harden to a large extent, it may then be idealized as a rigid-perfectly plastic,

which flows plastically at a constant stress. The theory of plane deformation of such materials is well developed by Hill [29].

A loaded body of rigid-perfectly plastic material comprises regions in which plastic flow is taking place and regions in which, on account of the assumption of rigidity, there is no deformation. The state of stress within the regions of flow can be represented by *slip line field*. The slip lines are drawn parallel to the directions of principal shearing stress at every point in the field, i.e. at 45° to the directions of principal direct stress.

Using the *slip line field theory*, Prandtl obtained the indentation pressure for a flat punch indenting a semi-infinite rigid-perfectly plastic solid by modeling it as a plane-strain problem and the indentation pressure is given by

$$p_m = (2 + \pi)\sigma_0^s, \quad (2.17)$$

where σ_0^s is shear yield stress. Hill's solution for this problem also yields the same value of indentation pressure. Both the Prandtl and Hill slip-line constructions are as shown in the Figure 2.7 [32].

Figure 2.7: Slip-line-field construction: (a) Prandtl's flow pattern, (b) Hill's flow pattern.

(b) The rigid-strain hardening plastic material

Because of strain hardening, the flow stress of the material increases with increasing indentation depth, and the plastic strains vary over the deformed region so that the amount of strain hardening varies from point to point. Tabor [4] observed that there is an effective strain ϵ_e beneath the indenter such that a relationship exists between the indentation pressure p_m and an effective flow stress σ_e corresponding to the effective strain. Thus, for a strain hardening plastic solid, which is governed by uniaxial power law of the form

$\sigma = \sigma_0 \epsilon^n$, the indentation pressure is

$$p_m = C \sigma_e, \quad (2.18)$$

where $\sigma_e = \sigma_0 \epsilon_e^n$. In the above equation, σ and ϵ are the uniaxial stress and uniaxial plastic strain, respectively, n is strain hardening exponent and C is a constant with a value of about 3. The effective strain is a function of the indenter geometry and indentation size.

(c) The elastic-perfectly plastic material

The cavity expansion theory proposed by Johnson [33] is often used to describe the indentation of an elastic-perfectly solid and the basic concepts are illustrated in Figure 2.8. The material immediately below the indenter is assumed to behave as an outwardly expanding core, which exerts a uniform pressure p_m on the surrounding material. Encasing the core is a region of ideally plastic material. Beyond this plastic region lies the elastic domain. This theory is based on the fact that the subsurface displacements produced by a blunt indenter are approximately radial from the point of first contact, with roughly hemi-spherical contours of equal strain.

Figure 2.8: Cavity model of an elastic-perfectly plastic indentation by a cone.

The volume of the material displaced during an increment of penetration is accommodated by the strains in the elastic domain. The indentation pressure p_m is assumed to be equal to the pressure within the outwardly expanding hemi-spherical core of radius a ,

where a is the radius of indentation. Outside the plastic core, it is assumed that the stress and displacement fields have radial symmetry and are the same as for an infinite, incompressible elastic, perfectly plastic body which contains a void of radius a under an internal pressure p_m . Using appropriate continuity conditions for the stresses and displacements at the boundary of the core and at the outer elastic-plastic boundary, Johnson [33] obtained an expression for the indentation pressure p_m as

$$p_m = \frac{2}{3} \left[2 + \ln \left(\frac{C}{\epsilon_0} \right) \right] \sigma_0, \quad (2.19)$$

where C is a constant which depends on the geometry of the indenter, and σ_0 and ϵ_0 are the yield stress and strain, respectively.

(d) *The general elastic-plastic material*

The solutions for the indentation of an elastic strain hardening plastic solids may best be obtained by numerical methods. In particular, the finite element method permits the simulation of indentation tests using more realistic constitutive equations and can provide detailed information regarding the displacement, stress and strain fields in the indented material. Finite element method has been used to obtain detailed solutions to indentation of an elastic-plastic solid by a circular cylinder [34], a cone [35], and a sphere [36]. All these analyses confirm the inadequacy of the slip-line field description of the deformation field beneath the indenter. Bhattacharya and Nix [37] found that the concept of a radial deformation field assumed by the cavity expansion theory is invalid for a cone or a wedge indenter with small included angle, and for materials with a high values of yield strain.

Matthews [38] has shown that the indentation pressure p_m for a perfectly plastic solid may be applied with approximation to a strain-hardening solid by replacing the yield stress σ_0 by an effective flow stress σ_e measured in simple compression at an effective strain ϵ_e , where for a material whose strain hardening is described by a power law relation

$$\sigma_e = \sigma_0 \left(\frac{\epsilon_e}{\epsilon_0} \right)^n. \quad (2.20)$$

For a spherical indenter of radius R , effective strain ϵ_e is given by

$$\epsilon_e = 0.28 \left(1 + \frac{1}{n} \right)^{-n} \frac{a}{R}, \quad (2.21)$$

where a is the contact radius.

2.2.3 Viscoelastic solids

The mathematical theory of viscoelasticity, though old in origin [39–41], has rapidly become the center of widespread attention in recent years because of the advent of polymers. In general, most engineering materials, including metals, are known to creep under sustained constant loads, and to relax when subjected to a constant strain.

In contrast to the theory of elasticity, which deals with materials that have a capacity to store mechanical energy, the theory of viscoelasticity is associated with those materials capable of dissipating mechanical energy as well as storing it. If a load is suddenly applied – but not impulsively so as to excite a dynamic response – an elastic material will respond instantaneously and reach a final state of deformation. A Newtonian viscous fluid responds to a suddenly applied loading state of uniform shear stress by steady flow process. There are, however, materials for which a suddenly applied and maintained state of uniform stress induces an instantaneous deformation followed by a flow process, which may or may not be limited in magnitude as time elapses. This behavior is known as viscoelastic. The governing differential equations in viscoelasticity are linear if infinitesimal strains are considered, whereas they become nonlinear if large displacements are involved.

The stress-strain relations for a linear viscoelastic material can be expressed in various ways, but the most common is to make use of the *creep compliance function* which expresses the strain response to a step change in stress or, alternatively, the *relaxation function* which expresses the stress response to a step change in strain.

Contact mechanics of viscoelastic materials

The contact of linear viscoelastic spheres in the absence of adhesion has been studied in detail by Ting [39, 40] and Graham [41] et al. If the creep compliance function $\Phi(t)$ and relaxation function $\Psi(t)$ are linear and continuous, then the stress and strain may be written as

$$\sigma(t) = \int_0^t \Psi(t - \tau) \frac{\partial \epsilon(\tau)}{\partial \tau} d\tau, \quad (2.22)$$

$$\epsilon(t) = \int_0^t \Phi(t - \tau) \frac{\partial \sigma(\tau)}{\partial \tau} d\tau. \quad (2.23)$$

The relaxation function $\Psi(t)$ specifies the stress response to a unit step change of strain; the creep compliance function $\Phi(t)$ specifies the strain response to a unit step change in stress. For particular materials, $\Psi(t)$ and $\Phi(t)$ may be deduced mathematically from appropriate spring and dashpot models or they may be obtained through experiments.

When a rigid spherical indenter is pressed into contact with a viscoelastic solid under the action of a constant normal force, the penetration of the indenter and the contact area both grow with time with a change in the distribution of contact pressure. A simple approach to this problem for finding the stresses and deformations in cases where the corresponding solution for a purely elastic material is known was suggested by Radok [27, 42].

It consists of replacing the elastic constant in the elastic solution by the corresponding integral operator from the viscoelastic stress-strain relations. If the deformation history is known, the stresses are found by replacing E^* in the elastic solution by the integral operator, expressed in terms of the relaxation function $\Psi(t)$. On the other hand, if the load or stress history is known, the variation in deformation is found from the elastic solution by replacing the constant $1/E^*$ by the integral operator involving the creep compliance $\Phi(t)$. It is known that this approach can be applied to contact problem provided that the contact area increases throughout the loading program.

The viscoelastic correspondence principle applied to the Hertz elastic theory gives the

variation of contact size $a(t)$ with load $P(t)$ as

$$a^3(t) = \frac{3R}{4} \int_0^t \Phi(t - \tau) \frac{dP(\tau)}{d\tau} d\tau. \quad (2.24)$$

For a single step load

$$P(t) = \begin{cases} 0, & t < 0, \\ P_0, & t > 0, \end{cases} \quad (2.25)$$

equation (2.24) becomes

$$a^3(t) = \frac{3R}{4} R P_0 \Phi(t), \quad t > 0. \quad (2.26)$$

For simple viscoelastic material models, the solutions are summarized below.

Simple viscoelastic models

Two basic models of viscoelasticity are the Kelvin Model and the Maxwell Model [31]. The Kelvin model represents the material through a schematic diagram consisting of a spring and a dashpot connected in parallel as shown in Figure 2.9(a), whereas the Maxwell model uses the schematic sketch in Figure 2.9(b) showing a spring and a dashpot connected in series.

For the Kelvin model, since a spring and a dashpot are connected in parallel, the total stress σ is the sum of the stresses in the spring and dashpot. The creep and relaxation functions are respectively given by

$$\Phi(t) = \frac{1}{E} \{1 - \exp(-t/T)\}, \quad (2.27)$$

$$\Psi(t) = E\Delta(t) + \eta\delta(t), \quad (2.28)$$

where $T = \eta/E$ is the relaxation time and

$$\Delta(t) = \begin{cases} 0, & t < 0, \\ 1, & t > 0, \end{cases} \quad (2.29)$$

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Figure 2.9: Viscoelastic material constitutive models: (a) Kelvin model, (b) Maxwell model, (c) three-parameter model.

$$\delta(t) = \dot{\Delta}(t). \quad (2.30)$$

Substituting the creep function $\Phi(t)$, i.e. equation (2.27), into equation (2.26) gives the variation in contact radius as

$$a^3(t) = \frac{3RP_0}{4E} \{1 - \exp(-t/T)\}. \quad (2.31)$$

For the Maxwell model, since a spring and a dashpot are connected in series, the total strain is the sum of the strains in the spring and dashpot. The creep and relaxation functions are respectively given by

$$\Phi(t) = \frac{1}{E} + \frac{t}{\eta}, \quad (2.32)$$

$$\Psi(t) = E \exp(-t/T). \quad (2.33)$$

Substituting creep function $\Phi(t)$, i.e. equation (2.32), into equation (2.26) gives the variation in contact radius as

$$a^3(t) = \frac{3RP_0}{4} \left\{ \frac{1}{E} + \frac{t}{\eta} \right\}. \quad (2.34)$$

Except for the above two simple viscoelastic material models, another most commonly

used one is the three-parameter-model shown in Figure 2.9(c), which composes by a Kelvin element in series with a spring. The creep and relaxation functions are respectively given by

$$\Phi(t) = \frac{1}{E_1} + \frac{1}{E_2} \{1 - \exp(-t/T_1)\}, \quad (2.35)$$

$$\Psi(t) = \frac{E_1}{E_1 + E_2} \{E_2 + E_1 \exp(-t/T_2)\}, \quad (2.36)$$

where $T_1 = \eta/E_2$ and $T_2 = \eta/(E_1 + E_2)$.

Substituting the creep function $\Phi(t)$, i.e. equation (2.35), into equation (2.26) gives the variation in contact radius as

$$a^3(t) = \frac{3RP_0}{4} \left\{ \frac{1}{E_1} + \frac{1}{E_2} \exp(-t/T_1) \right\}. \quad (2.37)$$

2.2.4 Numerical methods

Many indentation problems do not permit analytical solutions in closed form and require solution of the problems by a numerical method. Common problems of this sort are those that involve friction, complex geometry and nonlinear elastic properties. The problem is essentially to determine the distributions of normal and tangential traction, which satisfy the normal and tangential boundary conditions at the interface, both inside and outside the contact area whose shape and size may not be known at the outset.

The classical method, which has been applied to axisymmetric problems in which the shape of the contact area is known, is to represent the pressure distribution by an infinite series of known functions. The series is then truncated to satisfy the boundary conditions approximately. Although a continuous distribution of traction is obtained, this method is basically ill-conditioned and can lead to large errors unless the functions are chosen carefully.

Modern computing facilities generally favor a different approach in which continuous distributions of tractions are replaced by a discrete set of ‘traction elements’ and

the boundary conditions are then satisfied at a discrete number of points (the ‘matching points’). Some of the commonly used numerical methods for contacting problems are briefly reviewed below.

Matrix inversion method

The basic principle involves dividing the surface into a number of discrete elements. Some form of pressure distribution is then assumed to act on each element. Figure 2.10 shows three possible unit pressure distributions for a two-dimensional problem.

Figure 2.10: Discrete pressure elements: (a) concentrated forces, (b) uniform pressure, (c) overlapping triangles pressure.

The normal displacement at point i due to the loading at point j is expressed from the appropriate elasticity equation for the chosen unit loading. Hills et al [43] provide a useful summary of these traction elements. The displacement u_i at the point i due to the summation of the loading at each of the points j is expressed by a matrix equation of the form

$$u_i = \sum_{j=1}^n C_{ij} p_j. \quad (2.38)$$

The influence coefficient matrix C_{ij} is obtained from the appropriate expressions for the deflection caused by the chosen elemental pressure distribution. In many cases the region of contact will not be known initially and an iterative procedure is adopted. A region of contact is guessed and a set of displacements are determined from the interpenetration of the two bodies. The numerical matrix inversion procedure gives the corresponding pressure p . The total load is then found from the sum of the elemental pressures, i.e.

$$P = C \sum_{j=1}^n p_j, \quad (2.39)$$

where C is a constant depending upon the form and size of the pressure element. This method has been used extensively for the contact of rough surfaces [44–46]. Once the surface stress distribution is known, it is straightforward to determine the subsurface stress field.

Variational method

In some cases both shear and normal traction result in surface displacements, the matrix inversion method is very intensive in computational time. An alternative approach, which is more useful in these cases, is the variational method. The variational principle states that the true stress field within a solid, loaded at its boundary, exists when the complementary potential energy is at a minimum value. The complementary potential energy V^* can be expressed in terms of the stored strain energy and the force and displacement prescribed at the surface as

$$V^* = \frac{1}{2} \int_{i=1}^n p(u_{z1} + u_{z2})dS + \int_S p(h_i - \delta)dS, \quad (2.40)$$

where δ is the overlap of the two surfaces and h_i is their initial separation. S is the surface over which the pressure p acts, causing normal displacement u_z . The solution proceeds by determining the values of p which minimize V^* subject to $p > 0$. The minimization has been done using a simplex method [47] or by using a direct quadratic programming technique [48].

Finite element method

If the indentation problem involves nonlinear material behavior such as plasticity or the half-space assumption is no longer valid (e.g. when the dimensions of the body are not large compared with the region of contact or thin film is involved in the contact process), then it may be necessary to employ a Finite Element Method (FEM) for the analyses. A number of commercial finite element codes are currently available which are suitable for the solution of contact problems (e.g. ABAQUS, ANSYS, MARC, NASTRAN,

DYNA3D, RADIOSS).

The contacting bodies are divided into an array of either two-dimensional axisymmetric or planar elements or three-dimensional brick elements. A set of gap or contact elements are defined between the surface nodes which are likely to come into contact during loading. A friction coefficient may be defined for the contact. Appropriate materials constitutive models, boundary conditions, and nodal loadings are also specified.

Modern programs have graphical interfaces to assist in the generation of meshes. The ‘preprocessing’ program generates an input file to use with the main analysis program. Also available are ‘postprocessing’ packages, which generate graphical presentations of results such as stress or strain contours and deformed mesh shapes.

Typically, indentation problems involve distributed loads over a small area of contact. This results in rapid changes in stress in the elements surrounding the contact. Therefore, the mesh in this region must be refined adequately to describe these high stress gradients. Thus even a simple Hertz contact analysis need a fine mesh for accurate results. The nonlinearities associated with contact, stick-slip behaviors, and plasticity imply that the solution is loading-path dependent. This requires the applied load to be divided into a number of small steps and a solution is to be found at each iteration. These features can often lead to extensive computations.

The availability and ease of use of commercial FEM codes has led to their widespread adoption in both industrial and academic environments. Many fundamental tribological phenomena have been investigated by this method (e.g. elastic plastic indentation) [49–51].

As an alternative to the FEM, the Boundary Element Method (BEM) has been applied to contact problem by Andersson et al [52]. One of the main advantages is its unique feature of mesh reduction. As is well known, a common requirement of the FEM is the generation of a mesh in the problem domain, which is often the most costly and time consuming part of the solution process and is far from fully automated, particularly in the three-dimensional case. As a result, the BEM has become a method of choice for solving

problems with complicated domains, since only boundary discretization is usually needed rather than domain discretization. Significant mesh reduction is achieved when boundary discretization is used.

2.2.5 Experimental methods

As the indenter is pressed into the sample, both elastic and plastic deformation occurs. This results in the deformation of a hardness impression conforming to the shape of the indenter. During the withdrawal of the indenter, only the elastic portion of the displacement is recovered. This facilitates the use of an elastic solution in modeling the indentation process. Figure 2.11 shows a typical load-displacement curve [53]. δ is the contact depth and is defined as the depth of the indenter in contact with the sample under load. $\delta_{\max}$ represents the displacement at the peak load $P_{\max}$. δ_f is the final displacement after complete unloading. S is the initial unloading contact stiffness.

Figure 2.11: A typical load-displacement curve during the indentation test.

The hardness H is defined as the indentation load $P_{\max}$ divided by the projected contact area A of the indentation

$$H = \frac{P_{\max}}{A}. \quad (2.41)$$

The elastic modulus of the indented sample can be inferred from the initial unloading contact stiffness $S = dP/d\delta$. Based on relationships developed by Sneddon [28] for

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the indentation of an elastic half space by any punch that can be described as a solid of revolution of a smooth function, a geometry-independent relation involving contact stiffness, contact area, and elastic modulus can be derived as

$$S = 2C \sqrt{\frac{A}{\pi}} E^*, \quad (2.42)$$

where C is a constant that depends on the geometry of the indenter and E^* is the reduced elastic modulus, which accounts for the fact that elastic deformation occurs in both the sample and the indenter. The parameter E^* is given by

$$\frac{1}{E^*} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i}, \quad (2.43)$$

where E and ν are the elastic modulus and Poisson's ratio for the sample; E_i and ν_i are the same quantities for the indenter. To calculate the elastic modulus, the contact stiffness and the projected contact area need to be determined from the load-displacement curve.

In an experimental analysis of indentation problems, it is likely that the measurement of contact area is of practical interest. There are several experimental techniques for determining this parameter. Measurements of the electrical resistance between contacting surfaces can provide some information on the true area of contact. However, it is not possible to determine the real area of contact directly unless some assumptions are made on either the size or number of individual contact spots. If one of the contacting bodies is transparent to light, then the regions of contact can be observed directly. On the other hand, if both bodies are transparent, then a light beam may be directed through the interface. The beam will pass through regions of contact without deflection but will be scattered at air gaps. Contact areas then shown up as bright spots against a grey background [54]. A wave of ultrasound incident at an interface between two materials will be transmitted through region of contact and be reflected back at air gaps. This phenomenon can be used to investigate the true area of contact at an interface [55].

2.2.6 Indentation on layered solids

Thin films or coatings are widely used in technologies, such as microelectronic devices or wear and erosion protection. Examples include zirconia coating as thermal barriers on superalloys in engines [56], nitrogenated carbon layer on magnetic recording disks [57], and indium-tin oxide coatings on polyethylene terephthalate (PET) in optoelectronic and display devices [58].

The contact of solids, which have surface layers whose elastic properties differ from the substrate, is schematically illustrated in Figure 2.12 in which the indenter (E_2, ν_2) is in contact with the surface layer (E_1, ν_1) on substrate (E_3, ν_3). If the thickness h of the surface layer is large compared with the contact size $2a$ (i.e. $h \gg 2a$), then the substrate has little influence and the contact stresses are given by the Hertz theory.

Figure 2.12: Indentation on layered solid.

Stress and deformation analysis for the case of a rigid cylindrical indenter making contact with a half-space carrying an elastic surface layer with differing mechanical properties from the bulk have been carried out by a number of authors [59, 60]. The usual method of solution is to first calculate the local surface deformations from the degree of nominal overlap of the two surfaces and then to solve, either analytically or numerically, the stress-deformation integral equation, arriving at a consistent distribution of normal pressure at the interface. This can then be integrated to give the normal load. Gupta and Walowit [61], Kannel and Dow [62] obtained generalized solutions for elastic cylindrical indenters against layered elastic solids. Walowit and Pinkus [63], King and

O'Sullivan [64] analyzed the stress field associated with an elastic cylindrical indenter sliding with traction over an elastic half-space with single surface layer and this analysis was extended to multiple layers by Elsharkaway and Hamrock [65]. O'Sullivan and King [66] examined the case of a spherical indenter loaded sliding over a single layered surface. Results from the analysis of this sort can be validated and extended by finite element analysis [67–70].

Relatively little work has been done on the analysis of the elastic-plastic contact of layered surfaces despite the fact that plastic deformation is a major concern in the design and performance of engineering components. It is valuable to have a picture of the spatial distribution of the position of the initiation and subsequent development of the plastic zone. Based on an FEM analysis, it was found that coating thickness has a significant effect on the initiation and development of the plastic zone. Deformation maps, which define the deformation mechanism in terms of such parameters as maximum pressure and coating thickness, can be constructed [71–73].

In most coating-substrate systems, plastic deformation is initiated first in the substrate at the coating-substrate interface. Plastic deformation is not initiated in the harder coating until a large plastic zone has been developed in the substrate. Increasing the coating thickness and/or substrate strength increases the resistance of the composite to plastic deformation. When the substrate is relatively soft and the coating is relatively thin, plastic deformation is concentrated in the substrate. With increasing coating thickness and substrate strength, plastic deformation in the coating becomes more evident. This indicates the importance of depositing a sufficiently thick coating in order to optimize the tribological performance of coated system [74].

Failure of a hard coating-soft substrate system under tribological situations may be caused not by conventional wear but rather by debonding of the coating from the substrate, fracture of the coating, or even subsurface fracture. Accordingly, for their applications, the knowledge of their mechanical stability remains a critical aspect to analyze. For instance, residual stresses are induced because the deposition takes place at high temper-

ature and there is a mismatch in the thermal expansion coefficients or intrinsic stresses, induced in sputter deposited thin films due to lattice mismatch. When stresses reach high levels, they can induce spontaneous bucking or cracking of the layers. Therefore, to improve the properties of layered systems for intended mechanical applications, it seems obvious to investigate the coating failure mechanisms, when an external stress is applied to the system, and also to estimate relevant parameters for its mechanical stability.

2.3 Adhesion contact

It became evident that, for soft elastic solids such as rubber, the contact area could be larger than that predicted by Hertz theory of elastic contact, a contact area of finite size was seen at zero load and a tensile force was required to pull the surfaces apart. This is ascribed to the result of the competing forces of attraction and repulsion between individual atoms or molecules in both contact bodies [27].

Two ideally flat solid surfaces will have an equilibrium separation z_0 . At a separation less than z_0 they will repel each other and at a separation greater than z_0 they will attract. The variation of force per unit area as a function of separation is usually represented by a law of the form

$$p(z) = -\frac{A}{z^m} + \frac{B}{z^n} \quad m < n \quad (2.44)$$

as shown in Figure 2.13. In these circumstances it is clear that a tensile force - *the force*

Figure 2.13: Force separation curve and surface energy for ideal surface.

of adhesion - has to be exerted to separate the surfaces. The magnitude of the maximum tensile traction is large, but the effective range of action is very small. In view of the difficulty in measuring surface force directly, it is usual to measure the work 2γ required to separate the surfaces from $z = z_0$ to $z = \infty$ and to ascribe a surface energy γ to each newly created free surface. If the solids are dissimilar, the work to separate the surfaces become

$$w = \gamma_1 + \gamma_2 - \gamma_{12}, \quad (2.45)$$

where γ_1 and γ_2 are the intrinsic surface energies of the two solids and γ_{12} is the energy of the interface.

In order to model adhesion contact problems, different theories, such as Johnson-Kendall-Roberts (JKR) theory [7] and Derjaguin-Muller-Toporov (DMT) theory [8], are available in the literature [75]. In this section, the contact mechanics in the presence of adhesion is discussed, including their application, scope, and limitations.

2.3.1 The JKR theory

The Hertzian contact of a sphere with a plane is called a unilateral problem. For this case only compressive stresses exist in the contact area. In the presence of surface energy, the contact area under a given load is always greater than that predicted by the Hertz theory. The JKR model is schematically shown in Figure 2.14. A rigid frictionless sphere with radius R is brought into contact with the elastic homogeneous half-space, whose Young's modulus and Poisson's ratio are E and ν , respectively. The contact radius under the action of a compressive load P is a and the corresponding central displacement is δ .

In JKR theory [7, 22], the normal load P is made up of two terms: (i) an adhesionless load P_0 associated with the Hertz pressure

$$p_0(r) = \frac{3P_0}{2\pi a^2} \left[1 - \left(\frac{r}{a} \right)^2 \right]^{1/2}, \quad (2.46)$$

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Figure 2.14: The JKR theory: (a) contact pressure, (b) relation between load and displacement, (c) variation of contact radius with load.

and (ii) an adhesive load P_a associated with the flat punch indentation pressure

$$p_a(r) = -\frac{P_a}{2\pi a^2} \left[1 - \left(\frac{r}{a} \right)^2 \right]^{-1/2}. \quad (2.47)$$

The superposition of these two tractions is shown in Figure 2.14(a). A plot of the load against the displacement δ is shown in Figure 2.14(b). The application of the Hertzian load P_0 in the absence of adhesion follows the compliance curve from O to A . The adhesive (negative) force P_a is then applied, keeping the contact size a constant. This results

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in the linear compliance relation AC having the gradient $(dP_0/d\delta_0)_A$. From the aspect of thermodynamics, the Helmholtz free energy U_T of the system may be expressed by

$$U_T = U_E + U_S, \quad (2.48)$$

where U_E is the elastic strain energy in the system at point C in Figure 2.14(b), and U_S is the surface energy associated with a contact of radius a , i.e. $U_S = -\pi a^2 w$. The equilibrium value of a is then given by

$$\frac{dU_T}{da} = \left(\frac{\partial U_E}{\partial a} \right)_\delta + \frac{dU_S}{da} = \left(\frac{\partial U_E}{\partial a} \right)_\delta - 2\pi a w = 0, \quad (2.49)$$

where the term $(\partial U_E / \partial a)_\delta$ is the rate of change of elastic energy with a , keeping the displacement δ constant and is shown by the shaded area $CABC'$ in Figure 2.14(b). In the limit, the above term can be expressed as

$$\begin{aligned} \left(\frac{\partial U_E}{\partial a} \right)_\delta &= \frac{1}{2} (\delta_0 - \delta)^2 \frac{d}{da} \left(\frac{dP_0}{d\delta_0} \right) \\ &= \frac{1}{2} \left[\frac{P_a}{dP_0/d\delta_0} \right]^2 \frac{d}{da} \left(\frac{dP_0}{d\delta_0} \right). \end{aligned} \quad (2.50)$$

Substituting equation (2.50) in equation (2.49) gives

$$\frac{1}{4\pi a} \left[\frac{P_a}{dP_0/d\delta_0} \right]^2 \frac{d}{da} \left(\frac{dP_0}{d\delta_0} \right) = w. \quad (2.51)$$

When the solids are homogeneous and isotropic, the Hertz relationships apply, so that the compliance curve in Figure 2.14(b) is given by

$$P_0 = \frac{4E'a^3}{3R} = \frac{4}{3} E' R^{1/2} \delta_0^{3/2}, \quad (2.52)$$

where $E' = E/(1 - \nu^2)$ is plane-strain Young's modulus. Whereby

$$\frac{dP_0}{d\delta_0} = 2aE'. \quad (2.53)$$

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Substituting equation (2.53) in equation (2.51) gives

$$P_a = \sqrt{8\pi w E' a^3}. \quad (2.54)$$

So that the net contact load is P given by

$$P = P_0 - P_a = \frac{4E' a^3}{3R} - \sqrt{8\pi w E' a^3}. \quad (2.55)$$

This is the JKR relationship between applied load P and contact radius a . In the same way, the net displacement δ can be expressed as

$$\delta = \delta_0 - \delta_a = \frac{a^2}{R} - \sqrt{\frac{2\pi w a}{E'}}. \quad (2.56)$$

The pull-off force P_c , which is given by the maximum tensile (negative) value of P , is given by

$$P_c = \frac{3}{2}\pi w R. \quad (2.57)$$

Using the non-dimensional variables

$$\bar{P} = \frac{P}{3\pi w R}, \quad (2.58)$$

$$\bar{a} = a \left(\frac{4E'}{9\pi w R^2} \right)^{1/3}, \quad (2.59)$$

$$\bar{\delta} = \delta R \left(\frac{4E'}{9\pi w R^2} \right)^{2/3}, \quad (2.60)$$

the JKR relationship between applied load P and contact radius a reduces to

$$\bar{P} = \bar{a}^3 - \sqrt{2\bar{a}^3}. \quad (2.61)$$

The pull-off force is given in non-dimensional form by $\bar{P}_c = -0.5$. The contact radius at zero load is given by $\bar{a}_0 = 2^{1/3}$ and at pull-off by $\bar{a}_c = 2^{-1/3}$. The net elastic displacement

of the contact δ in the non-dimensional form is given by

$$\bar{\delta} = \bar{\delta}_0 - \bar{\delta}_a = \bar{a}^2 - \frac{2\sqrt{2}\bar{a}}{3}. \quad (2.62)$$

An alternative approach to the JKR analysis is through the concepts of linear elastic fracture mechanics due to Maugis [10]. The singularity at $r = a$ in the adhesive traction of equation (2.47) can be expressed by a model I stress intensity factor K_I as

$$K_I = \frac{P_a}{2a\sqrt{\pi a}}. \quad (2.63)$$

For an equilibrium ‘crack’ the strain energy release rate G is given by

$$G = \frac{K_I^2}{2E'} = w. \quad (2.64)$$

Eliminating K_I from equations (2.63) and (2.64) directly yields

$$P_a = \sqrt{8\pi w E' a^3}. \quad (2.65)$$

In a typical experiment the curved surfaces are brought into close together, whereupon they snap into contact at $a = a_0$. Increasing the compressive load P and then reducing it, the contact radius a and the displacement δ follow equations (2.61) and (2.62) reversibly, until the surfaces snap apart at the pull-off force P_c . Simultaneous measurements of P and a or δ can then be fitted to equations (2.61) and (2.62) to deduce E' and w .

2.3.2 The DMT theory

The adherence force of the JKR theory is independent of Young’s modulus. In 1975 Derjaguin, Muller and Toporov [8] gave their theory (DMT theory) in which adhesion forces act in an annular zone around the contact but without any change in the deformation contact profile. The main results of their theory were that the adherence force $2\pi w R$ is reached when the area of contact vanishes (no instability), that the radius of contact under

zero load is $a_0 = (3\pi wR^2/4E')^{1/3}$, and that the adhesion forces around the contact add to the applied load a force which decreases from $2\pi wR$ to πwR when the penetration increases.

Note that if the adhesion force equal to $2\pi wR$, instead of varying with penetration, the results would be

$$P = \frac{4a^3 E'}{3R} - 2\pi wR, \quad (2.66)$$

$$\delta = \frac{a^2}{R}. \quad (2.67)$$

2.3.3 JKR-DMT transition

At first thought, DMT theory and JKR are incompatible. However, comparing the two theories, Tabor [9] pointed out that the main defect of the DMT theory was to neglect the deformations due to adhesion forces around the contact, whereas that of the JKR theory was to neglect the adhesion forces outside the contact area. Tabor noted that the behavior should vary continuously from one limit to the other, depending on the elasticity parameter μ defined by

$$\mu = \left(\frac{Rw^2}{E'^2 z_0^3} \right)^{1/3}, \quad (2.68)$$

where z_0 is the equilibrium spacing between the contacting surfaces ($\approx 0.3nm$). The JKR model gives a good approximation, provided that the elastic deformation is large compared with the range of the surfaces forces, for which the elasticity parameter $\mu \gg 1$ (soft solids, large radius of curvature, large energy of adhesion). The DMT theory applies for $\mu \ll 1$ (hard solids, small radius of curvature, weak energy of adhesion).

By applying a Dugdale model of crack closure, Maugis [10] obtained the JKR-DMT transition (M-D theory). Also this JKR-DMT intermediate regime was explored in detail by Greenwood [11]. The adhesion forces are assumed constant and equal to σ_0 on an annulus of width $d = c - a$ around the contact until a separation h_0 is reached, whereupon it falls to zero (see Figure 2.15). This adhesive force σ_0 is selected to coincide with the minimum value of the assumed realistic interaction force (see Figure 2.16). The height h_0

Figure 2.15: The Maugis-Dugdale distribution of surface traction.

Figure 2.16: Force-separation relationship: Lennard-Jone and Dugdale approximation.

follows then using the condition $w = \sigma_0 h_0$. For a Lennard-Jones type potential integrated for a sphere-flat contact geometry, it can be shown that $h_0 = 0.971z_0$ [76].

The Maugis-Dugdale analysis is expressed in terms of a non-dimensional parameter λ defined by

$$\lambda = \sigma_0 \left(\frac{9R}{2\pi w E'^2} \right)^{1/3}. \quad (2.69)$$

It may be shown that $\lambda = 1.16\mu$, so that λ is an alternative and similar measure of the ratio of elastic deformation to the range of surface force.

Intimate contact is maintained over a central region of radius a ; adhesive forces of intensity σ_0 extend to a radius c . In the annulus $a < r < c$ the surfaces separate slightly from zero at $r = a$ to h_0 at $r = c$. This determines the magnitude of the adhesive force P_a at any given load P . The distribution of surface traction, as shown in Figure 2.15, contains

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two terms: (i) the Hertz pressure associated with a contact of radius a , as given by

$$p_0(r) = \frac{3P_0}{2\pi a^2} \left[1 - \left(\frac{r}{a} \right)^2 \right]^{1/2}, \quad (2.70)$$

and (ii) the adhesive (Dugdale) traction

$$p_a(r) = \begin{cases} -\frac{\sigma_0}{\pi} \cos^{-1} \left(\frac{2a^2 - c^2 - r^2}{c^2 - r^2} \right), & r \leq a, \\ -\sigma_0, & a \leq r \leq c. \end{cases} \quad (2.71)$$

The stress-intensity factor K_I , due to external loading alone, is that of the JKR theory, i.e.

$$K_I = \frac{1}{2a\sqrt{\pi a}} \left(\frac{4a^3 E'}{3R} - P \right). \quad (2.72)$$

The stress-intensity factor K_m , which corresponds to constant stresses acting on an exterior circular ring, is given by

$$K_m = -\frac{\sigma_0 a}{\sqrt{\pi a}} \left(\sqrt{m^2 - 1} + m^2 \tan^{-1} \sqrt{m^2 - 1} \right), \quad (2.73)$$

where $m = c/a$. It is to be noted that the singular stress disappear if $K_I = -K_m$. The adhesive traction for the Dugdale model falls to zero when the separation of the surfaces exceeds h_0 . Therefore, the equilibrium equation may be written as

$$\begin{aligned} \frac{3^{2/3} \lambda \bar{a}^2}{2} \left[\sqrt{m^2 - 1} + (m^2 - 2) \tan^{-1} \sqrt{m^2 - 1} \right] \\ + \frac{4\lambda^2 \bar{a}}{3^{2/3}} \left[\sqrt{m^2 - 1} \tan^{-1} \sqrt{m^2 - 1} - m + 1 \right] = 1. \end{aligned} \quad (2.74)$$

By using the non-dimensional variables $\bar{P}$, $\bar{\delta}$ and $\bar{a}$ defined previously, the net contact force becomes

$$\bar{P} = \bar{a}^3 - 3^{-1/3} \bar{a} \lambda \left(\sqrt{m^2 - 1} + m^2 \tan^{-1} \sqrt{m^2 - 1} \right) \quad (2.75)$$

and the total elastic compression becomes

$$\bar{\delta} = \bar{a}^2 - \frac{4}{3^{4/3}} \bar{a} \lambda \sqrt{m^2 - 1}. \quad (2.76)$$

For a given parameter λ and a given radius of contact a , m can be extracted from equation (2.74). It can then be substituted into equations (2.75) and (2.76) to obtain the equilibrium curves $\bar{a}(\bar{P})$, $\bar{a}(\bar{\delta})$ and $\bar{P}(\bar{\delta})$ as shown in Figures 2.17, 2.18 and 2.19 respectively.

Figure 2.17: Relation between contact radius $\bar{a}$ and applied load $\bar{P}$ for various λ .

Figure 2.18: Relation between contact radius $\bar{a}$ and central displacement $\bar{\delta}$ for various λ .

Figure 2.19: Relation between applied load $\bar{P}$ and central displacement $\bar{\delta}$ for various λ .

For a comparison of their applicability, Hertz theory, DMT theory and JKR theory are also expressed in non-dimensional forms and listed in Table 2.1.

Table 2.1: Non-dimensional load and displacement for different contact theories.

	Hertz	DMT	JKR	M-D
$\bar{P}$	$\bar{a}^3$	$\bar{a}^3 - \frac{2}{3}$	$\bar{a}^3 - \sqrt{2}\bar{a}^3$	$\bar{a}^3 - 3^{-1/3}\bar{a}\lambda \left(\sqrt{m^2 - 1} + m^2 \tan^{-1} \sqrt{m^2 - 1} \right)$
$\bar{\delta}$	$\bar{a}^2$	$\bar{a}^2$	$\bar{a}^2 - \frac{2\sqrt{2}\bar{a}}{3}$	$\bar{a}^2 - \frac{4}{3^{4/3}}\bar{a}\lambda \sqrt{m^2 - 1}$

2.3.4 Adhesion map for the elastic contact

Using the M-D theory, Johnson and Greenwood [77] have constructed an ‘adhesion map’ (see Figure 2.20) with co-ordinates elasticity parameter μ (or λ) and non-dimensional load $\bar{P}$ ($= P/\pi wR$). The parameter μ represents the ratio of the elastic displacement of the surfaces at the point of separation (pull-off) to the effective range of surface forces characterized by z_0 . The non-dimensional force $\bar{P}$ is a measure of the ratio of the load to the adhesive pull-off force. The boundaries for the different deformation regimes may be chosen as described below.

In the Hertz Zone, the adhesive force P_a may be neglected compared with the net

Figure 2.20: Adhesion map for elastic spheres [77].

compressive force P , i.e. $|P_a/P| \ll 1$. In the JKR Zone, the elastic deformation of the surfaces caused by the adhesive forces is large compared with their effective range of action h_0 , i.e. $|\delta_a/h_0| \gg 1$. In the DMT Zone, the elastic deformation due to the surface forces is small compared with their range, i.e. $|\delta_a/h_0| \ll 1$. In the Rigid Zone, for stiff materials at sufficiently light loads, elastic deformation becomes small compared with the range of surface forces, whereupon the solids can be assumed to be rigid. For this to be so, not only $|\delta_a/h_0| \ll 1$ is required, but also $|\delta_0/h_0| \ll 1$.

2.3.5 Adhesion contact of inelastic solids

In many practical situations the high intensity of adhesive forces gives rise to inelastic (plastic or viscoelastic) deformation of the solids, notably in the separation zone at the edge of contact. This influences the apparent work of adhesion and gives rise to a difference between loading and unloading of the contact, often referred as adhesion hysteresis. Johnson [76] has provided methodology for analyzing adhesion hysteresis.

The elastic analysis is based on the fracture mechanics principle that, for a change in radius of the contact at constant displacement, the elastic energy release rate G is equal to the work done against surface forces w , i.e. $G = w$.

Assuming that a fraction q of the elastic energy is dissipated, during separation (contact area decreasing), the energy released has to overcome the dissipation as well as the

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work of adhesion, i.e.

$$G' = w + qG'. \quad (2.77)$$

The apparent work of adhesion becomes

$$w' = G' = \frac{w}{1 - q} = k'w. \quad (2.78)$$

During approach (contact area increasing), G and w change sign, but the dissipation does not, i.e.

$$-G'' = -w + qG'. \quad (2.79)$$

The apparent work of adhesion becomes

$$w'' = G'' = \frac{w}{1 + q} = k''w. \quad (2.80)$$

It is obvious that more work is required to separate the surfaces than to bring them come together. This gives rise to the hysteresis loop. Figure 2.21 shows the relationship between normalized load $\bar{P}$ and normalized contact area $\bar{A} = \pi \bar{a}^2$ with a dissipation factor $q = 0.5$, $k' = 2$ and $k'' = 2/3$. The surfaces jump into contact at point A and, during compression, follow curve III to point B. Reducing the load to separate the surfaces cause

Figure 2.21: Adhesion hysteresis due to inelastic deformation.

the effective work of adhesion to be augmented. Thus, the contact area remains unchanged until curve IV is reached at point C, after which curve IV is followed to the enhanced pull-off point D. For a comparison, the JKR and Hertz relations are added into this figure as curves I and II.

Plastic Solids

When the elastic limit of one of the bodies is exceeded, the analysis of the preceding sections no longer applies. No satisfactory analysis exists for a truly elastic-plastic contact, but if the plastic deformation is severe, then the stresses given by the theory of rigid-perfectly plastic solids are found to provide a good approximation to the problem [30]. The mean contact pressure is given by

$$p_m = \frac{P}{\pi a^2} = 2.8\sigma_0 \cong H \quad (2.81)$$

where σ_0 is the yield stress in uniaxial compression test and H is the hardness.

During loading, there is a sharp discontinuity in slope at the edge of the contact area so that the effect of adhesion forces in augmenting the size of the contact will be negligible. During unloading, however, the situation is different. The release of elastic stresses leads to the contact surface having a slight crown, whose exact profile depends upon the pressure distribution at the end of loading. If the true pressure distribution is approximated by an elliptical distribution (Hertzian) having the same mean pressure, then the recovered profile will be a circular arc of radius R' given by

$$R' = \frac{4E^*a}{3\pi H} = \frac{4E^*P^{1/2}}{3(\pi H)^{3/2}}. \quad (2.82)$$

During the elastic unloading of clean smooth surfaces adhesive forces come into play. The plastically deformed sphere acts like an elastic sphere of radius R' . Thus the force required to separate the surfaces is given by $P_c = 3\pi wR'/2$ (using JKR approximation),

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i.e.

$$P_c = \frac{2wE^*P^{1/2}}{\pi^{1/2}H^{3/2}}. \quad (2.83)$$

Soft materials are usually very ductile. Thus, it is possible that an adhesive contact, established during loading, is capable of appreciable plastic extension before fracture. An adhesive contact between plastically deformed spheres is equivalent to a deep notch in large diameter bar or a ‘spot weld’ between two half-spaces. The mean tensile stress necessary to produce a ductile extension of the notch is given by the fully plastic indentation pressure in reverse, i.e.

$$p_m = -2.8\sigma_0 \cong -H. \quad (2.84)$$

Thus, ductile extension would be expected, if the hardness H is less than the critical stress p_{cm} for ‘brittle’ failure of the adhesive joint. Since

$$p_{cm} = -\frac{P_c}{\pi a_c^2} = -\left(\frac{4E^*H}{\pi a}\right)^{1/3}, \quad (2.85)$$

it follows that, for ductile extension, one obtains

$$H^2 < \frac{2wE^*}{\pi a}, \quad (2.86)$$

where a is the size of the contact established during fully plastic loading.

Recently, adhesive contact of elastic-plastic spheres was analyzed numerically [78, 79]. The results of Mesarovic and Johnson [78] are summarized as follows.

Elastic-plastic loading is modeled by a fine grid finite element analysis revealing that the contact pressure at the end of loading is approximately uniform. During unloading to the point of pull-off, the deformation is perfectly elastic. In the absence of adhesion, the contact size and pressure distribution during elastic unloading from a uniform pressure is found by the method of rigid punch decomposition. The pressure distribution approaches the Hertzian one as the contact size approaches zero. The distribution of adhesive traction is obtained in two ways. One is by using the singular distribution of linear elastic frac-

ture mechanics. The other is by using cohesive law, whereby a constant adhesive stress σ_0 acts between surfaces separated by less than h_0 . The non-dimensional pull-off force $\bar{P}_c$ has been found by the cohesive zone model to be a function of two non-dimensional parameters: χ ($\approx 1.38wE^*/p_0^2a_0$) and S ($= \sigma_0/p_0$). A decohesion map (Figure 2.22) is constructed whereby the parameter space (χ, S) is divided into the regions where decohesion is governed by competing physical processes.

Figure 2.22: Map of decohesion in space.

Viscoelastic solids

Theoretical models for contacts to viscoelastic materials in the absence of adhesion have been available since 1960s. In the early 1970s, Barquins and Maugis [80], Greenwood and Johnson [81] developed models that included adhesion, but viscoelastic effects were confined to the periphery of the contact. Schapery [82] and Hui et al [83, 84] have further put forward a model that accounts for linear viscoelastic response at all length and time scales. Later on, Johnson [85, 86] extended this theory to the case of an entire load-displacement cycle. In the late 1990s, several experiments demonstrated that viscoelastic effects are not confined to the contact periphery but include the entire contact zone. Johnson [85] and Unertl [87] have shown that crack tip and long-range creep phenomena generally occur on much different time scales depending on the characteristic relaxation time of the

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viscoelastic material and experimental measurement time, both of which are important. Recently Greenwood [88] has carried out an exact analysis of the opening and closing contacts of viscoelastic spheres. In this section, the Johnson's theory for bulk viscoelastic solid is described. Giri et al [13] have demonstrated that this theory provides a robust method to determine the thermodynamic work of adhesion from the load-displacement data obtained at sufficiently low loading rates.

In the elastic JKR theory [7], the pressure acting on the contact consists of two parts. One is the Hertz pressure due to the compressive load and is distributed smoothly over the entire contact. The other is due to the adhesive interaction, which is the most important at the contact periphery. For viscoelastic materials, surface energy w is replaced by its scaled value βw , so that JKR equation in non-dimensional form becomes

$$\bar{P} = \bar{a}^3 - \sqrt{2\beta\bar{a}^3}, \quad (2.87)$$

where $\bar{P} = P/3\pi wR$, $\bar{a} = a(4E_\infty^*/9\pi R^2 w)^{1/3}$ and E_∞^* is the long time effective modulus.

For the case of constant loading or unloading rates ($|d\bar{P}/dt| = \bar{P}_0/t_0$), differentiating equation (2.87) with respect to $\bar{a}$ leads to

$$\frac{d\beta}{d\bar{a}} = 3\sqrt{2\bar{a}\beta} - 3\left(\frac{\beta}{\bar{a}}\right) - \sqrt{\frac{2\beta}{\bar{a}^3}}A\Phi(\tau)\tau, \quad (2.88)$$

where $A = \frac{\bar{P}_0}{t_0} \left(\frac{36RE_\infty^* w^2}{\pi h_0^3} \right)^{2/3}$ is the loading rate parameter. During loading, $\beta = \beta_{cl}$ with

$$\beta_{cl} = E_\infty^* \Phi(\tau). \quad (2.89)$$

During unloading, $\beta = \beta_{op}$ with

$$\beta_{op} = \frac{1}{E_\infty^* \Phi(\tau)}. \quad (2.90)$$

Integrating equation (2.88) step-by-step gives the variation of β with $\bar{a}$. The variation of contact size with load can be obtained by using equation (2.87).

2.3.6 Adhesion measurement

Numerous testing techniques have been and are continuing to be developed to characterize adhesion. Adhesion test techniques [89] include: pull-off test, interfacial fracture test, blister test, scratch test, bending test et al. Pull-off tests are performed by measuring the forces and displacements involved in bringing the tip into contact and then out of contact with the sample surface. Pull-off tests are particularly informative when done under displacement control, which eliminates the snap-to-contact phenomena. This test provides information concerning the attractive forces sensed before contact, as well as the adhesive forces that resist breaking the contact. Interfacial fracture tests can be performed in different ways. The most common method to induce interfacial fracture via nanoindentation methods is through a scratch test, in which delamination occurs at the identified point. At this point, an abrupt change in lateral force and normal displacement are observed to film fracture. Alternatively, cross-sectioned samples can also be indented at the interface to induce delamination. Both methods use known forces and displacements to produce fracture, giving insight into the energetics of the delamination process.

From the experimental point of view, the Surface Force Apparatus (SFA) [15–17], Atomic Force Microscopy (AFM) [18–20] and Interfacial Force Microscopy (IFM) [90] are important instruments to evaluate molecular level adhesion. Chemical modification of the mica surface, replacing mica with thin, smooth polymer films, and adsorbing polymers on mica have enabled the SFA to be used for direct measurements of surface and interfacial energies. To enable optical measurements of contact area, the samples have to be thin, smooth, and transparent. These are stringent requirements for many materials. However, these have been met by experimentalists and produced direct measurements of surface energies between semi-crystalline and glassy polymeric films [91–93], as well as between glass and ceramic surfaces [94,95].

Recently, there has been increasing use of the contact mechanics experiments and analyses based on JKR theory (the so called ‘JKR experiments’) [12–14, 96–98]. The JKR experimental apparatus is illustrated schematically in Figure 2.23. A lens is placed

Figure 2.23: Schematic diagram of JKR experimental apparatus [98].

on a rigid transparent support that is fixed on a translating stage of an optical microscope. The lenses are compressed against the substrate that is put on the plate of an analytical balance. The microscope/balance combination enabled the simultaneous measurement of the contact radius and the corresponding load. The microscope operated in the reflection mode, requiring only one transparent material, either the substrate or the lens, in the contacting pair. The balance consists of an electromagnetic system maintaining the pan in a fixed position during load determination. The system is loaded under displacement control rather than load control. The balance is interfaced with a computer to record automatically the load variations.

In JKR experiments, typically two spheres, or one sphere and a flat, of either identical or different materials are brought into contact. Two regimes of the experiment are conducted – loading and unloading. Hysteresis may be observed between loading and unloading. A set of (a, P) data are collected and fitted to JKR equation (2.55) to obtain the best-fit values of E^* and w .

2.4 Summary

A comprehensive review of the adhesionless and adhesion contact of elastic, plastic and viscoelastic materials is given in this chapter. It covers analytical methods, experimental techniques and numerical procedures. In the studies of contact mechanics, the analyti-

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cal methods, experimental measurements and numerical analysis are complementary to each other, especially for nonlinear analysis. There are mature theories for solving the problems of bulk material indentation. However for layered-solid indentation, there are comparatively less results, especially for the thin compliant viscoelastic films, which are commonly used for MEMS applications.

Adhesion between solid surfaces becomes important with smooth surfaces, on a small scale and with light loads. The physical processes involved are manifold: interatomic forces, electrostatic forces, capillarity and so on. From the point of view of mechanics modeling, adhesive strength, whatever its physical origin may be, can be expressed by ‘work of adhesion’, which is the external work done to separate unit area of the adhering surfaces. For elastic adhesion contact, there are JKR theory, DMT theory and M-D theory. The JKR theory assumes that the region outside the contact zone where cohesive forces act is vanishingly small compared with the contact radius. On the other hand, the DMT theory takes such cohesive forces into account but assumes that they do not change the shape of the deformation profile outside the contact zone from that of the Hertzian profile. M-D theory is a unified model, which uses a cohesive zone model to connect the JKR theory and DMT theory, and to determine the applicability range of each theory. Inelastic deformation (plasticity and viscoelasticity) of the solids leads to ‘adhesion hysteresis’. The effect is to increase the work required to separate the surfaces. Viscoelastic deformation of the solids causes the effective work of adhesion to increase with speed of separation.

The theories described in this chapter form the basis for adhesion contact research. In the present proposed research project, the JKR theory will be extended to adhesion indentation research of elastic thin film. Also, the inelastic adhesion theory will be used in the adhesion research of viscoelastic thin film indentation.

Chapter 3

Adhesion Mechanics of Three-Layer Elastic System Representing SFA Geometry*

3.1 Introduction

Understanding the mechanical properties of materials often requires knowledge of the contact deformation mechanisms of that material. The deformation of a solid is a function of the applied load, elastic and plastic properties of the solid, surface roughness and surface energy. The degree of deformation provides useful information about the strength, compliance, and adhesion of materials.

Hertz first proposed the theory of contact between elastic solids [5]. No surface traction was assumed to occur between the solids. Hence, the surfaces would separate at zero load. Johnson, Kendall, and Roberts (JKR) [7] modified the Hertz theory by including surface energy in the interaction zone and predicted a greater contact size for a given load. JKR theory is applicable when the elastic deformation caused by the surface forces is large compared to the range of the surface forces. In addition, JKR theory assumes that the solids in contact are homogeneous, isotropic, and perfectly elastic.

JKR theory has been successfully used to model the adhesion between rubber elastomeric hemispheres [12–14]. In these experiments, an optical microscope measured the flattened contact radius as a function of the applied load as two spherical elastomeric hemispheres contact. Another technique used to directly measure the properties of sur-

*The experiments described here are conducted by collaborator Dr. P. M. McGuiggan at National Institute of Standards and Technology (NIST), Gaithersburg, Maryland, USA.

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faces is the Surface Force Apparatus (SFA) [15, 16]. It comprises thin sheets of molecularly smooth film of interest (such as mica or gold), glued to cylindrical glass lenses of equal radii, which are then pressed into elastic contact with their axes at right angles. The schematic diagram of the layered SFA system is shown in Figure 3.1. The resulting contact between the two crossed cylinders is geometrically equivalent to a rigid sphere on a flat layered half-space. A Hertz-type circular area of contact ensues and can be measured by direct observation of the optical interference fringes through the glass, glue and mica. The mica surfaces may be ‘dry’ or separated by molecularly thin films of fluid. In view of the smooth and intimate nature of the contact, adhesive forces are easily detectable.

Figure 3.1: Schematic diagram of the layered SFA (mica/epoxy/silica) system. The thin silver layers are used to produce optical interference fringes to measure the contact radius and thickness of mica and epoxy.

Previous measurements of the deformation of the surfaces showed a trend consistent with JKR theory, but JKR theory underestimated the measured contact radius by as much as 30% [91]. A more recent measurement under sliding conditions showed better agreement [99]. However, JKR theory assumes homogeneous, isotropic half-space, which is not the case for the layered materials in the SFA experiments. Thus, Johnson and coworkers [21] proposed a modified numerical version of JKR theory to analysis the adhesion of layered SFA system. An important parameter in the elastic deformation of layered materials is the elastic modulus. Since the contacting solids in the SFA experiments consist of mica/epoxy/silica layers having widely different elastic constants, it is expected that the effective elastic modulus depends upon the mica thickness, the epoxy thickness, the con-

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tact radius and the elastic moduli of the corresponding materials. Recent theoretical work on layered structures shows that the effective elastic modulus varies with layer thickness and contact size in the SFA experiments [21]. As the contact size increases under load, effective modulus of the system changes according to the amount of deformation. For small forces, effective modulus will be determined by the modulus of the top layer, i.e. the mica. As the load and contact size increases, effective modulus will be increasingly influenced by the substrate layers, the epoxy and eventually the silica glass substrate. The theoretical work further demonstrated that JKR theory can be modified to treat layered systems [21]. The modified theory predict the contact radius versus load dependence for a layered system has the same general JKR curve shape as that found in a homogeneous isotropic sample. The adhesion characteristics, including the pull-off force, were found to depend upon an adhesion parameter, as well as on the layer thickness and their elastic moduli. However, because the epoxy and mica layer thickness were not measured in previous SFA experiments, a direct comparison of this theory to experiments for layered structures has not been performed.

In the present experiments, the elastic moduli of mica and epoxy were measured by nanoindentation using spherical indenters. The SFA was then used to measure the adhesion and deformation of two thin mica sheets which were attached to a cylindrical silica support with an epoxy layer. The mica and epoxy thickness as well as the contact radius as a function of applied load in adhesive and non-adhesive contact, were measured by optical interferometry at the point of contact. By measuring all the experimental parameters, the validity of modified JKR theory for a layered structure could be experimentally examined.

3.2 Theoretical model

According to the JKR theory, the relationship between the contact load P and the contact radius a between two elastic homogeneous spheres of radius R_1 and R_2 is given by

$$P = P_0 - P_a = \frac{4E^*a^3}{3R} - \sqrt{8\pi a^3 w E^*}, \quad (3.1)$$

where $R = (1/R_1 + 1/R_2)^{-1}$, E^* is the effective modulus of the two spheres and is given by $E^* = [(1 - \nu_1^2)/E_1 + (1 - \nu_2^2)/E_2]^{-1}$, E_1 and E_2 are the modulus of each sphere and ν_1 and ν_2 are their respective Poisson's ratio, w is the work of adhesion, and P_0 and P_a correspond to the Hertz load (adhesionless) and the adhesive (flat punch) load respectively. In the absence of adhesion, the force reduces to P_0 (the Hertz value). It follows from equation (3.1) that the surfaces separate at the 'pull-off force' P_c which is obtained from $\partial P/\partial a = 0$ and given by

$$P_c = -\frac{3\pi R w}{2}. \quad (3.2)$$

The JKR theory can be considered locally as a rigid sphere of radius R indenting an elastic half-space with plain strain modulus $E' = E/(1 - \nu^2)$.

The above homogenous theory has been extended, using a finite element method for a surface consisting of two layers on a substrate indented by a rigid sphere [21]. Figure 3.2 schematically shows the simplified three layer SFA geometry. It comprises a glass substrate with mica and epoxy layers of thickness h_1 and h_2 . The corresponding elastic moduli and Poisson's ratios of the layers and substrate are respectively E_i and ν_i ($i = 1, 2, 3$). The adhesionless load P_0 and adhesive load P_a terms in equation (3.1) are evaluated separately. The adhesionless load P_0 (Hertz load) may be written in the non-dimensional form as

$$\frac{P_0 R}{E'_1 a^3} = f \left\{ \frac{a}{h_1}, \frac{h_2}{h_1}, \dots, \frac{E'_2}{E'_1}, \frac{E'_3}{E'_1}, \dots \right\}, \quad (3.3)$$

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and the adhesion (flat punch) term P_a may be represented in non-dimensional form by

$$\begin{aligned} \frac{P_a R}{E'_1 a^3} &= \left(\frac{2wR^2}{E'_1 h_1^3} \right)^{1/2} \left(\frac{h_1}{a} \right)^{3/2} g \left\{ \frac{a}{h_1}, \frac{h_2}{h_1}, \dots, \frac{E'_2}{E'_1}, \frac{E'_3}{E'_1}, \dots \right\} \\ &= \left(\frac{2wR^2}{E'_1 h_1^3} \right)^{1/2} \bar{g} \left\{ \frac{a}{h_1}, \frac{h_2}{h_1}, \dots, \frac{E'_2}{E'_1}, \frac{E'_3}{E'_1}, \dots \right\}, \end{aligned} \quad (3.4)$$

where $E'_i = E_i/(1 - \nu_i^2)$, $i = 1, 2, 3$. With the values of the thickness and moduli known, f and $\bar{g}$ are computed as functions of (a/h_1) . The net applied load P is then given by the summation of the Hertz load P_0 and adhesive load P_a as

$$\frac{PR}{E'_1 a^3} = \frac{P_0 R}{E'_1 a^3} - \frac{P_a R}{E'_1 a^3} = f - \alpha \bar{g}, \quad (3.5)$$

where $\alpha = (2wR^2/E'_1 h_1^3)^{1/2}$ is a non-dimensional measurement of the strength of adhesion.

When $w = 0$, $\alpha = 0$ and equation (3.5) reverts to the adhesionless Hertzian contact case.

Figure 3.2: Three layer SFA model and parameters.

3.3 Experimental measurements

3.3.1 Indentation of bulk material

a. Mica

The bulk elastic properties of the mica used in the experiment are measured from instrumented nanoindentation tests that were performed on a rigidly supported 500 μm thick piece of mica. A commercial nanoindenter (Nano Indenter II, Nano Instruments, Inc.) was used to record the force and displacement with a 21 μm radius spherically tipped diamond indenter loaded onto the mica surface. A range of forces from 1 mN to 90 mN resulted in indentation depths from < 20 nm to approximately 350 nm. The whole displacement was elastic; the mica did not deform permanently even at the highest forces and no residual impressions were observed. A Hertzian analysis of this elastic sphere-on-flat contact permits the determination of the quantity $E_1/(1 - \nu_1^2)$, where ν_1 is the Poisson's ratio for mica, if the material is assumed elastically isotropic. Although the isotropic assumption is not likely to be true for a material with mica's layered structure, the indentation experiment applies force along the same axis (perpendicular to the planes of the layers) as in the surface force experiments, thus can be considered valid. The value obtained for $E_1/(1 - \nu_1^2)$ for mica by this technique was $65 \text{ GPa} \pm 1 \text{ GPa}$. Although ν_1 could not be independently determined, it is likely to fall in the range $0.15 \leq \nu_1 \leq 0.25$ for a covalently bonded oxide, giving a value of $E_1 = 62 \text{ GPa} \pm 2 \text{ GPa}$.

b. Epoxy

The bulk elastic properties of the epoxy (E_2) were measured using an instrumented microindenter. Epon 1004 epoxy (Shell Chemical Company, Houston, TX) was preheated in a vacuum oven overnight to remove residual air bubbles and a thick epoxy layer (> 2 mm) was directly applied onto a solid sample stub. The epoxy was indented with a 3.28 mm diameter tungsten carbide sphere with a loading rate of approximately 0.5 N/s to a maximum load of 5 N, then a 5 s holding at peak load and an unloading rate of approxi-

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mately 1 N/s. The elastic modulus was calculated from the load-depth data using a linear least squares fit. Twenty five repeat measurements at different contact positions gave the elastic modulus of epoxy $E_2 = 3.0 \text{ GPa} \pm 0.04 \text{ GPa}$, if the Poisson's ratio ν_2 is assumed to be given by 0.495.

3.3.2 Surface force apparatus measurements

The SFA uses two molecularly smooth mica sheets bonded to silica cylindrical lenses with EponTM 1004 hot melt epoxy. A 10 nm thick silver film was first sputtered onto the silica cylinders and the hot melt epoxy was spread onto the silvered cylinders. Mica sheets with 50 nm silver films on the back (epoxy) side were bonded with the epoxy to each lens. The cylindrical lenses are mounted in the SFA with their cylinder axes at right angles, producing a contact that is geometrically equivalent to a sphere on a flat (Figure 3.1).

The mica thickness h_1 , epoxy thickness h_2 , contact radius a and cylinder radius R were all measured using multiple beam optical interferometry. Optical interference between the two 50 nm silver layers produced primary fringes of equal chromatic order allowing measurement of the mica thickness, h_1 . Interference between the 10 nm silver layer and the 50 nm silver layer produced secondary fringes, allowing measurement of the epoxy thickness h_2 at the point of contact. These were typically between 10 μm and 30 μm in thickness. In these experiments, the mica thickness was the same for the both cylinders. Since the epoxy thicknesses were not controlled to be the same on each surface, both thicknesses were measured. For this particular experiment, the measured mica thickness was $5.5 \mu\text{m} \pm 0.1 \mu\text{m}$; the epoxy thickness on one cylinder is $25.5 \mu\text{m} \pm 1.5 \mu\text{m}$, and on another cylinder was $16 \mu\text{m} \pm 1 \mu\text{m}$, respectively. The mean radius of the crossed cylinders was $26 \text{ mm} \pm 1.5 \text{ mm}$.

The radii of curvature of the mica surfaces when the cylinders were not in contact were determined from the shape of the primary interference fringes. Deformation of the surfaces when the surfaces were in contact was determined from the primary fringes. The contact radius, a , was measured directly from the amount of flattening of the deformed

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primary fringes. Prior to each measurement, the contact radius is maximized by scanning the contact. The measured contact radius was in the range $5 \leq a/h_1 \leq 15$.

The lower cylinder was mounted on a double-cantilever spring, which was used to apply a force to the crossed cylinders. The force was measured by noting the deflection of the spring, which had a spring constant of $1.2 \times 10^5 \text{ N/m} \pm 0.2 \times 10^5 \text{ N/m}$. Testing was performed in the closed chamber of the SFA which was purged with dry N_2 for at least 20 minutes prior to beginning the measurements and solid P_2O_5 was placed in the chamber to ensure a dry atmosphere.

The SFA experiments were performed by bringing the mica surfaces into van der Waals contact at zero applied force. The force was increased in a step-wise fashion and the contact radius was measured at the same time. The measurements continued until an applied compressive load of approximately 500 mN was reached. The load was then decreased until the surfaces spontaneously separated ('pull-off') at a negative load P_c . The time between loading steps was approximately one minute, for a total loading/unloading time of approximately 40 minutes with 6 hours to 12 hours between subsequent runs. All measurements were made at $23^\circ\text{C} \pm 2^\circ\text{C}$.

In order to determine the contact behavior of the mica surfaces when adhesive forces were absent, a droplet of 10^{-3} M KCl solution was placed between the mica surfaces. The interaction between mica surfaces immersed in 10^{-3} M KCl solution is repulsive due to a short range hydration force, thus the interaction would be expected to follow Hertzian mechanics. No adhesion was measured between the surfaces in the 10^{-3} M KCl solution, i.e. the surfaces separated when the applied force was reduced to zero, as expected from Hertzian contact theory. The shape of the contact zone under compressive force in adhesionless contact is more rounded (less defined) at the edges than in adhesion contact. As a result, the radius of contact is less distinct in adhesionless contact and could only be measured to within 5%, with even more uncertainty at small contact radius.

3.4 Finite element model

The bulk material properties from indentation and SFA experiments are summarized in Table 3.1 and used as input values to the finite element simulation of the SFA experiments. The Young's modulus for the cylindrical silica supports is taken from the literature to be 72 GPa [23].

The ABAQUS finite element (FE) program (ABAQUS, HKS Inc., Rhode Island, USA) is used for the linear elastic numerical analysis of the mica/epoxy/silica layered system shown in Figure 3.2. As the imposed displacement is very small, infinitesimal strains are considered. No geometry non-linear is included. The FE mesh and far-field boundary conditions are as shown in Figure 3.3. The layers are assumed to be perfectly bonded without any interlayer slip. A large domain, 100 – 150 times the contact radius, is discretized using axisymmetric 8-noded quadratic and 6-noded triangular elements. Triangular elements are used for mesh grading purpose. Hybrid elements should be chosen for the epoxy layer as its Poisson's ratio is close to the incompressibility limit of 0.5. A particularly fine mesh is required in the region close to the edge of contact radius $r = a$ in order to obtain reliable values of the stress intensity factor at that point. Stresses at the remote boundaries are negligible. The boundary conditions for the simulation are as follows. The nodes in the bottom side are fixed in the vertical (z) direction; the nodes on the axisymmetric axes are fixed in the radius (r) direction. The load is applied by given the displacements on the surface nodes in the contact range.

Table 3.1: The parameter values used in finite element simulation for SFA model

Layer material	Young's modulus (GPa)	Poisson's ratio	Layer thickness (μm)	Indenter radius (mm)
mica	62	0.21	5.5	26
epoxy	3.0	0.495	20	
silica	72	0.25	∞	

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Figure 3.3: Axisymmetric finite element mesh for indentation of layered SFA half-space.

To simulate the indentation, the normal displacement

$$u_z(r) = \delta - \frac{r^2}{2R} \quad (r \leq a) \quad (3.6)$$

is decomposed into $u'_z = \delta$ (flat punch profile) and $u''_z = -r^2/2R$ (parabolic profile), and applied separately. The displacement u'_z and u''_z give rise to load P' and P'' and to stress intensity factor K'_I and K''_I at $r = a$, respectively. The loads are found by summing the reaction forces at the nodes lying within the contact area. The stress intensity factors were found by an extension of the virtual crack extension method [21]. The displacement $u'_z(r)$ is that due to a flat punch in which the displacement δ' , the load P' and the stress intensity K'_I are in direct proportion. In an adhesionless contact there is no singularity at $r = a$, whereupon the adhesionless load P_0 is given by

$$P_0 = P'' - \left(\frac{K''_I}{K'_I} \right) P'. \quad (3.7)$$

Computing P_0 by equation (3.7) for a series of values a/h_1 and replacing P_0 into equation (3.3) yields the function $f(a/h_1)$. The function $g(a/h_1)$ is given directly by

$$\frac{P_a}{K_I a^{3/2}} = \frac{P'}{K'_I a^{3/2}} = g\left(\frac{a}{h_1}\right). \quad (3.8)$$

3.5 Mesh sensitivity analysis

To test the results for mesh sensitivity, the substrate material silica ($E = 72$ GPa, $\nu = 0.25$) was used to substitute the mica and epoxy layers to make the indentation as homogeneous one. Following the method introduced in previous section, the displacement was decomposed as constant displacement ($u'_z = 0.01 \mu\text{m}$) and quadratic displacement ($u''_z = -r^2/2R$, $R = 26$ mm). The constant displacement will be regarded as flat punch indentation; the combination of constant displacement and quadratic displacement profile using equation (3.6) will be regarded as spherical indentation. The results were compared with theoretical flat punch indentation and spherical indentation (Hertzian).

Figure 3.4 illustrates the errors of FE calculation of flat punch indentation and spherical indentation. The errors in both cases are less than 0.8% in the whole range of contact radii considered. These two verifications establish the validity of the finite element models with the original meshes to study the three-layer mica/epoxy/silica SFA geometry.

3.6 Results

The full finite element analysis for the three layer SFA half-space is used to compare with the experimental measurements.

The functions $f(a/h_1)$ and $g(a/h_1)$ calculated from the FE analysis are plotted in Figure 3.5 for a series values of non-dimensional contact radius a/h_1 . When $a/h_1 \ll 1$, the deformation is confined to the surface layer and $f(a/h_1)$ takes the value of $4/3$ given by equation (3.3) and $g(a/h_1)$ takes the value of $2\sqrt{\pi}$ given by equation (3.4). With increasing indentation size, $f(a/h_1)$ decreases in response to the compliance of the layer of epoxy, until the stiffening provided by the glass substrate becomes significant. A similar variation is found in the function $g(a/h_1)$. Further increasing the contact size, the effect of glass substrate becomes dominant. At last functions $f(a/h_1)$ and $g(a/h_1)$ approach the constant value $4k/3$ and $2\sqrt{\pi k}$, respectively, where $k = E'_3/E'_1$ represents the ratio of plain strain moduli of glass substrate to mica layer.

Figure 3.4: The error of FE calculation for (a) flat punch indentation and (b) spherical punch indentation.

Adhesionless load P_0 (Hertz load) is computed using equation (3.7), and Figure 3.6 gives a comparison of FE calculation with the experiment data. The FE calculation of Hertz load fits the experiment data very well. Replotting Figure 3.6 in the form of the applied Hertz load P_0 against (a^3/R) is as shown in Figure 3.7. If the solid were homogenous with a plain strain modulus E' , the Hertz theory predicts a linear relationship between P_0 and a^3/R with a slope of $4E'/3$ as shown by the dashed line in Figure 3.7. The upward curvature of the experimental measurements show that E' is not constant, but increases with depth of indentation as the stiffness of the silica substrate is increasingly felt. This effect is clearly shown by the theoretical variation of the effective modulus E'_e

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Figure 3.5: Functions $f(a/h_1)$ and $g(a/h_1)$ as a function of contact size a/h_1 calculated from FE computation for SFA model.

as a function of contact radius, which is analyzed in detail as follows.

Figure 3.6: Contact radius as a function of Hertz load and the comparison of FE calculation with experimental measurements.

3.6.1 Effective modulus

The effective modulus E'_e is defined as the modulus of a homogeneous isotropic half-space which would exhibit the same stiffness S ($= dP/d\delta$) as the layered system, i.e.

$$E'_e(a) = \frac{1}{2a} \frac{dP_0}{d\delta_0}. \quad (3.9)$$

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Figure 3.7: Hertz load P_0 as a function of a^3/R .

It is clear that the effective modulus E'_e is a function of the contact size a . The effective moduli are calculated using equation (3.9) by numerical differentiation of the compliance relationship $P_0(\delta_0)$. The effective modulus E'_e calculated from spherical tip indentation are plotted using the cross points in Figure 3.8. The effective modulus E'_e can also be calculated directly from the flat punch indentation, for which $dP/d\delta = P_a/\delta_a$, plotted using a solid line in Figure 3.8. For the experimental measurements for $5 \leq a/h_1 \leq 15$, and according to Figure 3.8, the modulus of the silica is strongly influencing the effective modulus. The values of the effective modulus as a function of experimental contact radius were calculated. The experimental values of E'_e are found by converting the variation of load with a (Figure 3.6) to variations with displacement δ_0 and hence the stiffness of the layered solids. The values of dP_0/da are derived from Figure 3.6 and the indentation penetration can be obtained by

$$\delta_0 = \frac{a^2}{R} B(a/h_1), \quad (3.10)$$

where $B(a/h_1)$ is a non-dimensional penetration function, which is defined by

$$\frac{\delta_0 R}{a^2} = B \left\{ \frac{a}{h_1}, \frac{h_2}{h_1}, \frac{E'_2}{E'_1}, \frac{E'_3}{E'_1} \right\}. \quad (3.11)$$

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The function $B(a/h_1)$ is calculated from the FE computation and is shown in Figure 3.9. The value of function $B(a/h_1)$ is almost a constant and equals to 0.62 when the non-dimensional contact radius is in the range of experimental measurements $5 \leq a/h_1 \leq 15$. It follows that

$$\frac{d\delta_0}{da} \approx \frac{2Ba}{R}. \quad (3.12)$$

Figure 3.8: Effective modulus E'_e as a function of non-dimensional contact radius.

Figure 3.9: The variation of non-dimensional indentation penetration $B(a/h_1) = \delta_0 R / a^2$ with the non-dimensional contact radius.

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Now the contact stiffness is given by

$$S = \frac{dP_0}{d\delta_0} = \frac{dP_0/da}{d\delta_0/da} = \frac{R}{2Ba} \frac{dP_0}{da}. \quad (3.13)$$

The effective modulus based on the definition equation (3.9) becomes

$$E'_e = \frac{S}{2a} = \frac{R}{4Ba^2} \frac{dP_0}{da}. \quad (3.14)$$

The effective moduli calculated from experimental data using above method are plotted using the open circle points in Figure 3.8. These experimental values of the effective modulus compare well with the theory.

3.6.2 Net contact load

Figure 3.10 shows the experimental measured contact radius versus applied load during the loading process in dry N₂ (circles) and 10⁻³ M KCl solution (squares). The data represent measurements for the adhesion and adhesionless case, respectively, for the same experimental contact position. As expected, Hertz (adhesionless) contacts have smaller radii at a given force than adhesion contacts, at least for the small forces used in these SFA experiments.

Figure 3.11 shows measurements of the contact radius versus force in dry N₂ for loading (filled symbols) and unloading (open symbols). As seen in the figure, a loading/unloading hysteresis is present. The value of a for the unloading curve is greater than or equal to that for the loading curve. Although the amount of hysteresis varied from run to run, there was a less than 1% variation in the value of the zero force contact radius, a_0 , measured on each subsequent loading curve after leaving the sample unloaded for 6 hours or more. The value of a_0 upon unloading, however, has an 8% variation for different runs. Since the contact radius at zero force on loading was similar for each run, no permanent gross plastic deformation of the epoxy occurred. The range of pull-off force P_c for different runs was measured to be between -15 mN and -25 mN. This corresponds to an

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Figure 3.10: Experimental measurements of contact radius versus load with and without adhesion.

adhesion energy within the range $120 \text{ mJ/m}^2 \leq w \leq 200 \text{ mJ/m}^2$ as calculated by using equation (3.2).

For the experimental run shown in Figure 3.11, the pull-off force on unloading give $w = 200 \text{ mJ/m}^2$ by equation (3.2). The average value of E'_e during the run, from the adhesionless test results shown in Figure 3.7 is about 23 GPa. The variation of contact size a with load during unloading, given by the standard JKR equation (3.1) for a homogeneous solid, is shown by the solid line in Figure 3.11. The fit with experimental measurements of the JKR curve, involving no adjustable constants, is both remarkably and surprisingly good.

For a layered solid the net load P as a function of the contact radius a is given by equation (3.5); it depends on the adhesion parameter α . For the thicknesses and moduli appropriate to the SFA, the FE computed values of this function are plotted in Figure 3.12 for a series of values of α . The experimental points from Figure 3.11 are added to the graph. Taking the values of w mentioned above gives $4 \leq \alpha \leq 5$, which is consistent with the experiments. However, the experiments are not a good fit to the shape of the curve and fail to show the expected increase in effective modulus at higher forces.

Using non-dimensional variables $\bar{P} = P/3\pi wR$ and $\bar{a} = a(4E'_1/9\pi wR^2)^{1/3}$, equa-

Figure 3.11: Experimental measurements of contact radius versus load with and without adhesion.

tion (3.5) reduces to

$$\bar{P} = \frac{3}{4} \bar{a}^3 (f - \alpha \bar{g}). \quad (3.15)$$

Curves of contact area $\bar{A} = \pi \bar{a}^2$ plotted against load $\bar{P}$ are presented in Figure 3.13. They are of the same general shape as JKR curve. The FE calculation with non-dimensional adhesion parameter $\alpha = 5.0$ is in good agreement with the unloading experimental data.

3.6.3 Pull off force

The values of the non-dimensional pull-off force $\bar{P}_c$ have been measured from the curves in Figure 3.12 and plotted against non-dimensional adhesion parameter α in Figure 3.14. The adhesion parameter α has a significant effect on the pull-off force $\bar{P}_c$, which ranges from -0.32 to -0.65 compared with the JKR value of -0.5 . Moreover from the theoretical curve for the pull-off force of Figure 3.14, a value of α between 4 and 5 suggests that $\bar{P} = -0.58$ compared with $\bar{P} = -0.5$ in the homogeneous case. These results will be discussed further in the next section.

Figure 3.12: Contact radius as a function of net load and the comparison of FE calculation with experimental measurements.

3.7 Discussion

The experiments with the SFA without adhesion have clearly shown the increase in effective modulus E'_e (Figure 3.7) due to the effect of the silica substrate, in agreement with the theory. However, when compared to adhesion case, measurements of contact radius with load, during unloading (Figure 3.11), follow the standard JKR relationship with a constant value of E'_e roughly equal to the average of the values found from the adhesionless test. This is a paradox which is also apparent in Figure 3.12, where the measurements in Figure 3.11 are compared with the theoretical predictions of the theory with adhesion. There is a good fit for small forces, but at larger forces the measured radius does not appear to respond to the increasing modulus of the layered solid found in the adhesionless case.

A possible reason for the greater difference with theory in the adhesion case could be viscoelastic effects in the epoxy layer causing adhesion hysteresis, which was observed to be greater in the presence of adhesion. In the range of these experiments, where the influence of the thin mica layer is small, the high peak of adhesive stress at the edge of contact will be transmitted to the epoxy layer with little attenuation. The jump into contact on loading and the pull-off on unloading, which are instantaneous with elastic materials, will show some viscoelastic delay. In a study of viscoelastic contact, it is shown that

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Figure 3.13: Non-dimensional contact area $\bar{A}$ as a function of non-dimensional net load $\bar{P}$ and the comparison of FE calculation with experimental measurements.

the pull-off force can be significantly enlarged compared with the JKR value [85, 86]. One consequence of the measured hysteresis is that two values of a are measured; one on loading and one on unloading, with the contact radius of the unloading curve always being greater than or equal to the contact radius of the loading measurements. In all cases, the value of a for the unloading measurements appears to be the only values which reliably fit JKR theory for homogeneous materials.

Repeated SFA measurements showed that no permanent gross plastic deformation of the epoxy had occurred. The time scale between measurement runs was 6 hours to 12 hours, and the epoxy appeared to relax within that time. The micro indentation experiments that were used to determine the elastic modulus of the epoxy were much faster (20 s to 30 s), and showed minimal hysteresis or plasticity. It has been assumed that the experiments are at equilibrium at each step during the SFA loading and unloading cycles, which may not necessarily be the case if the relaxation time is long [80].

According to finite element analysis, least error in the assumption of constant E'_e will occur for $0.6 < a/h_1 < 3$. This is where E'_e is at or near the minimum value and is determined largely by the epoxy modulus. For the adhesion case, the contact radius was in the range of $30 \text{ mm} < a < 80 \text{ mm}$, giving $5 < a/h_1 < 15$. Hence, it seems to be outside

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Figure 3.14: Non-dimensional pull-off force as a function of adhesion parameter.

the range where E'_e is relatively constant. The forces used in these experiments are higher than generally attained in SFA experiments due to the large spring constant used (i.e., 100 to 1000 times stiffer than normal). If more typical spring constants had been used, the assumption of relatively constant E'_e would be more reasonable.

The value of E'_e is larger than that previously measured by Horn et al [91] for the interaction of similar mica/epoxy/silica layered systems. In those experiments, the contact radius was within the range $0 < a/h_1 < 20$ and the effective modulus E'_e was found to vary from 2.5 GPa to 11.6 GPa. Since the previous experiments had different epoxy and mica thicknesses, E'_e was unique for each specific experiment. The varying values of E'_e indicate the strong dependence of E'_e on the layer thicknesses. Due to the large cylinder radius ($1.5 \text{ cm} < R < 2.7 \text{ cm}$) and the thin mica sheets ($h_1 < 6 \text{ mm}$) used in the surface forces experiments, combined with the soft epoxy under-layer, the contact radius in air at zero force is generally 5 times greater than the mica layer thickness. Hence, the mica plays a minimal role in the overall measured mechanical properties of the system; the mechanical properties are mostly dictated by the epoxy and the underlying silica surface, depending on the applied force.

At what point does the silica begin to influence the mechanical properties? This can be seen in Figure 3.15 where a two layer material is compared to a three layer system.

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The three layer case represents the SFA system; two layer case represents a mica layer on thick epoxy substrate. The thickness of the outermost mica layer (h_1) is the same in both systems but the modulus of the two layer system is lower than that for the three layer system. Hence, it appears that even at very low contact radii ($10^{-2} < a/h_1 < 1$), the silica substrate is significantly influencing the measured modulus. For $a/h_1 = 0.01$ where there begins to be a difference between the response of a $20\text{ }\mu\text{m}$ epoxy layer thickness and an infinite epoxy thickness, $a = 0.55\text{ }\mu\text{m}$. Hence, the silica is influencing the mechanical properties through $5.5\text{ }\mu\text{m}$ mica and $20\text{ }\mu\text{m}$ epoxy, a distance that is approximately 25 times the contact diameter. This value is much larger than the rule of thumb of 3 times the contact diameter that is frequently used. These results are the result of the large difference in the epoxy and silica moduli.

Another factor in comparing experimental and theoretical values is that the SFA measurements consist of two deforming surfaces (sphere on flat geometry) having epoxy thicknesses of $16\text{ }\mu\text{m}$ and $25\text{ }\mu\text{m}$. The theoretical calculations model this contact as a rigid flat punch pressed onto a 3-layer material having an epoxy thickness of $20\text{ }\mu\text{m}$ (the average value of the two epoxy thicknesses). This difference in the model from experiment may lead to slight differences from the measured contact, as was observed. In addition, the experimental measurement of the contact radius assumes the contact radius

Figure 3.15: Effective modulus for three layer case and two layer case.

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to be spherical, as is expected for crossed cylinders. If however, the cylinders were not at 90° , an elliptical contact would occur. In this case, an error would occur in the measurements of the contact radius. In addition, if the contact radius were not measured on a major axis, the measurements would further deviate from the theoretical predictions.

Another important conclusion to this work is in regards to the pull-off force. Homogeneous theory gives the non-dimensional pull-off force, $\bar{P}_c \equiv P_c/3\pi wR = 0.5$, resulting in $w = 2P_c/3\pi R$. In homogeneous JKR theory, the pull-off force depends solely on the mica surface energy and is independent of the contact dimension or modulus. As shown in Figure 3.14, three layer JKR analysis gives $\bar{P}_c \approx 0.58$ for $\alpha = 5$. Thus, $w \approx P_c/1.75\pi R$. This result was found to be relatively independent of the layer thickness ratio h_2/h_1 [21]. It is interesting to note that this value is intermediate to the JKR limit for homogeneous materials, where $w = 2P_c/3\pi R$, and the DMT limit, where $w = P_c/2\pi R$ [8].

The results have implications for other experimental techniques, such as atomic force microscopy and nanoindentation, where the contact radius can vary from 5 nm to 1,000 nm. These results show that even minute indentations ($a/h_1 > 10^{-2}$) can be influenced by the underlying materials and the measured effective modulus will be a composite response.

3.8 Conclusion

This chapter reports an experimental investigation of the micro and nano indentation of layered surfaces, both with and without adhesion. In particular, it investigates the contact mechanics of the Surface Force Apparatus (SFA) in which almost all the parameters of the apparatus are found by direct measurement. The experimental results are compared with finite element analysis (FEA) of layered solids.

Nano-indentation of bulk mica gave the elastic modulus of mica: $E_1 = 62 \pm 2$ GPa. Micro-indentation of bulk epoxy gave the modulus of epoxy: $E_2 = 3.0 \pm 0.04$ GPa.

Indentation using the SFA, with the mica surfaces treated to eliminate adhesion, gave values for the effective modulus of the system (mica and epoxy layers on a silica sub-

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strate), which were in good agreement with the theory, both in variation with depth and in absolute value.

Using the SFA in dry nitrogen to promote adhesion between the mica surfaces gave contact radius - force curves which were in reasonable agreement with theory. However the loading and unloading curves showed appreciable adhesion hysteresis presumably due to viscoelasticity of the epoxy adhesive layer. To a first approximation, the JKR relationship for a homogeneous solid could be fitted to the unloading curve.

The finite element analysis further showed that $w = P_c/1.75\pi R$ for the experimental geometry of the SFA at these conditions.

Chapter 4

Adhesion between Two-Layer Elastic System and Spherical Probe

4.1 Introduction

When the size of a machine or a device becomes smaller, the effect of adhesion, friction and wear on its performance becomes relatively large. The adhesive forces are important when the contacting bodies are small or compliant in nature. Thin compliant layered surfaces that are used as bearing surfaces in artificial joint replacements and micro electro mechanical systems (MEMS) are two examples, where adhesive forces have an influence on their performance. Tribological properties known in macrosystems are not always available for such compliant thin layered material systems. For example, adhesive force or pull-off force is negligibly small in macrosystems, but it becomes relatively larger than the external load in the nano-newton range.

Contact mechanics under the effect of adhesion due to long range atomic forces was theoretically analyzed by Johnson-Kendall-Roberts (JKR) theory [7] by extending the Hertzian analysis and was shown to be effective for explaining experimental results involving compliant elastomers. A problem arises when the JKR theory is applied to the contact between thin films as they cannot be treated as a semi-infinite elastic half-space as in the JKR theory. The contact radius relative to the thickness of the film is an important parameter characterizing the significance of confinement effect on the elastic and adhesive properties of the film.

The relevance of the JKR theory to contact mechanics problems is more significant due to the fact that the adhesive forces become more important as the stiffness and size

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of the contacting materials are reduced. Shull [100] recently reviewed the current state of the art on contact mechanics and the adhesion of soft compliant elastic and visco-elastic solids. But, his work is limited to the case of a compliant solid perfectly bonded to a rigid substrate. In this chapter, adhesion mechanics between a compliant elastic coating and a spherical indenter is investigated through a finite element (FE) method. Broadly it follows the computational approach proposed in Chapter 3 (to model the SFA geometry), but a two layer elastic half space is considered. The effect of the coating's Poisson's ratio is included in this work by considering both compressible (ν smaller than 0.5) and incompressible (ν close to 0.5) coatings. The effect of substrate stiffness on the adhesive forces is also estimated. Another novelty of this investigation is in finding the compliance of the system as a function of contact radius and finally in obtaining empirical expressions for the adhesive load for all the material systems considered.

4.2 Extension of JKR theory

The JKR theory is based on contact of homogeneous, isotropic solids, which is not applicable for the layered material adhesion analysis. A numerical version JKR theory was proposed to model the adhesive contact between a rigid sphere and a two-layer homogeneous half-space [22]. The model is shown in Figure 4.1. It comprises a layer of thickness h , which is perfectly bonded to an elastic half-space. The Young's modulus and Poisson's ratio of the thin layer and substrate are E_1, ν_1 and E_2, ν_2 , respectively. It is indented by a rigid spherical surface of radius R . Friction between the indenter and the surface of the layer is neglected at present. The radius of the contact circle and the elastic compression under the action of a compressive (positive) load P are denoted by a and δ , respectively. Following JKR method, the net load is separated into an adhesionless component P_0 and an adhesion component P_a . For the layered half-space, guided by equation (2.55), the adhesionless load P_0 and adhesive load component P_a may be expressed in non-dimensional form as

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Figure 4.1: Indentation of a two-layer half-space by a spherical indenter with adhesion (a) typical experimental setup (b) modeling approach.

$$F(x, k) \equiv F \left\{ \frac{a}{h}, \frac{E'_2}{E'_1} \right\} = \frac{3}{4} \frac{P_0 R}{E'_1 a^3}, \quad (4.1)$$

$$H(x, k) \equiv H \left\{ \frac{a}{h}, \frac{E'_2}{E'_1} \right\} = \frac{P_a}{\sqrt{8\pi w E'_1 a^3}}, \quad (4.2)$$

where $x = a/h$, $k = E'_2/E'_1$ and $E'_i = E_i/(1 - \nu_i^2)$, ($i = 1, 2$) is the plane strain Young's modulus. Similarly, guided by equation (2.56), the displacement δ_0 and δ_a due to P_0 and P_a separately may be expressed by

$$B(x, k) \equiv B \left\{ \frac{a}{h}, \frac{E'_2}{E'_1} \right\} = \frac{\delta_0 R}{a^2}, \quad (4.3)$$

$$C(x, k) \equiv C \left\{ \frac{a}{h}, \frac{E'_2}{E'_1} \right\} = \frac{P_a}{2a E'_1 \delta_a}. \quad (4.4)$$

Dividing equation (4.2) by equation (4.4) gives

$$D(x, k) \equiv \frac{H(x, k)}{C(x, k)} = \delta_a \sqrt{\frac{E'_1}{2\pi w a}}. \quad (4.5)$$

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Equations (4.1) and (4.2) are combined to give the net load P as

$$\begin{aligned}\frac{PR}{E_1' h^3} &= \frac{P_0 R}{E_1' h^3} - \frac{P_a R}{E_1' h^3} \\ &= \frac{4}{3} x^3 F(x, k) - \sqrt{\frac{8\pi w R^2}{E_1' h^3}} x^{3/2} H(x, k)\end{aligned}\quad (4.6)$$

and equations (4.3) and (4.5) are combined to give the net displacement δ as

$$\begin{aligned}\delta &= \delta_0 - \delta_a \\ &= \frac{a^2}{R} B(x, k) - \sqrt{\frac{2\pi w a}{E_1'}} D(x, k).\end{aligned}\quad (4.7)$$

Now introducing an adhesion parameter (a non-dimensional measurement of work of adhesion) $\alpha = (2wR^2/E_1'h^3)^{1/2}$, it is found that

$$\bar{a} = x \left(\frac{9\pi\alpha^2}{8} \right)^{-1/3}. \quad (4.8)$$

With the adhesion parameter α and other parameters $\bar{P}$, $\bar{a}$, $\bar{\delta}$ as defined previously, the net load (equation (4.6)) and central displacement (equation (4.7)) are simplified as

$$\bar{P} = \bar{a}^3 F(x, k) - \sqrt{2\bar{a}^3} H(x, k), \quad (4.9)$$

$$\bar{\delta} = \bar{a}^2 B(x, k) - \frac{2\sqrt{2\bar{a}}}{3} D(x, k). \quad (4.10)$$

Equation (4.9) directly gives the relation of applied load P and contact radius a , and equation (4.10) gives the relation of central displacement δ and contact radius a . The use of equations (4.9) and (4.10) to cancel out the contact radius a gives the relation between applied load P and central displacement δ . In practice, the load-displacement relationship is more relevant than the load-contact radius relationship, because it is comparatively difficult to measure the in-situ contact radius during the indentation test. For example, the micro or nano prob instruments, such as nanoindenter, can not record the load-contact radius relation but load-displacement relation.

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When $a/h \ll 1$ (i.e. $x \rightarrow 0$), deformation is confined to the thin film layer. Such that the adhesionless load P_0 and adhesion load P_a can be approximately expressed as

$$P_{0 \ x \rightarrow 0} \approx \frac{4}{3} \frac{E_1 a^3}{R(1 - \nu_1^2)}, \quad (4.11)$$

$$P_{a \ x \rightarrow 0} \approx \left(\frac{8\pi w E_1 a^3}{1 - \nu_1^2} \right)^{1/2}. \quad (4.12)$$

The values of function $F(x, k)$ and $H(x, k)$ now become

$$F(x, k)_{x \rightarrow 0} = \frac{3}{4} \frac{P_0 R (1 - \nu_1^2)}{E_1 a^3} \approx 1, \quad (4.13)$$

$$H(x, k)_{x \rightarrow 0} = P_a \left(\frac{1 - \nu_1^2}{8\pi w E_1 a^3} \right)^{1/2} \approx 1. \quad (4.14)$$

Similarly, when $a/h \ll 1$, functions $B(x, k)$ and $D(x, k)$ also approach unity. Equations (4.9) and (4.10) then reduce to the standard JKR relation in non-dimensional form, i.e. equations (2.61) and (2.62).

When $a/h \gg 1$ (i.e. $x \rightarrow \infty$), deformation is dominated by the substrate. Such that the adhesionless load P_0 and adhesion load P_a can be approximately expressed as

$$P_{0 \ x \rightarrow \infty} \approx \frac{4}{3} \frac{E_2 a^3}{R(1 - \nu_2^2)}, \quad (4.15)$$

$$P_{a \ x \rightarrow \infty} \approx \left(\frac{8\pi w E_2 a^3}{1 - \nu_2^2} \right)^{1/2}. \quad (4.16)$$

The values of function $F(x, k)$ and $H(x, k)$ now become

$$F(x, k)_{x \rightarrow \infty} = \frac{3}{4} \frac{P_0 R (1 - \nu_1^2)}{E_1 a^3} \approx k, \quad (4.17)$$

$$H(x, k)_{x \rightarrow \infty} = P_a \left(\frac{1 - \nu_1^2}{8\pi w E_1 a^3} \right)^{1/2} \approx \sqrt{k}. \quad (4.18)$$

However, when $a/h \gg 1$, functions $B(x, k)$ and $D(x, k)$ should approach unity and $1/\sqrt{k}$,

respectively.

For a range of a/h values in between the limits, the functions $F(x, k)$, $H(x, k)$, $B(x, k)$ and $D(x, k)$ can be evaluated by finite element method.

4.3 Numerical analysis

The numerical analysis follows the same method that is introduced in the three-layer SFA geometry (Chapter 3). An incompressible material response cannot be modelled with regular elements because the pressure stress in the element is indeterminate. If the material is incompressible, its volume cannot change under the loading. Therefore, the pressure displacement formulation is inadequate for any element with incompressible material behavior. Hybrid elements include an additional degree of freedom that determines the pressure stresses in the element directly. Hybrid elements are used when the material behavior is incompressible (Poisson's ratio = 0.5) or very close to incompressible (Poisson's ratio > 0.475).

FE computations have been carried out for material system representing (i) a compliant incompressible film on a rigid substrate, (ii) a compliant incompressible film on an elastic substrate, and (iii) a compliant compressible film on an elastic substrate. In the first case, the Poisson's ratio of the film is varied from 0.45 to 0.4999, to investigate its effect on the pull-off force calculations. For a comparison, the Poisson's ratio equal to 0.2 and 0.3 are also included. The incompressibility in the second case is modelled by setting the Poisson's ratio value to 0.4999, which is close to the incompressibility limit of 0.5. Although the numerical results are presented in non-dimensional form, the actual material properties used in the FE simulations are listed in Table 4.1. The radius R of the spherical probe was taken to be 20 mm. The thickness of the thin film h was set to $2.0 \mu\text{m}$ and the contact radius a was varied in the range of $0.02 - 2000 \mu\text{m}$, i.e. $10^{-2} \leq a/h \leq 10^3$.

Table 4.1: The material properties used in the FE calculations

	Case i	Case ii	Case iii
E_1 (GPa)	0.02	0.02, 0.2, 0.5, 2, 5	0.2, 0.5, 2, 5, 20
ν_1	0.2, 0.3, 0.45, 0.49, 0.495, 0.499, 0.4999	0.4999	0.3
E_2 (GPa)	Rigid	70	70
ν_2	—	0.2	0.2

4.4 Results and discussion

4.4.1 Relation between load and contact radius

Numerical values of the non-dimensional adhesionless load and adhesive load given by the function $F(x, k)$ and $H(x, k)$ (as defined in equations (4.1), (4.2)) are shown as data points in Figures 4.2 and 4.3 respectively. It is noted that for a thick layer or small indentation ($a/h < 0.5$), deformation is confined to the thin film layer and both $F(x, k)$ and $H(x, k)$ approach unity irrespective of the material constants (i.e. Young's modulus and Poisson's ratio) of the layer. The standard JKR model can be used to represent the variation of the load and contact radius by replacing the elastic properties of the half-space with that of the thin film.

With increasing indentation size (i.e. for a thin layer with large a/h) both $F(x, k)$ and $H(x, k)$ increase in response to the stiffness of the harder substrate. For the case of compliant incompressible thin film on a rigid substrate (case i), the values of function $F(x, k)$ and $H(x, k)$ are sensitive to the Poisson's ratio of the thin film ν_1 , especially when ν_1 is very close to 0.5 ($0.45 \leq \nu_1 \leq 0.4999$); while when the value of ν_1 is around 0.3, the effect of Poisson's ratio is not so clear. Because of the rigid properties of the substrate, the modulus ratio of substrate to thin film $k (= E'_2/E'_1)$ approaches infinity, thus theoretically, function $F(x, k)$ and $H(x, k)$ will continuously increase to infinity value, even

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Figure 4.2: FE computations of non-dimensional adhesionless load $F(x, k)$ as a function of contact radius x : (a) case i, (b) case ii, (c) case iii.

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Figure 4.3: FE computations of non-dimensional adhesion load $H(x, k)$ as a function of contact radius x : (a) case i, (b) case ii, (c) case iii.

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though practically it is impossible to reach that value. Whereas, for the case of compliant thin film (both incompressible and compressible) on an elastic substrate (case ii and case iii), the values of functions $F(x, k)$ and $H(x, k)$ are sensitive to the mismatch parameter k . Further large indentation depth, $F(x, k)$ and $H(x, k)$ approach k and $\sqrt{k}$ as expected. For the case ii, both $F(x, k)/k$ and $H(x, k)/\sqrt{k}$ approach unity when $x/k > 5$ (Figure 4.4). For the case iii, both $F(x, k)/k$ and $H(x, k)/\sqrt{k}$ approach unity provided $x/k > 25$ (Figure 4.5). In this regime, again, the standard JKR model applies for indentation of a half-space having the elastic properties of the substrate.

Figure 4.4: FE computations of (a) $F(x, k)/k$ and (b) $H(x, k)/\sqrt{k}$ for the case ii.

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Figure 4.5: FE computations of (a) $F(x, k)/k$ and (b) $H(x, k)/\sqrt{k}$ for the case iii.

In the numerical simulation, only a few representative values for k and a/h are chosen. In practice, the values of k are determined by the mechanical properties of the system under consideration. However, it is a tedious task to carry out finite element analysis for each and every material properties combination and geometry. Herein empirical equations are fitted to the numerical data in Figures 4.2 and 4.3 for practical reference. Unfortunately, it is not possible to fit a general expression for the functions $F(x, k)$ and $H(x, k)$ covering the entire spectrum of layer-substrate material systems and non-dimensional contact radius x . The empirical expressions of $F(x, k)$ and $H(x, k)$ are represented in Figures 4.2 and 4.3 as solid lines and are as given follow.

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For the case of compliant incompressible film ($\nu_1 \geq 0.49$) on rigid substrate (case i),

$$F(x, k) = 1 + \left[\frac{8}{x(1+x)^{1.5}} + \frac{6.8(1-2\nu_1)}{x} \right]^{-1}, \quad (4.19)$$

$$H(x, k) = 1 + \left[\frac{3.8}{x\sqrt{1+x}} + \frac{2.1\sqrt{1-2\nu_1}}{\sqrt{x}} \right]^{-1}. \quad (4.20)$$

For the case of compliant incompressible film ($\nu_1 = 0.4999$) on elastic substrate (case ii),

$$\log F(x, k) = \frac{k'}{2} \{ \tanh [A'(\log x + B')] + 1 \}, \quad (4.21)$$

$$\log H(x, k) = \frac{k''}{2} \{ \tanh [A''(\log x + B'')] + 1 \}, \quad (4.22)$$

where,

$$A' = 0.1006k'^2 - 0.7204k' + 2.7366, \quad A'' = 0.3280k''^2 - 1.3439k'' + 2.7903,$$

$$B' = 0.0253k'^2 - 0.3404k' - 0.1276, \quad B'' = 0.1160k''^2 - 0.7397k'' - 0.0291,$$

$$k' = \log k, \quad k'' = \log \sqrt{k}.$$

For the case of compliant compressible film on elastic substrate (case iii),

$$\log F(x, k) = k' \left\{ \frac{\tanh [A'(\log x + 0.60)] + 1}{2} \right\}^8 \quad (4.23)$$

$$\log H(x, k) = k'' \left\{ \frac{\tanh [A''(\log x + 0.75)] + 1}{2} \right\}^8 \quad (4.24)$$

where,

$$A' = 0.037k'^2 - 0.33k' + 1.2, \quad A'' = 0.18k''^2 - 0.70k'' + 1.2,$$

$$k' = \log k, \quad k'' = \log \sqrt{k}.$$

To give a sense of the accuracy of the fits, the calculated errors between the results of FEM and fitting equations are plotted in Figures 4.6 and 4.7. For most of the data points, the error is less than 4%. As seen from the Figures 4.2, 4.3, 4.6 and 4.7, the comparison between the numerical data and mathematical fit is very good for the practical range of

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Figure 4.6: The errors of fitting equations $F(x, k)$ compared with the FEM results: (a) case i, (b) case ii, (c) case iii.

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Figure 4.7: The errors of fitting equations $H(x, k)$ compared with the FEM results: (a) case i, (b) case ii, (c) case iii.

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non-dimensional contact radius x and the plane-strain modulus ratio k . It is also noted that with increasing the modulus ratio k , the fitting equations become less accurate. However, based on the case study, most of k values in the practical applications will lie in the cited regimes. The fit equations will become increasingly inaccurate as the non-dimensional parameters deviate from those investigated.

The variation of the contact radius $\bar{a}$ with the net load $\bar{P}$ for varying values of adhesion parameter α as given by equation (4.9) are shown in Figure 4.8 for all the three material systems considered. The solid lines are obtained by fitting spline curves to the FE computed data points, whereas the dotted lines are obtained from the empirically fitted curves of $F(x, k)$ and $H(x, k)$. It is to be noticed that for the practical values of α in the range of 1.0 – 1000.0, the comparison between the numerical data and empirical equations is very good. The load-contact radius curves of thin film system have the same shape as the standard JKR relationship, and these curves lie in between the two limiting JKR cases obtained with E' replaced by E'_1 and E'_2 . When α is small, the curve of load-contact radius is close to $\text{JKR}(E'_1)$. Increasing the value of α , the load-contact radius curve gradually approaches to $\text{JKR}(E'_2)$. There is a minimum load point in each load-contact radius curves. In fact, it is at this point that the surfaces under contact are pulled off during unloading process. In the process of adhesion measurements, the surfaces are brought into contact at zero load. The surfaces of two contact solids jump into contact at an initial contact size a_0 , because of the adhesion properties of two surfaces. Increasing the indentation load P (positive) slowly to a certain maximum value P_m , the contact size a also increases accordingly to a maximum value a_m . After that, the load P decreases gradually from P_m to zero, at which a finite size contact radius is still visible due to the surface adhesion force. A negative value of load P_c must be applied to pull the contacting surfaces off, at which the surfaces will separated abruptly. The contact radius corresponding to pull-off force is a_c . In the theoretical curves of load-contact radius shown in the Figure 4.8, there still are values when the contact radius is lower than a_c . In fact, this part will never happen in the experimental test. Because of the fully elastic properties of the layered system, the

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Figure 4.8: Variation of the non-dimensional contact radius $\bar{a}$ with net load $\bar{P}$ for a series values of adhesion parameter α : (a) case i, (b) case ii, (c) case iii.

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energy stored during the loading process is completely reversible, i.e. the loading process and unloading process will follow the same load-contact radius curve. If the thin film is not perfectly elastic one, some inelastic properties, such as viscoelasticity, will come forth and will have effect to the loading/unloading curves. For example, the hysteresis of loading/unloading may be clearly seen. This phenomena due to the viscoelastic properties of the thin film will be discussed in Chapter 5.

4.4.2 Relation between contact load and penetration

The numerical results of the non-dimensional displacement (penetration depth) δ_0 for spherical probe and δ_a for the flat punch given by function $B(x, k)$ and $D(x, k)$ (as defined in equations (4.3) and (4.5)) are shown in Figures 4.9 and 4.10 as data points. In these figures, the spline curves are used to smoothly connect the numerically simulated results.

It is noted that for small contact size or a thick layer (x is small), the deformation is confined to the thin film layer and all the non-dimensional functions $B(x, k)$ and $D(x, k)$ for different layered systems approach unity irrespective of the material properties (Young's modulus and Poisson's ratio). In a long range of non-dimensional contact radius $10^{-2} < x < 10^{-1}$, the values of flat punch displacement (i.e. function $D(x, k)$) are approximately equal to unity. However, for spherical displacement (i.e. function $B(x, k)$), the contact radius range in which the $B(x, k)$ values are approximately equal to unity is comparatively short. That means the spherical punch indentation is more sensitive to the stiffness effect of substrate than flat punch indentation provided that the contact radius is the same.

With increasing contact size, the central displacement function $B(x, k)$ of spherical indentation first decreases to a minimum value, then increases and finally approaches to a constant value of 0.5 and 1.0 for thin film on rigid substrate and on elastic substrate, respectively. At large values of contact radius a/h , for thin film on rigid substrate, the contact radius a is approximately equal to $\sqrt{2R\delta}$ rather than $\sqrt{R\delta}$, because of the non-deformable properties of the substrate. The value $\sqrt{2R\delta}$ is also close to $1.5\sqrt{R\delta}$ quoted by Shull and Crosby [101] in the axisymmetric adhesion test of pressure sensitive adhesives.

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Figure 4.9: FE computations of non-dimensional central displacement $B(x, k)$ as a function of contact radius x : (a) case i, (b) case ii, (c) case iii.

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Figure 4.10: FE computations of non-dimensional displacement $D(x,k)$ as a function of contact radius x : (a) case i, (b) case ii, (c) case iii.

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However, for thin film on an elastic substrate, the contact radius a is still approximately equal to $\sqrt{R\delta}$. At this regime, the effect of thin film to the whole layered system can be neglected. For the compressible film on elastic substrate (Figure 4.9(c)), the contact radius considered is not big enough to fully reflect the substrate behavior, but the trend is clear. The minimum values of the function $B(x, k)$ and the contact radii corresponding to the minimum $B(x, k)$ values depend on the material properties (i.e. mismatch parameter k and Poisson's ratio ν) made up of the layered systems. For a given contact radius, the larger the mismatch parameter k (Figures 4.9(b) and 4.9(c)) or Poisson's ratio (Figure 4.9(a)), the smaller the value of function $B(x, k)$, vice versa.

With increasing contact size a/h , function $D(x, k)$ decreases continuously. For the elastic substrate supported layered system, the function $D(x, k)$ will finally approach a constant value of $1/\sqrt{k}$, which can be theoretically deduced from the definition given by equation (4.5).

Based on the equations (4.9) and (4.10), the variation of load $\bar{P}$ with central displacement $\bar{\delta}$ is plotted in Figure 4.11 for a series of adhesion parameter values α . There is also a minimum load point in the load-displacement curve. In fact, it is the same pull off point with previously in load-contact radius curve, i.e. at this load, the contact surfaces are separated. During adhesion measurement, the surfaces are brought into contact at zero load, at which there is an initial central displacement δ_0 . Increasing the indentation load P (positive) slowly to a certain maximum P_m , the displacement δ also increases accordingly to a maximum value δ_m . After that, the load P begins to decrease from P_m to zero value, then continually to a negative value P_c , at which the surfaces are separated abruptly. The load-displacement curves are fully reversible during loading and unloading process, because of the elastic analysis of the layered system.

4.4.3 Initial contact and pull-off analysis

In the experimental contact measurement with adhesion, the surfaces will jump into contact at zero load. There is an initial contact size a_0 . In the loading phase, with increasing

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Figure 4.11: Variation of the non-dimensional load $\bar{P}$ with central displacement $\bar{\delta}$ for a series values of adhesion parameter α : (a) case i, (b) case ii, (c) case iii.

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load, contact radius and indentation displacement increase; during the unloading phase, in which the load decreases, the contact radius and indentation displacement decrease accordingly. There is a minimum load point in each load-contact radius curves and load-displacement curves. In fact, the minimum load is the pull-off force $\bar{P}_c$, at which the surfaces snap apart. The initial contact size and the pull-off force are normally interested to the researches, because from which the scale of adhesion energy is clear. The pull-off force can be determined from Figure 4.8 or Figure 4.11 easily. The contact radius at zero load can be obtained from Figure 4.8.

The pull-off force as a function of adhesion parameter α is plotted in Figure 4.12 using solid lines. It is noted that the value of non-dimensional pull-off force directly connects with the adhesion parameter α , the mismatch parameter $k (= E'_2/E'_1)$, and the Poisson's ratio of the thin film ν_1 . The non-dimensional pull-off force $\bar{P}_c$ has a range from -0.5 to -0.95 depending on the exact mechanical properties of the layered system: for a given material system, the normalized pull-off force $\bar{P}_c$ initially decreases with adhesion parameter and after a critical value of adhesion parameter, it starts to increase and finally reaches the JKR value of -0.5 asymptotically. For any given adhesion parameter α , the value of normalized pull-off force $\bar{P}_c$ deviates further away from the JKR value of -0.5 , when the mismatch parameter k is bigger. For the thin film system with a rigid substrate, the pull-off force is very sensitive to any small change of thin film Poisson's ratio.

The variations of contact radius at zero load $\bar{a}_0$ with the adhesion parameter α for a series values of material parameters are shown in Figure 4.13. It is noted that, for any given layered system, the non-dimensional contact radius at zero load decrease continuously from the JKR limit ($\bar{a}_0^{\text{JKR}} \approx 1.26$) to a certain constant value with increasing the adhesion parameter. The constant values are mainly decided by the system elastic mismatch parameter k . For a given adhesion parameter value, the bigger the mismatch parameter k , the smaller the contact radius.

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Figure 4.12: The non-dimensional pull-off force $\bar{P}_c$ as a function of the adhesion parameter α : (a) case i, (b) case ii, (c) case iii.

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Figure 4.13: The non-dimensional contact radius at zero load $\bar{a}_0$ as a function of the adhesion parameter α : (a) case i, (b) case ii, (c) case iii.

4.4.4 An empirical approach for the estimation of adhesive load

The contact behavior during an adhesion experiment can be expressed in terms of the contact compliance C by

$$P_a^2 = (P - P_0)^2 = 4\pi aw \left(\frac{d}{da} [C(x)] \right)^{-1}. \quad (4.25)$$

If compliance C and adhesionless load P_0 are known, the net load P can be obtained by using equation (4.25). Here, the objective is to determine accurate analytical expressions for the compliance C and adhesionless load P_0 as a function of the contact radius, through finite element simulations.

The finite element calculations of indentation by a flat punch gives the compliance $C(x)$ directly as δ_a/P_a . For thick layers, i.e. x is very small ($\approx 10^{-2}$), irrespective of the substrate modulus, the compliance is asymptotically given by the following expression [27]

$$C(x \rightarrow 0) \equiv C_0 = \frac{\delta}{P} = \frac{1 - \nu_1^2}{2aE_1}. \quad (4.26)$$

In the case of a thin film ($x \rightarrow \infty$) on an elastic substrate, the asymptotic compliance is obtained by replacing E_1 and ν_1 with E_2 and ν_2 respectively in equation (4.26), whereas for thin film on a rigid substrate the compliance is given by [102]

$$C(x \rightarrow \infty) \equiv C_\infty = \frac{(1 + \nu_1)(1 - 2\nu_1)h}{\pi a^2(1 - \nu_1)E_1}. \quad (4.27)$$

The compliance is identically equal to zero for an incompressible film of infinite thickness. But, the compliance is non-zero for finite thickness incompressible films.

For a certain range of material parameters E_1 , E_2 , ν_1 , and ν_2 , the variation of the computed non-dimensional compliance C/C_0 with the non-dimensional contact radius x is shown as data points in Figure 4.14.

For a compliant incompressible thin film on a rigid substrate (case i), the following empirical expression (shown as solid lines in Figure 4.14(a)) gives an excellent fit to the

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Figure 4.14: The variation of compliance of the layered system with contact radius: (a) case i, (b) case ii, (c) case iii.

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compliance data across the entire range of non-dimensional contact radius x values:

$$\frac{C_0}{C} = 1 + \frac{9\pi x}{32} \left[\left(2 + x^{3/2} \right)^{-1} + 2(1 - 2\nu_1) \right]^{-1}. \quad (4.28)$$

Shull and Crosby [101] have obtained a slightly different expression for the compliance of elastic coating on a rigid substrate as

$$\frac{C_0}{C} = 1 + \left[\frac{0.75}{x + x^3} + \frac{2.8(1 - 2\nu_1)}{x} \right]^{-1}. \quad (4.29)$$

The above equation is shown with dotted lines in Figure 4.14(a) for various values of Poisson's ratio. It can be seen that fit equation (4.28) for the compliance is slightly better than the Shull and Crosby's empirical relation, especially when the Poisson's ratio ν_1 is greater than 0.495 and $x > 2$.

For an incompressible film on an elastic substrate (case ii), with $k' = \log(E'_2/E'_1)$,

$$\log\left(\frac{C_0}{C}\right) = k' \left\{ \frac{\tanh[(\log(x) - 0.29k' + 0.062)(2.3 - 0.2k'))] + 1}{2} \right\}^{0.6}. \quad (4.30)$$

For a compressible elastic coating on an elastic substrate (case iii), with $k' = \log(E'_2/E'_1)$,

$$\log\left(\frac{C_0}{C}\right) = k' \left\{ \frac{\tanh[(\log(x) - 0.34k' + 0.51)(1.2 - 0.2k'))] + 1}{2} \right\}^2. \quad (4.31)$$

The variation of C_0/C with x calculated from empirical equations (4.30) and (4.31) are plotted as solid lines in Figures 4.14(b) and 4.14(c), respectively. It is clear that the empirical compliance equations represent the FE data very well.

The gradient of the adhesionless displacement (δ_0) versus load (P_0) curve also provides the instantaneous contact compliance. The compliance expression has to be substituted into equation (4.25) to obtain a closed form expression for adhesive load P_a . Notice that equation (4.25) requires further differentiation of the contact compliance with respect to the contact radius.

For the adhesionless indentation of a homogeneous elastic half-space by a rigid sphere,

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the indentation load, contact radius and indentation depth should be governed by the Hertzian relation. Whereas for layered thin film indentation, the adhesionless load P_0 and contact radius a will deviate from the Hertzian value. The function $F(x, k)$ provides the variation of Hertzian load (adhesionless load) for a given value of x and k .

In equation (4.25), now replacing the contact compliance C and adhesionless load P_0 by equations (4.28), (4.30), (4.31) and (4.19), (4.21), (4.23) respectively, the net load with adhesion can be deduced directly as a function of non-dimensional contact radius x . The variation of the non-dimensional contact radius with the non-dimensional net load obtained using this empirical relation is in good agreement with the FE calculations.

4.4.5 Contact traction distribution

It is useful to know the stress distribution within the contact zone between the loading probe and the compliant coating. The JKR contact pressure due to the Hertz load P_0 and adhesive load P_a at contact radius a can be represented as

$$p^{\text{JKR}}(r, 0) = \frac{3P_0}{2\pi a^2} \left[1 - \left(\frac{r}{a} \right)^2 \right]^{1/2} - \frac{P_a}{2\pi a^2} \left[1 - \left(\frac{r}{a} \right)^2 \right]^{-1/2}. \quad (4.32)$$

It is to be noted that the adhesive pressure is dominant at the periphery of the contact zone ($r \approx a$). In the non-dimensional form, the above pressure distribution can be represented as

$$\frac{\pi a^2 p^{\text{JKR}}(r, 0)}{P} = \frac{\sqrt{1 - \left(\frac{r}{a} \right)^2}}{2(1 - Z(x, k, \alpha))} \left[3 - \frac{Z(x, k, \alpha)}{1 - \left(\frac{r}{a} \right)^2} \right], \quad (4.33)$$

where $Z(x, k, \alpha) = (3\sqrt{\pi}\alpha H(x, k))/(2x^{3/2}F(x, k))$ and P is the net load given by equation (4.6). The normal pressure (traction) distribution across the contact radius is plotted in Figure 4.15 for different values of the non-dimensional contact radius a/h and the non-dimensional load P/P_0 for the case of compliant incompressible film on rigid substrate. The tangential traction is identically equal to zero as no friction condition is assumed between the loading indenter and the coating. For thin layers ($a/h \gg 1$), the magnitude of the stress singularity at the edge of the contact zone continues to decrease. The stress in

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this case is dominated by hydrostatic stresses that reach a maximum at the center of the contact zone and reduce to zero at the edges.

Figure 4.15: Pressure distribution within the contact zone for the case of a compliant incompressible film on a rigid substrate: (a) for different contact radius values x when the adhesion parameter $\alpha = 5$, (b) for different values of P/P_0 when $x = 1$ ($P/P_0 = 1$ indicates adhesionless contact).

4.4.6 Effect of indenter elastic modulus

In all the above analysis, the spherical probe has been modelled as a rigid body, whereas in a practical experimental situation, the elastic modulus of the indenter E'_3 is likely to be comparable with that of the substrate E'_2 . An exact treatment of indenter elasticity

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would require a full FE representation of the indenter, leading to an additional independent parameter E'_3/E'_1 . Instead, here an approximate correction to the above results to allow for the elasticity of the indenter is proposed.

Recall that the net load P at a contact radius a , when a rigid sphere of radius R in contact with an elastic half-space of plane-strain modulus E' under the presence of adhesion is given by

$$P = P_0 - P_a = \frac{4E'a^3}{3R} - \sqrt{8\pi w E' a^3}. \quad (4.34)$$

Here, an approximate correction to the above results to account for both thin layered system and elastic indenters is proposed. An equivalent plane-strain modulus $E'_{eq}(x, k)$ for the layered system representing a homogeneous elastic half-space at contact radius a , obtained by using equations (4.28) – (4.31) is found to be $[2aC(x, k)]^{-1}$. It follows that the contact between the equivalent half-space and an indenter of plane-strain modulus E'_3 will be governed by a combined modulus of E'_{co} such that

$$\frac{1}{E'_{co}(x, k)} = \frac{1}{E'_{eq}(x, k)} + \frac{1}{E'_3} \quad (4.35)$$

Now the constant modulus E' in equation (4.34) should be replaced by the combined modulus $E'_{co}(x, k)$ given by equation (4.35).

Using the elastic properties of the indenter as $E_3 = 70$ GPa, $\nu_3 = 0.2$, the computed values of the pull-off force $\bar{P}_c$ and the contact radius at zero load $\bar{a}_0$ are added to Figures 4.12 and 4.13 (the dash lines). The effect of the elastic behavior of the indenter appears to decrease the pull-off force value and the contact radius at zero load for a given adhesion parameter α .

4.4.7 Effect of friction

In the standard JKR theory of isotropic elastic half-space or modified numerical version JKR theory of layered system analyzed above, the friction operating between surfaces of materials is neglected. Although Hills [43] and Lin et al [102] assert that the effect of

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friction on the contact load-displacement will not be large, it is of practical significance to include the friction part in the analysis since frictionless contact is only an idealization. When two frictional dissimilar elastic spheres are brought into contact, the radial shear stresses will develop, perturbing the pressure distribution and producing a slip annulus. This slip will be resisted by frictional forces, which in turn will influence both the radial and vertical components of the displacements. For practical application, it is difficult to predict the slip annulus; however, it is easier to consider the totally bonded frictional case (no slip). Such that, any frictional contact will lie in these two limits, i.e. totally bonded frictional case and frictionless case. In this section the totally bonded frictional contact adhesion mechanics between compliant incompressible thin film and rigid spherical indenter is investigated. Finite element computations have been carried out for material system representing a compliant incompressible film on (i) a rigid substrate and (ii) an elastic substrate, as contacts with incompressible films likely to have frictional contact conditions. In the first case, the Poisson's ratio of the film is varied from 0.49 to 0.4999; and in the second case, the incompressibility is modelled by setting the value of Poisson's ratio to 0.4999, which is very close to the incompressibility limit 0.5. The obtained load-contact radius and load-displacement relations are compared with the frictionless case.

Numerical simulation including friction

To simulate the contact process including friction, the basic method used here is the same with the one previously introduced for frictionless contact problem. However this time, the horizontal displacement of surface nodes under contacting is set to be zero to simulate the fully bonded friction case. The normal surface displacement u_z is still separated into constant part u'_z and quadratic part u''_z . The stress arising from each displacement component are computed separately, and they give rise to stress intensity factors K'_I , K'_{II} and K''_I , K''_{II} , respectively.

To describe the local stress state at the contact edge, it is convenient to use local Cartesian coordinate system $(\hat{r}, \hat{z})$ centered at the contact radius $r = a$. The local stresses

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are in the state of plane strain, and are identical to that of a semi-infinite plane strain crack along the interface of indenter and thin film. The stress state near the contact edge must have the form

$$\sigma_{zz} + i\sigma_{rz} = \frac{(K_I + iK_{II})\hat{r}^{i\varepsilon}}{\sqrt{2\pi\hat{r}}}, \quad \hat{r} \rightarrow 0, \quad (4.36)$$

where $i = \sqrt{-1}$ and ε is a non-dimensional bimaterial parameter, which is defined as

$$\varepsilon = \frac{1}{2\pi} \ln \left(\frac{1 - \beta}{1 + \beta} \right). \quad (4.37)$$

In the above equation, β is Dundur parameter [103] related to layer and substrate shear moduli. When the indenter is rigid, ε is simplified as

$$\varepsilon = \frac{1}{2\pi} \ln(3 - 4\nu_1). \quad (4.38)$$

To avoid the oscillating singularity and simplify this problem, thin film is assumed to be incompressible ($\nu_1 \approx 0.5$). As the incompressible property considered for the thin film, the material parameter ε is very close to zero. For this case, the stress intensity factor K_I and K_{II} can be interpreted as

$$\sigma_{zz} = \frac{K_I}{\sqrt{2\pi\hat{r}}}, \quad \sigma_{rz} = \frac{K_{II}}{\sqrt{2\pi\hat{r}}}, \quad \hat{r} \rightarrow 0. \quad (4.39)$$

Furthermore, the stress intensity factor can be related to the energy release rate (the J-integral) for linear homogeneous elastic material through

$$G = \frac{1}{2E'}(K_I^2 + K_{II}^2). \quad (4.40)$$

The ‘G’ value can be computed using the J-integral option in ABAQUS finite element package. The summation of the normal nodal reaction forces on the surface gives the corresponding normal load P' and P'' due to u'_z and u''_z respectively. The adhesive force P_a is given directly by P' . In the absence of adhesion, the normal stresses σ_{zz} are smooth

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at $r = a$, so that P_0 is given by the combination of P' and P'' which satisfies the condition $K_I' + K_I'' = 0$. For the frictionless contact, because the tangential stress $\sigma_{\hat{r}\hat{z}}$ on the interface is identically equal to zero, stress intensity factor K_I is directly connected with J-integral. However, for totally bonded frictional contact, the relation between K_I and J-integral depends on the phase angle θ , defined by

$$\theta = \tan^{-1} \left(\frac{K_{II}}{K_I} \right) = \tan^{-1} \left(\frac{\sigma_{\hat{r}\hat{z}}}{\sigma_{\hat{z}\hat{z}}}_{\hat{r} \rightarrow 0} \right). \quad (4.41)$$

To calculate the phase angle, it requires a much finer mesh to resolve the stress distribution at distances substantially smaller than the thickness of the thin film [102]. The variations of phase angle θ are plotted against non-dimensional contact radius x in Figures 4.16 and 4.17 for thin film on rigid substrate and elastic substrate, respectively. The variation of $\theta(x, k)$ with the non-dimensional contact radius clearly shows that for incompressible thick films (or small contact sizes, $x < 0.1$) the strain-energy release rate is completely governed by the mode-I stress intensity factor.

Figure 4.16: FE computations of phase angle θ as a function of contact radius x for a compliant incompressible film on a rigid substrate due to the displacement (a) u_z' and (b) u_z'' .

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Figure 4.17: FE computations of phase angle θ as a function of contact radius x for a compliant incompressible film on an elastic substrate due to the displacement (a) u'_z and (b) u''_z .

The effect of friction on load and displacement functions

The numerical results of the non-dimensional adhesionless load and adhesion load given by function $F(x, k)$ and $H(x, k)$ (as defined in equations (4.1) and (4.2)) are shown in Figures 4.18 and 4.19. The numerical results of the non-dimensional central displacement δ_0 for spherical probe and δ_a for the flat punch probe given by functions $B(x, k)$ and $D(x, k)$ (as defined in equations (4.3) and (4.5)) are shown in Figures 4.20 and 4.21. In all these figures, the spline curves are used to smoothly connect the FE data points. The markers

Figure 4.18: FE computations of non-dimensional adhesionless force $F(x, k)$ as a function of contact radius x for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

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Figure 4.19: FE computations of non-dimensional adhesion force $H(x, k)$ as a function of contact radius x for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

Figure 4.20: FE computations of non-dimensional displacement $B(x, k)$ as a function of contact radius x for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

in these figures are only used to differentiate the different thin film systems. It is noted that for small contact size or a thick layer, the deformation is confined to the thin film layer and all those non-dimensional functions $F(x, k)$, $H(x, k)$, $B(x, k)$ and $D(x, k)$ approach unity irrespective of the material properties (i.e. elastic modulus, Poisson's ratio) and the contact surface properties (with or without friction). In this contact radius range ($a/h < 0.2$), the standard JKR theory can be used to represent the variation of the load and displacement by replacing the elastic properties with that of the thin film. According Hills [43], if the bodies are elastically similar or more precisely $[(1 - 2\nu_1)/\mu_1] - [(1 - 2\nu_3)/\mu_3] = 0$ (μ is

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Figure 4.21: FE computations of non-dimensional displacement $D(x, k)$ as a function of contact radius x for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

the shear modulus), no slip will occur. Because the thin film is incompressible ($\nu_1 = 0.5$) and the indenter is rigid ($\mu_3 \rightarrow \infty$), the friction case and frictionless case will have the same result in this range. With increasing contact size, the variation trends of functions $F(x, k)$, $H(x, k)$, $B(x, k)$ and $D(x, k)$ are the same for friction and frictionless cases. The friction effect to the load and displacement functions seems to be very small except for the transition region from thick film to thin film: the non-dimensional adhesionless and adhesion loads are slightly higher than that obtained from the frictionless case and the difference increases with increasing material mismatch parameter value k . The reason that the difference between bonded and frictionless cases is not great, except in the transition region, could be explained as follows. For a homogeneous half-space, a rigid indenter on an incompressible half-space gives zero shear stress, even for the bonded case. This is because normal stresses do not give tangential displacements. Thus for $a \ll h$ the layered system behaves primarily as a half-space of the incompressible layer material. On the other hand, for $a \gg h$ the system behaves much like a half-space of the substrate material. In either of those two limits the frictionless and bonded cases should and do give similar results to each other. Only for the intermediate values of x , which represent thick film to thin film limit, the results differ.

The effect of friction on load-contact radius relation

Based on the equations (4.9), the variation of non-dimensional load $\bar{P}$ with contact radius $\bar{a}$ is plotted in Figure 4.22 for a series of adhesion parameter α values. As seen from this figure, when the value of the adhesion parameter α is very small ($\alpha < 1.0$) or very big ($\alpha > 1000$), the difference between the friction and the frictionless cases is almost neglectable. Only in the transition range of $1 < \alpha < 1000$, the effect of friction to the load-contact radius relation is clear. It is around $\alpha = 10$ that the difference between friction and frictionless cases is the biggest. It also to be noted that the friction affects the normalized pull-off force $\bar{P}_c$ only slightly.

Figure 4.22: Variation of the non-dimensional contact radius $\bar{a}$ with load $\bar{P}$ for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

The effect of friction on load-displacement relation

The use of the equations (4.9) and (4.10) to cancel out the contact radius gives the variation of non-dimensional load $\bar{P}$ with central displacement $\bar{\delta}$ as plotted in Figure 4.23 for a series of adhesion parameter α values. For a given load, the effect of friction is to decrease the central displacement slightly.

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Figure 4.23: Variation of the non-dimensional load $\bar{P}$ with central displacement $\bar{\delta}$ for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

The effect of friction on pull-off force

The non-dimensional maximum tensile load $\bar{P}$, which is non-dimensional pull-off force $\bar{P}_c$ for a given adhesion parameter α can be read from load-contact radius (i.e. Figure 4.22) or load-displacement (i.e. Figure 4.23) graphs. The variation of normalized pull-off force $\bar{P}$ with adhesion parameter α is plotted in Figure 4.24. The pull-off force value depends strongly on the adhesion parameter and material properties: the value ranges in between the JKR limit of -0.5 and -0.95 for the range of material parameters considered in this study. There is no effect of friction between the probe and film on $\bar{P}_c$ when α is less than 0.2 . After that, $\bar{P}_c$ decreases with α till a critical value is reached, then the effect of friction is to increase $\bar{P}_c$ slightly with α .

The empirical equations of load and compliance

When fully bonded friction is considered, the empirical expressions for the non-dimensional adhesionless load $F(x, k)$, adhesion load $H(x, k)$ and compliance $C(x, k)$ as a function of non-dimensional contact radius x are provided here. The curves calculated from the empirical equations are represented in Figures 4.25, 4.26 and 4.27 as solid lines, respectively.

In these figures, the data points are calculated directly from the finite element simulation. These figures show that the empirical equations can represent the FE computations very

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Figure 4.24: Variation of the non-dimensional pull-off force $\bar{P}_c$ with adhesion parameter α for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

Figure 4.25: Empirical equation curves of adhesionless force $F(x, k)$ for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

well.

For the case of compliant incompressible film ($\nu \geq 0.49$) on rigid substance,

$$F(x, k) = 1 + \left[\frac{8}{x(1+x)^{1.8}} + \frac{6.8(1-2\nu_1)}{x} \right]^{-1}, \quad (4.42)$$

$$H(x, k) = 1 + \left[\frac{4}{x^{0.85}(0.5+x)} + \frac{2.3\sqrt{1-2\nu_1}}{\sqrt{x}} \right]^{-1}, \quad (4.43)$$

$$\frac{C_0}{C(x)} = 1 + \frac{9\pi x}{32} \left[(2+x^{1.8})^{-1} + 2(1-2\nu_1) \right]^{-1}. \quad (4.44)$$

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Figure 4.26: Empirical equation curves of adhesion force $H(x, k)$ for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

Figure 4.27: Empirical equation curves of compliance $C(x, k)$ for a compliant incompressible film on (a) rigid substrate and (b) elastic substrate.

For the case of incompressible film ($\nu_1 = 0.4999$) on an elastic substrate,

$$\log F(x, k) = \frac{k'}{2} \{ \tanh [A'(\log x + B')] + 1 \}, \quad (4.45)$$

$$\log H(x, k) = \frac{k''}{2} \{ \tanh [A''(\log x + B'')] + 1 \}, \quad (4.46)$$

$$\log \left(\frac{C_0}{C(x)} \right) = k' \left\{ \frac{\tanh [A'''(\log x + B''')] + 1}{2} \right\}^{0.6}, \quad (4.47)$$

where,

$$\begin{aligned}
 A' &= 0.055k'^2 - 0.53k' + 2.800, & B' &= 0.021k'^2 - 0.300k' - 0.110, \\
 A'' &= 0.430k''^2 - 1.700k'' + 3.300, & B'' &= 0.058k''^2 - 0.530k'' + 0.045, \\
 A''' &= -0.29k' + 2.80, & B''' &= -0.26k' + 0.09, \\
 k' &= \log k, & k'' &= \log \sqrt{k}.
 \end{aligned}$$

4.4.8 Experimental verification

Herein, the numerical predictions of contact load versus contact radius or displacement are verified by using published experimental measurements of two researchers.

Case study 1

Tardivat et al [104] have indented a $1.52 \mu\text{m}$ thin layer of polyisoprene with a deformable polyisoprene sphere of $912 \mu\text{m}$ radius. The thin layer was deposited onto a silicon wafer. The measured Young's modulus of polyisoprene film was 5 MPa and that of loading sphere was 1.94 MPa, and these values are quite low when compared to the silicon wafer Young's modulus of about 150 GPa. The polyisoprene is assumed be an incompressible elastomeric material with a Poisson's ratio of 0.499. The data points in Figure 4.28 shows the measurements of contact radius versus normal load. By assuming the contact geometry as an elastic incompressible film on a rigid substrate being indented by an elastic sphere, the numerical fit for contact load versus radius is shown in Figure 4.28 by a solid line. The work of adhesion obtained from this fit is 64 mJ/m^2 and it is within the experimental scattered value of about 60 mJ/m^2 reported by Tardivat et al (refer Figure 4.28(b)). It is to be noted that the range of contact radius of the experimental data is in between $65 - 110 \mu\text{m}$, giving a ratio of contact radius to thin film thickness of $40 - 70$. From Figures 4.2(a) and 4.3(a), it infers that the substrate has considerable effect on the load in this range of normalized contact radius (a/h). Except for the initial two loading points, the experimental data fits well with the numerical simulations.

Figure 4.28: Comparison between the experimental measurements of contact load versus radius with numerical predictions. Experimental data are taken from Tardivat et al [104]: a polyisoprene sphere of radius $912 \mu\text{m}$ indented an isoprene film of $1.52 \mu\text{m}$ thickness on a silicon wafer substrate.

Case study 2

Flanigan and Shull [105] have measured the elastic and adhesive properties of thin polymeric gel by indenting them with a glass indenter of radius 6 mm . A $360 \mu\text{m}$ thin layer of polymeric gel was formed by diluting a poly(methyl methacrylate) – poly(*n*-butylacrylate) – poly(methyl methacrylate) triblock copolymer with 2-ethyl hexanol, a selective solvent for the midblock. The plane-strain Young's modulus of the gel was measured to be 0.02 MPa . When compared with the gel layer modulus, the silica glass substrate and the indenter can be assumed to be rigid, as glass Young's modulus is around 80 GPa . The glass indenter was pressed into the gel and then retracted in order to obtain the loading and unloading data and these measurements were shown in the Figures 4.29(a) and 4.29(b) as data points for the load-contact radius and load-displacement relations, respectively. The hysteresis loop indicates considerable visco-elastic nature of the gel material. The numerical predictions for an incompressible layer on a rigid substrate are plotted as curves in these figures. These numerical approximations fit very well to the loading points, when the work of adhesion (w) is equal to 25 mJ/m^2 . The fact that the loading curve has points for negative loads suggests that the experiment was nearer to

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Figure 4.29: Comparison between the experimental measurements of contact load versus radius with numerical predictions. Experimental data are taken from Flanigan and Shull [105]: a glass sphere of radius 6 mm indented a polymeric gel of $360 \mu\text{m}$ thickness on a glass substrate. (a) load-contact radius, (b) load-displacement.

‘displacement’ rather than ‘load’ controlled, which reduces the snap on effect and makes it closer to the JKR theory for elastic solids. The totally bonded frictional condition corresponds to the case where there is no relaxation of the shear forces, whereas the frictionless condition corresponds to the case where the interfacial shear forces relax instantaneously. From the comparison results, it is clear that the experimental measurements correspond to the intermediate case, where the interfacial shear forces relax partially due to frictional sliding.

In conclusion it does appear that the small discrepancies with experiment under the condition of low and negative load are due to viscoelastic effects. This is entirely consistent with the findings of Johnson and Greenwood [86] .

4.5 Conclusion

Adhesion between a spherical indenter and thin compliant elastic film has been investigated. The adhesion characteristics, such as pull-off force, contact area at zero load, etc, depend on the adhesion parameter, the material properties (i.e. elastic modulus, Poisson's ratio) and the contact surface properties (with or without friction). For incompressible coating on a rigid substrate, by analyzing the numerically calculated compliance data across the entire range of non-dimensional contact radius, the empirical relations for the compliance and hence for the net load as a function of contact radius are obtained. For compliant elastic coating on an elastic substrate, the empirical equations for the adhesionless load and adhesion force by curve fitting, and hence the relations between the non-dimensional net load and non-dimensional contact radius are deduced. All these empirical calculations agree very well with FE data.

The pull-off force provides the most direct measurement of the surface energy. When the substrate is rigid, the non-dimensional pull-off force can be -0.95 , which is very different from the JKR value of -0.5 . Even though, in the standard JKR theory, the pull-off force $P_c = 1.5\pi wR$ does not include the elastic properties of the contacting solids, the deviation in pull-off force for a layered system from the JKR limit is due to the changing distribution of traction and deflection across the contact surface.

First, the finite element analysis assumes frictionless contact between the compliant coating and the loading sphere. Then, the effect of totally bonded friction is studied for incompressible film on elastic and rigid substrate. Any frictional contact will lie in these two limits. The effect of friction on the contact is not great, except in the transition region.

Both the standard JKR theory and revised numerical one assume that all the layers are perfectly elastic and that the surface forces giving rise to adhesion are fully reversible.

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However, most compliant polymeric coatings are likely to have viscoelastic material behavior. Crosby et al [106], Greenwood and Johnson [81] incorporated viscoelasticity by a rate dependent correction to the work of adhesion in the JKR equation. Crosby et al express the strain energy release rate as a function of ligament opening velocity as $G(v) = G_0[1 + (v/v^*)^n]$, where v is the speed of separation, v^* and n are found by experimentation. Greenwood and Johnson find G/G_0 from the creep compliance function of an assumed standard three-element linear viscoelastic model. In this respect, the compliance equations that have been fitted for the two layer material system is considerably more useful. The adhesion mechanics of thin viscoelastic coatings will be discussed in Chapter 5.

Chapter 5

Contact Analysis of Viscoelastic Thin Film Applied on Rigid Substrate

5.1 Introduction

In a typical ‘contact test’, a spherical tip indenter is brought into contact with a flat surface with a controlled load or controlled displacement, then unloading till the surfaces snap apart. In many situations, adhesion forces are extremely small compared with the force needed to deform the contacting material, so that the effects of adhesion force can be neglected, and the classic Hertz theory can be applied to model this problem. If the contacting materials are soft and show high surface energies, the surfaces are found to adhere: under load the contact area exceeds that predicted by the Hertz theory of elastic contact, a contact area of finite size is seen at zero load and a tensile force is required to pull the surfaces apart. This type of contact problem with adhesion can be theoretically analyzed by JKR (Johnson-Kendall-Roberts) theory [7].

In recent years, micro or nano probe instruments, such as Surface Force Apparatus (SFA), are widely used for adhesion analysis. In these experimental tests, either the instrument itself is made up of thin film structure or it is used to measure the thin film on the substrate. For the adhesion analysis of thin film system, the standard JKR theory is not applicable for its semi-infinite elastic half-space hypothesis. In order to model the contact problem of thin film system with adhesion, a numerical version of JKR theory is adapted by Johnson and coworkers [21, 22]. This numerical analysis method effectively considers the effect of adhesion force by introducing an adhesion parameter. The numerical version of JKR theory is further extended to different type of elastic thin film systems

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in Chapter 4. Through analyzing the numerical simulation results, the compliance of thin film system as a function of contact radius is deduced and finally obtained empirical expression for the adhesive load for all the material systems considered. The empirical calculations of load versus contact radius are in good agreement with the indentation experiments conducted by Tardivat et al [104] and Flanigan and Shull [105]. These previous research work assumes that all the layers are perfectly elastic and that the surface forces giving rise to adhesion are fully reversible. However, most compliant polymeric films are likely to have viscoelastic material behavior. During the indentation test, the hysteresis loop of loading and unloading processes is clear visible.

Theoretical models for the contact of bulk viscoelastic materials in the absence of adhesion have been available since the 1960s [39–41]. Maugis and Barquins [80] and Greenwood and Johnson [81] further developed contact models that included adhesion. More recently, Schapery [82] and Hui et al [83,84] obtained analytical solutions for the viscoelastic contact problem with adhesion by separating the contact process into closing contact and opening contact. A model for the whole closing/opening contact cycle of viscoelastic materials was proposed by Johnson [85,86]. However for the contact problem that a viscoelastic thin film is applied to a substrate, there are less theoretical models available, because of its mathematic difficulty to deduce a precise relationship between the load and deformation for the layered system. In this chapter the adhesionless and adhesion contact between the spherical indenter and linear incompressible viscoelastic thin film on a rigid substrate is analyzed by extending the empirical results of elastic thin film obtained in Chapter 4.

5.2 Viscoelastic properties of material

Although the viscoelastic properties can be measured by experimental method [107], analytical continuum mechanics modeling of rate-dependent materials is more effective to be mathematically tractable. The material behavior is actually governed by two independent functions, i.e. the shear and bulk moduli. But in practice, most materials of interest

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have time independent Poisson's ratio $\nu(t) \approx 0.5$, so that the time dependence of Young's modulus or shear modulus, or the quantity which appears in contact problems, the plane strain modulus E' (defined as $E/(1 - \nu^2)$), will be much the same [88, 108]. In this study, a linear viscoelastic three-parameter-model material is considered, which is illustrated in Figure 5.1. It comprises two springs and a dashpot. At very low strain rate the material deform elastically with a modulus E'_∞ (as given by $E'_0 E'_1 / (E'_0 + E'_1)$) represented by the two springs in series. At high strain rates, the deformation is again elastic with a modulus E'_0 represented by the single spring. At intermediate rates, the strain at time t after a step change in stress is given by the creep compliance function

$$\begin{aligned}\Phi(t) &= \frac{1}{E'_\infty} \phi(t), \\ \phi(t) &= 1 - (1 - \kappa) \exp\left(-\frac{t}{T_1}\right),\end{aligned}\tag{5.1}$$

where $\kappa = E'_\infty / E'_0$ and $T_1 = (1 - \kappa)\eta / E'_\infty$ is the relaxation time of the material. The stress at time t after a step change in strain is given by the relaxation modulus function

$$\begin{aligned}\Psi(t) &= E'_\infty \psi(t), \\ \psi(t) &= 1 - \frac{1 - \kappa}{\kappa} \exp\left(-\frac{t}{T_1}\right),\end{aligned}\tag{5.2}$$

where $T_2 = \kappa T_1$. The relaxation modulus function $\Psi(t)$ is plotted in Figure 5.1. It can be thought of as a time dependent modulus $E'(t)$. Note that creep and relaxation functions are related to one another through the Laplace transform L as the following expression

$$s^2 L[\phi(t)] L[\psi(t)] = 1,\tag{5.3}$$

where s is the Laplace transform variable.

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Figure 5.1: The relaxation function $\Psi(t)$ for the three-parameter-model of linear viscoelastic material.

5.3 Empirical results of elastic adhesion analysis

For the analysis convenience of next sections, the empirical results of incompressible thin film on rigid substrate are summarized here. The model analyzed in Chapter 4 comprises an elastic layer of thickness h , which is perfectly bonded to a half-space substrate. The Young's modulus and Poisson's ratio of the thin layer and substrate are E_1 , E_2 and ν_1 , ν_2 respectively. The thin layer is loaded normally by a rigid frictionless spherical surface of radius R . The contact radius under the action of a compressive (positive) load P is a and the corresponding central deflection is δ . For the layered system, the net load and central deflection are expressed as

$$P = P_0 - P_a = \frac{4E'_1 a^3}{3R} F(x, k) - \sqrt{8\pi E'_1 w a^3} H(x, k), \quad (5.4)$$

$$\delta = \delta_0 - \delta_a = \frac{a^2}{R} B(x, k) - \sqrt{\frac{2\pi w a}{E'_1}} D(x, k), \quad (5.5)$$

where $x = a/h$, $k = E'_2/E'_1$, and $E'_i = E_i/(1 - \nu_i^2)$, $i = 1, 2$ is the plane-strain Young's modulus. The functions $F(x, k)$, $H(x, k)$, $B(x, k)$ and $D(x, k)$ are evaluated using a finite element method. The empirical expressions for functions $F(x, k)$ and $H(x, k)$ have been obtained by analyzing the numerical results. For the special case of incompressible film

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($\nu_1 \geq 0.49$) on rigid substrate, the mismatch parameter k is replaced by the Poisson's ratio of thin film ν_1 . The functions $F(x, k)$ and $H(x, k)$ change to $F(x, \nu_1)$ and $H(x, \nu_1)$, which are given as follows

$$F(x, \nu_1) = 1 + \left[\frac{8}{x(1+x)^{1.5}} + \frac{6.8(1-2\nu_1)}{x} \right]^{-1}, \quad (5.6)$$

$$H(x, \nu_1) = 1 + \left[\frac{3.8}{x\sqrt{1+x}} + \frac{2.1\sqrt{1-2\nu_1}}{\sqrt{x}} \right]^{-1}. \quad (5.7)$$

In non-dimensional form, equations (5.4) and (5.5) are given by

$$\bar{P} = \bar{a}^3 F(x, k) - \sqrt{2\bar{a}^3} H(x, k), \quad (5.8)$$

$$\bar{\delta} = \bar{a}^2 B(x, k) - \frac{2}{3} \sqrt{2\bar{a}} D(x, k). \quad (5.9)$$

For $x \ll 1$, the deformation is confined to the thin film layer and $F(x, k)$ and $H(x, k)$ each approaches unity. It follows that equation (5.8) reduces to the JKR relation in dimensionless form given by

$$\bar{P} = \bar{a}^3 - \sqrt{2\bar{a}^3}. \quad (5.10)$$

The results of numerical version JKR theory for incompressible film on rigid substrate will be extended to viscoelastic film in the following sections. Firstly, the adhesionless contact of viscoelastic thin film is considered.

5.4 Adhesionless analysis

Conveniently, the analysis of loading/unloading contact cycle is separated into two phases i.e. closing and opening contacts, where closing contact corresponds to the contact process during which the contact size increases from zero to maximum value; whereas opening contact corresponds to the contact process during which the contact size decreases from maximum value to zero.

5.4.1 Closing contact

The problem of adhesionless contact on viscoelastic half-space has been considered by Ting [39] and Graham [41]. Johnson [27] has summarized the works of Ting and Graham in his contact mechanics monograph. The simplest solution methodology is to apply the correspondence principle of viscoelasticity, namely, there is a one-to-one correspondence between the stresses in the contact zone in the actual viscoelastic problem and in a reference elastic problem. If the deformation history is known, the load is found by replacing E' in the elastic solution by the integral operator, expressed in terms of the relaxation function $\Psi(t)$. On the other hand, if the load history is known, the variation in deformation is found from the elastic solution by replacing the constant $1/E'$ by the integral operator involving the creep compliance function $\Phi(t)$. For the adhesionless half space indentation, the fully elastic solution is given by the Hertz theory, which is expressed as

$$P = \frac{4E'a^3}{3R}. \quad (5.11)$$

The correspondence principle of viscoelasticity applied to the Hertz theory gives the variation of contact size $a(t)$ with load $P(t)$ to be

$$a^3(t) = \frac{3R}{4} \int_0^t \Phi(t - \tau) \left[\frac{dP(\tau)}{d\tau} \right] d\tau, \quad (5.12)$$

in which the load history is known.

For the adhesionless contact of viscoelastic thin film system, the treatment here is equivalent. The effects of stress relaxation can be accounted for by defining an elastic load $P^{el}(t)$, which depends only on the instantaneous modulus and the current value of the contact radius. The expression for this adhesionless load is identical in the form to the expression for the adhesionless part (P_0) of load given previously in equation (5.4)

$$P^{el}(t) = \frac{4E'_1 a^3(t) F(x(t), \nu_1)}{3R}. \quad (5.13)$$

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Incremental changes in the contact radius produce incremental changes in the elastic modulus, which is represented by the non-dimensional function $F(x(t), \nu_1)$. They also give rise to incremental changes in the elastic load. These contributions to the load then relaxation with time are described by the relaxation function $\Psi(t)$. The overall load is obtained as a superposition of the loads imposed at different loading times. This gives rise to the overall adhesionless load, which may be expressed in terms of a convolution integral as given by

$$\begin{aligned} P(t) &= \int_0^t \Psi(t - \tau) \frac{\partial}{\partial \tau} [P^{el}(\tau)] d\tau \\ &= \int_0^t \Psi(t - \tau) \frac{\partial}{\partial \tau} \left[\frac{4a^3(\tau)F(x(\tau), \nu_1)}{3R} \right] d\tau. \end{aligned} \quad (5.14)$$

Because $\Phi(t)$ and $\Psi(t)$ are related one another by equation (5.3), equation (5.14) can be inverted to give the following expression for contact radius $a(t)$

$$a^3(t)F(x(t), \nu_1) = \frac{3R}{4} \int_0^t \Phi(t - \tau) \left[\frac{\partial P(\tau)}{\partial \tau} \right] d\tau. \quad (5.15)$$

5.4.2 Opening contact

The method provided in the previous section is only applied to the contact problem in which the contact area is increasing throughout during the loading/unloading process. For the opening contact, at time t , Ting [40] showed that the load is independent of the contact history for the intermediate times between $t_1(t)$ and t and only depends upon the variation of contact size prior to $t_1(t)$, where $t_1(t)$ is identified as that previously when the contact size $a(t_1)$ is increasing and equal to $a(t)$. Hence equation (5.14) can still be used to find contact load at time t , by making the limits of integration between 0 and $t_1(t)$, i.e.

$$\begin{aligned} P(t) &= \int_0^{t_1(t)} \Psi(t - \tau) \frac{\partial}{\partial \tau} [P^{el}(\tau)] d\tau \\ &= \int_0^{t_1(t)} \Psi(t - \tau) \frac{\partial}{\partial \tau} \left[\frac{4a^3(\tau)F(x(\tau), \nu_1)}{3R} \right] d\tau. \end{aligned} \quad (5.16)$$

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Inverting equation (5.16) gives the contact radius $a(t)$ at controlled load $P(t)$ as

$$a^3(t)F(x(t), \nu_1) = \frac{3R}{4} \int_0^{t_1(t)} \Phi(t - \tau) \left[\frac{\partial P(\tau)}{\partial \tau} \right] d\tau. \quad (5.17)$$

5.4.3 Contact cycle

Herein, the loading/unloading contact cycle of incompressible viscoelastic thin film ($\nu_1 \approx 0.5$) on rigid substrate is studied. For the contact cycle test, a controlled load is assumed, i.e. the loading history $P(t)$ is known. As an example, a controlled sinusoidal load increasing from zero to a maximum P_m and then decreasing to zero according to

$$P(t) = P_m \sin\left(\frac{t}{T_{Pm}}\right) \quad (5.18)$$

is considered, where T_{Pm} is the loading cycle time. For the closing contact, the contact size is given by substituting the creep compliance function of equation (5.1) and the load history of equation (5.18) into equation (5.15). When the loading cycle time T_{Pm} and the relaxation time of the material T_1 both equal to a time constant T , the contact area is given by

$$\frac{a^3(t)F(x(t), \nu_1)}{a_{Pm}^3 F(x_{Pm}, \nu_1)} = \frac{1}{2} \left\{ (1 - \kappa) \left[\exp\left(-\frac{t}{T}\right) - \cos\left(\frac{t}{T}\right) \right] + (1 + \kappa) \sin\left(\frac{t}{T}\right) \right\}, \quad (5.19)$$

where a_{Pm} is the static contact size under load P_m . This relationship only holds up to the maximum value of $a(t)$, which occurs at $t = T_{am} \approx 0.66\pi T$ for $\kappa = 1/4$. For the opening contact ($t > T_{am}$), the load $P(t)$ depends upon the contact load history up to time t_1 only, where $t_1 \leq T_{am}$, and t_1 is governed by $a(t_1) = a(t)$. To find t_1 , equation (5.16) is used for the load. Since the load variation is known from equation (5.18), the value of t_1 corresponding to any given time t can be found by solving the following equation numerically

$$P_m \sin\left(\frac{t}{T_0}\right) = \int_0^{t_1(t)} \Psi(t - \tau) \frac{\partial}{\partial \tau} \left[\frac{4a^3(\tau)F(x(\tau), \nu_1)}{3R} \right] d\tau. \quad (5.20)$$

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Since the interval of integration of above equation lies within closing contact period, the contact radius $a(t)$ may be found from equation (5.19). Using the empirical equation (5.6) as the function $F(x, \nu_1)$, computations have been performed for the three-parameter incompressible viscoelastic thin film ($\nu_1 = 0.4999$) on a rigid substrate with $\kappa = 1/4$. The variation of contact radius a/a_{pm} through the cycle is shown in Figure 5.2 for different values of cycle time T_{pm} .

Figure 5.2: Variation of contact radius with load for the adhesionless contact cycle with a sinusoidal controlled load, $\kappa = 1/4$.

As another example, the cycle of controlled load increasing and decreasing according to the linear function

$$P(t) = \begin{cases} Vt, & t < T_{pm} \\ 2P_m - Vt, & t > T_{pm} \end{cases} \quad (5.21)$$

is considered, where $V = P_m/T_{pm}$. The calculation can be carried in a similar way as described above. The variation of contact radius a/a_{pm} through the cycle is shown in Figure 5.3 for different values of cycle time T_{pm} .

From Figures 5.2 and 5.3, it is observed that the contact size increases with increasing load. However, the maximum contact size does not coincide with the maximum load. The contact area continues to grow by creep even when the load starts to decrease. At a later stage in the loading cycle, the contact radii rapidly shrink to zero when the load is finally removed. It is also noted that the time T_{am} , when the contact area equals the maximum a_m ,

Figure 5.3: Variation of contact radius with load for the adhesionless contact cycle with a linear controlled load, $\kappa = 1/10$.

is different for different values of cycle time T_{pm} . This asymmetry in the cycle reflects the energy dissipation, which is a maximum when the cycle time T_{pm} is the same order as the relaxation time T_1 . Johnson [85] made a similar observation for the case of indentation of bulk viscoelastic half-space by a rigid sphere.

5.5 Adhesion analysis

The contact problems between bulk viscoelastic materials in the presence of adhesion force have been considered by Schapery [82] and Hui et al [83] for closing contact and Lin [84] for opening contact. Later Johnson [85, 86] provided an analysis method to extend the elastic JKR theory of adhesive contacts to the case of linear viscoelastic by introducing an apparent surface energy factor. Recently Greenwood [88] has carried out a more exactly analysis for the opening and closing contacts.

The infinite tension at the crack-tip in the JKR model leads to difficulties in viscoelastic analysis since it corresponds to an infinite strain rate for a moving contact boundary. The crack tip singularity has been removed in the contact problem by Maugis [10], by using the Dugdale crack in an elastic-plastic problem as an analogy. As shown in Figure 5.4, a tensile cohesive traction σ_0 is assumed to exist over a region of width d outside

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Figure 5.4: The steady-state contact (closing and opening) of the viscoelastic system with a Maugis-Dugdale distribution of adhesive traction.

the contact radius a . Within the ‘process zone’ d , the separation of the surfaces is changing from zero to h_0 , where h_0 is related to the surface energy by $w = \sigma_0 h_0$. The width d of the process zone or Dugdale zone is given by

$$d \cong \frac{\pi E' h_0^2}{4 w}. \quad (5.22)$$

If the cohesive zone is very small in comparison with the overall size of the contact zone, i.e. $d/a \ll 1$, the JKR equations apply very well.

In the case of the elastic JKR theory, the elastic energy (G) needed for steady-state crack is exactly equal to the surface energy w and is the same for closing and opening phases. Whereas, in the case of viscoelastic problem, the energy required for crack opening is more than crack closure due to viscoelastic dissipation. A precise definition of critical strain-energy release rate is not possible, due to its dependence on the rate of loading and unloading (V). Hence, Johnson [85] introduced an apparent surface energy factor β to account for the energy dissipation. The apparent surface energy factor is defined for the closing crack and opening crack as

$$\beta_{cl} = \frac{G_{cl}}{w} = 1 - q_{cl}(V) \leq 1, \quad (5.23)$$

$$\beta_{op} = \frac{G_{op}}{w} = \frac{1}{1 - q_{op}(V)} \geq 1. \quad (5.24)$$

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In the above definition equations (5.23) and (5.24), it is assumed that a fraction $q_{cl}(V)$ of the surface energy w and a fraction $q_{op}(V)$ of the elastic energy G are dissipated in the closing and opening crack phases, respectively.

In the purely elastic JKR theory, the relationship between load and contact size in non-dimensional form is given by equation (5.10). In the viscoelastic case, the surface energy is scaled by the apparent surface energy factor β . Hence the expression for the net load becomes

$$\bar{P} = \bar{a}^3 - \sqrt{2\beta\bar{a}^3}. \quad (5.25)$$

Schapery [82] provided the approximately expressions of apparent surface energy factor for closing contact and opening contact as

$$\beta_{cl} = \phi(t^*/T_1), \quad (5.26)$$

$$\beta_{op} = \frac{1}{\phi(t^*/T_1)}. \quad (5.27)$$

In the case of constant loading and unloading rates as given by equation (5.21), differentiating equation (5.25) with respect to non-dimensional contact radius $\bar{a}$ leads to the differential equation

$$\frac{d\beta}{d\bar{a}} = 3\sqrt{2\bar{a}\beta} - \frac{3\beta}{\bar{a}} - \sqrt{\frac{2\beta}{\bar{a}^3}} \frac{A}{v}, \quad (5.28)$$

where $A = (\bar{P}_m T_1 / T_{\bar{P}_m}) / (36Rw^2 / \pi h_0^3 E'_\infty)^{2/3}$ is the loading rate parameter and v is the non-dimensional peeling speed corresponding to the representative time t^* , which can be found from

$$\frac{t^*}{T_1} \phi\left(\frac{t^*}{T_1}\right) = \frac{d_0}{3VT_1} \equiv \frac{1}{v}. \quad (5.29)$$

Equations (5.28) and (5.29) can be solved simultaneously by numerical integration to find the variation of β and $\bar{a}$ for the whole contact cycle and hence. From equation (5.25) the variation of contact size with load can be determined.

For the case where an incompressible viscoelastic thin film is applied to a rigid substrate, the similar analysis method can be used. The equivalent interface fracture problem

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with a Maugis-Dugdale distribution of adhesive traction is shown in Figure 5.4 with the viscoelastic film of thickness h . By introducing the apparent surface energy factor β into the relationship of load and contact radius for thin film system, equation (5.8) becomes

$$\bar{P} = \bar{a}^3 F(x, k) - \sqrt{2\beta\bar{a}^3} H(x, k). \quad (5.30)$$

For the thin film system, the thickness of the film strongly influences the apparent surface energy. Huntley [109] examined the effect of the layer thickness h on apparent surface energy factor β for the opening contact. By means of a double integral involving the creep compliance and stress distribution along the film-substrate interface, Huntley obtained the approximate expression for effective specimen compliance and further showed that the apparent surface energy factor given for bulk viscoelastic solids may be modified, by including the thickness of the layer, approximately to become

$$\beta_{op} = \frac{\phi[\bar{h}\phi(t^*)t^*]}{\phi(t^*)}, \quad (5.31)$$

where $\bar{h} = h/d_0$, and d_0 is the length of Dugdale zone at zero peeling speed. Similarly, the apparent surface energy factor for closing contact maybe expressed as

$$\beta_{cl} = \frac{\phi(t^*)}{\phi[\bar{h}\phi(t^*)t^*]}. \quad (5.32)$$

It should be pointed out that the thickness h of thin viscoelastic thin film must be much greater than the maximum Dugdale zone length d_0/κ , i.e. $\bar{h} \gg 1/\kappa$. Using equations (5.29), (5.31) and (5.32), the variation of β_{cl} and β_{op} with non-dimensional peeling speed v is plotted in Figure 5.5 for different values of thickness parameter $\bar{h}$.

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Figure 5.5: variation of apparent surface energy factor β with non-dimensional peeling speed v for $\kappa = 1/10$.

Differentiating equation (5.30) with respect to $\bar{a}$ leads to the differential equation

$$\begin{aligned}
 d\bar{P}/d\bar{a} &= 3\bar{a}^2 F(x, k) + \left(\frac{9\pi\alpha^2}{8} \right) \bar{a}^3 \frac{\partial F(x, k)}{\partial x} - 3 \sqrt{\frac{\beta\bar{a}}{2}} H(x, k) \\
 &\quad - \sqrt{\frac{\bar{a}^3}{2\beta}} H(x, k) \frac{d\beta}{d\bar{a}} - \left(\frac{9\pi\alpha^2}{8} \right) \sqrt{2\beta\bar{a}^3} \frac{\partial F(x, k)}{\partial x} \\
 &= \frac{A}{v}.
 \end{aligned} \tag{5.33}$$

The simultaneous integration of equations (5.29) and (5.33) step-by-step gives the variation of apparent surface energy factor β with non-dimensional contact radius $\bar{a}$. Hence the variation of non-dimensional contact radius $\bar{a}$ with non-dimensional load $\bar{P}$ can be obtained by using equation (5.30). During closing contact $\beta = \beta_{cl}$ given by equation (5.32), while during opening contact $\beta = \beta_{op}$ by equation (5.31).

In a typical experiment such as nanoindentation, the commonly measured relation is the load-displacement rather than the load-contact size. As such, it is worthy to consider the load-displacement relation of the viscoelastic system during closing/opening contact cycle. For the purely elastic thin film system, the central displacement is given by equation (5.9). After being scaled by the apparent surface energy factor β , central displacement

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becomes

$$\bar{\delta} = \bar{a}^2 B(x, k) - \frac{2}{3} \sqrt{2\beta\bar{a}} D(x, k). \quad (5.34)$$

Having determined the variation of contact size with load previously, combination equations (5.30) and (5.34) directly gives the variation of displacement against the load.

The closing/opening contact cycles are calculated for incompressible viscoelastic thin film ($\nu_1 = 0.4999$) on rigid substrate for $\kappa = 1/10$. The controlled load is linear type based on equation (5.21). The variation of non-dimensional contact radius versus load is shown in Figure 5.6; and non-dimensional load versus displacement is shown in Figure 5.7.

In the calculations of loading/unloading cycles, the simultaneous integration of equations (5.29) and (5.33) is separated into two phases: closing contact and opening contact. In the closing contact phase, the integration has to be carried out in two steps. Firstly, it is integrated from the initial contact ($\bar{P} = 0$) of the indenter and thin film to the maximum load ($\bar{P} = \bar{P}_m$, the contact radius is $\bar{a}_{Pm}$). The initial condition for the first step needs to analyze the variation of apparent surface energy factor β with non-dimensional peeling speed v (Figure 5.5). It is noted that the reduction in the thickness of the viscoelastic layer attenuates the work of adhesion and, at high non-dimensional peeling speeds, causes a decrease of adhesion with speed. Initially increasing speed brings about an increasing volume of material into the high dissipation regime. This process continues until it is blocked by the rigid substrate. The dissipation regime cannot be wholly accommodated within the layer and is progressively excluded at higher speed. The work of adhesion then decreases with speed, during which the peeling is basically unstable. The initial condition becomes $\bar{a}(\bar{P} = 0) = (2\beta_{\min})^{1/3}$, where $\beta_{\min}$ is the minimum value of the apparent surface energy for a given thickness parameter $\bar{h}$. Secondly, it is integrated from previously obtained value $\bar{a}_{Pm}$ to maximum contact radius $\bar{a}_m$. In this step, because the load begins to decrease from the maximum value, the loading parameter A needs to change the sign. In the opening contact process, the integration can be directly carried out using the initial condition $\bar{a}(\beta_{op}) = \bar{a}_m$.

In Figures 5.6 and 5.7, the dash lines are the reversible relationship for elastic thin

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Figure 5.6: The variation of non-dimensional contact radius with load for closing/opening contact cycle of incompressible viscoelastic thin film on rigid substrate, where adhesion parameter $\alpha = 1.0$ and thickness parameter (a) $\bar{h} = 100$, (b) $\bar{h} = 1000$.

film corresponding to the adhesion parameter $\alpha = 1.0$. For the closing/opening contact cycle of incompressible viscoelastic thin film, the finite load adhesion hysteresis is clearly visible. When the surfaces first touch, they snap into contact at an initial contact size. With increasing indentation load the contact area grows and the value is less than that of the elastic thin film since $\beta_{cl} < 1$. When the load begins to reduce, the contact size continues to grow slightly until the surface begin to separate at the higher apparent surface energy associated with an opening contact. The contact size then decreases continuously

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Figure 5.7: The variation of non-dimensional displacement with load for closing/opening contact cycle of incompressible viscoelastic thin film on rigid substrate, where adhesion parameter $\alpha = 1.0$ and thickness parameter (a) $\bar{h} = 100$, (b) $\bar{h} = 1000$.

until pull-off occurs at an enhanced pull-off force. This property for the contact cycle of incompressible viscoelastic thin film on rigid substrate is the same with that for the bulk viscoelastic material described by Johnson [86]. The variation of enhanced non-dimensional pull-off force with loading parameter A is shown in Figure 5.8. For a given elastic adhesion parameter α , the pull-off force increases with loading rate parameter A and its value strongly depends upon the size of the assumed cohesive zone. To make sure that the differential equation (5.33) is solvable, the loading rate parameter A is limited up

Figure 5.8: The variation of non-dimensional pull-off force with loading rate parameter for incompressible viscoelastic thin film on rigid substrate.

to a special value ($A = 60$ and 250 for $\bar{h} = 100$ and 1000 respectively). It is noted that when thin film thickness is smaller compared with the length of Dugdale zone (i.e. $\bar{h}$ is small), the upper limit of loading rate parameter is also smaller.

5.6 Conclusion

The adhesionless and adhesion loading/unloading contact cycles of viscoelastic thin film are investigated by extending the empirical results of elastic thin film. The stiffness effect of the substrate is considered by the introduced non-dimensional adhesionless force and adhesion force. The load controlled adhesion experiment has been modelled by applying the results of fracture mechanics to the motion of the edge of circular contact region. An increase of contact size during loading and partial unloading process corresponds to closing contact, while a decrease of contact size during unloading process corresponds to opening contact.

For contact in the absence of adhesion, both controlled sinusoidal load and linear load are considered, separately. The closing contact of the cycle, i.e. during which the contact size is increasing, is straightforward to analysis by using viscoelastic correspondence principle. The analysis of opening contact, during which contact size is decreasing, is

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more difficult, whereas the load of opening contact is only dependent upon the closing contact history up to a time, at which the contact size is equal to the contact size of opening contact. The variation in contact radius versus time through such a ramp cycle is calculated. During the closing contact, the thin film system needs shorter time than bulk viscoelastic material to reach the same contact radius. This is because of the stiffness effect of elastic substrate. The contact size increases with increasing load. However, the maximum contact size does not coincide with the maximum load. The contact area continues to grow by creep even when the load has begun to decrease. For different values of cycle time, the time needed for the contact size to be maximum, is different. This asymmetry in the cycle reflects the energy dissipation, which is a maximum when the cycle time is the same order as the relaxation time.

For the contact in the presence of adhesion, both loading and unloading take place at a uniform rate. Johnson's non-dimensional adhesion analysis method for bulk viscoelastic spherical contact is extended to thin film viscoelastic system by considering the stiffness effect of substrate and introducing the apparent surface energy factor. The non-dimensional load-contact radius relation and load-displacement relation are calculated. Because of the adhesion force, the surfaces snap into contact at a finite initial contact radius. With increasing load, the contact size also increases and the value is smaller than that of the elastic solution. When the load begins to decrease, the contact size still grows slightly, and then decreases continuously, during which the value of contact size is greater than that of the elastic solution. The pull-off forces needed to separate the contact surfaces are enhanced based on different loading rate parameter.

Chapter 6

Spherical Indentation of Elastic Thin Film Applied on Plastic Substrate

6.1 Introduction

Thin films are used in applications such as protective surfaces in artificial joints, underfills in flip chip technology and coatings in optical equipment [58, 110–113]. In these applications, the films are made of special substances with properties which are different from the bulk material and can therefore be used to tailor many surface properties of the workpiece in a controlled fashion. It is noted that the protective coatings are normally harder than the protected bulk materials. On the other hand, processes such as nitriding and carburizing produce stiff elastic layers on a bulk plastic ferrous steel. When subjected to loading, the substrate may undergo a plastic deformation before the onset of any other failure. A good understanding of the plastic behavior is essential for improving the reliability and lifetime of thin film systems and devices based on thin film technologies.

To investigate the mechanical properties of a thin film system, the indentation test based on the contact mechanics is commonly used. Much work has been done in finding analytical and numerical solutions of various contact problems. Analytical solutions to different mechanical contact problems have been summarized by Johnson [27]. Sen et al [72] have studied a bilinear elastic-plastic contact problem involving a layered half-space indented by an elastic sphere using the finite element method. They investigated the differences in the elastic and elastic-plastic solutions of the contact pressure distribution for various layer thicknesses. Souza et al [73] analyzed the stresses, fracture and delamination during the indentation of hard elastic films on elastic-plastic soft substrates.

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The results indicated that the suppression of the constraint imposed by the interface resulted in a decrease in the stresses in the film. These studies focused mainly on the stress distribution, but the initiation of plastic yielding or load-displacement relation are not investigated. Recently, Mesarovic and Fleck [49, 50] simulated the contact of similar and dissimilar elastic-plastic spheres using the finite element method and constructed the indentation maps with axes of contact radius and yield strain. Their work did not further consider in the presence of thin film coating however. In the micro or nano force scale, adhesion force between the indenter and thin film may contribute to the deformation. To understand the action of adhesion force, the load-displacement or load-contact radius relation is necessary [22, 78, 79].

In this chapter, the finite element method is used to perform an accurate numerical study of the normal indentation of a stiff elastic thin film on a Ramberg-Osgood hardening elastic-plastic substrate by a rigid sphere. The effects of elasticity and yield strain of the substrate on the indentation mechanism are investigated. The indentation force and depth as a function of contact size in non-dimensional form for a series of yield strain are calculated. The initiation of plasticity in the substrate is examined. The indentation map is constructed to display the competing regimes of deformation mode of substrate, namely the elastic, elastic-plastic and plastic finite deformation modes.

6.2 Statement of the problem

The normal indentation of an elastic thin film applied on an elastic-plastic substrate by a frictionless rigid sphere is considered. It is assumed that the thin layer is perfectly bonded to the substrate and the layered half-space is sufficiently tough for indentation to proceed without crack formation or delamination. The geometric parameters associated with the spherical (Brinell) indentation are defined in Figure 6.1. A rigid sphere of radius R , loaded by a normal force P , indents the semi-infinite layered medium to a depth δ , corresponding to a contact radius a . It is further assumed that the thin film is a homogenous isotropic elastic solid, with Young's modulus E_1 , and Poisson's ratio ν_1 . The substrate is a homoge-

Figure 6.1: Indentation geometry of layered elastic-plastic solid.

nous isotropic elastic-plastic solid, with Young's modulus E_2 , and Poisson's ratio ν_2 . The plastic behavior of the substrate is governed by the Ramberg-Osgood deformation theory.

In one dimension, the hardening model of Ramberg-Osgood is described by the relation

$$\frac{\epsilon}{\epsilon_0} = \frac{\sigma}{\sigma_0} + \alpha \left(\frac{\sigma}{\sigma_0} \right)^n, \quad (6.1)$$

where $\alpha = 3/7$ is the yield offset (in the sense that, when $\sigma = \sigma_0$, $\epsilon_0 = (1 + \alpha)\sigma_0/E_2$) and n is the strain hardening exponent. The yield strength is defined by σ_0 and the yield strain by ϵ_0 . When $n = 1$, the constitutive law becomes fully elastic; when $n \rightarrow \infty$, the constitutive law stands for the elastic-ideally plastic and when n is in the transition range of above two limits, the stress-strain relation of constitutive law is shown in Figure 6.2. The uniaxial constitutive relation equation (6.1) is readily generalized to multi-axial stress states using Hooke's law for the linear term and the Mises stress potential and associated flow law for the nonlinear term. The focus of the current study is to obtain a general concept of relationship among different parameters such as $P, \delta, a, R, \epsilon_0$ etc.

6.2.1 The self-similarity solution of bulk elastic-plastic solids

If there is no elastic thin film on the elastic-plastic substrate, with increasing indentation depth δ , Hill et al [114] have showed that the indentation response becomes dominated by plastic flow around the indenter with elastic parameters becoming irrelevant. The non-dimensional contact size a/R and contact pressure $P/(\pi a^2 \sigma_0)$ then depend only upon

Figure 6.2: Ramberg-Osgood plastic hardening law.

non-dimensional depth δ/R and the strain-hardening exponent n . Hill et al [114] further determined that the overall solution has the properties of self-similarity as given by

$$\frac{P}{\pi a^2 \sigma_0} = c \left(\frac{a}{\epsilon_0 R} \right)^{1/n}, \quad (6.2)$$

where the parameter c depends only upon the value of n and the coefficient of friction at the contact interface and must be determined numerically for the nonlinear solids [115]. For the frictionless case of elastic-ideally plastic solid ($n \rightarrow \infty$), the value of c is approximately 3. In this chapter, the solution in the presence of elastic thin film is compared with the similarity solution for the bulk elastic-plastic solids.

6.2.2 The elastic solution for layered solids

If both the thin film and substrate are purely elastic ones, the relations among the non-dimensional load $3P/(4E'_1 \sqrt{R\delta^3})$ or $3PR/(4E'_1 a^3)$, the contact area $a^2/(\delta R)$ and the contact radius a/h are analyzed in Chapter 4. Here, E'_i is the plane-strain Young's modulus defined in terms of Poisson's ratio ν_i as $E'_i = E_i/(1 - \nu_i^2)$. Under the rigid spherical indentation of

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the layered system, the Hertzian contact load may be expressed as

$$F_1(x, k) \equiv F_1\left(\frac{a}{h}, \frac{E'_2}{E'_1}\right) = \frac{3}{4} \frac{P}{E'_1 \sqrt{R\delta^3}}, \quad (6.3)$$

or

$$F_2(x, k) \equiv F_2\left(\frac{a}{h}, \frac{E'_2}{E'_1}\right) = \frac{3}{4} \frac{PR}{E'_1 a^3}, \quad (6.4)$$

and the contact area may be expressed as

$$A(x, k) \equiv A\left(\frac{a}{h}, \frac{E'_2}{E'_1}\right) = \frac{a^2}{\delta R}, \quad (6.5)$$

where $x = a/h$ and $k = E'_2/E'_1$. It is to be noted that the equation (6.3) is related to equation (6.4) by equation (6.5). For thick layer indentations (i.e. a/h is smaller less than about 0.5), the deformation is confined to the surface layer and both $F_i(x, k)$ and $A(x, k)$ approach unity. For thin layer indentations (i.e. a/h is larger), the deformation is sensitive to the substrate, and $F_i(x, k)$ and $A(x, k)$ approach k and unity, respectively. Empirical expressions for $F_2(x, k)$ based on finite element analysis are obtained in Chapter 4 for a combination of film (compressible and incompressible) and substrate (rigid and elastic) systems. When the substrate shows plastic behavior, the solution deviates from the elastic solution. In this chapter, the extent of the deviation will be explored.

6.3 Finite element implementation

The finite element calculations were performed using the commercial finite element package ABAQUS (ABAQUS, HKS Inc., Rhode Island, USA). A typical mesh, comprising axisymmetric 8-noded quadratic elements (CAX8), is shown in Figure 6.3. The rigid contact surface option was employed to mimic the rigid indenter and the effects of finite deformation were included in the formulation. To carry out the finite element analysis for the indentation of a spherical indenter on the layered half-space, the axisymmetric dimensions in the half-space are modelled using the axes of r (direction 1) and z (direc-

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Figure 6.3: Typical element mesh for the indentation of layered elastic-plastic solid. The inset on the left shows the zoomed region underneath the loading spherical probe.

tion 2), respectively. By exploiting the symmetry due to the geometry and loading, only half of the layered media is discretized. Despite the differences in contact behavior of the mid-side and corner nodes, the second-order elements showed better convergence and accuracy than the linear elements. A refined mesh was required for area close to the contact in order to obtain a better resolution.

For the finite element analysis of contact problem, one possible error is due to the discrete increments in contact size. As indentation proceeds, the indentation depth δ increases, but, until the next node makes contact, the contact size a remains the same. Mesarovic and Fleck [49] investigated this effect and found that the middle increment from one contact node to adjacent node coming into contact represents the contact accurately. Herein, this method is used to find the contact size for a given displacement. For the accuracy of contact radius, controlled stepping of displacement was used to ensure at least 500 increments in every calculation.

Preliminary studies showed that the precise outer boundary conditions are unimportant for meshes if an outer artificial boundary with dimensions more than 100 times contact radius is used. Stresses at remote boundaries are negligible. To check the validity of

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the finite element solution, the stress distribution from numerical results obtained for the special case, in which the layer and substrate are occupied by the same elastic material, are checked against the known exact Hertzian solution. The difference between the numerical results obtained using the FEM and the corresponding analytical solution was found to be less than 1.0%. It may be concluded, therefore, that the finite-element mesh used in this work is an acceptable representation of an elastic semi-infinite solid.

6.4 Results and discussion

The finite element analysis was used to simulate the indentation of a layered system by a frictionless rigid sphere. The thin elastic film is assumed to be perfectly bonded to an elastic-plastic half-space. In elastic contact analysis, Johnson [27] showed that the solution can be represented by a single elastic property: the plane-strain elastic modulus $E' (= E/(1 - \nu^2))$. Although the results are reported in terms of E' , the used Poisson's ratio in finite element analysis is 0.3 for both the thin film and substrate.

6.4.1 Load, displacement, contact radius

For the frictionless indentation of a thin film on an elastic-plastic substrate, it is assumed that the thin film is 10 times stiffer than the substrate, i.e. $k(= E'_2/E'_1) = 0.1$. In the elastic contact of the layered system, the load is normally given in non-dimensional form as $3P/(4E'_1 \sqrt{R\delta^3})$ or $3PR/(4E'_1 a^3)$, contact area as $a^2/(\delta R)$ and contact radius as a/h . These non-dimensional groups are also selected here to show the relation among each other. Figures 6.4 and 6.5 shows the variations of non-dimensional load with contact radius. In Figure 6.4, the load is normalized by the displacement, i.e. function $F_1(x, k)$, while, in Figure 6.5, it is normalized by the contact radius, i.e. function $F_2(x, k)$. When the contact radius a/h is small, both functions $F_1(x, k)$ and $F_2(x, k)$ approach unity as the deformation is only confined to the thin elastic film. With further indentation, the substrate comes into effect, and the load begins to decrease (as the substrate is a compliant one when compared

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Figure 6.4: Relation between load and contact radius, where load is normalized by indentation depth, with $k = 0.1$ and (a) $n = 3$, (b) $n = 7$, (c) $n = 200$.

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Figure 6.5: Relation between load and contact radius, where load is normalized by contact radius, with $k = 0.1$ and (a) $n = 3$, (b) $n = 7$, (c) $n = 200$.

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with the thin film), but the normalized load curves first follow the solution of purely elastic layered system till the initial yielding happens in the substrate. The onset of plasticity in the substrate is a strong function of the substrate yield strain ϵ_0 . When E'_2/σ_0 is small, i.e. for a high yield strain value ϵ_0 , the solution is very close to the layered elastic system. Only in the large value regime of indentation depth or contact radius, the solution begins to deviate from the elastic layered one. When E'_2/σ_0 is large, i.e. for a lower yield strain value ϵ_0 , the initial yielding of the substrate is sensed at lower levels of indentation.

For the same yield strain value ϵ_0 , it is noted that the strain hardening exponent n will affect the normalized load, especially when the yield strain is small ($E'_2/\sigma_0 = 10^4, 10^5$). At the same contact radius, the bigger the strain hardening exponent n , the smaller the normalized load. It is easy to understand, because when the strain hardening exponent n is smaller, the strain hardening effect is much more sever (refer to Figure 6.2). After the beginning of initial yield of the substrate, for elastic-ideally plastic solid ($n = 200$), the increased load is only used to deform the elastic layer and to spread the plastic zone. However, for a strain hardening plastic material ($n = 3, 7$), the load is further used for the continuously increasing yield stress.

6.4.2 Contact area

The evolution of the normalized contact area $a^2/(\delta R)$, i.e. the function $A(x, k)$, with contact size a/h for a series values of substrate yield strain (ϵ_0) are plotted in Figure 6.6. For purely elastic layered solids, when the contact radius a/h is small, the function $A(x, k)$ approaches unity. With increasing contact size, function $A(x, k)$ firstly decreases to a minimum value, which depends on the mismatch parameter k , then increases and finally approaches unity for thin film case. It is clear from Figure 6.6 that the case of $E'_2/\sigma_0 = 10$ shows this behavior well when $a/h < 150$. From Figure 6.6, when $a/h \rightarrow 0$, the trend of function $A(x, k)$ for every curve is clear and correct, but the contact radius is not small enough to show the pure effect of the film alone. As noted from the elastic solution, increasing contact radius to around $a/h = 0.8$, the function $A(x, k)$ decreases to a minimum

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Figure 6.6: The variation of non-dimensional contact area $a^2/(\delta R)$ with a/h , where $k = 0.1$ and (a) $n = 3$, (b) $n = 7$, (c) $n = 200$.

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value. This value of a/h corresponds exactly to the solution of purely elastic layered solids for $k = 0.1$. From Figure 6.4, it is to be noted that, at $a/h = 0.8$, the plastic deformation of substrate has already begun for the case $E'_2/\sigma_0 = 10^4$ and 10^5 . Obviously, the plastic deformation will affect the minimum value of $a^2/(\delta R)$, and make it much smaller; but surprisingly it does not affect the corresponding contact radius a/h . The contact radius corresponding to the minimum value of function $A(x, k)$ still only depends on the mismatch parameter k . With further loading, the function $A(x, k)$ increases. However, because of the plastic effect of substrate, the values of $A(x, k)$ will gradually depart from the purely layered elastic solution. At this stage, the deformation of substrate includes both elastic and plastic components, which compete with each other. For the cases $E'_2/\sigma_0 = 10$ and 10^2 , the initial yield points are very clear in the plots; however, for other cases, the transition from elastic behavior to plastic behavior is smooth, i.e. no obvious turning points visible. With the spreading of plastic deformation zone, the plastic finite deformation comes into effect, and the value of normalized contact area $a^2/(\delta R)$ begins to decrease. The finite deformation zone is again strongly dependent on the yield strain of the substrate and contact radius.

The effect of hardening exponent n on the normalized contact radius $a^2/(\delta R)$ seems not clear except for the case of especially low yield strain, i.e. $E'_2/\sigma_0 = 10^5$. At the same contact radius and yield strain, the more severe the strain hardening (i.e. n is small) is, the higher the value of $a^2/(\delta R)$.

6.4.3 Average indentation pressure

For the indentation of bulk elastic-plastic solids, Johnson [33] found the degree of deformation depends upon the representative strain a/R and yield strain ϵ_0 . The degree of indentation can be represented by the non-dimensional parameter $aE'_2/(R\sigma_0)$ [49]. In the elastic stress analysis of homogenous media, the stresses are usually normalized by the maximum pressure of the contact. In the elastic-plastic regime, however, normalization by the yield strength σ_0 is more appropriate. In the similarity solution equation (6.2), the

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Figure 6.7: The variation of average pressure $P/(\pi a^2 \sigma_r)$ with $aE'_2/(R\sigma_0)$, where $k = 0.1$ and (a) $n = 3$, (b) $n = 7$, (c) $n = 200$.

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average indentation pressure can be scaled by the reference stress $\sigma_r = \sigma_0[a/(\epsilon_0 R)]^{1/n}$. It is very clear that when $n \rightarrow \infty$, $\sigma_r \rightarrow \sigma_0$. In this study, the average indentation pressure is normalized with respect to the reference yield strength of the substrate, and its variation with $aE'_2/(R\sigma_0)$ is shown in Figure 6.7 for a series value of yield strain and strain hardening exponent. For the case $E'_2/\sigma_0 = 10^4$, because the values of average pressure $P/(\pi a^2 \sigma_r)$ are too high for smaller values of $aE'_2/(R\sigma_0)$, the data points are not included in this plot. But the trend of those points can be deduced from the other E'_2/σ_0 cases.

In the elastic deformation regime, with increasing of $aE'_2/(R\sigma_0)$, the normalized average pressure $P/(\pi a^2 \sigma_r)$ also increases accordingly. As the contact size is increased further, the average indentation pressure $P/(\pi a^2 \sigma_r)$ continually increases for the cases of higher yield strain; while $P/(\pi a^2 \sigma_r)$ decreases to a plateau value for cases of lower yield strain. Ultimately, as the plastic finite deformation comes into effect, the average pressure increases continually with further indentation process.

The constant plateau stress value mentioned above, which corresponds to a similarity solution, strongly depends on the yield strain ϵ_0 and strain-hardening exponent n . For bulk elastic-plastic solids, the similarity solution c is about 1.1, 1.9 and 3 for the cases of $n = 3$, 7 and 200, respectively, and the similarity solution is independent of the yield strain ϵ_0 . In the layered system considered here, for different cases of yield strain and strain-hardening exponent of the substrate, the similarity deviates from the value c . The similarity solution is only limited to a range of yield strain. There is no similarity solution for the cases with a sufficiently high yield strain or otherwise with a sufficiently low yield strain. In that case, the meaning of ‘similarity solution’ for the layered solids is a little different from that for the bulk elastic-plastic solids. It is more suitable to call the similarity solution for the layered solids as ‘reference similarity solution’.

6.4.4 Load-displacement

Load-displacement relationship is the most important material characteristic during the contacting test using micro or nano prob instruments, such as nanoindenter. The plots

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Figure 6.8: The variation of load with indentation displacement, where $k = 0.1$ and (a) $n = 3$, (b) $n = 7$, (c) $n = 200$.

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of normalized load versus indentation displacement are shown in Figure 6.8 for a series of yield strain and strain hardening exponent. With increasing indentation displacement, the load also increases. For small values of δ/R , the load is a strong function of material mismatch parameter k and the strain hardening exponent n of the substrate. In the finite deformation regime, indentation load is independent of the material mismatch parameter. It is to be noted that an exponent value is used for the yield stress σ_0 , when to normalize the indentation load. The stress exponent is used to account for the effect of elastic thin film. For different values of strain hardening n , the power exponent of σ_0 is a little different.

6.4.5 Indentation map

From the above analysis of normal indentation of a layered solid made up of elastic thin film and elastic-plastic substrate by a frictionless sphere, it is to be noted that there are different regimes of deformation, i.e. elastic (thin film), elastic (substrate), elastic-plastic, similarity and plastic finite deformation. When the indenter tip comes into contact with surface of thin film, it appears that even at very low contact radii, the substrate is significantly influencing the measured load values. Such that the first two regimes are combined together as one elastic regime. The plastic similarity solution value varies for different substrate yield strain and strain hardening exponent. In fact, the plastic similarity solution is a fraction of elastic-plastic deformation process. A map giving the various regimes of deformation is shown in Figure 6.9. The abscissa is the normalized contact size a/R and the ordinate is the yield strain σ_0/E'_2 of the substrate. From the comparison of the indentation map for the cases of $n = 3$ and $n = 200$, it is to be noted that there is only a minor effect of strain-hardening rate on the location of the boundaries of the map.

The map shows the deformation evolution processes in indentation with increasing contact size a/R for any given prescribed value of substrate yield strain σ_0/E'_2 . For example, consider the case of $\sigma_0/E'_2 = 10^{-3}$ and $n = 200$. Elastic deformation of the film and substrate prevails up to a contact radius of $a/R < 1.7 \times 10^{-3}$. With further indentation,

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Figure 6.9: The indentation map of layered elastic thin film/elastic-plastic half space by frictionless sphere, where $k = 0.1$.

plasticity initiates in the substrate and both the elastic and plastic properties of the layered materials contribute to the deformation. This elastic-plastic deformation is noticed within $1.7 \times 10^{-3} < a/R < 0.15$. In partial of this regime ($10^{-2} < a/R < 4 \times 10^{-2}$), the contact pressure remains constant resembling that of a self-similar indentation geometry. Finally, the elastic properties of the film and substrate contribute negligible load to the strain-field beneath the spherical probe. When $a/R > 0.15$ in the finite deformation zone deformation is only controlled by the plasticity of the substrate.

The considerations on how to decide the boundaries for each regime are briefly described as follows. When the Von-Mises stress within the substrate is less than the yield strength σ_0 , the elastic regime pertains. After yielding, the substrate deforms elastic-plastically. The beginning position for plastic finite deformation is found from the non-dimensional contact area Figure 6.6. A reference similarity solution can be found from the average pressure Figure 6.7.

6.4.6 Stress distribution

Figure 6.10 shows the Mises stress distribution for the case $n = 200$, $\sigma_0/E'_2 = 10^{-3}$, where $k = 0.1$ and $a/R = 2 \times 10^{-3}$. From the indentation map Figure 6.9, when $a/R = 2 \times 10^{-3}$, the deformation is in the elastic-plastic regime. Figure 6.10 clearly shows the plastic and elastic regimes in the substrate.

It appears that the initiation of plasticity begins from the layer-substrate interface directly underneath the indenter. When the substrate comes into plastic deformation, the maximum equivalent Mises stress in the thin film is at least 5 times the yield stress σ_0 . Further indentation, the yield zone of substrate will expand gradually. It is also to be noted that the stress distribution is not continuous in the interface between thin film and substrate. In fact, the layer-substrate interface failure or interfacial delamination may occur due to the incontinuity of stress.

Figure 6.10: The distribution of Mises stress under the spherical indenter for the case $n = 200$, $\sigma_0/E'_2 = 10^{-3}$, where $k = 0.1$ and $a/R = 2 \times 10^{-3}$.

6.4.7 The effect of stiffness ratio

In previous analysis, the stiffness ratio of substrate to thin film (k) is set at 0.1. In the following, another value of $k = 0.01$ is considered to analyze its effect to load and contact area. The substrate strain hardening exponent n is assumed to be 7. The normalized load versus contact radius is shown in Figure 6.11. The trend is similar to that of when $k = 0.1$, except that for a given contact radius, the normalized load drops by about 10 times more than that for when $k = 0.1$. Comparing Figures 6.11 and 6.4(b) shows that, for the same yield strain value, the yield point for $k = 0.01$ occurs at a lower a/h than that of $k = 0.1$.

Figure 6.11: Relation between load and contact radius, where load is normalized by (a) indentation depth and (b) contact radius with $k = 0.01$ and $n = 7$.

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The curves of normalized contact radius $a^2/(\delta R)$ versus a/h are plotted in Figure 6.12. The minimum value of $a^2/(\delta R)$ is decided by the stiffness ratio k . It is to be noted that the minimum value of $a^2/(\delta R)$ is comparatively smaller than that obtained for $k = 0.1$ and the corresponding contact radius is $a/h = 0.6$. Once again, the plastic deformation affects the minimum value of $a^2/(\delta R)$ and makes it smaller, but it doesn't change the corresponding position of a/h . Comparing Figures 6.12 and 6.6(b) shows that the finite deformation for $k = 0.01$ occurs at a lower a/h than that of $k = 0.1$.

Figure 6.12: The variation of non-dimensional contact area $a^2/(\delta R)$ with a/h , where $k = 0.01$ and $n = 7$.

The variation of normalized load versus indentation displacement is shown in Figure 6.13 for a series of yield strain. When the indentation comes into the finite deformation regime, the curves collapse together. Comparing Figures 6.13 and 6.8(b), it is to be noted that the exponent value used for the yield stress σ_0 is different for the cases of $k = 0.1$ and $k = 0.01$. Both the strain-hardening exponent n and the stiffness parameter k affect the power exponent of σ_0 .

The indentation map in Figure 6.14, which shows the deformation evolution process from elastic regime to plastic finite deformation, gives a direct comparison for the cases of $k = 0.1$ and $k = 0.01$, when the strain-hardening exponent $n = 7$. The stiffness ratio k has an effect on the boundaries of indentation map. When the thin film is much more

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stiffer compared with the substrate, the plastic initiation and finite deformation occur at lower values of normalized radius a/h .

Figure 6.13: The variation of load with indentation displacement, where $k = 0.01$ and $n = 7$.

Figure 6.14: The indentation map of layered elastic thin film/elastic-plastic half space by frictionless sphere, where $n = 7$.

6.4.8 The effect of friction

In the above analysis, the friction between the indenter and thin film is neglected. It may be of practical significance to consider the frictional effect on the evolution process of plastic deformation. In practical indentation tests, it is difficult to control the degree of friction between indenter and layered half-space. Herein, the totally bonded frictional case is considered to gauge the sensitivity of the indentation response to friction level. Figure 6.15 shows the comparison of non-dimensional load for the frictionless and totally bonded frictional cases, when the strain hardening exponent n is 7. It is to be noted that the friction condition almost has no effect on the indentation load over the entire range of a/h considered. Although only two representative yield strain cases ($E'_2/\sigma_0 = 10^3, 10^4$) are reported here, selected calculations show that the broad conclusions do not change for other values of yield strain E'_2/σ_0 and strain-hardening exponent n .

Figure 6.15: The comparison of indentation load between frictionless and totally bonded frictional cases, where $n = 7$.

6.5 Conclusion

Accurate and detailed finite element studies on the indentation of an elastic thin film bonded to an elastic-ideally plastic substrate are carried out. The Ramberg-Osgood hard-

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ening law is used to model the behavior of the plastic substrate. The simulation results from FE are compared with that of the purely layered elastic system and bulk elastic-plastic solids. The substrate yield strain and strain-hardening exponent have strong influence on the relations for the indentation load, depth and contact radius. Compared with the similarity solution of bulk elastic-plastic solid indentation, there is only limited similarity solution regime, and the value of similarity solution depends on the yield strain and strain hardening exponent. The constructed indentation map plotted in $(a/R, \epsilon_0)$ clearly shows the contribution of various competing mechanisms to the indentation. When the indenter comes into contact with the thin film surface, the layered solid deforms elastically. With increasing indentation depth or contact size, the plasticity of the substrate begins to initiate. Then both the elastic and plastic properties of the substrate contribute to the deformation, and the plastic zone in the substrate expands gradually. Finally, the contribution from the elastic property is negligible, and deformation is controlled by the finite plastic flow. The effect of stiffness ratio on the deformation is analyzed. The stiffer the thin film is, the earlier the plastic initiation and finite deformation come. The friction operating between the indenter and thin film has no effect on the indentation load. The stress distribution is not continuous in the interface between thin film and substrate, which may lead to the layer-substrate interface failure or interfacial delamination.

Chapter 7

Conclusions and Scope for Future Research

7.1 Conclusions

The development of micro-probe instruments for the measurement of surface energy and other material mechanical properties on the micro/nano scale has led to the need for continuum mechanics models of the contact of a spherically tipped probe with the surface of a specimen in the presence of adhesive forces. In this research, the adhesion mechanics of layered thin film system indented by a spherical tip is investigated by extending the JKR theory. A numerical approach based on the finite element (FE) method is presented. The FE calculation effectively takes into account both adhesionless and adhesion contacts.

7.1.1 Elastic three-layer geometry

In the elastic three-layer geometry, the numerical version of JKR theory is used to carry out a specific numerical or theoretical verification for a surface force apparatus (SFA) experimental measurements. SFA comprises thin sheets of molecularly smooth mica glued to cylindrical glass lenses of equal radii, which are then pressed into elastic contact with their axes at right angles. In the SFA measurements, contact radius-load curves with and without adhesion are obtained by treating the mica surfaces. The elastic moduli of mica and epoxy are measured by nanoindentation using spherical indenter. The mica and epoxy thicknesses as well as the contact radius as a function of applied load in adhesive and non-adhesive contact, are measured by optical interferometry at the point of contact. The experimentally measured data, e.g. elastic moduli and thicknesses of mica and epoxy,

are used as input parameters in the finite element simulation. Then the FE results for the variations of contact radius with contact load are compared with the SFA experimental data.

The contact load of FE analysis is separated into adhesionless and adhesion parts, which are connected by the adhesion parameter. For the layered system, its mechanical properties can be represented by the effective modulus, which is a function of the contact size. When the surfaces come into contact, the deformation is small and confined to the mica layer. With increasing indentation size, effective modulus decreases in response to the compliance of the layer of epoxy, until the stiffening provided by the glass substrate becomes significant. Finally, the deformation is dominated by the glass substrate. The effective moduli measured from SFA experiment are in good agreement with that computed by FE simulations.

The SFA measured contact radius-load curves in the presence of adhesion are in reasonable agreement with the theory. However, the loading and unloading curves showed appreciable hysteresis due to the viscoelasticity of the epoxy glue layer.

The pull-off force P_c provides the most direct measurement of the surface energy, and is found to be a function of the adhesion parameter. For the experimental SFA geometry, the FE analysis shows that $P_c = 1.75\pi wR$, which is intermediate to the JKR limit for homogeneous materials ($P_c = 1.5\pi wR$) and the DMT limit ($P_c = 2\pi wR$).

7.1.2 Elastic two-layer geometry

For the elastic two-layer geometry, adhesion between a spherical indenter and thin compliant elastic film applied on a substrate has been investigated. The FE computations have been carried out for three material systems, i.e. (i) a compliant incompressible film on a rigid substrate, (ii) a compliant incompressible film on an elastic substrate, and (iii) a compliant compressible film on an elastic substrate.

During the modeling and calculation, the net load and displacement of indentation are decomposed into non-dimensional adhesionless and adhesion components. The variations

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of contact size a versus load P , load P versus displacement δ for a series of values of adhesion parameter α are computed for the above three layered systems. By analyzing the numerically calculated load and compliance data across the entire range of contact radius, the empirical relations for the compliance and hence for the net load as a function of contact radius are obtained. All these empirical expressions agree very well with FE data.

It is to be noted that, for a thick layer for small indentations ($a/h < 0.5$), deformation is confined to the surface layer and both the non-dimensional load and displacement approach unity irrespective of the material properties (i.e. Young's modulus and Poisson's ratio) and the contact surface properties (i.e. with or without friction). In this regime, the standard JKR theory can be used to represent the variation of the load and contact radius by replacing the elastic properties of the half-space with that of the layer. With increasing the indentation size, the solution deviates from JKR limit in response to the stiffness effect of substrate.

The minimum value of load for a given adhesion parameter represents the pull-off force P_c at which the surfaces snap apart. The pull-off force P_c and contact radius at zero load a_0 are complex functions of adhesion parameter α , thin film Poisson's ratio ν_1 and the stiffness ratio k . The maximum non-dimensional pull-off force $\bar{P}_c$ can be 0.95, which is very different from the JKR limit 0.5. It is believed that this difference is due to the changing distribution of traction and deflection across the contact surface. The effect of the elastic behavior of the indenter is to decrease the value of P_c and a_0 for a given adhesion parameter α .

The effect of friction to the deformation behavior of a layered system is analyzed. To avoid the oscillating singularity, the thin film is assumed to be incompressible. The friction effect on the load and displacement functions is very small except for the thick film to thin film transition region: the non-dimensional adhesionless and adhesion loads are slightly higher than that obtained from the frictionless case and the difference increases with increasing material mismatch parameter value or Poisson's ratio of thin film.

The empirical expressions for load versus contact radius are used to carry out experimental verification from the published researches.

7.1.3 Viscoelastic thin film on rigid substrate

The adhesionless and adhesion contact between the spherical indenter and linear incompressible viscoelastic thin film applied to rigid substrate is analyzed by extending the empirical results of elastic thin film. Most of the viscoelastic materials are incompressible having the Poisson's ratio close to 0.5. The viscoelastic behavior of thin film is modelled by the creep compliance and relaxation modulus functions.

For the adhesionless analysis of viscoelastic thin film, the contact cycle is separated into two phases, i.e. closing contact, during which the contact size increases from zero to maximum, and opening contact, during which the contact size decreases from maximum to zero. The viscoelastic corresponding principle is used to study the closing contact phase. While, the analysis of opening contact is more difficult, the load of opening contact is only dependent upon the closing contact history up to a time, at which the contact size is equal to the corresponding contact size of opening contact. The variation of contact radius versus time for linear and sinusoidal loading/unloading cycles is calculated and compared with the results of bulk viscoelastic material. With increasing load, the contact size increases accordingly. However, the maximum contact size is not coincident with the maximum load. The contact area continues to grow by creep even when the load has begun to decrease. The asymmetry in the cycle reflects the energy dissipation, which is a maximum when the cycle time is the same order as the relaxation time.

Johnson [85] has studied the adhesion contact between a rigid sphere and a viscoelastic half-space by applying the results of fracture mechanics to the motion of the edge of circular contact region under load control. This technique is extended to a viscoelastic film on a rigid substrate. The variation of non-dimensional contact radius versus load is calculated for a series of values of loading rate parameter. Because of the adhesion force, the surfaces snap into contact at a finite initial contact radius. With increasing load, the

contact size also increases and the value is smaller than that of the elastic solution. When the load begins to decrease, the contact size still grows slightly, and then decreases continuously, during which the value of contact size is greater than that of the elastic solution. The pull-off forces needed to separate the contact surfaces are enhanced based on different loading rate parameter. The hysteresis loop decreases with increasing loading rate.

7.1.4 Elastic thin film on plastic substrate

Hard and stiff thin films are deposited on to soft ductile substrates to improve the tribological performance. When subjected to a normal load, the substrate may undergo plastic deformation before any other failure triggers. A finite element method is used to perform an accurate numerical study of the normal indentation of an elastic stiff film on a Ramberg-Osgood hardening elastic-plastic substrate by a rigid sphere. The solution is compared with the similarity solution of the bulk elastic-plastic solids and purely layered elastic solids. The effects of yield strain, strain hardening of the substrate, and film to substrate modulus ratio on the indentation response are explored. The indentation force and depth as a function of contact size in non-dimensional form for a series of yield strains and strain hardening are calculated. The evolution process of layered solids from elastic deformation to finite plastic deformation is shown as indentation map. When the indenter comes into contact with the thin film surface, the layered solid deforms elastically and follows the solution of purely elastic-layered solid. With increasing indentation size, the plasticity of substrate comes into effect, and the solution deviates from the elastic layered one gradually. For small yield strain and/or large strain hardening exponent values, the plasticity of the substrate is observed at lower indentation depth. The similarity solution is only limited to a special range of substrate yield strain values. The stiffer the thin film is, the earlier the plastic initiation and finite deformation come. The stress distribution is not continuous in the interface between thin film and substrate, which may lead to the layer-substrate interface failure or interfacial delamination.

7.2 Key contributions from the author

The JKR theory was successfully extended to the adhesion analysis of elastic layered thin film system. The empirical equations for contact load versus contact area, contact compliance as a function of contact radius for different combinations of layered elastic systems were deduced based on the finite element simulation results. These empirical equations can be directly used by experimentalists to fit in their experimental measurements of contact load and area. The author has demonstrated the use of the empirical equations by comparing published experimental results with the theory.

The empirical results of the elastic thin film system were further extended to the viscoelastic thin film applied on a rigid substrate. This method provided a possible solution to analyze the adhesion problem of viscoelastic thin film. Contact load versus contact area curves for loading and unloading stages are obtained for a range of loading rates.

An indentation map for an elastic thin film on a plastic substrate system was constructed in the space of contact radius and substrate yield strain. The indentation map provides a clear representation of elastic, elastic-plastic and finite strain deformation regions during the indentation process. The effect of substrate yield strain and strain - hardening on the indentation map are explored.

7.3 Suggestion for further research work

In this thesis, the adhesion mechanics between a spherical probe and layered solids is comprehensively studied. The considered models include purely elastic three-layer and two-layer systems, incompressible viscoelastic thin film applied to rigid substrate, elastic harder thin film on elastic-plastic substrate. Despite the models and results reported in the literature and this thesis, there are many open issues in modeling the adhesion of layered solids. A few of these issues are discussed in this section.

In the adhesion study of elastic layered solids, the considered thin films are compliant than the substrate and indenter curvature is large. Both of these assumptions justify the

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usage of the JKR theory. But, when the elastic stiffer thin films are deposited onto the elastic substrate, it will violate JKR assumptions and the DMT theory should be considered. DMT theory applies for hard solids featuring low surface energies and spheres with small curvature. Maugis-Dugdale theory forms an intermediate bridge to connect the JKR theory and DMT theory. In JKR theory, the adhesion part of net load can be represented by force due to the flat punch indenter. However, for DMT and Maugis-Dugdale theories, the possible expressions for adhesion force are complicated. Layered structures with hard (stiff) films usually develop high residual stresses in compression or tension in the films or abrupt stress changes at the film-substrate interfaces. High or discontinued stresses can significantly affect the stability of the systems depending on the loading conditions.

Indentation size effects (ISE) have been observed for both elastic and plastic indentation in the micro and nano-indentation regimes. It is often observed that hardness increases as indentation size decreases, even for tests of homogeneous materials. This problem has been attributed to fundamental characteristics of the material. Traditional continuum mechanics theories are unable to predict this effect and it would be worth noting this inherent drawing back of all these theories, which may be relevant in interpreting the nano and micro-indentation tests. The traditional continuum models can be extended to non-local and other quasi-continuum analyses by include the indentation size effects in the future work.

In the contact analysis (with and without adhesion) of viscoelastic thin film, only the case of incompressible viscoelastic thin film applied on rigid substrate is studied, because two preliminary conditions to use corresponding principle and Johnson's methods are: the film is incompressible and on a rigid substrate. At present, no accurate analytical solution is available for this problem. Through an apparent surface energy factor, the method in this thesis is to extend the empirical solution from elastic layered solid. Another possible method for the adhesion analysis of viscoelastic thin film system is to directly use finite element simulation process, in which the adhesion surface force will be modelled by appropriate cohesive elements. This method can eliminate the limitation of the method

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presented in this thesis. Any combination of thin film and substrate is possible. The cohesive element is introduced recently in ABAQUS version 6.5 to model the behavior of adhesive joints, interfaces in composites, and other situations where the integrity and strength of interfaces may be of interest. The constitutive behavior modelling of the cohesive element is the essential for a successful analysis. Despite the constitutive response of cohesive element can use a traction-separation description, the accurate initial response and the succeeding damage evolution law must be chosen with discretion.

The indentation process of stiffer thin film applied on elastic-plastic substrate is analyzed in this thesis. The elastic-plastic behavior of the substrate is modelled by the deformation theory Ramberg-Osgood plasticity model. It is, in fact, a nonlinear elastic model; but at a limit state when all of a specimen or structure is responding plastically, this model is a useful equivalent representation of the plastic response. At present stage, the adhesion surface force operating between the surfaces of indenter and film is neglected. In order to include the adhesion behavior, the unloading process and stress distribution should be analyzed. Mesarovic and Johnson [78] have studied the adhesive contact of elastic-plastic spheres. They assumed that during unloading to the point of pull-off the deformation is predominantly elastic. The contact size and pressure distribution during elastic unloading, in the absence of adhesion, is found by the method of rigid punch decomposition. The distribution of adhesive traction has been found in two ways, i.e. using the singular traction distribution of linear elastic fracture mechanics and using a step cohesive law. However, in the presence of elastic thin film, the deformation behavior of layered solid is complicated. The unloading process and stress distribution should be studied comprehensively.

In the theoretical study of adhesion mechanics, it has been tacitly assumed that the surfaces of contacting bodies are topographically smooth; that the contact surfaces follow precisely the gently curving nominal profiles. In consequence contact between them is continuous within the nominal contact area. The asperities on the surface of very compliant solid may be squashed flat elastically by the contact pressure, so that a perfect

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contact is obtained throughout the nominal contact area. However, contact between solids surfaces is discontinuous and the real area of contact is smaller than the nominal contact area. Such that, the adhesion due to the surface traction between the contacting solids will be much smaller than that predicted by the presented theories. Even though the asperities on a rough surface are randomly arranged, it is advisable to model them as a series of regular waves with Weierstrass surface profile, e.g. a sinusoidal one. A clear analysis should focus on the effect of surface roughness on the indentation measurements. This would provide the plastic analogue to the Greenwood and Williamson [116] elastic analysis, in which surface roughness is idealized as a collection of discrete asperities, with a Gaussian height distribution, and (in the simplest model) identical curvatures.

Another factor that affects the stress distribution and adhesion mechanism in the layered solid is the interfacial bonding. In most of the theoretical analysis, it is assumed that the interfaces between the thin film and substrate are perfectly bonded. In fact, fracture and delamination between the film and substrate are challenging issues in the contact measurement process. It is mainly due to a poor adhesion strength and the accumulation of residual stress in the film due to various process conditions. A good understanding of the interfacial properties is absolutely necessary to consummate the adhesion mechanics of thin film. The detailed chemical analyses of debonded areas or contact surfaces perhaps using EDX would have a great help to understand the adhesion mechanism.

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