Measurement of granular contact forces using frequency scanning interferometry

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Measurement of Granular Contact Forces Using Frequency Scanning Interferometry

By

Mohammad Shahril Osman

A Doctoral Thesis

Submitted in partial fulfillment of the requirements for the award of

Doctor of Philosophy of Loughborough University

December 2002
To

My lovely wife Norhayani Ismail
Abstract

The propagation of stress within a granular material has been studied for many years, but only recently have models and theories focussed on the micromechanical (single grain) level. Experiments at this level are still rather limited in number. For this reason, a system using optical techniques has been developed.

The substrate on which the granular bed is assembled is a double layer elastic substrate with high modulus epoxy constituting the top layer and silicone rubber as the bottom layer. In between the two layers, gold is coated which acts as a reflective film. To design the substrate, a Finite Element Analysis package called LUSAS was used. By performing a non-linear contact analysis, the design of the substrate was optimised so as to give a linear response, high stiffness, deflection in the measurable range, and negligible cross-talk between neighbouring grains. Fabrication and inspection techniques were developed to enable samples to be manufactured to this design.

The deformation of the gold interface layer is measured using interferometry. The interferometer utilised a frequency tunable laser which acts both as the light source and the phase shifting device. The optical arrangement is based on the Fizeau set-up. This has removed several problems such as multiple reflections and sensitivity to vibration that occurred when using a Mach-Zehnder configuration. A fifteen-frame phase shifting algorithm, was developed based on a Hanning window, which allows the phase difference map to be obtained. This is then unwrapped in order to obtain the indentation profile. The deflection profile is then converted to a single indentation depth value by fitting a Lorentzian curve to the measured data.

Calibration of the substrate is carried out by loading at 9 different locations simultaneously. Spatial and temporal variations of the calibration constants are found to be of order 10-15%. Results are presents showing contact force distributions under both piles of sand and under face-centred cubic arrangements of stainless steel balls. Reasonable agreement was obtained in the latter case with both the expected mean force and the probability density function predicted by the so-called ‘q’ model.
The experimental techniques are able to measure small displacements down to a few nanometers. To the best of my knowledge these experiments are the first to employ the interferometer method in attempting to measure the contact force distribution at the base of a granular bed.
Acknowledgements

Firstly, I would like to express my highest gratitude to my supervisors, Prof. J.M Huntley and Dr. R.D Wildman for their guidance, support, patience, discussions, comments and enthusiastic academic support over my time in Loughborough University.

I am also very grateful for the support from my family as well as my friends for their understanding throughout my years of studies in Loughborough.

My highest appreciation towards all the members of the Structural Integrity Research Group, namely Dr. C.R Coggrave, Dr. P Ruiz, Dr. A. Davilla and Dr. T.W Martins for their rewarding discussions and friendship.

I also like to express my thanks towards the Department of Mechanical Engineering and University Malaysia Sarawak (UNIMAS) for the financial support.

Lastly I would like to express my highest appreciation and gratitude to my lovely wife, Norhayani Ismail for her great patience and support throughout my studies.

Mohammad Shahril Osman
Loughborough University
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INTRODUCTION

Granular materials have a range of unique characteristics that are different from either solids or liquids. One of these phenomena is the significant dip at the centre of a conical sandpile which has been shown experimentally to be up to 60% of the height of the pile [1, 2]. For this reason it is interesting to measure the pressure distribution at the bottom of the pile with a spatial resolution comparable to the mean grain diameter, to provide further insight into the behaviour of this type of material. Furthermore, results derived from experiments such as these would provide a greater understanding of the interaction between microscopic and macroscopic laws. The experiments described in this thesis however will use ball bearing as the indenter. The studies is important for theoretical physicist in trying to validate models, experimentalists trying to measure small deflection as well as potentially companies trying to develop an improved powder handling facilities.

The experiments described in this thesis employ an optical technique that is sensitive and is able to measure the grain-wall indentation to accuracy of the order of 1 nanometer. An interferometry technique has been developed based on the Fizeau set-up using a tuneable wavelength laser as a phase-shifting device. The set-up has the advantage that it is a common path interferometer which is less susceptible to environmental error with a good signal to noise ratio.

The substrate developed for this particular experiment is a double-layer elastic substrate with gold film evaporated in between the layers as a reflective mirror. The top layer is composed of a relatively high modulus epoxy and the bottom layer of a low modulus clear silicone rubber. To optimise the substrate, it is essential to design a sample that will match key criteria specified below, involving combinations of material elastic properties and thickness.

A Finite Element Analysis package is utilised in order to model the substrate. The package is a windows-based software programme called LUSAS. The design of the substrate was modelled on several criteria. Amongst these criteria are ensuing a linear force deflection curve and maximising the deflection of the gold layer for a
given indentation depth. The curve should be as broad as possible within the contact region while at the same time avoiding a crosstalk between one point and another. Once this is achieved, methods for fabricating the substrate were investigated.

The fabrication of the silicone layer is carried out by manufacturing an in-house spin-coater. Previous methods have been based on pouring the silicone liquid in between two glass discs [3]. This is an unfavourable method as it may damage the glass and the silicone itself. The spin-coater method also provides a smooth surface with relatively uniform thickness. Once the bottom layer is manufactured, the gold film is then evaporated in between the layers by means of a vacuum evaporation chamber. The top layer is manufactured by pouring the epoxy liquid onto the gold film and covering the layer with a clear and smooth PTFE film. A smooth-surface weight is then applied to exert an equal distribution of pressure. Once cured, the PTFE film is peeled to reveal a smooth surface double layer elastic sample.

The experiment employs a New Focus tunable laser that acts as the light source and the phase shifting hardware. The interferometer set-up was designed and constructed to include the objective lens and the rotating diffuser in order to expand the beam. The interference pattern, which were produced from the gold and the glass-air interface, are recorded by the camera which were connected to the Sun workstation for analysis.

The sample is then calibrated by means of stacking ball bearings with a diameter of 4 mm on top of one another. An initial phase map without load is acquired before the experiment is performed. Ball bearings are stacked successively and for each additional weight, a measurement is taken. Through this procedure, wrapped phase maps are obtained. The measurements obtained from the wrapped phase maps are subtracted from the initial phase map to give a wrapped phase difference map. This is then unwrapped in order to remove any phase discontinuities by adding or subtracting an integral multiple of $2\pi$.

The unwrapped phase map is then analysed to yield a displacement map which reveals the points of indentation. This experimental value is then fitted with mathematical equations in order to quantify the value of the depression. From the analyses it was found that the Lorentzian equation provides a good fit to the
experimental data. For each ball bearing, the value of the depth is noted and plotted against the number of ball bearings. This results in the force-deflection calibration curve that is needed when the layered and sandpile experiments are commenced.

The chapters in this thesis are structured as follows: Chapter 1 discusses the current research regarding stress propagation within granular materials giving details of work to date that has been carried out in this field.

Chapter 2 briefly discusses the limited experimental work that has been carried out in this area. This chapter also outlines certain interferometer techniques so as to give an overview of the experimental method.

Chapter 3 discusses the fringe pattern analysis. In this chapter the details of phase shifting algorithms used in order to calculate the phase are discussed. The phase shifting technique results in a wrapped phase map which in turn is unwrapped to correct the phase discontinuities.

Chapter 4 describes the work of the Finite Element Analysis in order to obtain an optimised elastic substrate. This includes a description of the design used to obtain the right material composition and thickness.

Chapter 5 discusses the experimental arrangement for both the Mach-Zehnder interferometer and the tuneable laser. This includes the methods developed in calibrating the piezo-electric translator (PZT) and the tuneable wavelength laser. The methods employed for the calibration, the layered experiment and the sandpile experiments are discussed.

Chapter 6 describes the fitting process as well as the comparison between the Lorentzian and the Gaussian Equations. In this chapter, several analyses, including the visco-elasticity and the friction-wall effects of the calibration rig are also investigated. The automation of the analyses to obtain the central positions of the indentations are also discussed.

Chapter 7 discusses the results obtained from the calibration experiments. This also includes the results of a single layer silicone cast on a glass and a clear Perspex. The analysis of the layered and sandpile experiments is presented and finally in Chapter 8, conclusions and suggestions for future work on this project are given.
CHAPTER 1

STRESS PROPAGATION IN GRANULAR MATERIALS

1.0 Introduction

The mechanics of granular or powder materials are different from those of liquids and gases. In certain cases, they show behaviour similar to that of liquids or of gases and in some cases they do not. Powders have significant commercial importance, as many materials tend to be in granular form. This is an issue particularly with regard to the storage of heaps, storage within the pharmaceutical industry, the construction of highways and dams, landslides and snow avalanches etc.

One of the factors that affects storage is the pressure distributions, for example, pressure distributions under a sandpile have a unique characteristic that a local minimum is located at the centre. This phenomenon has yet to be explained. Several approaches have been put forward. To name a few, the approaches are continuum theories, microscopic models, statistical approaches and computer simulations.

The problem of stress propagation within granular media depends to a large extent on the contacts, both grain-grain and grain-wall. The understanding of these contacts will provide an overview of the force network involves within the granular media.

The subject of modern contact mechanics started by the work of Hertz [4]. He formulated several formulae based on simple assumptions to give a profile between the surface and the indenter. The mathematical equations put forward were used in order to resolve the forces into its normal and tangential component. This provides a distinct solution though many have generalised the solution to incorporate all the different shapes.

The behaviour of the granular media was also investigated by introducing microscopic model. Many have adopted using circular shapes such as disks. This also
includes packing the disk into pyramidal shape, hexagonal shape and many more. Most of these works assumed frictionless bodies and this give rise to an incomplete picture of the problem. To ensure stability, the effect of friction is essential. However, the studies which include friction tend to be highly mathematically complex.

In some research, works using statistical model can also give an attractive solution. An example is the ‘q’ model which is used to predict the force distribution in static bead packs.

Another approach is using continuum theories. This approach is based on solving the simultaneous equations of stress continuity. For example, given three unknowns, one need three equations in order to solve for the unknowns. However, in granular media, there are two equations with three unknowns. A closure equation is needed to complete the solution. This prompted models that depend heavily on the assumptions in order to achieve the closure equation.

All these methods are based on theoretical assumptions. The predictions from each method needs verification. To achieve this, experimental studies are needed. The literature on experimental work in this field is however rather scarce. This chapter will give an overview of the work undertaken to date in trying to give solutions to the problem.
1.1 Experimental Work

The most important experiment work for measuring the force distribution under a heap is that of Smid and Novosad [1]. They measured vertical and shear stress distributions at the base of a cone shaped bulk material. The aim was to determine the profile of vertical pressures and shear stresses in the base of the heap. Intuitively, one would expect the pressure to be a maximum at the centre. However, the result showed that there is a significant depression in the middle of the heap and the neighbouring positions attain pressures that are maxima as in Figure 1.0.

The dip at the centre implies that part of the weight is transmitted to the outer regions of the heap. The dip is also observed to be a characteristic feature during the filling process right from the start. The vertical pressure and shear stress increase linearly with the height of the heap. The shear stress and the friction condition is affected by the way the material is poured. The friction factor however for the base and the bulk material is not constant and decreases from the circumference towards the centre of the heap. It also decreases with the height of the heap [5].

![Pressure Cell Position vs Pressure (Pa)](image)

Figure 1.0: Results from Smid and Novosad Experiment [1] for a sandpile.
One significant drawback with these experiments is that the pressures only provide average stresses and therefore cannot give insight into the microscopic mechanism for the observed pressure distribution. Furthermore the experiment used pressure cells located at a finite number of positions which will only give a limited spatial resolution. More recently, experiments have been carried out to measure the individual contact forces.

Shear stress is potentially important, but was not measured in this project due to experimental difficulties in doing so. It could be necessary to measure tangential displacement components, which would require Moiré interferometer.

An experiment using a simple elasto-optical method was used to measure the normal force distribution under a conical pile [2]. The experiments also revealed a dip in the normal stress. The measurement was based on recording contact diameters of steel balls between the pile and an elastomeric substrate as shown in Figure 1.1. Through suitable calibration experiments it was possible to measure contact forces to an accuracy of 2%. A suitable bearing diameter was chosen so that the spatial resolution was comparable to the grain size. The problem with this approach is that the experiment is rather slow and involves large displacements (~10μm) which may affect the stress distribution. The relationship between the contact diameter and load employed an extended version of Hertz's equation which takes into account surface adhesion and gives the following relationship between the radius of contact area, \( a \) and the load, \( P \):

\[
\left( P - \frac{4Ea^3}{3R(1-\nu^2)} \right)^2 = \frac{16\pi\gamma Ea^3}{(1-\nu^2)}
\]  

where \( E \) = Young's modulus
\( \nu \) = Poisson's ratio
\( R \) = Radius of ball bearing
\( 2\gamma \) = energy required to separate unit area of interface

Equation (1.0) can be rewritten as

\[
P = Aa^{3/2} + Ba^3
\]  

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where A and B are the material constants to be obtained by the calibration experiment.

Figure 1.1: Contact between steel ball and silicone rubber for the elasto-optical experiment [2].

The contact area method has also been used to examine the force distributions inside two dimensional granular systems. The experiment is based on contact area trace measurement using disks to determine the force that act at the point of contact [6] in two-dimensional (2-D) geometries. The disks were initially placed randomly and then compressed. The results from such experiments showed that strong particle to particle contact forces within the packing are exponentially rare. The experiments also showed the robust property of the normal contact forces confirming the results of the numerical simulations on these distributions inside a 2-D packing. The simulation data and experimental test shows that direct measurement of the contact area gives relatively good agreement in analysing granular assemblies.

The mechanical properties of static granular 3-D assemblies were investigated experimentally in Reference [7]. Results of experiments based on probing the response of static granular assemblies to a local stress perturbation known as the Green’s Function were presented. The response function showed one peak centred vertically below the point of application of the force. The results from the experiments show qualitative agreement with the prediction of the elastic theory although the two separate peaks predicted by the hyperbolic model was not observed.
For all granular assemblies, it was found that the width at half of the amplitude of the response function increased linearly with the depth.

A method that measures the normal force using a high precision electronic balance was presented in [8]. The high sensitivity of the experiment made it possible to measure the individual beads at the base. The measurement of the weight, however, was based on changing the probe height of the balance. The technique was able to measure the force without any external loads applied i.e. the weight of the media itself, with a controlled perturbation occurring during the measurement. The result of the high sensitivity technique was found to give a good agreement with statistical model [9, 10].

None of the experimental techniques described above provide a fully ideal solution to the measurement problem. An ideal technique would offer wholefield (rather than pointwise information), use substrates of infinite stiffness and exhibit a non-hysteretic response. Interferometry does not appear to have been used previously in the granular materials community and yet it offers distinct advantages over other techniques, in particular the possibility of constructing very stiff sensors. Relevant literature to the problem of measuring contact forces interferometrically is described in Chapter 2.
1.2 Continuum Theories

Over many years, the static distribution of stresses in a granular sample has been analysed by means of determining the relations between stress and strain in a model sample. In many cases, the problem can be simplified by assuming that the grains have 'froze' together.

An example for this case is a silo filled with grains. The filled silo is shown in Figure 1.2 [11, 12]. The stress is measured with gauges at the bottom and in general is found to be much smaller than the hydrostatic pressure $\rho g H$ which is present in a liquid ($\rho$ is the density, $g$ is the gravitational acceleration and $H$ is the column height).

The model was put forward by Janssen where the assumption is that the horizontal stresses in the granular media ($\sigma_{xx}, \sigma_{yy}$) are proportional to the vertical stresses giving:

$$\sigma_{xx} = \sigma_{yy} = k_j \sigma_{zz} = -k_j p(z) \quad (1.2)$$

where $k_j$ is a phenomenological coefficient and $p = -\sigma_{zz}$ is the pressure.

The important point is the friction between the grains and the vertical walls where the walls endure a stress $\sigma_{rz}$. The equilibrium condition for a horizontal slice of grain, of area $\pi R^2$ and height $dz$, gives:

$$-\rho g + \frac{\partial p}{\partial z} = \frac{2}{R} \sigma_{rzi=R} \quad (1.3)$$

where $r$ is the radial coordinate and $z$ is measured positive towards the bottom.

Janssen assumes that everywhere on the walls, the friction force has reached its maximum allowed giving:

$$\sigma_{rz} = -\mu_f \sigma_{rr} = -\mu_f k_j p \quad (1.4)$$

where $\mu_f$ is the coefficient of friction between grains and wall.

Incorporating equations (1.3) and (1.4), yields:

$$\frac{\partial p}{\partial z} + \frac{2\mu_f}{R} k_j p = \rho g \quad (1.5)$$

This introduce a characteristic length:
\[ \lambda = \frac{R}{2\mu_k k_j} \]  

(1.6)

and leads to pressure profiles of the form:

\[ p(z) = p_\infty [1 - \exp(-\frac{z}{\lambda})] \]  

(1.7)

with \( p_\infty = \rho g \lambda \). Near the surface (\( z < \lambda \)) the pressure is hydrostatic (\( p \sim \rho g z \)), but at larger depths (\( z > \lambda \)), \( p \to p_\infty \), i.e. the weight is mainly carried by the walls.

There are a few assumptions that are open to some questions. The model assumes the \( x, y, \) and \( z \) axes to be the principal axes of the stress tensor. In actual fact the off-diagonal components \( \sigma_{xz}, \sigma_{yz} \) are also required. It is also entirely arbitrary to assume full mobilisation of the friction as in equation (1.5). In fact, any value \( \sigma_r/\sigma_r \) would be acceptable. Janssen model is simple and gives a crude approximation to some of the main features of stress distribution in silos.

Figure 1.2: A silo filled with granular material. The material falls slightly under its own weight, by an amount \( u \). The width of the silo has been exaggerated to display the expected profile of \( u \) in the model.
For a static heap, it was proposed in Reference [13] that stress propagates according to local rules that depend on the construction history. The way a pile is constructed determines the behaviour of the stress. There are a few ways to construct a pile such as filling from a point source or by distributed force as shown in Figure 1.3 [14]. Once a pile is constructed, and no slip occurs, the pile has 'perfect memory' where the construction history determines the organisation of the grains [15]. The constitutive relation depends on this construction history which is needed to close the problem [13].

![Diagram of Point Source and Distributed Filling](image)

**Figure 1.3: Method of constructing a granular pile [14].**

The effect of construction history is found experimentally in Reference [16, 17]. The experiments in [17] employ three methods of filing the particles; i) fixed height point source; ii) slowly moving point source; and iii) distributed source. The filling method based on localised source at fixed height yield stress profiles with a stress dip near the centre. For the other two methods mentioned, no stress dip is found. The pressure profile was also found to scale linearly with the pile height [16]. It was also observed that the progressive formation of the pile by successive small avalanches leads to a pressure dip.

The equations governing the forces resemble the conservation laws [18]. If an assembly has been formed, forces are generated at the point of contacts. The

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approximation taken is that the friction forces present avoid slipping and the grains have rigid but not exactly spherical shapes.

In [19, 20], the derivation of equations to solve the problem were put forward. These are based on the simple solvable problem of stress transmission through a static granular material when the grains are perfectly rigid. The problem is yet to be solved systematically but all approaches taken to date appear to involve the introduction of same arbitrary rule.

A key requirement of stress continuity is that forces must balance. For a three-dimensional (3-D) granular material the equations are:

\[ \frac{\partial \sigma_{x}}{\partial t} + \frac{\partial \sigma_{x}}{\partial r} = \alpha \left( \sigma_{xx} - \sigma_{n} \right) \]

\[ \frac{\partial \sigma_{y}}{\partial t} + \frac{\partial \sigma_{y}}{\partial z} = g - \alpha \sigma_{yz} \]

\[ \frac{\partial \chi \sigma_{ij}}{\partial z} = 0 \]

where \( z \), \( r \) and \( \chi \) are cylindrical polar coordinates, with \( z \) as the downward vertical. \( r = 0 \) is the symmetry axis, so that \( \sigma_{rz} = \sigma_{rx} = 0 \), \( g \) is the force of gravity per unit volume, and \( \sigma_{ij} \) is the usual stress tensor which is symmetrical in \( i \) and \( j \) [13]. The equations for two-dimensions (2-D) are obtained by setting \( \alpha = 0 \) and suppressing equation (1.8c) which gives:

\[ \frac{\partial \sigma_{x}}{\partial t} + \frac{\partial \sigma_{x}}{\partial z} = 0 \]

\[ \frac{\partial \sigma_{y}}{\partial t} + \frac{\partial \sigma_{y}}{\partial z} = g \]

In two-dimensional (2-D) problems the fact that there are two equations and three unknowns of the stress tensor \( \sigma_{zz}, \sigma_{m}, \) and \( \sigma_{tz} = \sigma_{m} \), means that a closure equation is needed to calculate the stress distribution.

A number of continuum theories have been proposed to explain the stress characteristics in granular material. The main differences between the models is the choice of the constitutive relation needed to close the problem. Each of the models has different assumptions.

Material in the Incipient Failure Everywhere (IFE) model means that material is at the point of slip failure at all points in the granular bed. By choosing a yield criterion, this gives the missing constitutive equation. For a sandpile, however this is
only true at the surface and not inside the bulk [13]. This means that away from the free surface, IFE is not strictly valid [2]. A stress plateau is predicted rather than the experimentally observed dip [15].

The Fixed Principal Axis (FPA) hypothesis is that the principal stress axes are fixed at the time of the burial and unaffected by subsequent loading. Each element 'remembers' its construction history and fixes the orientation of the stress that the element can support [13]. The stress propagates along a nested set of archlike structures [15]. The direction of these characteristics is the same everywhere and coincides with the principal axes. There is no shear force acting between one arch and its neighbour. The normal force exist as to sustain the arches [21].

The Oriented Stress Linearity (OSL) model leads to linear relationship between stress components. The co-ordinate system is tilted and the direction of the propagation remains fixed. The direction of the stress paths remains unaffected by distant load. In continuum limit, these directions become characteristics of the stress propagation equations, which OSL takes to be fixed [15, 21].

The model proposed by Edward & Oakeshot is based on the assumption of a pile consisting of nested arches and each arch supports its own weight. The vertical stress decreases towards the centre due to the smaller arches that exist there. The load is transmitted unevenly due to this type of arching mechanism. The disadvantage of this model is that the dip is over predicted. There is no downward force at the centre. The arch is parallel to the free surface and the outermost arches are incomplete [15].

The Radial Stress Field (RSF) hypothesis means that there is no intrinsic length scale in the problem of a sandpile under gravity. It assumes that the principal axes of the stress tensor have a fixed angle of inclination to the vertical [13]. RSF predictions fit the experimental data better than FPA. RSF scaling also rules out finite deformation [15]. The validity of RSF is discussed in [22]. The RSF hypothesis does not take into account the fact that grains can move at any points of the free inclined surface except at the bottom boundary.

S. Savage [12] discussed the case of a 2-D heap with a different support plane. For a 2-D heap with a rigid support plane, no dip were observed in the experiment. If however, the support is slightly deformable, the stress field may change and the dip
may occur. In a 3-D case, the results are extremely sensitive to the details of the deposition procedure.

Variational closure models have been proposed to describe the stress distribution and the arch formation in sandpiles with a rigid base. The model considers a 2-D rectangular pile granular block with a vertical force applied at the top block. The results show that the stress field changes due to rearrangement of granular packing at the microscopic level. The stress minimum, however, does not appear for a wide range of the model parameters [23].
1.3 Microscopic Theories

The understanding of granular materials can be further understood by investigating the microscopic behaviour within the pile. As mentioned earlier, the construction history will determine the packing structure, which influences the stresses in granular media. Most of the analysis is performed by means of constructing 2-D or 3-D piles of frictionless smooth discs.

The stress distribution for a pyramid-shaped hexagonal packed granular pile in 2-D as shown in Figure 1.4 was presented by D.C. Hong [24]. For stability, the floor support for the pile was assumed to be rough which provided necessary friction to the grains. From this set-up, the vertical component of the force acting on each grain at the bottom layer was found to be identical. The drawback of this is that in granular packing, it is packed in a random manner with voids or vacancies.

![Figure 1.4: A hexagonally packed granular pile. Each grain is identified with the matrix $a_{ij}$.](image)

The effect of these voids or vacancies in this type of pile was investigated in [25]. In a hexagonal array of vacancies, the pile was stable and the force distribution can be calculated. The load acting on each grain at the bottom was also found to be identical. The vacancies form a series of arches, which were found to modify the stress distribution locally but not globally.
Figure 1.5 shows a similar 2-D model for analysing the force structure in 2-D equilateral pile of perfectly hard, identical cylinders. Particles were put into a grid system and forces resolved in the horizontal and vertical directions. The normal force was shown to be constant for all particles along the base. Comparisons were made between analytical and computer simulations and showed that the dip in the normal stress at the base of the sandpile occurs when additional shear stress in the horizontal components exists within the pile [26].

Reference [27] proposed a symmetrical pyramid pile of ten smooth disks as shown in Figure 1.6. The difference between this model with [24] is the fact that the disks is supported by the two walls. The disk support interaction is smooth and the contact force acts inwardly along the normal at the point of contacts. The approach is to set-up equilibrium equations along the contact, using a top down algorithm. The simulation shows that the equilibrium force distribution is not unique but a function of how the pile is constructed. The contact force is found to be sensitive to small geometrical changes.

Another attempt by the same authors using a triangular pile of particles interconnected by linear springs that is subjected to gravity, was looked at. The aim of this study was to calculate the deformation distribution within the pile. The deformation patterns depend critically on the relative strength of the springs. This
method, however, did not replicate the key behaviour of the results of Smid and Novosad [28].

Another pyramidal piling of 2-D disks was presented in [29]. The disks were under the effect of gravity and in absence of friction. The work presented was based on numerical studies using 'spring like' contacts under compression between disks. The authors concluded that the lattice orientation and the characteristics of the supporting surface have a very important impact on the physical properties of the pile.

The works by [24, 26-28], provide the analysis for a 2-D pile as shown in Figure 1.7 [30]. The model has \( p-1 \) layer of discs with each having a diameter \( d \). Figure 1.7 shows \( p = 10 \) and a non-orthogonal coordinate systems \( (i, j) \) with the axes along the surface diagonal is used. The main assumption is that: 1) the contacts are frictionless; and 2) there are no horizontal contacts. The pile is stable when the floor on which it is standing is rough, similar to Reference [24] and thereby capable of providing a horizontal force inward to the centre of the heap. The normal force components acting along the \( i, j \) axes are denoted by \( I \) and \( J \) respectively and no tangential components are present due to the assumption of frictionless contact. Resolving the forces acting on grain \( i, j \), which are \( I(i,j) \), \( I(i+1,j) \), \( J(i,j) \) and \( J(i,j+1) \) both horizontally and vertically yields in two recurrence equations:

\[
I(i+1,j) - I(i,j) = \frac{W}{2\sin \theta} \tag{1.10a}
\]
\[ J(i,j+1) - J(i,j) = \frac{W}{2 \sin \theta} \]  \hspace{1cm} (1.10b)

where \( W = mg \) and \( \theta \) is the angle of repose.

From equations (1.10a) and (1.10b), the force distribution throughout the pile is obtained as:

\[ I(i,j) = (i-1) \frac{W}{2 \sin \theta} \]  \hspace{1cm} (1.11a)

\[ J(i,j) = (j-1) \frac{W}{2 \sin \theta} \]  \hspace{1cm} (1.11b)

The total vertical component of force on a disc is proportional to \((I+J)\) and since \(i+j=p\), for the bottom layer, equations (1.11a) and (1.11b) show that the vertical component of force on the bottom layer is uniform across the pile.

Figure 1.7: 2-D model of granular pile. The pile consists of \(p-1\) layers (in this figure \(p=10\)) [30].
The 2-D model can be extended in 3-D for the case of hexagonally close packed in ABCABC ... structure [30]. Three axes i, j, k, are now required and the arrangement is shown in Figure 1.8. This gives I(i,j,k), J(i,j,k), K(i,j,k) as the contact forces acting on particle (i,j,k) as described earlier. The angle $\theta$ between the axes and the horizontal is chosen to be less than $\cos^{-1}\left(\frac{1}{\sqrt{3}}\right)$, the perfectly close-packed value and hence no horizontal forces exist between the particles in a given layer. In 3-D, each particle experiences six contact forces, three from above and three from below. Resolving the forces vertically and horizontally yields recurrence relations for I, J and K as follows:

$$I(i,j,k) = (i-1) \frac{W}{3\sin \theta}$$  \hspace{1cm} (1.12a)

$$J(i,j,k) = (j-1) \frac{W}{3\sin \theta}$$  \hspace{1cm} (1.12b)

$$K(i,j,k) = (k-1) \frac{W}{3\sin \theta}$$  \hspace{1cm} (1.12c)

The particles in a given layer have $i + j + k = $ constant, and therefore $I + J + K$ is also constant. The vertical force is proportional to $I + J + K$ and as in the 2-D case, there is also a constant value across a given layer in the pile.

![Figure 1.8: FCC Structure (ABCABC...) of identical spheres [30].](image)
Another investigation for a 3-D pyramidal pile problem using identical, stiff and frictionless spheres is presented in [31]. The pyramid is of an equilateral triangle base with face-centered cubic structure. As described earlier, work by other authors has shown that in 2-D, the pressure profile at the bottom is uniform. This is also observed in 3-D. The method used was able to calculate the normal and shear force between any two neighbouring particles.

In real sandpile, this perfect structure and unique coordinate direction will not exist. The model from [30] can be further extended by retaining the idea of the forces being transmitted at an angle to the vertical and eliminating the directions of the FCC pile. This is shown in Figure 1.9a. The force is assumed to be transmitted uniformly through a conical shell, rather than the three directions of the FCC structure. The mass element \( dM \) is supported by a conical shell and exerts pressure on the floor over an annular ring. The load per unit length of the ring is proportional to \( dM/R \), where \( R \) is the distance from \( dM \) to the ring. The pressure distribution is calculated by a volume integral over the entire cone. Another equivalent approach is to consider a single point on the base and to add up the forces due to all the mass elements that contribute to the force at that point. All of these elements lie on an inverted cone (denoted cone B) as shown in Figure 1.9b. The stresses, \( \sigma_{ij} \), at \( x_0 \) along the diameter of the base can be written as:

\[
\sigma_{ij} \propto \int_{\text{area}} \frac{X_{ij}}{R} \, dA
\]  

(1.13)

where the integral is over the surface area of cone B that lies within the sandpile (cone A), \( R \) is the distance between \( x_0 \) and a point on the cone, and \( X_{ij} \) is a function to resolve the force into normal or shear stresses as required. \( dA \) is the small element of area containing mass \( dM \), lying at polar coordinates \((r, \phi)\). \( z_0 \) is the height of the cone and \( r_0 \) is the radius measured at the base. \( \alpha \) is the angle of repose of the pile and \( \theta \) (greater than \( \alpha \)) is the angle of inclination of the contributing shell. Equation (1.13) can be rewritten (by using the fact that \( R \propto r \) and \( dR \propto dr \)):

\[
\sigma_{ij} \propto \int_{-\pi}^{\pi} \int_{0}^{r_0} X_{ij} R \, dR \, d\phi = \int_{-\pi}^{\pi} X_{ij} r_0 \, d\phi
\]  

(1.14)
assuming that $X_{ij}$ is independent of $R$. $r_c = r_c(\phi)$ is the locus of the line of intersection of the two cones. In an FCC structure, the force vectors pointed directly along the diagonals. In the conical-shell model, the force due to a point on the contributing cone acts along the line joining it to the vertex of the cone (the point on the base of the pile.

The form of $X_{ij}$ for the normal component is:

$$X_{zz} = \sin \theta$$

(1.15)

and for the shear component along the diagonal it is:

$$X_{xz} = \cos \theta \sin \phi$$

(1.16)

The shear component perpendicular to the diagonal is $\sigma_{xy} = 0$, due to the symmetry of the problem. The equations of shear and normal stresses at the base of a conical pile can thus be written as:

$$\sigma_{zz} = \kappa \int_{-\pi}^{\pi} r_c \sin \theta \, d\theta$$

(1.17)

$$\sigma_{xz} = \kappa \int_{-\pi}^{\pi} r_c \cos \theta \cos \phi \, d\phi$$

(1.18)

where $\kappa$ is a normalisation constant.

The lowest stress at the centre will be produced when $\theta = \alpha$, since this is the case of the weight at the centre of the pile being supported as far away from the centre. The integral then gives the normalised stress distribution as:

$$\sigma_{zz} = (1 - (x_0/r_0)^2)^{1/2}$$

(1.19)

The maximum of this distribution occurs at $x_0 = 0$. This model therefore fails to produce a pressure dip. Extension of the model by allowing propagation over a range of angles, rather than just $\theta$ also does not result in a dip. This is due to the fact that the solution is just a linear combination of the basic conical-shell solution. From this analysis, the transmission of force in a sandpile in general was concluded to be non-axisymmetric, presumably as a result of an anisotropic network of contacts formed during the formation of the pile.
Figure 1.9: Model for force propagation in a randomly packed medium. a) forces propagate through a conical shell rather than down the three directions of the FCC structure. b) All points of cone B contribute to the pressure at point O on the base of the pile. The symbols are explained in the text [30].

This normal and shear stress of the model was compared with the experimental data in order to compare the model's prediction. Equation (1.17) and (1.18) were fitted with Smid and Novosad [1] data by scaling the data for the correct $\kappa$ while varying $\theta$, until the least-square error was obtained. Figure 1.10 shows the data along with the best-fit curves obtained with a $\theta$ of 0.989 rad (56°). This can be
compared with the value for a perfect FCC structure in which a diagonal makes an angle of 54.7° to the horizontal. This is a simple model where the functional fit appears to be reasonable, although the central dip in the data is not reproduced.

Figure 1.10: Fits of 'conical-shell' model stress profiles to normal and shear stress measurements obtained by Smid and Novosad [1]. (a) Sand, normal stress; (b) sand, shear stress.
For a real sandpile, several features are different from the model presented. The packing in a pile is random and not regular. Friction in this type of model is assumed to be zero or a fixed coefficient at all contact points, whereas in a granular pile it is not. Also the forces are constrained to be 2-D.

Packing in an array of ordered 2-D parallel cylinders was studied experimentally in [32]. The cylinders used exhibited several defects such as deformation along their axis, they were not perfectly cylindrical, they had small differences in diameter, etc. These geometrical defects can be considered as weak and responsible for the heterogeneity of the spatial distribution of intergranular stresses. The arrays were found to form irregular force networks. The experiment proved that the macroscopic law is very sensitive to the network.

An experimental study of uniaxial compression of a mixture of 'hard' and 'soft' cylinder is presented in [33]. Macroscopic stress-strain laws depend on the geometrical and compositional heterogeneity. The largest stress paths prefer to pass through large grains. Repartition of the stress is inhomogeneous. For a uniaxial compression, the links follow the principal stress direction. This heterogeneity in the stress is due to weak geometrical heterogeneities. However at microscopic level, the stress transmission is localised at the contact.

A study of network forces in a static assembly of hard, frictionless, spherical beads of random size is discussed in [34]. The aim was to investigate the transmission of the forces. In frictionless particles, the transmission of forces is shown to be isostatic. However, by including the friction, it is shown to be in elastic behaviour. The elasticity is due to the contacts that provide minimal constraints.

The force perturbation in a 2-D pile of frictionless arising from lattice distortions is discussed in [35]. The model is an extension work from [24, 36] where the discs of uniform size are arranged on a regular lattice, which predicts a uniform normal stress distribution at the base of the pile. The analysis is applied to two problems; 1) deformable grains that undergo Hertzian deformation at the points of contact and; 2) a pile containing a gradient in particle size from the centre to the free surfaces. The former results in the pressure dip and the latter produce a dip if the large particles are placed at the centre.
In [37], contacts in 2-D frictionless packed beds under isotropic load were identified. The distribution of contact force in confined granular at rest is heterogeneous. There is a disordered set of intergranular contacts that leads to force chains, arching effects and characteristic lengths $l$ appearing. As the compaction proceeds, forces gradually localise into strongest bonds. The force distributions are robust properties of static granular media under uniaxial compression. There is no dip but the distribution is consistently described by a functional form.

Molecular Dynamic (MD) simulations were used by Luding, and these focused on the properties of granular systems in the absence of friction [38]. The model is a frictionless 2-D spherical particles with a linear spring and a linear dashpot active on contact. Stress chains and arching were examined as these depend on the friction. The normal force result at the bottom was found to be similar to [24, 30] where it is constant.

Reference [36] construct a discrete element computational code, also known as discrete element code (DEM), which solves the equations of motion that describe particle to particle interaction. The code is applied to a 2-D triangular pile of discrete particles. The model replicates results from Smid and Novosad for the vertical and shear stress at the base. The depression or dip in a sandpile appears to grow with the size of the pile. The model suggests that the stress at the apex should become constant within a certain distance of the centre.

A 3-D DEM code was use in Reference [39]. The model use slightly deformable spheres. The results show that homogeneous particle did not produce a dip in the normal stress. The dip was found if the particles were different is sizes.

Numerical technique for a granular media is performed in Reference [40, 41]. Constitutive theories are needed to model a complex 3-D granular pile. This provides a qualitative analysis for a 3-D stress-strain behaviour [40]. At macroscopic scale the results in [41] shows a qualitative agreement with the mechanical bahaviour of sand observed in experiments. Numerical simulation permits an analysis of the evolution of internal variables associated with the micromechanical processes occurring at the particle scale.
In summary, the microscopic behaviour within the pile shows that the force distribution depends on the surface of the pile, the geometrical defects and the hardness of the material and how the pile is constructed. All of these contribute on how the stress is determined at the bottom of the pile. A number of the model discussed found that there is a uniform force at the base of the pile. This clearly shows that will be no dip observed. It is also worth mentioning that the model use frictionless material with perfect structures where the granular pile will exhibit friction and in random state.

1.4 Statistical Microscopic Models

The dominant physical mechanism which leads to force chains is the inhomogeneities of the packing which causes unequal distribution of weight. A statistical approach to explaining the force inhomogeneities in static bead packs, the so-called 'q' model was put forward in [42].

The model considers a regular lattices of sites as shown in Figure 1.11. Each site i in layer D is connected to exactly N sites j in layer D+1. The model only considers the vertical component of the forces and friction is ignored. The model leads to a force distribution that decays exponentially as a power law at large forces [42]. The stress distribution can be obtained by propagating the force from top to bottom. The drawbacks of this model are: that it takes no account of the constraints imposed by horizontal force balance; the torque balance on each grain does not have the right conservation laws at the microscopic level; and there is no clear procedure for connecting the lattice constant in the model to a physical length scale.
Figure 1.11: Schematic diagram showing the paths of weight support for a 2-D system for the 'q' model where each site transmit its weight to exactly one neighbour below.

An improved approach over the 'q' model is the 'α' model which incorporates relevant force and torque balance constraints into the lattice approach of the 'q' model. This provides a natural connection between the lattice constant and the grain size. The stress propagates to the left and right with increasing depth [43].

A disordered 3-D pack experimental study of monodisperse soda lime spheres based on carbon paper techique is presented in [10]. The results showed that the first few layers were hexagonally packed and further up the packing became random. This showed that the distribution was an exponential distribution and consistent with the 'q' model. No dip was found from the results but they gave good fit over the full range of forces with good agreement with the 'q' model.

Statistical properties of the force network were investigated in [44] and the model used is shown in Figure 1.12. The contacts are at an angle of 60° to each other. The contact force was found to be highly disordered with large fluctuations. The friction forces were locally biased and this describes the force propagation in a granular assembly.
A stochastic lattice model that includes landslides is presented in \cite{44, 45}. The granular material was a mixture of small and large grains. The resulting piles were observed to have alternating layers with large and small grains. Both mixtures had the tendency to segregate in different region.

A heap model employing basal rods which were stacked up and at random was investigated \cite{46, 47}. The heap was in static equilibrium as the forces exerted were resisted by friction between the floor and the rods. The randomness introduced did not remove the overall structural effects the large uniform stress across the base.

Another attempt was to construct cylindrical rods of indeterminate size but equal lengths placed in layers \cite{48}. The so-called 'Slack contact' analysis was introduced where it is assumed that not all of the rod contacts are used to transmit the force. Nevertheless, the number of slack contacts within a heap can be use as a measure of the degree of arching. Both attempts however, proposed by D. F Bagster, did not give a realistic picture of the heap and the results were not conclusive.

Computer generation based on the rod modelling was then analysed in \cite{49}. The aim was to produce a large number of heaps and focus on individual particles and the force balance on a microscopic scale. The result, however, was not consistent with experiment as the construction history determines the stress behaviour in a pile and the algorithm for the computer simulation did not take into account this effect.

An investigation using rectangular sectioned blocks was used to model a pile as shown in Figure 1.13. Using the blocks model, a characteristic dip was observed.

Figure 1.12: Arrangement of disc for investigating force network \cite{44}. 
The graph plotted showed ‘M’ shaped profiles. This suggests that the mechanical behaviour may be independent of the particle shape [47].

![Figure 1.13: Rectangular block in a heap.](image)

Using the heap as in Figure 1.13, a computer program was then used to analyse the block and to calculate the force distribution at the base [50]. The computer analysis showed that the result depended on the heap size. If the size of the heap was small, a peaked profile was produced and increasing the size resulted in ‘M’ shaped profiles.

1.5 Summary

All of these approaches aimed to understand granular behaviour inside a pile and to explain why granular material has different behaviour which is difficult to predict. It is clear that for a pile, the behaviour depends on how it is constructed i.e. the construction history. This determines the stress chains that exist within the granular pile.

The experimental work attempts to measure the pressure profiles at the base of a pile. The main experimental works include the use of pressure cells located at a finite number of positions over the pile and the measurement of contact diameter to determine the force distributions. These methods give pointwise information and are unable to provide wholefield information that could explain the behaviour at the base of a pile.
The continuum theories put forward constitutive relations in order to solve the simultaneous stress equations by proposing the closure equations. These closure equations are proposed by suggesting models with different arbitrary assumptions.

Microscopic approaches are based on constructing 2-D and 3-D models of either frictionless spheres or cylindrical rods. The approaches provide models with perfect structure with fixed coordination number which is not the case for a real sandpile. Real sandpiles exhibit friction since the geometry of the sand is not constant throughout the pile.

The ‘q’ model predicts the stress propagation using a statistical approach. The theories is based on regular lattice which leads to force distributions that decays as a power law at large forces. This approach however, ignores the friction effect that exists in a granular pile.

It is clear from the above review that most of the works involving granular materials are geared towards theoretical and numerical studies, and often using spherical discs as a 2-D and 3-D model. There is however, not enough experimental work in this field particularly in the measurement of forces at the microscopic (single grain) level. Such experimental work is important, both to validate the models put forward by various researchers, and to give insight into the behaviour of real granular assemblies.
CHAPTER 2
INTERFEROMETRY AND ITS APPLICATION TO CONTACT FORCE MEASUREMENT

2.0 Introduction

Many works in granular media have involved theoretical studies that span from statistical methods, numerical techniques and computer simulation. The studies in this field still have to be verified by means of experimental work. The research conducted by different groups have involved different methods with the same aim in mind, namely to explain the behaviour of granular material.

The measurement of force is usually carried out by using a precision scale or by means of other force measuring equipment. Using this equipment, the weight measurement is determined by placing down the appropriate load and reading can then be observed.

Direct measurement techniques in this field have posed several challenges. Among them is the measurement of the force at a discrete point within the granular material without causing any perturbation. If perturbation occurs, this may cause changes in the arrangement of the powder. The change in geometry within the granular powder will then cause the forces within the heap to be altered.

As mentioned in the previous chapter, the way in which the heap is constructed in an experiment is important and will determine the stress network and hence the stress profiles at the base. If the powder is displaced during the experiment, the measurement will reflect these changes. However, in some cases, there are some experiments [8] which measure the forces with little perturbation, provided that this disturbance is small and controlled.

Frequently the measurements involve the need to acquire data simultaneously at many points at the base of the powder. Reference [1] includes an example of the use of such transducers. Transducers such as strain gauges must be placed at several points which can become very costly if a number of precision instruments is required.
Alternatively, one can make use of an optical measurement technique which is able to provide whole-field information.

In this experiment the sample employed is a double layer elastic substrate, and ball bearing is used as the indenter. The ball bearing will be in contact of the substrate and the weight will cause the indentation. The indentation will cause the substrate to conform to the indenter, which will be detected by the interferometer. This indentation is a type of contact problem where Hertz [4] has put forward analytical solutions to predict the displacement.

The experimental work in this project has employed interferometry techniques. This is due to the method being non-contact and whole-field with high sensitivity. For an interferometer, the accuracy is dependent on the light source used for illumination and can be a small fraction of the wavelength (for example a He-Ne laser has a wavelength of about 633 nm). This means that the required grain displacements at the wall can be under 1 μm, meaning that the system is at least an order of magnitude stiffer than the elasto-optical method used by Brockbank et al [2]. Stiff walls are preferable to compliant walls to avoid distortions to the force networks. The technique is also non-contacting which has the advantage of no disturbance occurring while measurements are taken.

This chapter will briefly discuss the main experimental configurations for optical interferometers and review of some experimental work on contact force measurement by interferometry. An overview of the contact mechanics of layered substrates will also be discussed.
2.1 Methods of Measurement

Smid and Novosad [1] carried out amongst the first experimental works in this field. The method used in this experiment made use of strain gauges. The gauges were placed at intervals at the base of the heap. The sand was poured and measured by means of the gauges. The drawback of this technique is that it only provides an average value at the finite positions of the gauges. Furthermore, this technique yields only average macroscopic values. For microscopic values one would have to measure individual contact forces.

Another experiment made use of carbon paper being indented with beads or circular disks [6, 9, 10]. A normal force applied to the beads makes an indentation on the carbon paper and leaves behind marks of various sizes and darkness. The force is then determined after interpolating the calibration curves which are obtained by applying a predetermined mass on a bead or disk. Experiments have revealed that the area and the darkness of the mark increase monotonically with the normal component of the exerted force [10].

The use of contact area to determine the forces is discussed in [6]. This experiment involved constructing a uniaxial cell for studying 2-D random granular packing bounded by four rigid walls. A vertical displacement was imposed by means of an external force which compressed the packing. The contact area traces were then determined by capturing an image. To determine the force, the calibration involved the relationship between the contact force and the contact area. This was done by placing a disk in between two half disks under an axial force. A thin sheet of carbon paper was placed between the disks. A range of forces was applied to the disk to determine the contact area.

Measuring the normal force under a granular bed using a high precision electronic balance was introduced in [8]. Figure 2.0 illustrates this experiment.
Figure 2.0: Experimental set-up from Reference [8]. Shown here are the granular system and the container, the aluminum table, the probe and the balance. Carbon paper is placed between the granular packing and the table. Forces at the bottom of this system are traced by sliding the granular system over the table and the probe.

The system consists of a cylinder filled with glass spheres while the probe is a flat pointed needle attached to a pressure sensor. The balance has a feedback mechanism that maintains the vertical position of the probe and thus measures the force. The probe movement is small and it is well suited for pressure measurement. Changing the probe height could change the perturbation on the granular material in a systematic and controlled manner. This can then explore the possibilities of measuring the response to small disturbances.

It is apparent that measuring force within the pile requires force transducers that are able to measure with sufficient spatial resolution. Manufacturing these force transducers is certain to induce high cost even though the data acquired is not enough.
A method is then needed in order to attain ample experimental data capable of measuring individual points without introducing perturbation.

To achieve this a simple elastooptical method [2] was proposed that measures the normal force distribution under three-dimensional (3D) conical piles. The experiment measured the elastic deformation of a transparent silicone sample with a hexagonal close-packed monolayer of ball bearings placed on top. The ball bearings acted as a blanket of pressure sensors. The circular contact diameter will relate directly to the normal force acting on the ball bearing.

The experimental method in [2] can then be extended into employing techniques based on optical methods. The system based on this method is called the interferometer method which can vary depending on application. Amongst the advantages of optical methods is that they yield whole-field information without any contact being made with the sample. The measurement is an accurate one with a typical accuracy of up to several nanometer.
2.2 Contact Mechanics

The problem of contact between the ball bearing and substrate is not a straightforward problem. Various aspects regarding the loading and the contact radius need to be considered to find the stress for the structure. Figure 2.1 shows a contact between a sphere and soft elastic layer. From this geometry, work on this problem has resulted in many mathematical methods being developed to try to quantify the stresses and pressure at the substrate level.

Among the first satisfactory analyses of stress for contact of two elastic solids is that of Hertz [4]. He formulated equations for the displacements in the solid. Several assumptions were made for the formulation; 1) the surfaces are continuous and non-conforming; 2) the strains are small; 3) each solid can be considered as an elastic half-space; 4) the surfaces are frictionless. Hertz Theory gives the assumed pressure distribution, between contacting surfaces as follows:

\[ p = p_0 \left( 1 - \left( \frac{r}{a} \right)^2 \right)^{1/2} \]  

(2.0)
where \( a \) is the contact radius, \( r \) is the radial distance from the loading axis and \( p_o \) is the maximum pressure. The theory, however, is restricted to frictionless and perfectly elastic solids, and for finite samples, the results are true only near the indentation. It is well known that Hertzian is inherently non-linear in terms of the force-displacement curve. However, provided that measurements are taken at a limited range from the loading point, an approximately linear response can be expected. This is the case for the samples being developed for the experiments described in this thesis.

The area of contact based on Hertzian theory was investigated in [52]. The results gave a good agreement with the Hertzian theory when the diameter of the contact circle was measured. It was also noted that the effect of the indenter elasticity on contact radius appeared to be less than that given by Hertz's prediction.

A 3-D contact problem for a frictionless indentation of a rigid punch into an elastic cone was solved analytically in [53]. The method presented allows one to find simultaneously the normal contact pressures and the unknown contact area. The results of the analysis depend on various parameters of the problem area. These parameters are force, settlement, moment, and size and shape of the contact area.

Another approach in analysing the contact mechanics is by using Finite Element Method (FEM). The practicality of using this method is that FEM offers the possibility of simulating the deformation and stresses around the indentation edges under various indentors. Reference [54-57] present results and analysis of using FEM to analyse the contact problem. This can also be extended to include FEM analysis for layered structure which will be discuss in the subsequent section. The analysis of FEM, however, depends on the boundary conditions applied toward the model geometry.

The Hertzian equations were used in [54] to validate the results achieved from Finite Element analysis between two elastic solids. These were then compared with the direct experimental measurement. Characteristic curves were then obtained so as to give a comparison between the three methods. This gives a close agreement between Finite Element and the Hertzian theory. The Finite Element and the experimental results were estimated to be in agreement within 5%. This is probably due to the Tresca criterion adopted for the plasticity behaviour and the error
associated with the yield stress of the experimental material in the Finite Element analysis.

Reference [55] present results using FEM of elastic-plastic indentations using various indenters. The analysis found that the elastic-plastic indentation is a complex problem that depends on the elastic modulus (where \( E \) is the Young's modulus), the resistance to the plastic deformation and the post yield strain hardening. Elastic deformation only occurs only at the beginning of the indentation. The displacement of the material depends on the shape of the indenter, material and the initial state of the material.

### 2.2.1 Contact Mechanics of Layered Substrates

The contact analysis discussed in previous sections has been extended into the analysis layered systems. Layered systems have many practical applications, which involves constructing highways, dams etc. The calculated stress distribution is needed in order to optimise the design of layered systems. This section will give an overview of several works on layered systems.

Burmister [58] put forward a general theory of stress and displacement in layered systems. The assumptions were as follows; 1) the two layers are homogeneous, isotropic, elastic materials which obey Hooke's Law; 2) the top surface is infinite in length but finite in depth; 3) the bottom layer is infinite in length and depth; 4) the top surface is free from shear stress and normal stress; 5) stress and displacement for the bottom layer must be equal to zero at infinite depth; and 6) both surfaces are continuously in contact. Using these assumptions, he then solved equations for both layers to find the stress and displacement using Bessel's functions.

M.J. Jaffar [51, 59] presented a rigorous analysis for the axisymmetric frictional contact problem involving a thin elastic bonded layer and a rigid indenter of arbitrary profile. The method showed that friction has a significant influence on the results. The analysis was based on numerical studies with two types of problem considered: complete contact and incomplete contact. For complete contact, the radius of the contact region is known and this gives a known pressure value. With an
incomplete contact, the radius is unknown and the pressure will be zero at the contact boundary.

Reference [60] presented results for a given contact and stress resulting from application of a pressure distribution on a double layered half plane or its indentation by a body of prescribed shape. The stress states at the surface and within the structure are associated with a number of different combination of materials. The solution shows that it is not possible to have a comprehensive parametric analysis of the double layer substrate contact problem due to the number of geometric and material parameters and the non-linear relationship between these parameters. It is however possible to obtain and present concisely results of the extent of contact, surface contact pressure and interfacial stresses.

A non-linear model of contact between a rigid sphere and a thin elastic layer bonded on a rigid plane is presented in [61]. The model found that the area of contact between the sphere and the film varies linearly with the depth of penetrations of the sphere considering that the penetrations are much smaller than the layer thickness.

Reference [56], provides FEM results for a layered medium under a rigid and deformable indenter. The results show that the contact pressures in the elastic regime are much higher than for the deformable indenters. The FEM analysis also showed that varying the material properties of the indenters will affect the point of yielding of the layered medium.

The same authors then present FEM results for another layered medium by using the rigid and the deformable indenter again, but the top layer is a strain hardened substrate. The results show that the contact area is reduced and there is an increase in the central and peak contact pressures near the contact edges. As the plastic deformation proceeds, the radial and hoop stresses at the interface of the two layers shows a large increase in the compressive stress within the contact region.

The experiment employs a double layer elastic substrate as the sample (shown in Figure 4.0 pg. 81). In order to simulate the contact problem, FEM is employed so as to give prediction of the indentation. This will be use in designing the sample so as to optimised the design of the sample.
2.3 Interferometric Techniques

There are many types of interferometer with different arrangements. These configurations are able to measure physical parameters such as strain, displacement, vibration or deformation.

Generally interferometers can be an 'out of plane' such as Michelson and Mach-Zehnder Interferometers; ‘in plane’ such as Moiré; and common path interferometers such as the Fizeau Interferometer. The following subsection will discuss briefly a few important optical arrangements.

2.3.1 The Michelson Interferometer

The specimen for this particular experiment is a flat polished surface. A typical arrangement for this interferometer is shown in Figure 2.2 [62]. This configuration is able to measure the displacement component normal to the specimen. Light from the laser passes through the lens onto the beam splitter. The beam splitter (BS) then splits and recombines the beam. The Piezo-electric Translator (PZT) acts as the phase-shifting device.

![Michelson Interferometer Arrangement](image)

Figure 2.2: Michelson Interferometer Arrangement. The system includes a light source, lens (L), beam splitter (BS) and photo-detector array.
2.3.2 The Mach-Zehnder Interferometer

The Mach-Zehnder Interferometer can also be considered a two beam interferometer. Figure 2.3 shows a simple optical arrangement for this interferometer.

![Mach-Zehnder Interferometer Diagram](image)

Figure 2.3: A simple optical arrangement of the Mach-Zehnder Interferometer depicting the beam being split by a beam splitter (BS). BS<sub>1</sub> splits the beam and BS<sub>2</sub> recombines the beam with the mirror (M) redirecting the beam. The fringes are localised at O [63].

In this set-up, the advantages are that the separation of the two beams can be controlled as desired, and the test section is transversed only once. Furthermore, white light fringes can be obtained and localised on the same plane as the test section. The adjustment of this interference however is rather complex as either the mirrors or the beam splitter should be provided with tilt adjustments for the horizontal and vertical axes.

Mach-Zehnder Interferometer are useful in measuring tilt, strain and displacement. This interferometer was initially used at the beginning of the project. The system includes a polariser, half-wave plate, fibre optics to deliver the beam and a piezo-electric translator (PZT). Chapter 5 will discuss in details this configurations.
The two beams are however susceptible to environmental disturbance air turbulence and vibration may disrupt the measurements. This may cause erroneous information being detected that may be reflected within the results. To improve this, a more robust configurations is needed in order to measure the displacement.

2.3.3 The In-Plane Interferometer

An example of an in-plane interferometer is the Moiré Interferometer. Figure 2.4 shows an in-plane configuration of the interferometer. A beam-splitter divides the beam into two and these then illuminate the specimen at equal and opposite angles $\theta$ to the optic axis. The two beams are then recombined through diffraction at the specimen surface.

Figure 2.4: In-plane Interferometer. The beam is split using a beam splitter. The beam is then directed on to the specimen. Interference is occurs through diffraction which is then detected by means of the photo-detector array [62]. In Moiré, the deformed surface will include a grating.

Moiré Interferometry yields a contour map of an in-plane deformation field. The interferometer uses a coherent light and produces two beams of optical interference fringe pattern of high contrast. In this interferometer, a grating is applied to the surface of the specimen which is deformed together with the underlying
specimen [64]. The virtual reference grating interacts with the deformed specimen grating to form an interference pattern.

2.3.4 Elastohydrodynamic Experiments

Elastohydrodynamic experiments have been carried out by a number of groups which allows the measurement of the thickness of the film separating two contacting bodies. The most important application for elastohydrodynamics is to determine the lubrication film thickness in gears and rolling element bearings. Figure 2.5 shows a typical experimental set-up for elastohydrodynamics experiments [65].

Figure 2.5: An elastohydrodynamic experimental set-up used to measure the lubrication film thickness between the steel ball and the glass disc [65].

The glass disc was mounted in a steel disc supported by the large ball bearing. Both the glass disc and the ball bearings are connected to an electrical stepper motor as both are allowed to rotate. The glass disc was made semi-transparent with a chromium layer on the side facing the steel ball in order to reflect the illumination.
light, as shown in Figure 2.6. The light reflected from the chromium layer and the steel ball will give the interference pattern which was magnified by the microscope and recorded by the camera.

![Diagram of light reflection and interference](image)

**Figure 2.6:** Light being reflected from the chromium layer and the steel ball which provides the interference pattern [66].

The elastohydrodynamic experiments in recent years are able to measure these films down to just 1 nm [67]. The method however, needs high magnification in measuring the film thickness. This restricts the application into a small field of view for a given experiment. Furthermore, the analysis of the interferograms is difficult to automate since phase-shifting is not possible in a common path interferometer of this type with such a thin layer between the two reflective surfaces. One semi-automation approach using colour information is described by Marklund [66].
2.3.5 Shack-Hartmann Sensor

Shack-Hartmann sensors is a wavefront sensors which has become more available for the past 20 years. These sensors were initially used to improve the images of satellites taken from earth.

A common Shack-Hartmann sensor consists of a microlens array or lenslet array and a CCD camera [68-70]. A more recent work in Reference [71] shows a possibility of replacing the microlens with liquid crystal displays, which would offer a more flexible approach.

![Figure 2.7: Principle of Shack-Hartmann sensor [71].](image)

Figure 2.7 shows the principle of Shack-Hartmann sensors. This sensor divides the wavefront surface into a number of beams by the sun-apertures of the lenslet array. Each sub-apertures provides a separate focus on the detector of a CCD camera which shows as a spot. The displacement $(\Delta x, \Delta y)$ of the spot position are then measured with respect to the reference position of a plane wave. This can be defined as a 2-D field of partial derivatives of a wavefront $w$: 
\[
\begin{bmatrix}
\frac{\delta}{\delta x} \\
\frac{\delta}{\delta y} \\
\frac{\delta}{\delta y'}
\end{bmatrix}_w = \frac{1}{f} \begin{bmatrix}
\Delta x \\
\Delta y
\end{bmatrix}_y
\]  

(2.1)

where \( f \) is the focal length and \( i \) and \( j \) indicate the rows and columns of a microlens within the array.

The wavefront is then determined by the discrete field of partial derivatives using a least square fit \([72, 73]\). This then enables the required parameter to be extracted for analysis.

The Shack-Hartmann sensor will be used as part of the interferometer with the measurement of these spots can be performed rather easily by using commercial software available. The measurement using techniques however, will render a limited spatial resolution, as wide sensor will be required in order to be able to measure the whole area of displacement. Furthermore, the ability of measuring a whole-field information can be easily performed by using Fizeau interferomery techniques.
2.3.6 The Common Path Interferometer

A typical common path interferometer is the Fizeau Interferometer. The simplest application for this type of interferometer is the testing of the flatness of an optical surface. This is due to the demand in many optical industries, for example, for mirrors, to be flat and finished within a few nanometres. Figure 2.8 shows the basic arrangement of the Fizeau Interferometer.

![Diagram of Fizeau Interferometer](image)

Figure 2.8: Basic configurations of the Fizeau Interferometer used to determine the flatness of a surface [74]. The beam-splitter (BS) is only used to direct the beam towards the camera and is not part of the interfering paths.

In this method, two flat glass surfaces are placed in contact one another. Interference fringes will appear at their interface. If one of the surface is the 'test plate' which is optically flat, the fringes will form a contour map of the other surface with a contour spacing of half a wavelength. If the surface being tested is spherical, the fringes will appear as a series of concentric rings.
Applying finger pressure to the center of the surface being tested constitutes a simple check to determine the shape of the material. If the fringes move inward toward the center, the surface is concave and if they move outward, it is convex. If necessary, tilt fringes can be introduced by applying pressure at a point on the edge of the surface.

In Figure 2.8, the configuration uses a beam splitter in order to direct the beam towards the camera and is not part of the interference path. However, in certain modified Fizeau Interferometers there is no need to use a beam splitter. This is due to the fact that the beam-splitter introduces aberrations to the beam. This then leads to the coherent noise and also reduces the amount of intensity detected by the detector.

To mitigate these problems, a simple configuration based on the Fizeau set-up was proposed in [75]. The configuration proposed simplifies the optical alignment simpler without the use of a beam-splitter. However, this has the disadvantage that the beam needs to be off-axis. A tunable laser is used as the phase shifting device by means of tuning the wavelength.

This experiment is aimed at developing an experimental technique based on the Fizeau set-up, since this method is able to measure surface displacement and the arrangement proposes to be simpler than that in [75]. The experiment does not make use of a beam splitter, thereby reducing aberration and yielding an improved signal to noise ratio. The laser employ for the experiment is a tunable laser, which also use as a phase shifting device by means of tuning the wavelength. The configuration of this laser is discussed in subsequent section. The method constitutes a common path interferometer, it is also less susceptible to environmental error such as vibration and air turbulence. Chapter 5 will discuss the experimental arrangement.
2.3.7 Tunable Laser Configuration

The laser use for the experiment is of New Focus Vortex Tunable which are developed based on Littman-Metcalf design [76, 77]. This design is used in the Vortex Laser. The laser has a coherence length of 10m. The design uses a diffraction grating at grazing incidence to provide wavelength selectivity. Figure 2.9 shows the modified Littman-Metcalf configuration used by the laser. The high quality anti-reflection (AR) coating on the front facet of the diode is essential to the performance of the laser.

![Figure 2.9: Modified Littman-Metcalf configuration for the Vortex Laser [78].](image)

The AR coating turns the diode into purely a gain element. A collimating lens directs the output of the diode across a diffraction grating at grating incidence. The end mirror of the laser cavity reflects the first diffraction order of the grating to provide feedback.

Dispersion provided by the grating allows the laser to operate as only one cavity mode, resulting in a very narrow linewidth. The zero diffraction order of the grating serves as the output beam of the laser.
The angle between the grating and the end mirror determines the laser wavelength. Tuning is achieved by varying the angle using a piezoelectric actuator to rotate the end mirror. Continuous tuning for mode hop free behaviour requires selecting an appropriate rotation point.

Discontinuous tuning characterised by periodic mode hop results from two competing wavelength-selection constraints, the mirror-grating angle and the laser-cavity length. The laser cavity length, \( L \), defines a discrete set of possible wavelengths or modes, \( \lambda_Q \), that the laser can operate on as given by the equation:

\[
L = Q \lambda_Q/2
\]  
(2.2)

where \( Q \) is an integer. The grating equation is:

\[
\lambda = \Lambda \left( \sin \theta_i + \sin \theta_d \right)
\]  
(2.3)

where \( \Lambda \) is the groove spacing of the grating, \( \theta_i \) is the incident angle of the laser beam and \( \theta_d \) is the diffracted angle of the laser beam. Rotation of the end mirror causes the parameters in both equations to change. An appropriately selected point of rotation synchronizes the two, such that the cavity length remains the same number of half-wavelengths long as the mirror is being rotated. When this occurs, mode-hop free tuning is achieved. When this condition is not met, the laser wavelength will periodically hop from one mode to the next (e.g. \( Q \) to \( Q+1 \)) as the laser is tuned. The mechanical design of the Vortex laser is advertised as providing truly a mode-hop free tuning.

The Vortex controller provides current, voltage and temperature controls to the laser head, as well as manual and computer controlled input and output interfaces. The low noise current supply drives the diode in the laser head, controlling the laser wavelength. The temperature controller regulates the laser-cavity temperature, providing a stable output wavelength.
2.4 Summary

Various experimental techniques have been proposed in the literature in order to obtain the force distribution underneath a granular pile. The main challenge in measuring granular media is to obtain whole-field experimental results without having to perturb the granular pile, which requires stiff substrates. Interferometry is therefore an ideal experimental technique because of its excellent sensitivity. Some of the main interferometry configurations have been outlined in this chapter.

In measuring the force distribution, one can use a suitable rigid sphere in contact with the elastic substrate. This area of study is known as contact mechanics based on the pioneering work by Hertz [4]. Many mathematical models have subsequently been developed for more complex geometries although these normally require some form of numerical studies.

Another approach is the FEM, whereby the indentation process is simulated within computer software in order to predict the outcome of the indentation. From this analysis, parameters such as stress or strain can be obtained. FEM will be used as a tool to simulate the indentation of layered substrate in order to design the elastic substrate. Chapter 4 will discuss this analysis in greater detail.

The technique used in the experiment is an interferometer technique based on a Fizeau set-up as this is a common path interferometer and it is usually used to determine the surface flatness of optical components. This idea is exploited in the experiment in order measure the displacement due to the indentation. The optical arrangement will be discussed in Chapter 5.
CHAPTER 3
FRINGE ANALYSIS TECHNIQUES

3.0 Introduction

The measurement technique used throughout this thesis is the interferometry method. This enables physical parameters such as deformation, strain, refractive index, vibration and surface profile to be measured. The method uses the phenomenon of optical interference, which involves the superposition of two coherent beams of light, leading to constructive and destructive addition of rays.

This technique produces a two-dimensional intensity profile (or interferogram) which has been phase modulated. The information needed is encoded in a fringe pattern which, in turn, needs to be analysed. This chapter will discuss the methods presented in the literature, as well as the analysis used for the experiments described in Chapter 5.

The fringe phase can be calculated either by shifting the fringes through a known phase change or by Fourier Transformation. In both cases, the phase will lie between $-\pi$ and $\pi$. This is called the wrapped phase. The wrapped phase is then further processed by restoring the unknown multiple of $2\pi$ at each pixel, a technique known as phase unwrapping.

There are two main forms of analysis on fringe patterns namely intensity and phase analysis. The intensity-based technique was among the first methods, to be used, and this occurred significantly before the introduction of phase analysis. The intensity-based technique is carried out by digitizing the fringes and trying to track fringes from one pixel to another. The limitations of this are the difficulty in getting a good fringe numbering on the intensity data, because of an unpredictable or complicated interferogram; poor reliability in the presence of noise and the fact that data are obtained only at the fringe maxima and minima.
Most interferometry techniques require the beam to be split into two: the test and reference beams. A schematic of this type of interferometer is shown in Figure 3.0. A phase difference is then introduced between these beams. The beams are then recombined to fall onto a charge coupled device which then measures the intensity distribution.

![Figure 3.0: Schematic diagram of the Interferometer.](image)

Phase analysis methods can give direct measure of phase information, which is directly proportional to the displacements when deformation measurement are measured. Furthermore, this method gives more precise information than the intensity-based technique of typically ten to a hundred times higher precision.

Phase measurements can be performed by either a temporal or a spatial method, which both involve a phase shift between the test and reference beams. The phase information in the temporal method is contained in the time sequence of the phase shifted images. For spatial methods the phase shift is recorded spatially separated images falling simultaneously on one or more image sensors. Alternatively, a spatial carrier technique can be used, which does not employ a phase stepping device.

There are many ways to the phase: by moving a mirror, tilting a glass plate, moving a grating, rotating a half wave plate or use of an electro-optic modulator. The most common technique is the use of a piezo-electric translator (PZT). The PZT will push a mirror or glass wedge by a controlled amount when a known voltage is applied. The PZT is placed in one of the two beams in the interferometer system.
The measurement process is usually applied to determine some physical change between the initial and final state. The interferograms are then computed as to give the phase change between them. The initial state may take the form of a test surface or undeformed state of the specimen. Each phase value will lie between \(-\pi\) and \(\pi\), the phase change values will then lie in the range of \(-2\pi\) to \(2\pi\). These values are usually wrapped back onto the principal range of \(-\pi\) to \(\pi\) before the unwrapping process.

As already stated, the phase shift techniques will result in the fringe patterns in the estimated wrapped phase lying in the range of \(-\pi\) and \(\pi\). These are the values modulo \(2\pi\) of the true phase change. The process of phase unwrapping is therefore required so as to remove the \(2\pi\) phase discontinuities. This is done by addition of the correct integral multiple of \(2\pi\) to each phase value. It is equivalent to assigning fringe orders in the intensity-based fringe analysis.

In order to unwrap correctly for a given phase distribution, the original phase function must be sampled in accordance with Shannon sampling theorem i.e. two samples per cycle. If this requirement is satisfied, the true phase change between two neighbouring sample points then lies in the range \(-\pi\) to \(\pi\). The unwrapping process will then be able to give the absolute phase difference between neighbouring wrapped phase.

The unwrapping process becomes difficult when the absolute phase difference between neighbouring pixels is greater than \(\pi\). Such undersampling may be caused by intensity noise, discontinuities of surface, and speckle decorrelation. In recent years many algorithms have been developed that are able to correctly identify such problem areas within an image with varying levels of success.

The algorithm can be classified as either temporal phase unwrapping or spatial phase unwrapping. This is in accordance of either the phase unwrapping is performed along the time axis or spatial axis respectively.

The physical quantity of interest can then be extracted, which involves conversion of the unwrapped phase map. For a displacement field, this involves a simple scaling of the unwrapped phase map. For an out of plane interferometer, this is given by:
u_z(x, y) = \frac{\lambda \Delta \phi(x, y)}{4\pi} \quad (3.0)

where \( u_z \) = displacement, \( \lambda \) = wavelength and \( \Delta \phi \) = unwrapped phase change, and \( x \) and \( y \) are coordinated representing position on the sample surface.

Taking into account the optical magnification, the lens distortion and \( x \) and \( y \) can be related to pixel coordinates by the pixel size of the CCD camera.

### 3.1 Phase Shifting

Generally it is impossible to obtain a unique phase distribution from a single interferogram as the positive displacement cannot be distinguished from the negative displacement without further information. The usual method of solving this problem is to add to the phase function a known phase ramp, or carrier which is linear in time.

Common phase shifter devices are the PZT or a tunable diode laser in an interferometer with unequal path lengths. There are two ways that a measurement can be carried out. This is either phase stepping or phase shifting. Phase stepping is when the phase is held constant for the integration time of the detector device, and then increased by a known phase increment ready for the next frame. Phase shifting on the other hand is when the phase is modulated during the detector’s integration time. This means that the phase is shifted using the modulator in order to produce a linear ramp throughout the exposure time. This results in lower intensity modulation than the phase stepping. This is due to the cosinusoidal fringe signal being convolved with the time window duration, equal to the exposure time of the camera. The subsequent processing steps are, however, the same for both phase step and phase shift methods.

In either cases the phase is shifted by a known amount between the intensity measurements. The intensity at measurement or frame \( j \) can be written as:

\[ I_j = I_0[1 + \gamma_0 \cos(\phi + \alpha_j)] \quad (3.1) \]

where \( I_0 \) = background intensity, \( \gamma_0 \) = fringe visibility, \( \phi \) = wavefront phase, \( \alpha_j \) = phase shift for frame \( j \). All the terms in equation (3.1), except for \( \alpha \), are functions of \( x \) and \( y \).
In equation 3.1, the three unknowns are $I_o$, $\gamma_o$ and $\phi$, which require a minimum of three measurements to determine the phase. The phase shift between each frame taken can be any value between 0 and $\pi$.

These techniques are relatively simple. However, these measurements are affected by several errors that contribute to inaccuracy. Among these errors are phase-shifter miscalibration, non-linearity effects, vibration and air turbulence.

The vibration and air turbulence can be minimized by isolating the system on a vibration free table and shielding the beam from air turbulence. The choice of the phase stepping algorithm to solve equation 3.1 can influence the sensitivity towards error. The following will discuss several algorithms commonly used in the literature.

### 3.1.1 Three-Frame Technique

This algorithm requires three interferograms to be recorded [79]. The phase shift is $\frac{\pi}{2}$ and it is convenient to write, $\alpha_1 = \frac{\pi}{4}$, $\alpha_2 = \frac{3\pi}{4}$, $\alpha_3 = \frac{5\pi}{4}$. The intensity can then be expressed as:

\[
I_1 = I_o \left[ 1 + \gamma \cos \left( \phi + \frac{\pi}{4} \right) \right] = I_o \left[ 1 + \sqrt{2} \gamma \left( \cos \phi + \sin \phi \right) \right]
\]

\[
I_2 = I_o \left[ 1 + \gamma \cos \left( \phi + \frac{3\pi}{4} \right) \right] = I_o \left[ 1 + \sqrt{2} \gamma \left( -\cos \phi - \sin \phi \right) \right]
\]

\[
I_3 = I_o \left[ 1 + \gamma \cos \left( \phi + \frac{5\pi}{4} \right) \right] = I_o \left[ 1 + \sqrt{2} \gamma \left( -\cos \phi + \sin \phi \right) \right]
\]

This gives the phase at each point as:

\[
\phi = \tan^{-1} \left( \frac{I_3 - I_2}{I_1 - I_2} \right)
\]
3.1.2 Carré Technique

This technique is independent of the amount of the phase shift [80]. This requires four interferograms being shifted by $\alpha$. Since this method is independent of the phase shift, it is not sensitive to miscalibration of the phase shifter.

The equation from the intensity measurements gives:

\[
I_1 = I_0 \left[ 1 + \gamma \cos \left( \phi - \frac{3\alpha}{2} \right) \right] \\
I_2 = I_0 \left[ 1 + \gamma \cos \left( \phi - \frac{\alpha}{2} \right) \right] \\
I_3 = I_0 \left[ 1 + \gamma \cos \left( \phi + \frac{\alpha}{2} \right) \right] \\
I_4 = I_0 \left[ 1 + \gamma \cos \left( \phi + \frac{3\alpha}{2} \right) \right]
\] (3.4)

The phase shift can be calculated from equation (3.4) as:

\[
\alpha = 2 \tan^{-1} \left[ \frac{3(I_2 - I_3) - (I_1 - I_4)}{\sqrt{(I_2 - I_3) + (I_1 - I_4)}} \right] \\
\] (3.5)

with the phase at each point given by:

\[
\phi = \tan^{-1} \left\{ \frac{\tan \left( \frac{\alpha}{2} \right) \left[ (I_1 - I_4) + (I_2 - I_3) \right]}{\left[ (I_2 + I_3) - (I_1 + I_4) \right]} \right\}
\] (3.6)
3.1.3 Four-Frame Technique

The Four-Frame Technique requires four recorded interferograms [81]. This is a common algorithm with a phase shift of $\frac{\pi}{2}$ giving $\alpha_1 = 0$, $\alpha_2 = \frac{\pi}{2}$, $\alpha_3 = \pi$, $\alpha_4 = \frac{3\pi}{2}$. The equations are then given as:

\begin{align*}
I_1 &= I_o \left[ 1 + \gamma \cos \phi \right] \\
I_2 &= I_o \left[ 1 + \gamma \cos (\phi + \frac{\pi}{2}) \right] = I_o \left[ 1 - \gamma \sin \phi \right] \\
I_3 &= I_o \left[ 1 + \gamma \cos (\phi + \pi) \right] = I_o \left[ 1 - \gamma \cos \phi \right] \\
I_4 &= I_o \left[ 1 + \gamma \cos (\phi + \frac{3\pi}{2}) \right] = I_o \left[ 1 + \gamma \sin \phi \right]
\end{align*}

The phase is therefore given by:

$$\phi = \tan^{-1}\left( \frac{I_4 - I_2}{I_1 - I_3} \right)$$

3.1.4 Five-Frame Technique

An algorithm to minimize phase calibration errors using a phase shift of $\frac{\pi}{2}$ is the Five-Frame Technique [82]. This gives a phase shift of $\alpha_1 = -\pi$, $\alpha_2 = -\frac{\pi}{2}$, $\alpha_3 = 0$, $\alpha_4 = \frac{\pi}{2}$, $\alpha_5 = \pi$. The intensity measurements are:

\begin{align*}
I_1 &= I_o \left[ 1 + \gamma \cos (\phi - \pi) \right] = I_o \left[ 1 - \gamma \cos \phi \right] \\
I_2 &= I_o \left[ 1 + \gamma \cos (\phi - \frac{\pi}{2}) \right] = I_o \left[ 1 + \gamma \sin \phi \right] \\
I_3 &= I_o \left[ 1 + \gamma \cos \phi \right]
\end{align*}
\[ I_4 = I_0 \left[ 1 + \gamma \cos \left( \phi + \frac{\pi}{2} \right) \right] = I_0 \left[ 1 - \gamma \sin \phi \right] \]

\[ I_5 = I_0 \left[ 1 + \gamma \cos \left( \phi + \pi \right) \right] = I_0 \left[ 1 - \gamma \cos \phi \right] \]  

(3.9)

The phase can then be calculated as:

\[ \phi = \tan^{-1} \left[ \frac{2(I_2 - I_4)}{2I_3 - I_5 - I_1} \right] \]  

(3.10)

Two sets of images are acquired both for the before and after the experiment. At each set of images, the following comparisons can be made. The first and the fifth frame should look the same because there is a \(2\pi\) phase shift between them. The first and third have complementary intensities because there is a \(\pi\) phase shift. This \(\pi\) phase shift also occurs between the second and fourth frames and the third and fifth frames. However, due to the presence of error, there may be differences between them.

This method has a significantly greater tolerance for mis-calibration: the phase errors vary as the square of the mis-calibration factor rather than a linear variation that occurs when using the Four-Frame method.

### 3.1.5 “2+1” Technique

This technique is used when the frame is taken in a fast manner in the presence of vibration and air turbulence [83, 84]. The quick data recording is important when long path lengths are tested i.e. the mirrors are not in the same table as the system.

The optical system does not need to be isolated. The first two frames are taken quickly with a \(\frac{\pi}{2}\) phase shift. This minimises the vibration and air turbulence. The
third frame is the average of the two frames with a $\pi$ shift between them. The intensity is given as:

\[ I_1 = I_o \left[ 1 + \gamma \cos \phi \right] \]

\[ I_2 = I_o \left[ 1 + \gamma \cos \left( \phi - \frac{\pi}{2} \right) \right] = I_o \left[ 1 + \gamma \sin \phi \right] \]

\[ I_3 = \frac{1}{2} \{ I_o \left[ 1 + \gamma \cos \phi \right] \} + \frac{1}{2} \{ I_o \left[ 1 + \gamma \cos \left( \phi + \pi \right) \right] \} = I_o \] (3.11)

This can be used to calculate the phase as:

\[ \phi = \tan^{-1} \left( \frac{I_2 - I_3}{I_1 - I_3} \right) \] (3.12)

### 3.1.6 Scanning Phase Shift Technique

This technique can be used if there is the presence of vibration and air turbulence. The technique however needs a large collection of data frames with random phase shifts [85]. The intensity, as given in equation 3.1 with a phase shift $\alpha_j$ is of random values between 0 and $2\pi$. For each data frame, the maximum and minimum intensity is determined. When the number of frames becomes large, the intensity reaches maximum and minimum values of $I_{\text{max}}$ and $I_{\text{min}}$.

This can then be used to determine $I_o$, where:

\[ I_o = \frac{I_{\text{max}} + I_{\text{min}}}{2} \] (3.13)

\[ \gamma = \frac{I_{\text{max}} - I_{\text{min}}}{2I_o} \] (3.14)

Giving the phase as:

\[ \phi = \cos^{-1} \left( \frac{I_1 - I_o}{\gamma I_o} \right) \] (3.15)
3.1.7 Phase Errors

Errors in the phase measurement are primarily due to systematic and random errors. The sources of systematic errors are 1) miscalibration of the phase shifting device; 2) high harmonics in recorded intensity and; 3) vibration and air turbulence.

Error cause by miscalibration of phase shifting device may cause the phase value to deviate from the real phase. If a constant calibration error is present, the phase shift can be written as:

$$\phi' = \phi(1 + \varepsilon)$$  \hspace{1cm} (3.16)

where $\phi$ is the desired phase shift, $\phi'$ is the actual phase and $\varepsilon$ is the normalised error.

Several approaches have been developed to reduce the influence of this type of error [82, 86-92]. The phase error can be reduced by choosing an improved window function in the time domain to reduce the effect of spectral leakage (the height of the side bands) in the frequency domain. The Five Frame Algorithm [82] is an example of this type of algorithm having one extra sample with $M = N + 1$, with $N = 4$ where $M$ is the total number of frames required for the phase shifting algorithm and $N$ is the number of frames per-cycle for the phase shifting algorithm. However in the presence of harmonic content, it is advisable to increase the number of samples to a minimum of $M = 2N - 2$.

The measurement from the intensity signal may not be a pure sinusoidal wave. This may be due to high harmonics present in the recorded signal. This is due to non-linearities in the photo detector array and the multiple beam interference due to reflections.

In sampling, the harmonics with a frequency greater than the Nyquist frequency, $k_t = N/2$, will be aliased in the range of $-N/2$ to $N/2$. This may interfere with the frequency at which the phase is calculated i.e. $k_t = 1$. Harmonics components with a frequency $q$ times the fundamental will produce corresponding spectral peaks at $k_t = jN \pm q$, hence significant phase errors can be expected if $N = q \pm 1$. Therefore, the phase shifting algorithm should be chosen such that $N > q_m + 1$, where $q_m$ is the maximum frequency component in the recorded intensity signal normalised by the carrier frequency.
In order to get a good measurement, the optical set-up must be isolated from vibration and shielded from air turbulence. To reduce vibration, the optical set-up should be assembled on a vibration isolated table. The optical paths need to be covered so as to reduce air turbulence. de Groot [93, 94] has developed numerical methods to calculate the frequency response of the root mean square (RMS) phase error from any phase-shifting algorithm. The largest phase error is produced at a vibration frequency equal to twice the fundamental carrier frequency. Most phase shifting algorithms are equally sensitive to vibration at this resonant frequency.

Several solutions have been developed in recent years. In general, if the sampling rate is as high as possible, with respect to vibration frequency, the effects of this error may be reduced. Alternative methods include the introduction of hardware such as a feedback loop to stabilise the phase function [95]. High speed cameras or pulsed lasers which are capable of freezing the motion of the frame will often allow data to be obtained under high vibration levels.

Random errors will be present in the measurement and can be caused by intensity noise due to variations in the laser output power, random variations in the illumination and poor signal modulation. For an optimum phase shifting algorithm in the presence of random intensity errors, the minimisation of error is due to the number of intensity samples taken. This gives the average standard deviation in phase scaling to be $1/\sqrt{M}$, and the choice of sampling coefficients has a limited effect [90].

### 3.1.8 General Algorithm

The phase shifting algorithms are usually written as the arctangent ratio between the two combinations of the intensity values. The phase can be written as [96]:

$$\phi = \tan^{-1} \left[ \frac{\sum_{i=0}^{M-1} I_i b_i}{\sum_{i=0}^{M-1} I_i a_i} \right]$$  \hspace{1cm} (3.17)
where \( b_i \) and \( a_i \) are the real sampling coefficients and \( I_i \) is the intensity at the \( i^{th} \) frame.

The Four-Frame Technique is an example of the \( N \) frame technique. This means \( N \) frames are recorded with a phase shift of \( \alpha_j = \frac{2\pi}{N} \). If however it is necessary to eliminate the error, due to high harmonics, the algorithm of \( N > j + 1 \) frame can be used [97].

In minimizing phase shift calibration errors, \( M = N + 1 \) frame techniques can be used. The Five Frame techniques is an example of this sort of algorithm where the first and the last frame is similar due to a \( 2\pi \) phase shift. In view of this, the Five-Frame technique has greater tolerance to phase shift error than the Four-Frame Technique.

However, the \( N + 1 \) frame technique will not work well if both phase shift miscalibration and high harmonics are present. The algorithm best suited for this case is the \( M = 2N - 2 \) [97, 98].

Table 3.0 shows some of the most important phase shifting algorithms [62]. Algorithm 1 and 2 are the \( N \) frame technique. Algorithms 2 and 3 are the Four-Frame and Five-Frame techniques as discussed in previous sections. Algorithms 3 and 5 are the corresponding \( N + 1 \) algorithms. Algorithms 4 and 6 are examples of methods that are insensitive to high harmonics and linear phase shift error [82, 97]. Algorithms with large value of \( M \) are useful; for smooth - wavefront interferometer when accurate results are required. It is also useful to remove the miscalibration of phase shifting. The accuracy and the robustness of the different algorithms is shown and explained in detailed in Reference [86, 87, 90, 99].
Table 3.0: Phase Shifting Algorithm [62, 100]

<table>
<thead>
<tr>
<th>Algorithm</th>
<th>N</th>
<th>M</th>
<th>( b_i )</th>
<th>( a_i )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>3</td>
<td>3</td>
<td>( \sqrt{3} (-1,0,1) )</td>
<td>2,-1,-1</td>
</tr>
<tr>
<td>2</td>
<td>4</td>
<td>4</td>
<td>0,-1,0,1</td>
<td>1,0,-1,0</td>
</tr>
<tr>
<td>3</td>
<td>4</td>
<td>5</td>
<td>0,-2,0,2,0</td>
<td>1,0,-2,0,1</td>
</tr>
<tr>
<td>4</td>
<td>4</td>
<td>6</td>
<td>0,-2,-2,2,0</td>
<td>1,1,-2,-2,1,1</td>
</tr>
<tr>
<td>5</td>
<td>6</td>
<td>7</td>
<td>( \sqrt{3} (-1,3,3,0,-3,-3,1) )</td>
<td>3(-1,-1,1,2,1,-1,-1)</td>
</tr>
<tr>
<td>6</td>
<td>6</td>
<td>10</td>
<td>( \sqrt{3} (-1,-3,-3,1,6,6,1,-3,-3,-1) )</td>
<td>1,-1,-7,-11,-6,6,11,7,1,-1</td>
</tr>
</tbody>
</table>

3.1.9 Fourier Transform Method

This technique involves introducing a spatial carrier frequency \( f_o \) into the fringe pattern [101]. The fringe pattern can be written as:

\[
I_j(x,y) = a(x,y) + b(x,y) \cos [ \phi(x,y) + 2\pi f_o x ] \tag{3.18}
\]

where \( a(x,y) \) is \( I_0 \), \( b(x,y) \) is \( I_0 \gamma_0 \), which are the background intensity and contrast functions respectively, \( \phi(x,y) \) is the phase to be calculated, and \( f_o \) is the spatial-carrier frequency. In many cases \( a(x,y) \), \( b(x,y) \) and \( \phi(x,y) \) vary slowly compared to the spatial frequency.

If an interferogram is recorded, omitting the \( x \) and \( y \), the fringe can be written as:

\[
I_j = a + c e^{2\pi f_o x} + c^* e^{-2\pi f_o x} \tag{3.19}
\]

where \( c = \frac{1}{2} b e^{i\phi} \) and * is the complex conjugate.

Equation (3.18) is then Fourier Transform. The Fast-Fourier Transform (FFT) algorithm can be used on this to give:

\[
I(f) = A(f) + C(f_x - f_o) + C^*(f_x + f_o) \tag{3.20}
\]
where the capital letter denotes the Fourier spectra.

![Figure 3.1: Schematic of Fourier Transform of equation (3.20).](image)

Since $\alpha, \beta$, and $\phi$ are slowly varying compared to the spatial carrier, the Fourier transforms terms are separated by the carrier frequency, as shown in Figure 3.1. Using either of the two side lobes, for example $C(f_x-f_0)$, and translating by $f_0$ toward the origin, the single term $C(f_x)$ is obtained.

The next step is then to take the inverse Fourier Transform of $C(f_x)$ with respect to obtain $c(x,y)$. The phase is then determined by:

$$\phi = \tan^{-1} \frac{\text{Im}[c]}{\text{Re}[c]}$$

(3.21)

The advantage of using this technique is that it needs only one image to be recorded. The drawback is that the accuracy is limited due to the unwanted variations of $\alpha$ and $\beta$. Hence this technique is most suitable for regular fringe patterns with little contrast variations across the image and where it is difficult to record sequences of phase shifted images, for example when studying dynamic events.

In recent years, interferometry method using tunable light source to measure surface profile has been proposed [75, 102-105] as the method is able to give high resolution measurements and a large absolute measuring range. The method employs the use of FFT method for the wavelength shift. Reference [103] present a surface measurement using a tunable diode laser with an external cavity based on Littman-
Metcalf [76] design which are capable to tune the wavelength at a very high speed (less than 1 s) without having mode hop. The success of this method enable the successful measurements of smooth and rough surface with small and large heights, in level and in tilted positions.

3.1.10 Design of Phase Shifting Algorithms Using Window Function

The experimental sample uses a double layer elastic substrate as discussed in Chapter 4. This gives rise to problems such as back reflections from the unwanted surfaces. These reflections cause phase errors in the form of higher harmonics. Two sets of images were captured and subtracted to give the phase difference map in order to view this error. Ideally one would expect a uniform shade of grey images. However this is not the case as the phase difference map exhibits these errors. This results in the need to design a phase shifting algorithm that is able to minimise this effect.

Figure 3.2 shows a wrapped difference map using the Five Frame method from Table 3.0. The figure clearly shows traces of phase errors due to the higher harmonics. The calculated root mean square (rms) in the corresponding displacement fields of the order of 21 nm was found to be very large that may affect the results from the experiments.

To overcome this, the algorithm must have high number of frames per cycle of the carrier, i.e. a large N value. Figure 3.3 is the wrapped phase difference map using the Ten Frame method. It shows that the errors are still present in the wrapped difference phase map with a rms to be of the order of 6 nm. This error is still relatively large and will affect the outcome of the end results.

de Groot has shown how one can use the concept of data sampling windows to create customised phase shifting algorithms with arbitrarily large values of N [106, 107]. In an extreme example of this, he proposed the 101-frame phase shifting algorithm [107]. The sampling coefficients were based on equation (3.17) and can alternatively be expressed as:
\[ \phi = \tan^{-1}\left( \frac{\text{Im}(z(t))}{\text{Re}(z(t))} \right) \]  

(3.22)

where \( z(t) \) is defined as:

\[ z(t) = \left[ \sum_{t'=0}^{M-1} a(t') I(t + t') + i \sum_{t'=0}^{M-1} b(t') I(t + t') \right] \times \exp(-i\Delta \alpha t) \]  

(3.23)

This can be expressed as a window function as follows:

\[ a(t) + ib(t) = w(t)e^{-i\Delta \alpha t} \]  

(3.24)

where \( t = 0, 1, 2 \ldots , M-1 \)

\[ w(t) = \text{the windowed function} \]

The proposed window function to be applied is the Hanning Window [108, 109]. The sampling coefficients were then evaluated using the window in the form of:

\[ w(t) = \frac{1}{2} + \frac{1}{2} \cos \left( \frac{2\pi}{M} \left( t - \left( \frac{M-1}{2} \right) \right) \right) \]  

(3.25)

\[ t = 0, 1, \ldots, M-2 \]

and \( M - 1 \) frames are used [108]

Figure 3.4 is the wrapped phase map using the Fifteen Frames – Hanning Window. It shows a much smoother surface compare with using the Ten Frame and Five Frame Algorithm as shown by the colourbar in going from Figure 3.2 to Figure 3.4. The rms error is about 1.3 nm. This algorithm will be used throughout the experiment.
Figure 3.2: Phase Difference Map for Five Frame Algorithm.

Figure 3.3: Phase Difference Map for Ten Frame Algorithm.
Figure 3.4: Phase difference map for Fifteen Frame Algorithm – Hanning Window.

3.1.11 Phase Shifting Hardware

The most common phase shifter in an interferometer is a piezo-electric translator (PZT) primarily due to low cost and low drive requirement. Low voltage PZTs can move the required fraction of a μm with only a few volts. This therefore can be driven directly from the output of a digital to analogue converter.

The approach involves reflecting either the object beam or the reference beam from a PZT mounted mirror. If the beam is incident along the normal to the mirror, a movement of $\frac{\lambda}{2}$ will produce a $2\pi$ phase shift. It is worth mentioning that the PZT will induce a small lateral translation in the beam which may cause simultaneous intensity changes especially if there is a spatial filter following the phase shift device. A PZT driven glass wedge can also be employed to avoid this unwanted lateral translation or tilt of the beam [110]. Optical fibres can also be used to construct a compact fibre-based phase shifting interferometer [111, 112].
Interferometry based on diode lasers is also an attractive method. Changes in the injection current cause small changes in the wavelength. By arranging an unequal path length e.g. interference from successive layers of multi-layered structure, the phase shifting can be carried out without the use of mechanical translator device.

The problems that may be encountered are; a) the mode hopping of the laser and b) the simultaneous changes in the laser intensity. However, the phase shifting algorithm can be designed to be insensitive to such changes [87]. External cavity modulated diode laser has also become more available in recent years which offer greater tuning range without mode hops. In the experimental set-up discussed in the subsequent chapter, a New Focus Vortex Tunable laser is used as the wavelength tuning device and the light source.
3.2 Phase Unwrapping

The previous section describes a range of methods for determining the phase term in an interferogram. The process of converting this information into a continuous function is called phase unwrapping. The phase is determined from an arctangent function of the image intensity. The arctangent function returns a phase value lying in the range $-\pi$ to $\pi$, known as the wrapped phase. As a result, there are $2\pi$ phase discontinuities in the measured phase map. The wrapped phase then undergoes an unwrapping process in which an integral multiple of $2\pi$ is added at each pixel. Figure 3.5 shows the unwrapping process.

![Figure 3.5: Unwrapping process. The wrapped phase is converted by addition of integral multiple of $2\pi$ to restore the phase absolute value.](image)

The process is carried out by unwrapping the phase along a path through the data. This path can either be along a spatial axis or along a time axis. The basic principle is to estimate the phase gradient between two adjacent pixels in an image or between two successive phase values. If the gradient value exceeds the threshold of $\pi$, then a phase discontinuity is then assumed to lie between these two points and the phase jump is then corrected by adding or subtracting $2\pi$, according to the sign of the phase gradient. The unwrapped phase at any point is given by $\phi + 2\pi N_e$, where $N_e$ is the fringe order counter.
A successful unwrapping process leads to the correct identification of the discontinuities. If the experimental data have a low signal to noise ratio, the detection of the discontinuities can become difficult. If the fringe pattern contains defects, it is not always possible to unwrap the phase and maintain the correct phase relation. These defects can be structural, such as cracks, holes, bubbles or others related to the interferometric technique. High density fringe patterns could also cause unwrapping errors. An increasing density in the fringe pattern cannot usually be avoided when the sample analysed is deformed.

It is then important to consider a good unwrapping algorithm. The difference between these algorithms is the strategy employed to recognize discontinuities and error sources. The main factor is the determination of the integration path, because the phase difference between two points is independent of the integration path chosen, as long as it does not cross the invalid data.

The method for unwrapping can be described as a path dependent method. The simplest method is 'sequential linear scanning' [113]. This method scans sequentially through the data, line by line. At the end of each line, the phase difference between the current line and the pixel on the line below is determined. The line below is then scanned in the reverse direction. The two dimensional data array is treated like a folded one dimensional data set.

'Multiple Scan Directions' involve unwrapping by scanning sequentially through the data [114, 115]. At each point, the phase gradient is measured in two directions up and left. The approach depends on the error checking procedures working correctly. If the errors are let through then a phase discontinuity can propagate through the rest of the array.

The 'Spiral Scanning' technique uses the central pixel as the starting point [116]. The phase gradient is then measured between the current pixel with unmasked 3 x 3 neighbourhood pixels that have been unwrapped. The process is repeated by spiralling through the array around the growing central area of the unwrapped data.

To avoid phase errors propagating through the data array, it is important to unwrap the regions of 'good' pixel data first. These data are unlikely to propagate errors. The 'bad' data pixels with high measurement uncertainty are then unwrapped.
but the propagation of errors is confined to small regions. This technique is called 'Pixel Queuing' [117]. The method however depends on the definition of 'good' or valid data.

The image being unwrapped can also be segmented into regions containing no phase ambiguities [118, 119]. This is known as 'Unwrapping by Regions'. Smaller regions are taken at the edges of the larger regions and compared with larger regions as a whole if there is no phase jump between them. The advantage of this technique is that the small region will only permit small decisions about the phase jumps when analysing more complex data images.

A basic problem with these techniques is that they do not address the fundamental cause of the path dependence within a 2-D phase map, namely the presence of so called residues (also known as discontinuity sources, poles and phase singularities in the literature). Figure 3.6 illustrates this problem when unwrapping spatially in 2-D. Given the phase at pixel P, the phase at any other pixel (e.g. Q) can be unwrapped by counting the number of $2\pi$ discontinuities along any path (either A or B) linking the two points. In Figure 3.6a, all paths chosen give the same answer which is $6\pi$. However in Figure 3.6b, noise in the centre of the field of view has caused a break in one of the $2\pi$ discontinuities, forming two 'discontinuity sources' (points 1 and 2). If point Q is to be unwrap relative to point P, then a spatial unwrapping by path B would indicate that $4\pi$ should be added, whereas the correct answer (path A) is the $6\pi$. Thus local noise can propagate to cause global phase errors [120].
Figure 3.6: Illustration of spatial phase unwrapping, where the phase at Q relative to P is obtained from the number of the $2\pi$ discontinuities crossed by a path linking the two points: a) noise-free case and; b) noise at the centre of the field causes unwrapping errors dependent on the path taken.

A family of techniques known as the branch cut method has evolved in which barriers to unwrapping (the branch cut) are placed between residues of opposite sign. The simplest of these is the ‘nearest neighbour’ algorithm [120]. This method requires that for a given phase at a pixel $(x_0, y_0)$, the phase at any other point $(x, y)$ in the image should be defined uniquely, independent of the unwrapping path. The integral of the phase jumps should be zero. A non-zero value indicates an area in which residues occur. The sign of the integral is defined as charged and it tends to be in pairs of opposite signs called dipoles. Isolated sources may occur near the boundary and cut lines are placed between dipoles in the phase map or from a monopole to the boundary. Each residue is allowed to be at one end of a cut, with the other end attached to a source of the opposite sign, or the boundary of the phase map.

The next step is to minimise the cut-length. Several different cut placement routes will in general give the same minimum cut length. The choice of route will affect only the wrapped phase in the region containing the corrupted phase.
information. For the correct cut distribution, this method requires that the separator between dipoles is always larger than the spacing between the sources making up the dipoles.

This algorithm was developed further to give the 'modified nearest neighbour algorithm' [121]. The main feature of this algorithm is that the residues that form a dipole are grouped together before a simple rule are applied to break them up. This then split the group to leave clusters of individual dipoles. Both approaches are based on a local search method and the results, whilst an improved on the basic nearest neighbour algorithm, can still give long and physically unacceptable cuts as shown in Figure 3.7a.

An alternative approach is based on a graph theory method called the 'minimum cost matching' [122] algorithm. The algorithm will find the global minimum cut-length even for high concentrations of residues. This algorithm however may require more processing time to converge. The main cause is the specification of boundaries to the region of continuous phase. Boundaries to the continuous phase may present along crack lines, running through the middle of the surface, and this will make it difficult to identify. Figure 3.7b shows the branch cut distributions using this method. It clearly shows a better result than the modified nearest neighbour method.

![Figure 3.7: Branch cut distributions to unwrap a Brazilian test specimen. (a) The modified nearest neighbour and (b) the minimum cut length method [100].](image-url)
Another approach is to segment the wrapped phase into small rectangular regions, or tiles. Each region is unwrapped individually and to reduce any phase inconsistencies at the edges, the region are phase shifted. Regions with good values at the edges are then merged to form larger regions in an iterative manner until the whole image is reconciled. Various criteria have been used to decide which tiles should be merged first in order to maximise confidence in the unwrapped phase. One approach using minimum spanning trees [123] uses a weighting mechanism that is robust both in dealing with spike noise at the pixel level and dealing with phase inconsistencies due to aliasing and surface boundaries at higher level.

Reference [124] has proposed cellular automata techniques and consists of simple and discrete mathematical systems. For example a 2 x 2 pixel array, evolves in discrete time step based on local neighbourhood rules. Global behaviour is performed in an iterative strategy and many iterations is usually required to achieve convergence. A successful unwrapping is dependent on a suitable choice of unwrapping boundaries.

Another class of unwrapping method takes a global ‘fitting’ approach to determine the optimum unwrapped phase solution. These algorithms minimise the error term $\varepsilon^p$, using the so-called $L^p$-norm minimisation criterion. For a 2-D unwrapping problem, this can be stated as:

$$\varepsilon^p = \sum_{x=0}^{N_x-2} \sum_{y=0}^{N_y-1} |\hat{\phi}(x+1,y) - \hat{\phi}(x,y) - W[\Delta \hat{\phi}_w(x+1,y) - \Delta \hat{\phi}_w(x,y)]|^p$$

$$+ \sum_{x=0}^{N_x-1} \sum_{y=0}^{N_y-1} |\hat{\phi}(x,y+1) - \hat{\phi}(x,y) - W[\Delta \hat{\phi}_w(x,y+1) - \Delta \hat{\phi}_w(x,y)]|^p$$

(3.26)

where $W$ is the wrapping operator defined as:

$$W(\phi) = \phi - 2\pi \text{NINT}(\phi/2\pi)$$

(3.27)

and $\phi_w$ is the wrapped phase and NINT denotes rounding to the nearest integer.

The global minimisation approach differs from the path-dependent method in that it does not directly identify residues present in the wrapped phase map. In particular case is when $p = 2$, which corresponds to a least squares optimisation and can be solved by application of discrete cosine transform or fast Fourier transform [125].
The drawbacks are that a local noisy region can adversely influence the unwrapped phase map on a global scale unless pixel weighting is applied [126]. The algorithm can be improved by introduction of weighting factors to take account of region of poor data [125]. The drawback of this is that the algorithm is now iterative and the computational time will increase.

3.3 Summary

This chapter has introduce several phase shifting techniques methods used to obtain the interference phase from intensity distributions which results in obtaining the required physical quantity. In experiments, both the random and systematic errors are present and the design of these algorithms must minimise these errors. The most significant problem is the presence of high harmonics errors and hence the algorithms used must be of high N number. To further reduce this error the design of the algorithm is based on a window function called the Hanning window. From the design, the project will employ a Fifteen Frame Hanning Window as the phase shifting algorithm.

The phase unwrapping process is an important step in a whole-field optical technique. A summary of the important techniques has been given. The approach used throughout is the branch cut method in which barriers are placed between the residues of opposite sign to guarantee a path-independent unwrapping.
CHAPTER 4

FINITE ELEMENT ANALYSIS

4.0 Introduction

In this chapter, the design of the sample for the project is discussed. Before any experiments can proceed the sample which is the double layer elastic substrate needs to be manufactured. It is then essential to design the substrate in order to know the material and thicknesses for both top and bottom layer of the substrate.

The experiment uses a double layer elastic substrate cast onto a rigid glass base. The top layer is a high modulus epoxy material and the bottom layer is a low modulus silicone rubber. Between the two is a layer of gold which forms one of the two reflecting surfaces giving rise to the interference pattern.

To design the substrate, Finite Element Analysis (FEA) was use so as to optimise the desired characteristics. The FEA is able to predict the displacement of the double layered substrate after being indented by an indenter.

The modulus of the top layer must be sufficiently high, such that the indentation will give a measurable displacement at the interface. If the modulus of the top layer is too high, no displacement curve will be detected.

The substrate also depends on the thickness of the top and bottom layer. If a layer is too thick, it will increase the stiffness of the material and hence displacement may be too small to be measured accurately. If a layer is too thin, the indentation which occurs may penetrate the layers which may damage the sample and will not give any reasonable displacement curve. The measurable range of the experimental set-up is from a few (or few tens of) $\frac{\lambda}{2}$ (where $\lambda$ = wavelength of the laser) down to an expected minimum of a few nm (determined by the noise level of the phase measurement.

The deformation of the substrate needs to be localised. This is to ensure that the deformation occurring from one indentation will not affect the displacement
caused by the neighbouring indenter. However, the displacement curve should be as broad as possible within the allowable distance so as to ensure the fringes can be resolved by the camera.

The response of the interface between the top layer and the bottom layer also need to be checked to ensure a linear response. The ratio of the deflection for the top and bottom layers needs to be analyses to ensure that the deflection value on the interface will be as close as possible to that of the indenter. Optimisation of the sample is to be made in terms of the material combinations and thicknesses for both top and bottom layers.

The analysis is performed using a Windows based finite element solver package call LUSAS MODELLER. The physical model and the geometry of the analyses will be introduced in the subsequent sections. A number of simulation approaches were employed to tackle the problem and these will be discussed in detail.
4.1 MODEL GEOMETRY

4.1.1 Elastic Substrate

The schematic diagram of the sample for the experiment is shown in Figure 4.0. The whole of the elastic substrate is cast on to a glass which is approximated here as a rigid surface. The total thickness is denoted by $T$, and $t_1$ and $t_2$ are the thicknesses of the top and bottom layers respectively.

The material used for the analysis is PL3, PC10, thin glass and silicone rubber (Sylgard 184). PL3 and PC10 are respectively a high-modulus and low-modulus epoxy resin. Glass is chosen as a high modulus material and silicone is a transparent low modulus rubber. These materials were chosen because they are all transparent, can be cast or otherwise manufactured in thin layers and over a wide range of modulus values.

![Figure 4.0: A schematic of the elastic substrate for the experiment.](image)

The notation is given in the text.

The method involved trying out several materials for the analyses as to find the most suitable material combinations for the experiment. The top layer must be a compliant material with the bottom layer so that the deformation from the top layer will influenced the behaviour of the bottom layer. The top layer must be of a sufficiently stiff material that can conform to the deformation. Table 4.1 shows the

---

$^8$ PL3 and PC10 are supplied from Measurement Groups Ltd
$^9$ Glass is supplied from Econoglaze
$^8$ Silicone is supplied from Dow Corning Group
material combinations for the analysis, in order top layer – bottom layer. Table 4.2 gives the material properties used for the analysis.

Table 4.1 Material Combinations for the analysis.

<table>
<thead>
<tr>
<th>Material Combination</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL3 – Silicone</td>
</tr>
<tr>
<td>PL3 – Glass</td>
</tr>
<tr>
<td>PL3 – PC10</td>
</tr>
<tr>
<td>Glass – Silicone</td>
</tr>
<tr>
<td>Glass – PL3</td>
</tr>
<tr>
<td>Glass – PC10</td>
</tr>
<tr>
<td>PC10 – Silicone</td>
</tr>
<tr>
<td>PC10 – Glass</td>
</tr>
<tr>
<td>PC10 – PL3</td>
</tr>
<tr>
<td>Silicone – Silicone</td>
</tr>
</tbody>
</table>

Table 4.2: Material Properties for the material.

<table>
<thead>
<tr>
<th>Material</th>
<th>Young’s Modulus, $E$ MPa</th>
<th>Poisson’s Ratio, $\nu$</th>
</tr>
</thead>
<tbody>
<tr>
<td>PL3</td>
<td>1.4</td>
<td>0.42</td>
</tr>
<tr>
<td>PC10</td>
<td>3100</td>
<td>0.40</td>
</tr>
<tr>
<td>Glass</td>
<td>80 000</td>
<td>0.25</td>
</tr>
<tr>
<td>Silicone</td>
<td>0.179</td>
<td>0.45</td>
</tr>
</tbody>
</table>

PL3, PC10 and Glass material properties are obtained from the supplier data-sheet. Silicone material properties are obtained from Reference [2].
4.1.2 Simulation Methods

The geometry is 2-mm long in horizontal direction and the thickness is varied. Figure 4.1 shows the geometry of the substrate. This length size was chosen as the indenter is of 1mm radius. This is to see the effect of the deformation towards the neighbouring grains.

Boundary conditions were applied to characterise the problem area. Each surface side of the model is given a label, which corresponds to the boundary conditions applied. The substrate lies on an approximately rigid surface. The boundary conditions for each surface are as follows:

Surface 1: Rigid base, \( U_x = 0, U_y = 0 \), Fixed surface
Surface 2: Left edge, \( U_x = 0, U_y = \text{free} \), Allow to move in y direction
Surface 3: Right edge, \( U_x = 0, U_y = \text{free} \), Allow to move in y direction
Surface 4: Top Surface. Load applied dependent on type of analysis

where \( U_x = x \) displacement component
\( U_y = y \) displacement component
Once the boundary conditions were applied, the material properties can then be assigned to the appropriate layer for the analysis to proceed. The material properties are shown in Table 4.2.

The analysis used is in axisymmetric form. This is to reduce a 3-dimensional (3D) problem to a 2-dimensional (2D) one. The main positive outcome of this is that the analysis can then be performed much faster. For the load applied on surface 4, the value is divided by a factor of $2\pi$ so as to transform the load for the axisymmetric analysis.

The accuracy depends on the mesh size and thus it is important to choose the best one. Two types of mesh were used, coarse and fine. The coarse size was used as to check that the deflections were in approximately the right range. Once the results were in the correct range, a fine mesh was then used for that particular analysis.

Computation based on the fine mesh will take longer. This is because, using a fine mesh, more simultaneous equations are generated and hence a longer period is needed to solve them. A typical period of time to analyse the fine mesh using the nonlinear model is about 2-4 hours and 15-30 minutes for the coarse mesh. The disk space required for the analysis is also large which is about 20 Mb for the fine mesh and about 5Mb for the coarse mesh. To illustrate the coarse and fine mesh, Figure 4.2 and b shows the difference between the two with a 2mm by 1mm model size. Figure 4.2a shows the coarse mesh of 0.05mm mesh size having 800 elements and Figure 4.2b shows the fine mesh of 0.02mm mesh size with 5000 elements.
Figure 4.2a: Coarse mesh of 0.05mm mesh size. The model has 800 elements.

Figure 4.2b: Fine mesh of 0.02mm mesh size. The model has 5000 elements.
4.2 Solution Methods

In this section, the different load states for the Finite Element Analysis are discussed. These approaches start off as linear analysis with point load. The next step is distributed load, ball-loading analysis and non-linear ball loading. The subsequent sections discuss these approaches in details.

4.2.1 Point Load

Figure 4.3 shows a single point load applied at the left edge of the top surface (surface 4). The assumption is that the contact radius is sufficiently small that it can be treated as a single point.

![Figure 4.3: Load is applied at top left corner for Point Load Analysis.](image)

The analysis allowed the influence of mesh size and boundary conditions to be checked by comparisons with the theoretical equations for the point load [4]. Such equations assume an infinite half plane, but can be expected to be valid at points
much closer to the load application point than the layer thickness. This analysis assumes that the contact area is small enough that it can be considered as a point load.

4.2.2 DISTRIBUTED LOAD

The distributed load was then used to analyse a more realistic problem area of the contact than the point load. The contact on the surface usually occupies a small area. Each area then contributes to the load which is distributed throughout the area. Figure 4.4 shows the model for the analysis, where further nodes at the top surface are given load values.

Figure 4.4: Distributed Load at the top surface.
The distributed load is generated using theoretical Hertzian equations in [4].

The pressure distribution is given as:

\[ p = p_0 \left(1 - \left(\frac{r}{a}\right)^2\right)^{1/2} \]  
\[ p_0 = \frac{3P}{2\pi a^2} \]  
\[ a = \left(\frac{3PR}{4E^*}\right)^{1/3} \]  
\[ \frac{1}{E^*} = \frac{1-v_1^2}{E_1} + \frac{1-v_2^2}{E_2} \]

where \( P \) = load
\( p_0 \) = maximum pressure
\( a \) = contact radius
\( r \) = radial distance from the origin
\( R \) = indenter radius
\( E_1 \) = Young's Modulus of top layer, \( v_1 \) = Poisson's ratio of top layer
\( E_2 \) = Young's Modulus of bottom layer, \( v_2 \) = Poisson ratio of layer modulus

\( E_1 \) and \( v_1 \) are taken as steel where \( E_1 = 209000 \) MPa, \( v_1 = 0.30 \) and \( E_2 \) and \( v_2 \) are taken as PL3 where \( E_2 = 1.4 \) MPa, \( v_2 = 0.42 \). The radius of the indenter, \( R \) is taken as 1 mm. Fixing the radius of contact as \( a = 0.12 \) mm and rearranging equation 4.3 gives \( P = 0.0039 \) N. Substituting \( P \) into equation 4.2 gives \( p_0 = 0.1299 \) MPa. Taking \( r \) as having steps of 0.02 mm interval gives the pressure distribution, which is then converted to load, and then applied to surface 4, as shown in Figure 4.4. The addition of the load gives 0.0037010 N, where, for each load value it is converted to an axisymmetric value. This calculation is then repeated for the other material combination as they have different Young's Modulus and Poisson's ratio.

In this analysis, the effect of material combination is analysed as shown in Table 4.1 in section 4.1.1. Table 4.3 shows the thickness of each layer for the analysis. The ratio of the layer thickness is given as:
\[ \alpha = \frac{t_1}{t_2} \]  

(4.5)

where \( t_1 \) = top thickness and \( t_2 \) = bottom thickness.

The analysis then proceeded by using each material combination from Table 4.1 and using all the thickness ratios in Table 4.3. This will gives up to 60 simulations to be performed by the software. From these analyses, several material combinations could then be eliminated for further investigation.

Table 4.3: Thickness for each layer for the analysis and the \( \alpha \) value

The total thickness \( T = 1.00 \) mm

<table>
<thead>
<tr>
<th>Top Layer Thickness ( t_1 ) (mm)</th>
<th>Bottom Layer Thickness ( t_2 ) (mm)</th>
<th>( \alpha = \frac{t_1}{t_2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.10</td>
<td>0.90</td>
<td>0.10</td>
</tr>
<tr>
<td>0.17</td>
<td>0.83</td>
<td>0.20</td>
</tr>
<tr>
<td>0.25</td>
<td>0.75</td>
<td>0.30</td>
</tr>
<tr>
<td>0.33</td>
<td>0.67</td>
<td>0.50</td>
</tr>
<tr>
<td>0.50</td>
<td>0.50</td>
<td>1.00</td>
</tr>
<tr>
<td>0.67</td>
<td>0.33</td>
<td>2.00</td>
</tr>
</tbody>
</table>
4.2.3 NON-LINEAR BALL LOADING

The nature of the analysis is a non-linear one. To improve the modelling of the ball-substrate contact, a new geometry was employed, consisting of the double layer substrate and a stiff quarter section of a ball indenter, as in Figure 4.5 a and b.

The ball was moved up by 0.1 mm. Two loadcases are used for this analysis. The first loadcase brought the quarter ball bearing into contact with the substrate, as in Figure 4.5a. After contact was made, the load was then applied as shown in Figure 4.5b. The boundary conditions for the substrates were discussed in section 4.1.2.

The quarter section of the ball bearing is modelled on top of the surface, i.e. surface 4. The quarter ball bearing is given a load at the top left edge corner (shown in Figure 4.5b).

The quarter section in Figure 4.5 acts as the indenter for the analyses. Figure 4.6 shows this section with each surface given a label. The following are the boundary conditions applied:
Surface 1: Not assigned, as the loading is determined when this surface is placed on the substrate top surface
Surface 2: Left edge, \( U_x = 0 \), \( U_y \) free, Allow to move in y direction
Surface 3: Load is assigned dependant on the analysis

The geometry as in Figure 4.5 is a contact analysis problem. A feature in LUSAS called a ‘slideline’ is invoked, as this provides the option to identify the problem area as a contact problem. This is done in order to assign the line of contact between the substrate and the quarter ball bearing. Subsequent sections will discuss the ‘slideline’ in detail.

The geometry of the substrate in Figure 4.5 is divided into 4 sections. The area of interest has a high mesh density, as this is the region of contact. Moving away from the contact, the mesh size increases. This will improve the computation time and reduces the disk space needed for the analysis.

The model as in Figure 4.5 involves a non-linear analysis. This is because the contact radius between the quarter ball bearing and the surface of the substrate is an
unknown. This is invoked in the software by choosing the non-linear option. The computation then proceeds in an iterative manner. The analysis times depend on the mesh size. For a 0.05 mm mesh size, run times of 10 – 15 minutes were typically required. For a 0.02 mesh size, the run times were longer, about 2 – 4 hours.

Figure 4.5a: Fine mesh for contact analysis (displacement). First loadcase, to bring the quarter ball bearing into contact of the substrate.
Figure 4.5b: Fine mesh for contact analysis (load). Second loadcase where the load is applied onto the top surface of the substrate.

Figure 4.6: The section of the model that acts as the indenter.
4.2.4 Element Types

Once the model has been chosen for analysis, it is then important to use the appropriate element for the modelling to proceed. In the software package LUSAS, there are many different types of element that are available; these range from bar elements through those such as thermal elements, each having their own particular application.

The analysis carried out here is in two-dimensional (2D) and also in axisymmetric form. The types of elements are then restricted to a few. For this analysis, 2D continuum elements are used to model the structures and these also allow for the fact that the problem modelled is axisymmetric.

The software allows the use of triangular and quadrilateral elements. These elements can then either have 4 or 8 nodes for the quadrilateral elements and either 3 or 6 nodes for triangular elements as shown in Figure 4.7. The greater the number of nodes the greater the accuracy of the model. However, a greater number of nodes also increases the length of time taken to obtain the results.

The type of elements that was used in the models was QAX8 – a 2D solid axisymmetric elements. When slidelines are to be used, it is recommended that lower order are employed. However, the software will automatically constrain the displacements of the slideline nodes so that they behave in a linear manner [78].
4.2.5 Slidelines

In Figure 4.5, the geometry is modelled as a separate body from the substrate. The problem now becomes a contact problem. A method has to be employed to ensure correct contact between the two surfaces. LUSAS utilises a slidelines function to achieve this. Slidelines define the contacting surface for the problem. The slidelines are split into pairs, masters and slaves, which are the opposing lines/surfaces as shown in Figure 4.5a.

There are a number of different types of slidelines that can be invoked, these include:

1. Friction: Used for constant or intermittent contact
2. No Friction: Used for contact problems but ignores the effect of friction between the surfaces
3. Null: Used for a straightforward linear analysis.

Figure 4.7: Axisymmetric elements available for the analysis.
When using the slidelines function it must be noted that some elements may not work if the analysis is performed. For slidelines to work, generally a fine mesh is required where the two surfaces are contacting.

The analysis employed for the non-linear ball loading uses the penalty method. Figure 4.5 shows that the density of elements is highest in the region of contact.

The penalty method allows problems such as ball-loading to be solved. This due to the problem having an unknown contact radius between the indenter and the substrate.

The penalty method works by the use of a penalty factor that is multiplied by the penetration and gives the force required to prevent penetration. The procedure involves a screening procedure that eliminates those nodes, which need no detailed consideration.

A radial 'contact zone' is defined around each local node and contact is only possible if the current node is located within this zone. The contact zone radius is a measure of the maximum distance between any two neighbouring contact nodes. It has a different value for each slideline surface and is continuously updated throughout the solution as the segment lengths change. The relationship is defined as:

\[ r = p \cdot d \]  \hspace{1cm} (4.6)

where \( r \) is the zonal contact radius, \( p \) is the zonal detection parameter and \( d \) is the maximum distance between any two neighbouring contact nodes.

The zonal contact detection parameter controls the degree of radial overlap between neighbouring contact zones. The analysis used the default value of 10/9 to ensure the zones overlap. If it is set to less than 0.5, undetected penetration may occur. The zonal parameter may be set to a large value. This however, would be required when the displacements in the contact area are expected to be greater than the largest contact segment length in any increment. For the analysis, the default value was found to be suitable and was used throughout.

The stiffness for each contact node is based upon the material and geometric properties of the elements, which are immediately adjacent to each of the contact nodes. The stiffness scale factor is the input for the master and slave surfaces. This
factor controls the degree of inter penetration experienced in the contact zone. For the analysis, the default values were used. Larger values will reduce penetration and may cause chattering at the interface. The values for the stiffness coefficient were taken as 1 for both master and slave surface.

The analysis was performed by using two load cases as in Figure 4.5. The first load case acts as a ‘dummy’ support condition to the indenter which will permit a small amount of deformation and hence generate contact elements which will ‘join’ the two bodies together. The ‘dummy’ support is then released in the second load case to allow the complete deformation to take place.
4.3 Results

In this section, the results of the analysis performed using the model explained in Section 4.2 are discussed. This includes the comparison of the mesh, the graphs plotted for both the surface and interface and the dimensionless analyses performed. The subsequent section discusses this in further detail.

4.3.1 Mesh Accuracy

The model in LUSAS are defined in terms of geometric features which are sub-divided into finite elements for solution. This process is called meshing. The mesh datasets contains information about element type (as discussed in Section 4.2.4), element discretisation which controls the density of the mesh and the mesh types which controls the grid of the mesh.

As mentioned earlier, the analysis was done with both a coarse and a fine mesh so as to optimise the time spent on the project. It is important to check the results from the analysis with a theoretical Hertzian equation [4].

The results were compared by taking the deflection on the left edge corner of the model. In this analysis, the distributed load gave 13% discrepancy between the FEA and theoretical Hertzian equations. The mesh size used for the analysis, as in section 4.2.2, was 0.02mm. The time taken for the analysis was about 45 minutes. When the mesh size was reduced to 0.01mm, the time taken was 3 hours. The size 0.02mm was used as it gave good enough results (2-3% difference) to allow certain material combinations to be eliminated.

The accuracy for the geometry shown in Figure 4.5, with mesh size of 0.02mm, gives a range of 1.6% – 2.5% discrepancy between the results obtained by FEA and by the Hertzian equation. The analysis was performed using silicone as the material with the load of a single ball bearing having 63mg. Low loads were used in order to have a small contact radius compare with the depth of the layer for which Hertzian loading are valid. The contact radius was found to be less than 0.06mm with
the depth having 1 mm thickness. The results were compared using the deflection at the left edge corner of the elastic substrate.

4.3.2 DISTRIBUTED LOADING

Several analyses were performed using the distributed geometry as in section 4.2.2. This included varying the material combination and analysing it at different thicknesses, i.e. different \( \alpha \) ratios.

Surface and interface deflections were plotted for each material combination. Figure 4.9 shows a plot of PL3 – Silicone at \( \alpha = 1 \).

![Figure 4.9: Displacement against distance for PL3 – Silicone combinations where \( t_1 = 0.5\text{mm}, t_2 = 0.5\text{mm}, \alpha = 1 \).](image)

Figure 4.8: Displacement against distance for PL3 – Silicone combinations where \( t_1 = 0.5\text{mm}, t_2 = 0.5\text{mm}, \alpha = 1 \).

This figure shows that the deflection of the interface is much less than that of the surface, which, which is an undesirable feature if one are trying to maximise the stiffness of the substrate. The plots of displacements against distance for the other material combinations are in Appendix A.
The plots are discussed briefly as follows.

1. PL3 – Silicone
The graphs show curves of both interface and surface. As the thickness of PL3 increases, the surface plot shows a decrease in value. This is because an increase in thickness would make the material more rigid, hence the value of the deflection decreases. The curves also converge approximately 1mm away from the load.

2. PL3 – Glass
Glass is a high modulus material compared to PL3. Hence the interface plots yield small values which can be considered close to zero. This behaves like PL3 on top of a rigid base. Increasing the thickness of PL3 increases the surface depression.

3. PL3 – PC10
PC10 is also a stiff material compared to PL3. The graphs are as predicted and behave in a similar manner to PL3 – Glass. This means the interface curve plot can be assumed to be almost zero.

4. Glass – Silicone
The glass used in this analysis is assumed to be thin. The deformation of the surface and interface is similar at all $\alpha$ values. The problem is that the deflection does not tend to zero at $x \sim 1$mm, thereby causing interference with adjacent contacts.

5. Glass – PL3
PL3 is a low modulus epoxy with much lower E value compared with glass. The behaviour, as expected, was similar to the Glass-Silicone combination.

6. Glass – PC10
The ratio of moduli between glass and PC10 is about 26. The deformation is small, in the range of $10^7$ mm, which is too low to be measured by an interferometer. This is
due to both materials having a high Young's Modulus. The curve also shows that the
depression gives a broad value, for large \( \alpha \), before converging to zero.

7. PC10 – Silicone
The curves for this material combination are relatively broad. An increasing \( \alpha \) value
would also cause both plots to deviate from each other.

8. PC10 – Glass
Generally the behaviour at a high \( \alpha \) is similar to that shown by PL3 – Glass. This
combination can be considered as an epoxy on top of a rigid substrate.

9. PC10 – PL3
The curves do not converge as quickly as in PL3 – Silicone. At a high \( \alpha \), it takes too
long for \( U_y(x) \) to reach zero. This affects the neighbouring grains, which is not
desirable.

10. Silicone – Silicone
This combination can be considered as one elastic substrate on its own. The plot of
the interface is the plot at several heights. As the force is the same, the surface plots
for all \( \alpha \) values show the same curve. At an increasing \( \alpha \), the interface is nearer to the
rigid base, i.e. the boundary conditions, and the interface deflection value decreases.
A second dimensionless parameter was therefore introduced. The definition of the parameter is as follows:

\[ \beta = \frac{U_Y(0)}{U'(0)} \]  

(4.7)

where \( U_Y(x) \) and \( U'(x) \) are respectively the surface and interface deflection curves. A value of \( \beta \) close to 1 is desirable to maximise the measurable deflection for a given grain movement.

Figure 4.9: \( \alpha \) against \( \beta \) for PL3 – Silicone combination.

Figure 4.9 shows a plot of \( \alpha \) against \( \beta \) and illustrates the effect of the varying thickness ratio. The best \( \beta \) ratio is a value close to 1. This is to minimise the surface deflection for a given interface deflection. This is to detect the localised deformation
at the interface which will be recorded in the resulting fringe pattern. Graphs of $\alpha$ vs $\beta$ for other materials are plotted in Appendix B.

From the surface and interface plots, several material combinations can be eliminated. Among these are Glass – Silicone, Glass – PL3, Glass – PC10 and PC10 – PL3. All of these have broad curves, which do not converge to zero over a 2-mm distance. This means that the deflections will disturb the next ball bearing and may therefore make the results incorrect.

PL3 – Glass, PL3 – PC10 and PC10 – Glass result give small interface displacement values. The interface plot can be considered as close to zero. If the interface does not deflect, there will be difficulties in detecting the deformation from the interferometer.

Based on the $\alpha$ against $\beta$ plot, a few more material combinations can be eliminated. PL3 – Glass, PL3 – PC10 and Silicone-Silicone are eliminated because the $\beta$ ratio is far too large and clearly is not near to 1.

The possible material combinations that give the most promising results are PL3 – Silicone and PC10 – Silicone. These two material combinations will be further analysed so as to investigate which of the two is the best combination as well as to give the best thickness.
4.3.3 Non-Linear Ball Loading

The analysis to achieve the optimised substrate was then carried out using the non-linear ball loading model. The model is shown in Figure 4.5 and it consists of a section of the indenter and the substrate. Initially the indenter is moved down to be in contact with the indenter. This is then followed by the indentation procedure. Figure 4.10 shows the model employed for this type of analysis.

From the previous analysis, the probable material combination is either PL3-Silicone or PC10 – Silicone. To chose the best combinations and to find the right thickness, the analysis is performed by varying the thickness of either the top or bottom layer. The analysis was performed initially, by setting $\alpha$ as 1, with both top and bottom having 0.5 mm in thickness. By varying $\alpha$ the analysis is performed in order to determine the best thickness. Table 4.4 and Table 4.5 shows the main results for the overall thickness for the analysis for both PL3-Silicone and PC10-Silicone respectively. In Reference [2], a single ball bearing has a weight of 63 mg. Therefore, the analysis was performed using 20 ball bearings that represent the weight of stacking the ball bearings on top of one another.

In order to analyse the results, several parameters that need to be considered when choosing the substrate design are (a) the measurable range which is about $\frac{\lambda}{2}$ (where $\lambda$ is the wavelength) of the laser, (b) the value of $\beta$ which needs to be as close as possible to 1 and (c) the distance over which the deflection curve moves back to its original position.
To assist in the second of these criteria, a third dimensionless parameter is defined here:

\[ \theta = \frac{U_y(0)}{\left(\frac{\lambda}{2}\right)} \]  

(4.8)

The wavelength of the laser is about 635 nm. The typical deflections should be of the order of the range of a few \( \frac{\lambda}{2} \) as \( \frac{\lambda}{2} \) corresponds to a single fringe. The value of \( \theta \) should therefore be of order one. The value should not be more than 10, as this will create unwrapping errors. It is worth mentioning than the interference fringes must also be sampled at least twice per fringe in order to be able to give a correct measurement. This is based on Shannon’s Sampling Theorem.

To check the size of the indentation profile, \( \sigma \) is introduced, which is defined as the distance of the curve away from the point of the indentation at which \( U'_y(r) \)
reaches \( \frac{U_y(0)}{2} \), i.e. when the maximum deflection falls to half of the maximum value.

The profile of the indentation needs to be as broad as possible within the allowable distance. The analysis substrate length is 2 mm. Hence the suitable range should be about of half the indenter radius, where the indenter radius is 1 mm. This is to ensure that the profile will give a good measurable value by the system.

Another parameter is \( \Delta \) which is defined as the distance away from the point of deflection when the value is \( \frac{1}{20} \) of its maximum value. This value is chosen as a check that the profile will converge back to its original level and hence would not perturb neighbouring points.

The values can then be tabulated for the thicknesses chosen for the analyses. Table 4.4 shows the results for PL3 – Silicone and Table 4.4 shows the results for PC10 – Silicone.

Table 4.4: Results of PL3 – Silicone.

<table>
<thead>
<tr>
<th>PL3 – Silicone Thickness (mm)</th>
<th>( \alpha )</th>
<th>( \theta )</th>
<th>( \beta )</th>
<th>( \sigma ) (mm)</th>
<th>( \Delta ) (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.4 – 0.2</td>
<td>2</td>
<td>36.7</td>
<td>2.9</td>
<td>0.3</td>
<td>0.7</td>
</tr>
<tr>
<td>0.4 – 0.3</td>
<td>1.3</td>
<td>79.9</td>
<td>1.75</td>
<td>0.35</td>
<td>0.75</td>
</tr>
<tr>
<td>0.3 – 0.5</td>
<td>0.6</td>
<td>93.3</td>
<td>1.64</td>
<td>0.35</td>
<td>0.8</td>
</tr>
<tr>
<td>0.4 – 0.5</td>
<td>0.8</td>
<td>66.67</td>
<td>2</td>
<td>0.4</td>
<td>0.9</td>
</tr>
<tr>
<td>0.5 – 0.5</td>
<td>1.0</td>
<td>46.67</td>
<td>2.7</td>
<td>0.45</td>
<td>1.05</td>
</tr>
</tbody>
</table>

The analysis based on PL3 – Silicone gives a rough guide as to achieve the suitable combination and thickness. From Table 4.4, \( \theta \) values seem to be very large, which is not desirable as it will gives errors when the unwrapping process occurs. Increasing PL3 thickness, \( \beta \) increases further than one, with decreasing \( \theta \). \( \sigma \) values indicate a rather narrow distance from the point of indentation. This means the profile
is not broad. The indentation profile also moves back to zero at a distance of about 1 mm which is desirable. Reducing PL3 thickness will reduce $\theta$ values but $\beta$ will not be as close to 1. Based on these analyses, it shows that stiffer material than PL3 is needed as the top layer. The results also show that PL3 – Silicone does not fit the criteria.

Table 4.5: Results of PC10 – Silicone.

<table>
<thead>
<tr>
<th>PC10 – Silicone Thickness (mm)</th>
<th>$\alpha$</th>
<th>$\theta$</th>
<th>$\beta$</th>
<th>$\sigma$ (mm)</th>
<th>$\Delta$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1 – 0.5</td>
<td>0.2</td>
<td>8</td>
<td>1.04</td>
<td>0.85</td>
<td>*</td>
</tr>
<tr>
<td>0.2 – 0.5</td>
<td>0.4</td>
<td>3.3</td>
<td>1.10</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>0.3 – 0.5</td>
<td>0.6</td>
<td>2.7</td>
<td>1.12</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>0.05 – 0.1</td>
<td>0.5</td>
<td>10</td>
<td>1.07</td>
<td>0.35</td>
<td>0.9</td>
</tr>
<tr>
<td>0.05 – 0.2</td>
<td>0.25</td>
<td>14.7</td>
<td>1.02</td>
<td>0.45</td>
<td>1.05</td>
</tr>
<tr>
<td>0.05 – 0.4</td>
<td>0.125</td>
<td>21</td>
<td>1.08</td>
<td>0.55</td>
<td>1.25</td>
</tr>
<tr>
<td>0.05 – 0.05</td>
<td>1.0</td>
<td>25</td>
<td>1.01</td>
<td>0.575</td>
<td>1.30</td>
</tr>
<tr>
<td>0.1 – 0.4</td>
<td>0.25</td>
<td>7</td>
<td>1.04</td>
<td>*</td>
<td>*</td>
</tr>
<tr>
<td>0.1 – 0.05</td>
<td>2</td>
<td>2.7</td>
<td>1.12</td>
<td>0.5</td>
<td>1.2</td>
</tr>
</tbody>
</table>

* - the profile did not converge back to the surface

The results from the analyses for PC10 – Silicone proved to be better than for PL3 – Silicone. This is due to the stiffness of PC10 which is higher than PL3. From Table 4.5, increasing the PC10 thickness reduces $\theta$, but $\beta$ value increases. If the silicone thickness increases, both $\theta$ and $\beta$ increases. This shows that the silicone thickness should be less than the top thickness. In some cases, it was also noted that the deflection curve seems unable to move back to its original position i.e. the values for $\sigma$ and $\Delta$ are more than 2 mm.

In achieving the thickness for both top and bottom layers, the number of analyses performed were much greater than suggested both Table 4.4 and Table 4.5.
Both tables show the key results when considering the combinations and thickness. The analyses of PC10 – Silicone were able to give better results for all the parameter mentioned above. The analyses show that a good material combination is PC10 – Silicone with a thickness of PC10 of 0.1 mm and silicone rubber of 0.05 mm.

PC10 – Silicone will therefore be use as the elastic substrate for the experiment. The $\theta$ value is 2.7 with $\beta$ of 1.12 which is close to one. The $\sigma$ is about 0.5 mm away from the indentation which is about the correct range. The $\Delta$ values is 1.2 mm which is less than the size of the substrate.

Once the optimised substrate is determined, the analyses then proceed by increasing the number of ball bearings such that the load is equivalent to stacking the ball bearings on top of one another. The ball bearing used is a 4 mm ball bearing having mass of 0.2608 g.

The analysis is performed based on the weight of 1, 2, 4, 6, 8, 10, 12, 14, 16, 18 and 20 ball bearing. Figure 4.11 is the surface and interface plot for 20 ball bearings. The figure shows that the interface behave in accordance with the top depression apart from $U_Y^I(0)$, as this is the centre of indentation. The depression also shows that moving away from the point of indentation, the deformation deflect back in less than the indenter radius, which confirms that no crosstalk between adjacent ball bearings.

For each FE analysis, the maximum deflection, $U_Y^I(0)$ at the interface is determined and recorded. Graphs of deflection against the number of ball bearings are then plotted as shown in Figure 4.12. The graph shows a linear response between the maximum deflection and increasing number of ball bearings. This is compared with the force-deflection curve of Hertzian loading for the case of an elastic material with modulus chosen to give the same deflection as the double layer substrate at a load corresponding to 10 balls. The force deflection curve from the Hertzian loading shows a non-linear relationship. This further confirms the use of double layer substrate for the experiment.
Figure 4.12: Surface and interface plot for 20 ball bearing for PC10 – Silicone. The interface and surface responses show similar deflections magnitudes, indicating one of the key design criteria has been met. Furthermore, moving away from the point of indentation, the plot indicates a negligible crosstalk between adjacent ball bearings.

Figure 4.13: Comparison between the deflection against the number of ball bearings. The graph reveals a linear relationship for the FE analysis which corresponds to the double layer elastic substrate and the non-linear relationship for the Hertzian loading.
4.4 Summary

The experiment employs a double layer elastic substrate as the sample on which indentation will occur. This chapter described the FE analysis performed in order to achieve the optimised material combination and thicknesses of the sample. The main analysis performed was the distributed load and the non-linear ball loading.

The analysis performed was an axisymmetric one in order to reduce a 3D analysis into a 2D one. This will reduce the computational time required for the analysis. The accuracy of the results depends on the mesh size as a coarse mesh will provide an approximation of the solution and increasing the amount of mesh points will result in higher accuracy. However, increasing the number of mesh points will also result in increasing computational time.

The material chosen for the analysis was PL3 and PC10 of low and high modulus epoxy respectively, silicone rubber and a thin glass. The analysis was performed in two steps. The first was to determine the best material combination. This was achieved using the distributed load. There were two material combination shortlisted for the next analysis which were PL3 – Silicone and PC10 – Silicone. The two combinations were then further analysed using the non-linear method in order to analyse the optimised thickness.

The design of the substrate is based on several criteria. Amongst these criteria are ensuing a linear force deflection curve and maximising the deflection of the gold layer for a given indentation depth. The curve should be as broad as possible within the contact region while at the same time avoiding a crosstalk between one point and another.

From the analysis, it was found that the optimised design was PC10/Silicone having a thickness of 100 µm/50 µm respectively. This optimised substrate was then used to determine the force-deflection curve between the sample and the Hertzian loading. The analysis was performed by increasing the load applied such that it corresponds to the ball bearings being stacked to one another.

At each analysis, the maximum deflection was then recorded. This was then compared with the Hertzian deflection at each loading. From the analysis, it shows
that the sample force-deflection curve gives a much more linear response compared to the force deflection curve from the Hertzian loading. The results will provide a further confirmation of the use of double layer substrate.
CHAPTER 5

EXPERIMENTAL SET-UP

5.0 Introduction

The experimental method employed in this project is based on interferometry techniques. This chapter will give the outline of the experimental arrangements. As mentioned in the previous chapter, the methods employed will provide the deformation measurement of the interface between the two layers. Two interferometer techniques will be discussed, namely Mach-Zehnder Interferometer and Fizeau Interferometer.

The initial set-up was the Mach-Zehnder Interferometer. The interferometer was used as this experimental set-up was available within the Structural Integrity Research (SIR) group. The description of the set-up including the calibration of the piezo-electric translator (PZT) will be discussed in greater detail in the following sections.

There were several drawbacks in this set-up. Among them are the multiple reflections coming from the elastic sample and the necessary beam splitter. To improve this, several methods can be applied. These include using a wedge at the back of the sample glass so as to prevent the back surface reflections from entering the camera lens. Alternatively a laser having a tunable wavelength can be used for the experiments that avoid the used for a beam splitter and PZT.

The second interferometer set-up is based on using a tunable laser. This involved designing a new set-up for the wavelength-tuned laser. The set-up is based on a Fizeau Interferometer which is preferable to the first as it is a common path interferometer. This method also gives better results for measuring the deflection as the noise from high harmonics was reduces to rms 1.3 nm. Subsequent subsections will discuss this in greater detail.
This chapter will also outline the sample preparation for the experiments. The sample for the experiments is a double layer elastic substrate. Gold is coated in between which acts as the reflective mirror. The bottom layer is silicone rubber while the top layer is high modulus epoxy resin of PC10. From the Finite Element Analysis (Chapter 4), it was found that the expected required thickness for the top and bottom layers is 100 μm and 50 μm respectively.

For both set-ups, the calibration procedure will be discussed in order to give the relationship between the applied voltage and the phase step. In the Mach-Zehnder Interferometer the calibration involves calibrating the PZT. The tunable laser involves calibrating the laser with the applied voltage. This will then determine the phase for a given voltage applied.

The initial experiments are the force-deflection calibration experiments as the sample needs to be calibrated before any experiments can proceed. Once the force-deflection curve for the sample is determined, the layered and the sandpile experiments can then proceed. The details of the sample preparation and experimental works will be discussed in the following sections.

5.1 Sample Preparation

The experiment employs a double layer elastic substrate, which was cast on a rigid glass disc. Two types of glass disc were used, 50 mm diameter with 4 mm thickness and 150 mm diameter with 4 and 6 mm thickness.

The fabrication of the layer involves, firstly casting the silicone layer, which was followed by evaporating the gold layer and finally fabricating the top layer. The silicone is Sylgard 184, Dow Corning and the top layer is epoxy PC10. Gold is used as the reflective film as it will bond better with silicone better than Aluminum.

To ensure bonding between the glass disc and the silicone rubber, the glass surface needed to be coated with a layer of silicon primer (Dow Corning 92-023). The primer was applied using a pipette, continuously from one edge of the glass disc to the other. The glass disc was kept vertical to obtain a uniform distribution of the liquid. Once coated, the primer was then allowed to cure for 15 minutes at 50°C.
The silicone rubber was mixed with its curing agent and an accelerator (QFC 3-6559, Dow Corning) in the ratio of 10% and 5% by mass respectively. It would have been possible to cure the polymer without the accelerator but polymerization might not have completed and the time taken to cure would have been too long. After mixing thoroughly, the mixture was degassed using a vacuum dessicator so as to remove the bubbles that occurred during the mixing process.

The bubble-free mixture was then poured on to the glass disc. This was then coated using a spin-coater machine, manufactured in-house as shown in Figure 5.0. The silicone was spin-coated on to the glass sample with a thickness of 50 µm. To achieve this, for a 50 mm diameter glass disc, about 0.5 g of silicone was spin-coated for 45 seconds at 1500 rpm. For a 150 mm glass disc, 3 g of silicone was coated for 120 seconds at 1200 rpm. The silicone was then allowed to cure overnight at 60°C.

The Talysurf and several microscope methods were investigated to determine the thickness of the cured layer. The Talysurf method involves contacting the surface with a stylus which can scratch the surface. This was not desirable as the surface needs to be smooth. The microscope method was eventually chosen to determine the thickness, as the method is non-contacting.

Figure 5.0: Spin Coating Machine use for coating silicone onto the glass.
Two methods were investigated, namely the Nikon interferometric microscope and Olympus microscope. The Nikon interferometric microscope has a micrometer eyepiece which allows the measurement of the average spacing $d$ between the monochromatic Fizeau fringes observed with an interference filter (wavelength, $\lambda = 514$ nm). Removing the filter, two sets of white light fringes can be seen which corresponds to the top and bottom surfaces. However, due to the silicone being 100 $\mu$m, only a single fringe was observed. This was due to the relatively thick silicone being measured. The task of finding the second fringes proved to be very difficult. The method in practice only allows a very thin sample to be measured (less than 10 $\mu$m) and was therefore not used further.

The second method employed an Olympus microscope. This method involves making a mark at the glass surface with a coloured pen before coating. After coating the silicone, another mark of a different colour is then applied to the silicone. The microscope is then brought into focus on the first mark. By adjusting the microscope finer knob, with 1 $\mu$m accuracy, the second mark is then brought into focus. The thickness is then given by:

$$t = d \times n$$

(5.0)

where $t$ is the thickness, $d$ is the measured displacement of lens relative to the sample and $n$ is the refractive index for silicone (n for silicone is 1.4).

To check the thickness, Figure 5.1 shows a graph of the thickness against distance for a 150 mm diameter glass disc at 1200 rpm for 120 seconds using 3g of silicone. This technique will be employed throughout the fabrication of the sample.

To enhance the fringe visibility, a gold coating was evaporated onto the silicone once it had cured. The gold was coated using an Edwards Vacuum Evaporator machine. The layer thickness was controlled by a film thickness monitor (Edward FTM2) consisting of a 6MHz oscillating crystal placed in the vacuum chamber. As the gold was deposited, the oscillating crystal frequency shift was proportional to the amount of material being deposited. About 1 g of gold was then evaporated giving roughly 3 nm of thickness.

Once the gold is evaporated, the second layer of the substrate can then be manufactured. The top layer is a transparent epoxy resin PC10 from Measurement
Group. This process needs to be performed straight after the evaporation process. Leaving the gold for more than 15 minutes would result in the surface being difficult to be bonded.

![Graph of Thickness against Distance](image)

Figure 5.1: Graph of measured thickness against distance along the diameter of the glass disc.

The resin and hardener were mixed in the ratio of 1:0.15 by weight at room temperature. Mixing was done using a spatula, stirring it gently to avoid any bubble formation. This mixture was then poured onto the gold layer. A PTFE film was then applied onto the mixture to spread PC10 evenly on to the surface. The PTFE film used was MR-1 non-perforated film of 25 μm thickness supplied by Aerovac Systems Ltd. A flat surface weight was then applied to the PTFE. This was to ensure a flat surface of PC10 once cured. For 50 mm diameter discs a weight of 40 g was applied and for the 150 mm diameter discs a weight of 160 g was applied to give a thickness
of 100 μm. This was then allowed to cure for about four hours at room temperature. Once cured, the PTFE was peeled away to give a double layer substrate. Figure 5.2 shows a typical sample used for the experiments.

Figure 5.2: Double layer elastic substrate that is use as the sample for the experiment.
5.2 Mach-Zehnder Interferometer

In the initial part of the project, Mach-Zehnder Interferometer was used. This section will describe the experimental arrangement. Figure 5.3 shows a schematic diagram of the set-up.

Figure 5.3: Schematic Diagram of Mach-Zehnder Interferometer.

The system includes two 45° glass wedges $W_1$ and $W_2$, placed across one of the beam paths. $W_1$ is translated by a Physik Instrument P.820.10 piezo electric translator (PZT), a low voltage device with a specified displacement of 15 $\mu$m for 100 V of applied voltage. The PZT is driven by an digital to analogue (D/A) converter interfaced to a Sun Sparcstation. A second identical wedge $W_2$ brings the beam parallel to its original. In this way, no beam tilts are produced if the PZT tilts during
translation and there is no lateral beam translation, provided that the wedge is angled correctly in the beam at an angle of 63.7° [3].

The beam passes through a polariser and onto a half wave plate which allows the polarising plane to be rotated. The beams are then split into two, namely the reference and object beam using a polarising beam splitter.

The beams are delivered by two single-mode optical fibres (Fiber Optic Delivery System, Point Source Ltd). At the output fibre ends, the beams are launched to a second beam splitter which at the end combines both beams to form a fringe pattern. The beam from the object beam is directed to the glass sample. The light leaving the output of the object beam is reflected from the sample surface before being recombined with the output of the reference beam, thereby forming an interference fringe pattern.

Before any experiment can be performed, it is important to align the beam so that the correct polarisation and the maximum light are achieved. The system involves splitting the beam into two. This may result in power loss. The power loss was quantified using a Power Meter. It was found that 24% of the input power is lost in the optical fibres.

The alignment process uses a launch adjuster. It involves firstly inserting the adjuster with the small aperture furthest from the laser holding the adjuster in position using the tension screws and adjusting screws LA and RA to achieve maximum light transmission. Once achieved, the process is repeated but with the small aperture of the adjuster nearest to the laser and by adjusting screws LB and RB screws. The fibre optic is then inserted into position by adjusting the tension screw. Figure 5.4 shows the end of the laser with the aligned optical fibre.

The light emitted from each fibre also needs to be in the same polarisation state. The optical fibres have two axes, fast and slow. The light needs to be linearly polarised along one of the axes. It is then necessary to make sure that the light is polarised in one direction (e.g. vertical) both at input and output fibre ends.

The process of checking the polarisation involves two stages. To check the input end, a polariser is put in front of the output end to give a vertically polarised light. The fibre is tapped gently and the input end is rotated to get a stable beam. To
check the output end, the polariser is again used at the output end to give a vertically polarised light. The output end is then rotated to give a maximum optical transmission. This indicates that light coming out from the fibre output end is vertically polarised light.

The fringe patterns are recorded with a Kodak ES 1.0 CCD camera, which is connected to the SunSparc Station. The camera has a maximum frame rate of 30 frames/sec. The photo detector array of the camera has 1024 by 1024 pixels and has been used together with a Nikkor 135-mm lens with a maximum aperture of f2.8. Extension tubes may be used to increase the magnification and reduce the field of view. Two 31-mm extension tubes will give the pixel size of 15 μm by 15 μm. as measured on the specimen.

![Figure 5.4: Laser end showing the screws for the optical fibre alignment.](image)

There are several disadvantages when employing this experimental set-up to measure the deformation. The measurement of surface deflection of the sample will involve the interference pattern to be formed between the reflection from the gold film and the reference beam. However, since the substrate consists of multiple layers,
multiple reflections are detected by the interferometer. These multiple reflections come primarily from the bottom surface of the glass.

Figure 5.5: Schematic diagram of the sample depicting the multiple reflections coming from the sample.

These multiple reflections can be further described with an aid of a diagram as shown in Figure 5.5. The figure shows the double layer sample cast onto a glass which shows three reflections coming from the sample. The amplitude reflection coefficient can then be calculated using Fresnel equations. This is defined as:

$$r_\perp = \frac{n_i \cos \theta_i - n_t \cos \theta_t}{n_i \cos \theta_i + n_t \cos \theta_t} \quad (5.1)$$

where $n_i$ and $n_t$ is the refractive index of the incident and the transmitting media and $\theta_i$ and $\theta_t$ are the angles of incident and transmission respectively.

Table 5.0 shows the refractive index value for air, glass and silicone. To calculate the amplitude reflection coefficient, the angle can be assume to be zero. Equation (5.1) can then be reduced to:

$$r_\perp = \frac{n_i - n_t}{n_i + n_t} \quad (5.2)$$

By substituting the values from Table 5.0 into equation (5.2) will give the amplitude reflection coefficient for each interface. This is tabulated in Table 5.1.
Table 5.0: Refractive index values for air, glass and silicone.

<table>
<thead>
<tr>
<th>Material</th>
<th>Refractive Index (n)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air</td>
<td>1.0</td>
</tr>
<tr>
<td>Glass</td>
<td>1.5</td>
</tr>
<tr>
<td>Silicone</td>
<td>1.4</td>
</tr>
</tbody>
</table>

Table 5.1: Amplitude reflection coefficient for air-glass and glass-silicone interface.

<table>
<thead>
<tr>
<th>Reflection Coefficient</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>$r_{\text{air-glass}}$</td>
<td>0.2</td>
</tr>
<tr>
<td>$r_{\text{glass-silicone}}$</td>
<td>0.03</td>
</tr>
</tbody>
</table>

From these values, it shows high amplitude reflections coming from the air-glass interface.

A simple solution to overcome this problem is to attach a glass wedge to the bottom glass surface by using an index matching grease. The glass wedge will reflect the back surface reflections away from the camera lens so that the interferometer will not detect these reflections. Figure 5.6 shows a schematic diagram of the back surface with a wedge being attached.

![Figure 5.6: Schematic diagram of a bottom surface of the glass with a wedge being attached.](image)

Attaching the wedge causes further problems, however, as air bubbles are created within. This again will cause more errors to be introduced during the experiments. It was however, possible to attach a small glass wedge on to a 50 mm glass disc without the presence of air bubbles but the task prove to be more difficult
with a bigger diameter of glass wedge in order to accommodate the 150 mm glass disc.

It is therefore difficult to utilise this experimental set-up for the purpose of this experiment. It was proposed to modify the experimental arrangement in order to accommodate a Fizeau set-up with tunable laser, which is more suitable for measuring surface deflections. This is described in Section 5.3.

5.2.1 PZT Calibration

A piezo-electric translator is used as a phase stepping device. This requires a calibration procedure to find the relationship between the driving applied voltage and the phase step as employed in Reference [127]. The average S of the squared difference between a reference fringe pattern Io, and a fringe pattern with the voltage V applied, I, can be calculated as:

\[ S = < (I - I_o)^2 > \]  \hspace{1cm} (5.3)

S can be fitted by the function:

\[ S(V) = A + B \cos [\alpha(V)] \]  \hspace{1cm} (5.4)

where A and B are constants and \( \alpha(V) \) is the phase shift due to V. \( \alpha(V) \) can be approximated by:

\[ \alpha(V) = a + b(V - V_o) + c(V^2 - V_o^2) \]  \hspace{1cm} (5.5)

where \( V_o \) = Voltage for the reference frame.
S(V) is fitted as shown as in Figure 5.7. The constant can then be obtained and the value is shown as:

\( a = -9.995 \text{ rad} \)
\( b = 2.430 \text{ rad/Volts} \)
\( c = 1.289 \times 10^{-2} \text{ rad/Volts}^2 \)
The second order term, $c$, is small, therefore the phase approximates as a linear function of the applied voltage. The full quadratic is plotted in Figure 5.8.

![Plot of second order polynomial fit $S(V)$ of mean square intensity against voltage. The ‘o’ represent the experimental data and ‘-’ represent the fit.](image)

Figure 5.7: Plot of second order polynomial fit $S(V)$ of mean square intensity against voltage. The ‘o’ represent the experimental data and ‘-’ represent the fit.

![Phase shift $\alpha$ against applied voltage V. This is a good approximation to be a linear function.](image)

Figure 5.8: Phase shift $\alpha$ against applied voltage $V$. This is a good approximation to be a linear function.
5.3 Tunable Laser Set-up

In recent years, tunable laser has been used to measure surface profilometry [75, 102-105]. The design of the interferometer in this experiment was based on the proposed experimental set-up in Reference [75].

The experimental arrangement proposed in Reference [75] used a diode laser as the light source and the phase shifting device. To avoid mode hop the laser is controlled using a thermo-electric cooler. The beam is then delivered using a single mode polarisation-preserving (SMPP) fiber. The set-up does not make use of a beam splitter which made the arrangement and optical alignment simpler. A rotating diffuser is placed in the focal plane. The interference pattern is detected by the CCD camera, where the image is transferred to a computer for analysis.

The main experimental set-up for this experiment was based on a tunable wavelength laser that incorporates elements of the previous set-up. The arrangement is based on a Fizeau set-up as a common path interferometer. With this arrangement, there is the possibility to exclude the use of PZT and the beam splitter as there is no need to split the beam and the laser is able to tune the wavelength in order to shift the phase. Furthermore, the glass-air interface reflection, which previously was the cause of spurious fringes, is now used as the reference wave.

The laser chosen for these experiments is a New Focus Vortex Tunable laser 6005 with a 635.05 nm wavelength and a 0.09 nm tuning range with the rated power output is 4 mW. Figure 5.9 is a schematic diagram of the set-up.

The beam from the laser passes through a polariser and is then passed through a 40x objective lens so as to expand the beam. The beams then passes through a rotating diffuser, a ground glass connected to a 12V DC motor, which is housed in a casing as shown in Figure 5.10.

The rotating diffuser (40 mm in diameter) is used to minimise the coherent noise in the interferometer. The diffuser has the rotational speed of about 600 rpm and is used in order to scatter the beam. This gives the speckled image as shown in Figure 5.11, which is removed by rotating the diffuser. The rotation gives the average of the speckle leaving behind the required image. Figure 5.12 shows an image of an
interference pattern caused by a glass wedge when the diffuser is rotated. The use of a diffuser in this way also significantly reduces optical noise produced by scattering from dust and scratches within the optical system.

Figure 5.9: Schematic Diagram of Tunable Laser Set-up.

Figure 5.10: The rotating diffuser attached to 12V motor.
Figure 5.11: Speckled image when diffuser is static.

Figure 5.12: Image achieved on rotation of the diffuser. The interference pattern is from a glass wedge.
The diffused beam is then directed on to the sample by means of the two mirrors through a 500 mm field lens. The interference pattern is produced between the reflective gold and the back surface of the glass for the double layer elastic substrate. This interference pattern is subsequently detected by the CCD camera and digital images transferred to a Sun workstation as before.

As mentioned earlier, the design of this experimental set-up integrates the previous set-up with certain modifications. To achieve this a laser rig base has been designed. This laser rig base holds the Spindle & Hoyer micro bench equipment necessary for securing the objective lens and the mirror. Figure 5.13 shows the optical arrangement of the set-up.

The laser has two operating modes namely constant-power mode and constant-current mode [78]. The constant-power mode adjusts the laser current to maintain a stable power output. In this mode, the power and the laser frequency (wavelength) can be varied. However, operating in this mode involves having an external beam splitter as part of the set-up. The beam splitter is needed in order to sample the laser output to give an estimate of the power output. By having a feedback loop, the laser output power can then be stabilised.

On the other hand, the constant-current mode involves modulating the laser frequency (wavelength) by using an externally generated signal from D/A to modulate the voltage of the piezo-electric actuator which controls the laser frequency within the laser. This mode is possible by directly applying the signal from the D/A to the laser controller.

The first-mentioned method is unfavourable for this particular experiment as it involves using an external beam splitter. This may cause problems when recording the images during the experiment as it may introduce multiple reflection within the beam. The latter method is more favourable as it does not involve any further hardware and the D/A converter was available from the previous set-up. For this reason constant-current mode was used throughout the experiments.
5.3.1 Tunable Laser Calibration

The tunable wavelength laser is an alternative method for shifting the phase. The configuration of this laser is based on the Littman-Metcalf design as discussed in Section 2.3.6. As the phase is given as:

$$\alpha = \frac{4\pi}{\lambda} t$$  \hspace{1cm} (5.6)

where $\lambda$ is the wavelength and $t$ is the thickness of the glass that corresponds to the unequal path length. Differentiating the phase with respect to the wavelength gives the phase change as:

$$\delta \alpha = -\frac{4\pi}{\lambda_0^2} t \delta \lambda$$  \hspace{1cm} (5.7)

where $\lambda_0$ is the nominal operating wavelength.

From this equation, the phase shift is achieved by tuning the wavelength.
Before the experiment is initiated, the laser needs to be calibrated so as to give a relationship between the phase and the voltage applied. The laser is tuned by means of applying voltage from \(-2.25\text{V}\) to \(+2.25\text{V}\) using the D/A converter. The power output of the laser varies when tuning the wavelength, which results in fluctuations in recorded intensity. A small variation of the output power of the laser was noted which was recorded at about 0.2mW. Hence the calibration of the laser is different from that mentioned in Reference [127].

A laser calibration method which is insensitive to the intensity fluctuations is necessary for this experiment. The method proposed here is a novel technique that takes into account these fluctuations.

It is essential for the recorded interference pattern; 1) to have a constant number of fringes across the image; 2) the fringes of which must be well-aligned with the chosen data (in this case of either columns or rows) and 3) to yield an image which has not reached saturation value.

The calibration for the experiment uses a glass mirror as this produces a clear fringe pattern in accordance with the above-mentioned criteria. A typical image of this pattern is shown in Figure 5.12. The image has a well-defined number of fringes, well-aligned across the columns and without having reached saturation.

The calibration involves applying the voltages in step of 0.1V leading to 90 images for the analysis. At each image, taking the intensity value and subtracting from it the mean value for each row or column gives:

\[ I_i = I_o - I_{\text{mean}} \] (5.8)

where \(I_o\) is the intensity value and \(I_{\text{mean}}\) is mean value of the row or column.

Part of one cross-section of the image after applying this equation (5.8) is shown in Figure 5.14.
Figure 5.14: Cross-section of the interference pattern after applying equation (5.8).

Figure 5.15 shows the absolute intensity value of the Fourier Transform. Two peaks are seen in the positive frequency range. One is due to the fringes and the other (higher frequency) is probably due to multiple reflections. The peak of the low frequency is the required value that was then found and recorded.

Plotting the phase from each of these maximum values for the low frequency part (i.e. the peak near $kx = 10$ on Figure 5.15) against the applied voltage gives the graph shown in Figure 5.16. The form of the phase gives a sawtooth function with a discontinuity of about $2\pi$ for about every 0.5V. This is corrected by the unwrapping process so as to recover the unwrapped function as shown in Figure 5.17. This shows that the phase is a continuous function for a given voltage. The relationship between phase and voltage can then be approximated using equation (5.5). The constant value and gradient of the graph can then be calculated.
Figure 5.15: The plot of the absolute intensity value of the Fourier Transform. ($k_x$) is in units of fringes across the region analysed (100 pixels from Figure 5.14).

Figure 5.16: Phase value against the voltage applied.
Figure 5.17: Corrected phase for each of the voltage applied. This shows a relationship between the phase and the voltage applied.

The tunable laser calibration used a mirror of 8.85-mm thickness. The calculated constants for the glass mirror were found to be:

\[ a = -2.022 \text{ rad} \]
\[ b = 0.0824 \text{ rad/Volts} \]
\[ c = 2.08842 \times 10^{-4} \text{ rad/Volts}^2 \]

The phase values from the graph in Figure 5.17 are then scaled so as to accommodate the different thickness of the glass in the actual experiments. The glass for the granular beds experiments is of 6 mm thickness. The value is therefore obtained by simple scaling as:

\[ \alpha_g(V) = \frac{t_g}{t_m} \alpha(V) \]  

(5.9)

where \( \alpha_g = \) scaled phase, \( t_g = \) glass sample thickness, \( t_m = \) mirror thickness
Figure 5.18 shows the scaled phase value. This gives the constant values as:

\[ a = -1.371 \text{ rad} \]
\[ b = 0.0558 \text{ rad/Volts} \]
\[ c = 1.41587 \times 10^{-4} \text{ rad/Volts}^2 \]

![Diagram showing phase vs. piezo-voltage](image)

**Figure 5.18**: The scaled value of the phase is shown in red.

As mentioned earlier the laser power output was found to fluctuate. Severe fluctuations can occur if the laser undergoes a mode hop. This may occur when the laser heat sink is not properly in place. To achieve a satisfactory result, the tuneable laser needs to be stabilised in terms of temperature which will give a stable power output.

The laser operating temperature is about 16°C with a typical power output of about >3mW. The laser is designed such that it sits on an aluminium base which is a good heat conductor and acts as the heat sink of the laser. To further improve the heat path, the laser platform which sits on the laser base rig was also made out of aluminium. A heat sink compound was applied between the laser and the laser platform to achieve a good heat sink.
These steps are necessary in making sure that the laser is working properly as it is sensitive with temperature change. When these steps are not properly in order, a typical instability of about 1-1.5mW is recorded. Figure 5.19 shows the relationship between the phase and the voltage for the laser undergoing three mode hops through the entire tuning range.

![Figure 5.19: Phase against the voltage with severe mode hop.](image)

The laser was manufactured by New Focus Ltd, which guarantees that the laser will not show any mode hop for the whole tuning range. However, this was not the case as shown in Figure 5.19. This causes difficulties in trying to calibrate the laser.

As mentioned in Chapter 3, the experiment will employ 15-frame algorithm having $\frac{\pi}{4}$ phase step which is needs a total of $\frac{14\pi}{4}$ phase shift. The mode hop shown in Figure 5.19 however is repeatable. An attempt was then made to calibrate the laser in one of the ranges i.e. from $-1$ V to $0.5$ V. This however prove to be a difficult task as mode hops were still present.
The laser was then returned, as it does not meet the required specification. It was then reported that the laser was having diode problem and a complete rebuild was necessary. The rebuild process took three months to complete.

5.4 Experimental Work

In this section, the details of the experimental works are discussed. The main experimental work in this project is the calibration. This is the first step as it gives the relationship between the load and the resulting deflection depth. Once this is achieved, layered experiments and sandpile experiments can then proceed.

The calibration used ball bearings that act as the indenter. This is due to the ball bearing having a relatively smooth surface. This also enables the results to be obtained from the sandpile experiments. Allowing the granular media to make direct contact with the sample itself will result in difficulty in determining the indentation force due to the random variation in size and shape.

5.4.1 Calibration Experiments

4 mm steel ball bearings were used as the load. The ball bearings were stacked on top of one another as shown in Figure 5.20. Up to 20 ball bearings were used for this experiment. The experiment gives a profile between the depression and the load applied for the substrate discussed in the previous chapter.

The use of a clear monolayer silicone rubber and a 6 mm thickness of perspex were also investigated. These provided further confirmation of the benefits of using the double layer substrate. The results are presented in Chapter 7.

For this experiment, a rig has been designed specifically for the calibration. The sample size is 150 mm in diameter, hence it is possible to calibrate at several different positions or points.

The rig consists of a sample holder, calibration rig holder and the ball bearing holder as shown in Figure 5.21. The sample holder holds the sample in horizontal
positions with a 50-mm diameter circle drilled through the centre. This hole allows the beam from the laser to reach the sample for the experiments.

The ball bearing holder has an array of 9 x 9 openings as shown in Figure 5.22. The diameter of the hole is 4.2 mm. This gives the ball bearing a clearance of about 0.1-mm at each side. This is to avoid slack when stacking the ball bearings. If the clearance is too small, the effect of friction may affect the results and the weight of the ball bearing may be supported by the wall of the opening. If the clearance is too high, the balls will not stack vertically within the cylindrical hole. This will be discussed further in Chapter 6. Once the sample has been calibrated, other experiments can then proceed.

![Diagram](image)

Figure 5.20: Ball bearings stacked on top of each other using calibration rig.
Figure 5.21: Arrangement of the calibration rig.

Figure 5.22: Ball bearing holder. The rig has a 9 by 9 array of holes with each opening having a diameter of 4.2 mm.
5.4.2 Layered Experiments

Many works have put forward theoretical studies using discs [24, 25]. These contain works with layered structures. The most popular is the hexagonal packing. To achieve this, a hexagonal template was manufactured. Figure 5.23 shows this configuration with the ball bearings in place in a hexagonal template. One layer contains 265 ball bearings.

The experiment is performed by placing a layer and measuring the force distribution. Once a layer is arranged, another layer is placed on top based on face-centered cubic (fcc) arrangements. This is achieved by carefully placing the ball bearings on a ABCABC... structure. Figure 5.24 sows a diagram of this structure. Measurements are taken once each layer is placed.

Figure 5.23: Hexagonal template with a layer of ball bearings (265 ball bearings perlayer). Another layer will be placed on top in a face-centred cubic manner.
5.4.3 Sandpile Experiments

This experiment measures the contact forces at the base of the pile for a given weight. Figure 5.25 shows a schematic diagram of the set-up.

The way in which the sand is poured determines the history. This will be expected to give a direct impact on the force distribution at the base. For the sandpile experiments, the sand was poured from a single point i.e. point source. The sand was poured in stages with a measurement of the deflection profile being taken at each stage.
The experiment used ball bearings as the layer intermediate between the sand and the sample. To avoid the sand pouring in between the ball bearings, cling film was used as to hold the sand in place. The weight from the sand was then be transmitted onto the ball bearings. This was then detected by the interferometer.

5.5 Summary

The experiment employed a double layer elastic substrate. This chapter describes the procedures developed to manufacture the substrate and to assess the layer thickness. Silicone was spin-coated as the bottom layer and epoxy PC10 was cast as the top layer. Gold film was evaporated in between which acts as the reflective film.

The project initially employed a Mach-Zehnder Interferometry technique. This experimental arrangement was modified to accommodate a Fizeau set-up, as this set-up is able to measure surface displacement by forming the interference pattern between the gold film and the back surface of the glass.

The laser used is a tunable wavelength laser, which acts as both the light source and the phase shifting device. The laser was tuned using constant-current mode by means of applying the voltage. A method for measuring the phase shift as a function of applied voltage was developed. Despite the manufacturers guarantee, mode hops were consistently revealed by this method and a complete rebuild of the laser proved necessary.

Finally, a calibration procedure was developed by indenting the sample with ball bearings. The ball bearings were stacked on top of one another using a calibration rig. The calibration rig was built such that it has multiple openings for a multi-contact experiment, thereby allowing variations of elastic response across the substrate to be assessed.
CHAPTER 6

METHOD OF ANALYSIS

6.0 Introduction

In this chapter, an analysis of the methods employed to achieve the experimental results will be presented. The experiment yields an indentation curve, the values of which are taken purely from the experimental results. Therefore, a method to quantify these results is needed so as to provide a meaningful value when estimating the depression. For example, the calibration experiments involve stacking the ball bearings on top of one another. The value of the indentation depth for each successive ball bearing is needed in order to give a calibration curve.

The indentation yields a curve with a corresponding depth which returns to its original position upon moving further from the indentation. It is important to fit these experimental data in order to quantify the experimental results and reduce several thousand datapoints to a single number. The fitting process involves finding a suitable mathematical equation with the constant that will provide the above-mentioned value.

Initially two mathematical equation were put forward, namely the Lorentzian and Gaussian equations. In each method, trials were performed based on Finite Element results. These equations are then applied with authentically calibrated results. This is done in order to find the equation that will best fit the experimental data. A subsequent section will discuss in greater detail the fitting analysis.

In this chapter, several checks concerning the experiments will also be discussed. These include the visco-elastic effect of the sample and the wall friction of the calibration rig. The visco-elastic effect of the sample needs to be checked as the deflection may vary over time due to material creep. In this event the results from one experiment to another may vary correspondingly and may cause problems in trying to analyse the data. In the calibration experiment, a rig was built in order to stack the ball bearings. It is important to take into account the effects imposed by the friction
wall of the rig during the experiment. This is done in order to ensure that the whole weight of the ball bearing is supported by the sample itself rather than by the rig wall.

Once a suitable equation has been determined, the fitting procedure can be used towards acquiring the depth value from the calibration results as well as the results from a layered and sandpile experiment. In calibration experiments for a given sample, up to nine locations or points can be investigated with each point comprising a vertical stack of up to 20 ball bearings. This means that for a given sample, up to 180 analyses is needed. This constitutes a time-consuming analysis and hence it is necessary to automate the analysis by means of image processing tools.
6.1 Fitting the Equations

The data yielded by the experiment is a set of displacement on a 2D. Firstly it is necessary to find a suitable equation that provides a good approximation to the data. Two equations present themselves as potentially suitable for the quantification of the experimental data. These are the Lorentzian and the Gaussian equations and are formally presented below.

The Lorentzian:

\[ f_L(x,y) = \frac{D}{1 + Er^2} \]  \hspace{1cm} (6.0)

where \( f_L(x,y) \) is the displacement at \( x \) and \( y \), \( D \) is the depth, \( E \) is the inverse square width and \( r \) is defined as:

\[ r = (x^2 + y^2)^{1/2} \]  \hspace{1cm} (6.1)

The Gaussian:

\[ f_G(x,y) = D \exp \left( -\left( \frac{r}{E} \right)^2 \right) \]  \hspace{1cm} (6.2)

where \( f_G(x,y) \) is the displacement at \( x \) and \( y \), \( D \) is the depth and \( E \) is the \( \frac{1}{e^2} \) radius i.e. the radius at which the displacement drops to \( \frac{1}{e^2} \) of its peak value.

Each of these mathematical equations were applied to the Finite Element (FE) results as follows. For illustration Figure 6.0 shows a FE result for 10 ball bearings.

In order to carry out a two-dimensional fit (2D), one dimensional (1D) values need to be converted. This is done by interpolating the 1D values for each \( x \) and \( y \) coordinate on a 360° plane in order to achieve a 2D result. This is accomplished by using a software called MATLAB by invoking a command called 'interp1' that will interpolate the FE data. Figure 6.1 shows the results after interpolation of the 1D data.
Once this is achieved, both equations were then fitted using a function within MATLAB called ‘fminsearch’ which is a nonlinear constrained optimisation. Using this simulated data, the application of both the Lorentzian and the Gaussian equations can be undertaken. This is shown in Figure 6.2 and Figure 6.3.

![Graph showing depth vs distance for a 10 ball bearing result](image)

Figure 6.0: Finite Element plot for 10 ball bearing. The plot is the interface plot between the epoxy (PC10) and the silicone.
Figure 6.1: Two-dimensional plot of FE values after interpolating the 1D data.

Figure 6.2: Fitting the FE data using the Lorentzian equation.
Figure 6.3: Fitting the FE data using the Gaussian equation.

As can be seen from these plots, the obvious difference involves the width of the indentation curve. Furthermore, it is necessary to investigate the results yielded by both equations if a slope is applied along the \( x \) and \( y \) axes as well as the effect of introducing small disturbances or noise to the data. This is because the experimental data may also contain a tilt i.e. a slope on either the \( x \) and the \( y \) axis. The tilt is accounted for the following equation:

\[
f(x,y) = A + Bx + Dy
\]  

where \( A \) is the constant, \( B \) is slope in the \( x \) direction and \( C \) is slope at \( y \) direction.

This equation is then incorporated into those of (6.0) and (6.2) yielding the respective Lorentzian and Gaussian equations as follows:
The Lorentzian equation:

\[ f_L(x, y) = A + Bx + Cy + \frac{D}{1 + Er^2} \]  \hspace{1cm} (6.4)

The Gaussian equation:

\[ f_G(x, y) = A + Bx + Cy + D \exp \left(-\left(\frac{r}{E}\right)^2\right) \]  \hspace{1cm} (6.5)

Tilt values are then added into the FE data from Figure 6.1 and an example is shown in Figure 6.4.

In analysing the experimental values, the effects caused by errors due to background noise need to be simulated and in order to do this, random errors are incorporated into the 2D plot. Figure 6.5 shows the plot of these random errors with the standard deviation of 0.0001 mm.

These values are then fitted with equations (6.4) and (6.5). Figure 6.6 shows the Lorentzian fitting and Figure 6.7 shows the Gaussian fitting. Comparing the differences between the two plots proved to be a difficult task. Values for a single line passing through the centre of the curve were plotted in order to reflect the difference. Figure 6.8 shows FE values; FE values with random error and tilt effects; Lorentzian values and Gaussian values.

Generally the two equations result in reasonable agreement with respect with the tilt effect applied. The Lorentzian equation seems to be in agreement with both the FE with tilt and FE with tilt and random plot yielding a 4% difference between the actual indentation and that from the best fit curve. The Gaussian however, does not seem to provide the required depth value which amounts to a discrepancy of 18%. From this analysis, it is clear that the Lorentzian is a better choice in analysing the experimental data than the Gaussian.
Figure 6.4: FE values incorporating tilt effects in $x$ and $y$ direction.

Figure 6.5: The incorporation of random noise effect to tilted FE values.
Figure 6.6: Lorentzian – fitted displacement profile.

Figure 6.7: Gaussian – fitted displacement profile.
Figure 6.8: Comparisons between the tilting FE, the Lorentzian, the Gaussian, and the FE with tilt and noise effect. The plot shows the Lorentzian gives an agreeable fit compared with the Gaussian.

Both equations were then subjected to rigorous analysis with the calibration experimental data. Figure 6.9 shows a typical cross-sectional plot for comparisons between the two curves and one of the profiles measured under a load of 20 ball bearings. It is obvious that the Lorentzian provides a better fit than the Gaussian. From these experimental data, it estimated that the Lorentzian yields about 1-2% differences with the Gaussian gives about 13-15% differences.

The analysis found that the Lorentzian equation yields a reliable fit with the experimental data. This will provide a good estimation of the depression value for a given indentation. The Lorentzian equation will be employed throughout the subsequent analysis.
Figure 6.9: Comparisons between the Lorentzian curve, the Gaussian curve and typical experimental data. The plot reveals that the Lorentzian equations yield a better approximation to the experimental data.
6.2 Calibration Weight

Section 5.4.1 mentions the calibration rig built in order to stack the ball bearings on top of one another. This procedure is carried out for calibration purposes as it provides a depth value for a given weight of a particular number of ball bearings.

The weight of the ball bearing must be supported by the sample itself. The friction caused by the wall may cause part of the transmitted weight to be supported by the wall rather than by the sample. With this in mind, it is important to take this effect into account. A simple experiment was therefore carried out and to assess the importance of wall friction.

This experiment was carried out by stacking the ball bearings on top of one another in the same manner as during the calibration experiment, but positioning the calibration rig on top of a scale rather than the sample. This provides a direct measurement of the weight of the ball bearings. Readings are taken for the first and each successive ball bearing and the experiment is repeated several times. Figure 6.10 shows a plot of the reading. The scale used is a highly sensitive digital one scale with an accuracy of 0.0001g.

From the plot it can be seen that the discrepancy between the measurements and actual weights reflects a small deviation of less than 2% with a single ball bearing having a weight of 0.2608g. This result shows that the weight of the ball bearings will be supported by the sample and not by the calibration rig-wall. The effect of the calibration rig-wall can therefore be considered small.
Figure 6.10: Graph of weight against number of ball bearings. The plot is a good representation of the actual values, indicating that the frictional wall effect of the calibration rig is negligible.
6.3 Visco-elastic Check

The sample used for this experiment is an elastic substrate. For this reason it is important to check the visco-elasticity of the substrate during the experiment; a procedure which ensures that the elastic modulus of the substrate will not change significantly over a period of time.

The experiment is repeated using the same load over a period of time. The ball bearings are loaded onto the sample and the same load is then left on the sample for periods of 1, 10, 30, 45, 60 and 120 minutes.

To compare the results achieved for each of the different time periods, a cross-sectional plot passing through the centre of the curve at each interval is shown in Figure 6.11. For each curve the value of the depression i.e. constant $D$, is noted and compared with each of the other values. The values of $D$ were calculated over a region of radius 0.8 mm centred on the point of maximum deflection. Table 6.0 gives the tabulated values of the constant $D$.

Although the plot indicates significant systematic changes over time for radii exceeding about 0.5 mm, the calculated $D$ value is not changing significantly (just 2.8% from 1 minutes to 2 hours). This means that this effect too can be considered small and negligible.

<table>
<thead>
<tr>
<th>Table 6.0: Constant $D$ (depth value) over time.</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Time (Minutes)</strong></td>
</tr>
<tr>
<td>-------------------</td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>10</td>
</tr>
<tr>
<td>30</td>
</tr>
<tr>
<td>45</td>
</tr>
<tr>
<td>60</td>
</tr>
<tr>
<td>120</td>
</tr>
</tbody>
</table>
Figure 6.11: Plot for a single line passing through the centre of the curve for each time period in order to check the influence of the visco-elastic effect. The results show that over time the deflection within a central region will not change significantly.
6.4 Automation of Analysis

The calibration of the sample involves a ball bearing with a diameter of 4 mm. These are stacked upon one another in order to obtain the calibration curve required for the experiment. For a given sample and using a multiple contact calibration rig, up to 9 points of indentation are possible.

At each point, the depth depression for each ball bearing is needed in order to plot the calibration curve. This means that 180 analyses can be drawn from an experiment using 9 points with 20 ball bearings at each point. An experiment involving this amount of analysis is time-consuming and it is necessary to automate the results.

In general terms, the automation of this analysis lies in trying to find the valid indentation points that yield the co-ordinates of the $x$ and $y$ positions. The $x$ and $y$ values constitute the centre co-ordinates of the indentation which are the maximum values of the depression. Once these co-ordinates are found, the fitting process using the Lorentzian equation can be applied at each successive ball bearing in order to quantify the depression.

The analysis was performed by means of a software programme called MATLAB. This particular software makes use of 'Image Processing Tools' that make it possible to analyse the experimental data. The analysis illustrated here is based on one of the typically calibrated experimental results. Figure 6.12 shows the typical unwrapped phase map for 10 ball bearings, showing 9 points of indentation.

The analysis is performed in several steps. The first step in identifying the positions of $x$ and $y$ is to threshold the image. By thresholding the image, positions with values of less than half of the maximum value are considered as '0'. Positions with values greater than or equal to half the maximum value are considered valid points which denote as '1'. Figure 6.13 is a representation of the image after thresholding these values.

Once this is achieved, it is important to check the image for areas or clusters with high values and which are thus considered invalid points due to non-occurrence of indentations. This is due to the background noise that is generated during the
experiment or due to defects in the sample itself. Figure 6.13 shows these areas of noise which can be removed by using the function called ‘imfeature(area)’ within the ‘Image Processing Tool’. Since these regions are usually small, this effect can be removed by demarcating a valid area size. In this example, an area with a value of less than 10 pixels will be removed.

Once these clusters removed, it is then important to dilate the valid points. This is to ensure that within the valid clusters there are no points that are considered void or have been removed during the previous step. Figure 6.14 shows the image after removing the unwanted clusters.

A function called ‘imfeature(Centroid)’ is then invoked to yield the location of the clusters’ central points. The final step involves checking the position of these valid points such that there will be no overlap between them. By using the following equation:

\[(x_i - x)^2 + (y_i - y)^2 < d^2\]  \hspace{1cm} (6.6)

where \(x, y\) is the centre of indentation at a particular point, \(x_i, y_i\) is the centre of indentation at a neighbouring point, and \(d\) is the distance between two subsequent central points.

If the distance is less than \(d\), the two neighbouring points are considered as rogue points and are removed from the analysis. Care must be taken in analysing these points as this is not a straightforward analysis. In analysing rogue points such as these, the best method is manual input of the centre of indentation and control of the fitting process in order to achieve the indentation curve.

This analysis yields an array of \(x\) and \(y\) positions, which are at the centre of the cluster. Table 6.1 gives the values of the \(x\) and \(y\) position for this particular example. Once this is achieved, each \(x\) and \(y\) position is then fed into the fitting equation. This analysis is performed by looping each point, to achieve the depth value for each ball bearing.
A typical manual analysis will take up to 1-2 hours. By using this method, the analysis becomes increasingly autonomous and efficient. The method is a useful one and is used in analysing the results of the Layered and the Sandpile experiments.

Figure 6.12: An unwrapped phase map for 10 ball bearings above each of 9 points of calibrated indentation. The unwrapped phase map shows noise in the form of defects due to the sample.
Figure 6.13: The image after thresholding the values from Figure 6.12. The image shows several areas with noise considered to be invalid points.

Figure 6.14: The image after removing unwanted clusters. Nine indentation points are visible, yielding the x and y co-ordinates.
Table 6.1: \(x\) and \(y\) co-ordinate in terms of pixel position for the above-mentioned analysis.

<table>
<thead>
<tr>
<th>(x) co-ordinate (pixels)</th>
<th>(y) co-ordinate (pixels)</th>
</tr>
</thead>
<tbody>
<tr>
<td>76.97</td>
<td>316.83</td>
</tr>
<tr>
<td>103.05</td>
<td>554.67</td>
</tr>
<tr>
<td>127.84</td>
<td>808.16</td>
</tr>
<tr>
<td>333.35</td>
<td>274.21</td>
</tr>
<tr>
<td>361.03</td>
<td>538.30</td>
</tr>
<tr>
<td>387.02</td>
<td>780.97</td>
</tr>
<tr>
<td>569.22</td>
<td>253.46</td>
</tr>
<tr>
<td>607.16</td>
<td>499.08</td>
</tr>
<tr>
<td>627.30</td>
<td>754.64</td>
</tr>
</tbody>
</table>
6.5 Summary

The experiment employs ball bearings, which act as the indenters on to the sample. The indenter causes depression of the sample that will be detected by the interferometer. The amount of the depression needs to be quantified in order to determine the force being applied. The analysis revealed that the Lorentzian equation yields a good fit with 1-2% difference with the experimental data. This equation is applied throughout the analysis of the experimental results.

Before any experiment can proceed, it is important to analyse the effect of the friction of the calibration rig-wall and the visco-elasticity of the sample over time. This is to ensure that both of these effects will not perturb the outcome of the results.

The experimental results to check the effect of the friction of the calibration rig-wall reveals that these effects can be considered small and negligible. The visco-elasticity of the sample was analysed by applying a similar load over time. This analysis shows that the effect of visco-elasticity is also a negligible one, provided that the fitting is carried out within a sufficiently small radius of the load point.

As the analysis of the experiment involves applying the Lorentzian equation at each indentation point, it is important to set up the analysis to be autonomous. For example, for the calibration experiment involving multiple contact, where at each point up to 20 ball bearings were applied, the analysis becomes time-consuming. The method described here was developed to be able to analyse the results in an efficient and automated way.
CHAPTER 7

EXPERIMENTAL RESULTS

7.0 Introduction

This chapter will discuss the results of the experiments described in preceding chapters. There are three main sets of experiments which have been performed in this project namely, the calibration experiments, the sandpile experiments and the layered bed experiments.

The calibration experiments are performed to achieve the calibration curve needed in order to estimate the forces applied due to the indentation. These experiments involve stacking up to 20 ball bearings at nine locations for a given sample. In this section, alternative substrate consisting of just a sheet of Perspex and a single layer of silicone on glass as the sample will also be discussed. This also includes the reasons why both surfaces are unfeasible compared to the double layer substrate.

The results of the sandpile experiments are then analysed by plotting using the Lorentzian equation. The measurements acquired for the sandpile are currently limited to one field of view: although the plan was originally to mount the interferometer on a movable translation stage, this has not been achieved to date. Nevertheless, the results are put forward and discussed in the following sub-section.

The layered experiments involve placing the ball bearings in a layered structure to form a bed of uniform depth. A total of five layers were constructed with the ball bearing is placed in a face-centred cubic arrangement. The analysis involves comparing the measured force from the experiment with the fraction of the force applied within the analysed field of view. This result is also analysed statistically and compared with the Coppersmith ‘q’ model [42].
7.1 Calibration Experimental Results

The calibration experiment is the first experiment as it is intended to calibrate the sample in order to provide a relationship between the force applied and the depression. The results from this experiment will provide a force-deflection curve which will be used in analysing the outcome of the layered and the sandpile experiments.

These experiments were also employed as a comparison in order to ascertain the performance of the sample. The sample for this experiment is the double layer elastic substrate, the design of which is described in Chapter 4. Two further samples were analysed: a single layer of silicone on glass and a 6-mm sheet of Perspex so as to give alternative materials for carrying out the experiment. Both alterations would have been easier to manufacture than the layered substrate. However, the results show that the two chosen materials behaved less well than the double layer substrate. Subsequent section will describe this in greater detail.

7.1.1 Single Layer of Silicone on Glass

The silicone use in this experiment is equivalent to the silicone of the double layer substrate. The layer is fabricated in the same way as the silicone of the double layer substrate discussed in Section 5.1. Once the layer is coated using the spin-coater, the layer is cured and the experiment is carried out. Figure 7.0 shows the diagram of the layer cast on a high modulus glass base.

![Diagram](image)

Figure 7.0: A single layer of silicone cast on glass.

The experiments were carried out using the procedure discussed in Section 5.4.1. The silicone layer was 10 \( \mu \text{m} \) thick. Figure 7.1 shows the unwrapped phase
map for 10 ball bearings stacked vertically. The image shows nine points of indentation which have a small contact points.

Further analysis of this image reveals signs of unwrapping errors at certain locations of indentation. An example of this is at Point A in as shown in Figure 7.1. This is magnified in Figure 7.2. Figure 7.3 is the corresponding wrapped phase map of point A which is shown to highlight these errors. The image shows that there are residues occurring at points 1 and 2 as well as points 3 and 4. The large separation between residues 3 and 4 is not expected in a phase field which is sampled in accordance with Shannon's sampling theorem, and suggests some global phase discontinuity is present.

The experiment was repeated several times in order to assess the repeatability of these discontinuities and the results show the errors to be present in every analysis. The cause of this discontinuity may be due to the difference in phase change between a silicone-air interface and a silicone-metal interface. In the former case one would expect a phase change close to $\pi$ compared to a value close to 0 for the latter.

A procedure to remove this discontinuity was therefore attempted. This is achieved by multiplying the phase by a factor of 2. This is then re-wrapped and finally unwrapped again to achieve the correct phases. Regions whose phase changes by $\pi$ due to the change in contact material will then have a change of 2$\pi$ which is removed by the rewrapping process. The unwrapping process employs the addition map of each successive ball bearing to provide the required unwrapped phase map rather than the usual unwrapping procedure. This gives the unwrapping process to be:

$$\Delta \alpha_N^u = \Delta \alpha_{1,0}^u + \Delta \alpha_{2,1}^u + \ldots + \Delta \alpha_{N,(N-1)}^u$$

(7.0)

where $\Delta \alpha_{i,i-1}^u$ is the unwrapped phase change produced by loading from (i-1) to i ball bearings and N is the maximum number of ball bearing.

Figure 7.4 shows the unwrapped phase of point A. The image shows that the methods are able to remove this discontinuity.

The depth value for the analyses was also observed and the depth against the number of ball bearings was plotted. Figure 7.5 shows the plot with the presence of the discontinuity error and hence showing the depth value to be inconsistent and
erratic. Figure 7.6 shows the plot after removing the discontinuity error. In both plots a general curve is produced and shows that the depression for each ball bearing varies non-linearly with applied load.

Figure 7.1: Unwrapped phase map converted to mm for 10 ball bearings (silicone thickness is 10 μm). The image shows nine locations of indentation.

Figure 7.2: Magnified region near point A.
Figure 7.3: Wrapped phase map near Point A. Residues occur at points 1-2 and points 3-4.

Figure 7.4: Unwrapped phase map converted to mm near point A after correcting the discontinuity.
Figure 7.5: Depth against the number of ball bearings. The unwrapping errors cause inconsistencies in the calculated D value.

Figure 7.6: Depth against the number of ball bearings. The D values are more consistent after removing the unwrapping errors. However the single layer sample calibration results have a non-linear relationship between the depth and the load.
The diameter of the ball-silicone contact was found to be small. Further experiments were carried out by increasing the magnification of the field of view. However, the problems discussed earlier were still apparent. This results in difficulties in trying to achieve reliable the force-deflection curves.

In summary, the results using single layer of silicone have shown that unwrapping errors will occur and plotting the force-deflection curve results in a nonlinear response. For this reason, this sample was not pursued for further experiments.

7.1.2 Perspex

The Perspex employed in these experiment was a clear 6-mm thick plate. The experiment involved the same procedure as with the calibration experiment described in Section 5.4.1. This is to determine the feasibility of using such material for future analysis.

As in previous sections, the indentation diameter caused by the ball bearing is small, hence the experiment employed a 210-mm lens so as to increase the magnification of the acquired images. Figure 7.7a-e shows the wrapped phase difference map for 2,4,6,8 and 10 ball bearings indenting at four points on the Perspex.

The results show small contact points at higher loads. At lower loads, for example for 2 ball bearings, the wrapped phase map does not give any visible indication of indentation. This is due to the Perspex having high modulus (Young’s modulus of Perspex is 3GPa).

The wrapped phase difference map also shows a high background noise. The rms values of this noise ranges from 2nm to 5nm, which was found to be large and is easily seen from Figure 7.7. This may be due to the multiple reflections detected by the camera as the Perspex is a clear sample. The noise causes difficulties in detecting the contact points using the automated analysis described in Section 6.4.

The results using the Perspex show that the material is not suitable as the problems encountered can be prevented by using the double layer substrate. However,
the result perhaps shows potential in future analysis if a similar material can be found having an elastic modulus an order of magnitude or so lower and if the upper surface were to be coated with a durable highly reflective layer. The nonlinear response encountered with the silicone layer is likely to be a problem with this type of sample, however.

Figure 7.7a: Wrapped phase difference map for 2 ball bearings.

Figure 7.7b: Wrapped phase difference map for 4 ball bearings.
Figure 7.7c: Wrapped phase difference map for 6 ball bearings.

Figure 7.7d: Wrapped phase difference map for 8 ball bearings.
Figure 7.7e: Wrapped phase difference map for 10 ball bearings.
7.1.3 Double Layer Elastic Substrate

In this section, the results of the calibration experiment using the optimised double layer elastic substrate are discussed. This sample is chosen as it removes the problems encountered with the two samples discussed in the preceding sections. The sample was designed using Finite Element Analysis so as to optimise the material combination and the thickness as described in Chapter 4. The analysis suggested that the sample should consist of PC10 as the top layer and silicone rubber as the bottom layer with thicknesses of 100µm and 50µm respectively.

Figure 7.8 shows a wrapped difference map for 20 ball bearings with multiple contacts. The analysis involves determining the centre of the indentations described in Section 6.4. Once the locations are determined, the experimental data is fitted with the Lorentzian equations as successive ball bearings are added at each point. The experiments were repeated at least five times. For each experiment, graphs can be plotted for the measured depth against the number of ball bearings. These can of course be converted into depth against the force by converting the weight of the ball bearings into force values these are shown in Figure 7.9a-e.

The reason for carrying out many tests in this way is to check the repeatability of the force values. In doing so, the precise value of the deflection is not important as long as the values agree with each other when the experiments are repeated. However, Figure 7.9a-e shows that the results for the calibration are not consistent. It was found that the inconsistencies were typically between 10% to 15% of the maximum force. Potential reasons for these inconsistencies were therefore investigated to try to improve repeatability.

Initially it was thought that the effect of the wall friction of the calibration rig might contribute to these results. However, a simple experiment discussed in Section 6.2 eliminates this possibility. Several other potential cause methods were then checked. The top surface of the sample have tangential forces acting. To reduce this effect, the experiment was then repeated by spraying the top surface with PTFE spray. The experiment was also repeated in a ‘clean’ environment as dust or other particulates might have an effect on the results.
Though all these methods were put in place, the results however still showed inconsistencies. Nevertheless, these results were employed to achieve the calibration curve. The experiments were repeated five times and each having nine indentation points allowed 45 slopes to be determined. The slope was determined after the plot was converted into depth against the force as the ball bearing was measured to have 0.2608g in weight. This then gives the mean value of the slope to be 0.0019523 mm N\(^{-1}\) with standard deviation of 0.00041676 mm N\(^{-1}\). This figure was used in subsequent experiments to convert measured indentation depth values to force values.

![Diagram of indentation points and measurements](image)

Figure 7.8: Wrapped phase difference map for 20 ball bearings. The image depicts nine locations of indentation. Figure above shows the position of the image with respect to the sample.
Figure 7.9a: Depth against force (N) for Experiment 1.

Figure 7.9b: Depth against force (N) for Experiment 2.
Figure 7.9c: Depth against force (N) for Experiment 3.

Figure 7.9d: Depth against force (N) for Experiment 4.
Figure 7.9e: Depth against force (N) for Experiment 5.
7.2 Sandpile Experiments

The sandpile experiment was achieved by pouring 200-g of sand at 50-g intervals from a point source (funnel). The sample was covered with a layer of ball bearings which will provide the indentation due to the weight of the sand. A layer of 'cling film' was placed in between the sand and the ball bearings so as to avoid any sand pouring in between the ball bearings. This results in the formation of an arch whereby the size increases as the sand is poured. Figure 7.10a-d shows a wrapped phase difference map at each interval.

The image obtained from this experiment is not a complete picture of the whole sample. This result is achieved from a single point of view as the sample mount is placed on a static base. The next stage of the project will involve placing the interferometer on a translation stage which will allow detailed measurements of the spatial variation of contact force to be carried out. Nevertheless the results from the wrapped phase map is then unwrapped to correct the $2\pi$ integral which is then multiplied with equation (3.0) to obtain the displacement map.

From the displacement map, at each weight of the sandpile, the centre location of the indentation was determined using the method described in Section 6.4. Once these points were determined, the displacement map was then fitted with the Lorentzian equation. 3D images of the indentation are then plotted as shown in Figure 7.11a-d. The individual indentations due to the weight of the sandpile are clearly visible. The displacement map images were smoothed by convolutions with the kernel in equation 7.1 in order to reduce the noise.

\[ f = \frac{1}{9} \begin{pmatrix} 1 & 1 & 1 \\ 1 & 1 & 1 \\ 1 & 1 & 1 \end{pmatrix} \] (7.1)

Forces due to the weight of the sand are transmitted onto the ball bearings which causes the indentation. The results show the peaks increases as the weight of sand increases, which shows that the method is viable to detect the forces at the bottom of a granular pile, despite the forces being significantly lower than those used
in the earlier calibration experiment (maximum indentation was 45.1nm). Interestingly, several of the balls have very low loads on them, which is suggestive of the presence of internal arches.

Figure 7.10a: Wrapped phase difference map for 50g of sand.

Figure 7.10b: Wrapped phase difference map for 100g of sand.
Figure 7.10c: Wrapped phase difference map for 150g of sand.

Figure 7.10d: Wrapped phase difference map for 200g of sand.
Figure 7.11a: 3D plot for 50-g sandpile. The image has square pixel size of 0.03 mm. The position of the image is shown at the top right hand side of the page.

Figure 7.11b: 3D plot for 100-g sandpile. The image has square pixel size of 0.03 mm. The position of the image is depicted as shown at the top right hand side of the page.
Figure 7.11c: 3D plot for 150-g sandpile. The image has square pixel size of 0.03 mm. The position of the image is shown at the top right hand side of the page.

Figure 7.11d: 3D plot for 200-g sandpile. The image has square pixel size of 0.03 mm. The position of the image is depicted as shown at the top right hand side of the page.
7.3 Layered Experiments

The layered experiment involves placing the ball bearings in a hexagonal close packed arrangement. Subsequent layers are then placed on top of the previous layer in order to achieve a face-centred cubic arrangement (commonly known as ABCABC packing – see Figure 5.24 in Section 5.4.2). Five layers were arranged in this form with each layer having 265 ball bearings. At each layer, the position of the indentation will be the same as the experiment only involved placing one layer on top of one another in the face centred cubic arrangement. Figure 7.12a-e shows the result in the form of displacement map for all of the layers. Figure 7.12a and b shows small indentation suggesting low load but nevertheless the indentation values were extracted since the points of indentation are the same for all five layers.

Figure 7.12 yields 25 indentation points and at each indentation point the Lorentzian equations are fitted to provide the depression value. This is then converted to force values by means of the calibration curve. The first analysis carried out was to check that the sum of the measured forces from the experiment should be equivalent to the fraction of the total force applied within the field of view of the sample for a given layer. This gives:

\[
\sum F_m = \frac{\text{Number of Contacts in the field of view}}{\text{Total number of contacts}} \times \text{Total Force}
\]  

(7.2)

where \( \sum F_m \) = sum of the measured force

For each layer, the sum of the measured force from the experiment is estimated and compared with the calculated fraction of the total force. The results of this analysis are tabulated in Table 7.0.

The results from Table 7.0 shows that in general, the measured and the calculated forces are in agreement with one another except for layer 1 and layer 2. Layer 1 and layer 2 shows a significantly large percentage difference which may due to the low forces detected at this end. However, increasing the number of layers will involve more forces, which yield lower percentage differences at higher number of
layers. This indicates the method shows a good agreement between the measured and calculated average forces.

Table 7.0: Comparisons between the measured force and the calculated force.

<table>
<thead>
<tr>
<th>Layer</th>
<th>Measured Force (N)</th>
<th>Calculated Force (N)</th>
<th>Percentage Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(\sum F_m)</td>
<td>(0.06591)</td>
<td>27.45%</td>
</tr>
<tr>
<td>Layer 1</td>
<td>0.04782</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Layer 2</td>
<td>0.10039</td>
<td>0.13232</td>
<td>24.13%</td>
</tr>
<tr>
<td>Layer 3</td>
<td>0.20400</td>
<td>0.19848</td>
<td>2.78%</td>
</tr>
<tr>
<td>Layer 4</td>
<td>0.26993</td>
<td>0.26464</td>
<td>2.00%</td>
</tr>
<tr>
<td>Layer 5</td>
<td>0.32606</td>
<td>0.33079</td>
<td>1.43%</td>
</tr>
</tbody>
</table>

The force values resulting from each added layer can be analysed statistically in order to compare the results with a statistical model namely the 'q' model which has been put forward by Coppersmith et. al [42]. The reference proposed the weight distribution function that is independent of the number of layers to be:

\[ P_N(v) = \frac{N^N v^{N-1} e^{-Nv}}{(N-1)!} \]  \hspace{1cm} (7.3)

where \(P\) is the weight distribution, \(N\) is the number of sites (\(N = 3\) in this case since the ball bearing is placed in face-centered cubic arrangement), and \(v\) is the normalised weight given as:

\[ v = \frac{w}{D} \]  \hspace{1cm} (7.4)

where \(w\) is the weight or force of the ball bearings and \(D\) is the layer number.
For each layer, a histogram was of the number of contacts within a given force range. These force ranges can be normalised using equation (7.4) and Figure 7.13a-e shows the histogram plot for each layer. The probability function for each bin of the histogram is then determined using:

\[ P_i = \frac{N_i}{\Delta v \sum N_i} \]  

(7.5)

where \(N_i\) is Number of contact at each bin and \(\Delta v\) is the bin size.

Both the theoretical and the experimental probability functions are plotted in Figure 7.14a-e. Figure 7.15 then represents the combinations of all the results from the five layers with the theoretical curve since equation (7.3) is not dependent on the number of layers.

The normalisation of the force values yields a possibility of combining the entire histogram plot of Figure 7.13a-e into one plot. Figure 7.16 shows the combination of the plot that reveals the number of contact against the normalised force. Applying equation (7.5) and estimating the mean error, a curve can be plotted as shown in Figure 7.17. Figure 7.17 also include the theoretical plot in order to compare the results.

The experiment only measured up to five layers and the analysis reveals that the curve shows a general trend similar to the theoretical curve. This show that the method suggests here is suitable in testing the 'q' model theories. The large experimental errors can be reduce by repeating the experiment \(N_i\) times and the errors will decrease by \(\sqrt{N_i}\); where \(N_i\) is the number of experiment performed.
Figure 7.12a: Displacement map for 1 layer.

Figure 7.12b: Displacement map for 2 layers.
Figure 7.12c: Displacement map for 3 layers.

Figure 7.12d: Displacement map for 4 layers.
Figure 7.12e: Displacement map for 5 layers.
Figure 7.13a: Histogram plot for the number of contact against the normalised force for one layer.

Figure 7.13b: Histogram plot for the number of contact against the normalised force for two layers.
Figure 7.13c: Histogram plot for the number of contact against the normalised force for three layers.

Figure 7.13d: Histogram plot for the number of contact against the normalised force for four layers.
Figure 7.13e: Histogram plot for the number of contact against the normalised force for five layers.
Figure 7.14a: Force distribution against normalised force for layer 1. The '*' denotes the theoretical and 'o' denotes the experimental values.

Figure 7.14b: Force distribution against normalised force for layer 2. The '*' denotes the theoretical and 'o' denotes the experimental values.
Figure 7.14c: Force distribution against normalised force for layer 3. The '*' denotes the theoretical and 'o' denotes the experimental values.

Figure 7.14d: Force distribution against normalised force for layer 4. The '*' denotes the theoretical and 'o' denotes the experimental values.
Figure 7.14e: Force distribution against normalised force for layer 5. The ‘*’ denotes the theoretical and ‘o’ denotes the experimental values.

Figure 7.15: Combining the force distribution plot for the five layers.
Figure 7.16: Histogram plot of the force for the five layers.

Figure 7.17: Comparisons between the theoretical and the experimental force distributions for the layered experiment.
7.4 Summary

The results of the experiments performed have been presented and discussed in this chapter. The main experiments were the force-deflection experiments needed to calibrate the sample. This procedure was also performed with different sample which was a single layer of silicone on glass and a clear Perspex plate. This was performed so as to give a comparison with and the results from the double layer substrate.

The results from the single layer of silicone shows that unwrapping errors were present. This was thought to be due to the change in phase of reflected light from the silicone-steel relative to the silicone-air interface during the loading. The force deflection curve also shows a non-linear relationship between the depth and the force. The results from the Perspex plate shows that there was a high background noise occurring. This was attributed to the multiple reflections coming from the top surface of the Perspex. It was found that the noise was in the range of 2 nm to 5 nm. With these problems, it was clear that the double layer substrate is the best sample for the experiment.

The force-deflection experiment performed on the double layer elastic substrate reveals linearity between the force and the deflection. However, repeating the experiments shows an inconsistency of the results. This was found to be in the range of 10%-15%. Reasons for this effect were investigated, but no satisfactory solutions were found. Even though the calibration experiments showed this lack of repeatability, it was used as the calibration curve by taking an average of the whole results.

A sandpile experiment was performed on one single position and up to 200g of sand was poured in steps of 50g from a point source (a funnel). The experiment was performed at a single location due to the static nature of the experimental arrangement. The next step in the project will be to position the interferometer on to a translation stage. The analysis reveals qualitatively promising results, as the displacement profile showed some evidence of arching and increasing the amount of sand increased the value of the peaks.
Layered experiments were performed using ball bearings stacked in a face-centered cubic packing arrangement. The weight of the specific area measured was then compared with the calculated fraction of the force applied over the same area. This reveals a good agreement at higher load, suggesting that the method developed will be able to analyse the wall force deflection. The high percentage difference at the first two loads may possibly be due to the magnitude of the forces applied under these conditions.

The results were also compared with a theoretical equation, namely the 'q' model. The rather small numbers of contacts studied made it difficult to obtain highly accurate experimental probability distributions. However, averaging over all the curves resulted in a composite plot that shows promising results as the trend of this plot shows similar behaviour to the theoretical plot. The experiment needs to be repeated as well as increasing the number of points. This again will involve attaching the interferometer onto a translation stage. This will form part of the future work of the project.
Granular materials have many unique characteristics that are different from those of liquids and solids. These characteristics are not fully understood due to the physical nature of the material. One of these characteristics is the dip at the centre of a conical sandpile. Most materials are in one way or another in granular form at some stage of manufacturing process and this has prompted prolific research in this field.

Theoretical works in powder mechanics have put forward many articles in trying to explain these phenomena. Among them is the continuum approach, macroscopic and microscopic analyses, statistical works, computer simulations and many more. Experimental work to date however, seems to be rather limited.

The project is a pioneering experimental work using optical techniques in order to be able to measure the force distribution beneath the granular pile. The span of the projects covers: a summary of powder behaviour put forward by various authors in the literature; Finite Element analysis for modelling the optimised substrate; fringe analysis and the development of the phase shifting algorithm; improving the method to fabricate the sample; designing the laser set-up; calibration experiments; and performing the layered and the sandpile experiments as well as the analysis of the results.

Finite Element analysis was employed in order to determine the material combinations and the thickness of both the top and bottom layer of the elastic substrate. The substrate consists of epoxy PC10 as the top layer and silicone rubber as the bottom layer, with gold evaporated in between that acts as the reflective film. The design was based on several criteria. These were: a) to achieve a linear force deflection curve; b) to maximise the deflection of the gold layer for a given indentation depth; c) to give a curve which is as broad as possible within the contact
region and d) to avoid crosstalk between one point and another. The required thickness was found to be 100 µm for PC10 and 50 µm for the silicone rubber.

The sample was fabricated by firstly manufacturing the silicone layer. This was achieved using a 'spin-coater' machine that was manufactured in-house. Gold was then evaporated on top of the silicone which is the reflective film for the experiment. The top layer was cast by pouring the epoxy on to the gold and the layer was form by placing a smooth PTFE film which allows a smooth surface weight to be placed on top in order to compressed epoxy into the appropriate thickness.

The interferometer is based on a Fizeau arrangement as this is able to measure the deflection due to the force applied. This will provide the method needed in order to detect the indentation as the interferometer is a common path one. An interference pattern will occur between the beams reflected from the gold and the glass-air interface which are detected by the camera.

The laser employed is a tunable wavelength laser. This will acts as both the light source and the phase shifting hardware. This method was chosen because it is the only practical method of incorporating the phase shifter in such a common path interferometer.

The phase shifting algorithm employed is the fifteen-frame technique. This is used in order to reduce the background noise detected during the experiment. This enables the noise to have rms values of less than 1nm.

The sample was then calibrated by means of indentation with ball bearings. These ball bearings were stacked on top of one another at particular points. This was achieved by manufacturing a calibration rig in order to achieve a vertical stack as well as to achieve multiple contacts for the calibration of the sample. The calibration experiments were also used to check the feasibility of using other materials as the sample. Two types of material were used, namely a monolayer of 10 µm of silicone rubber on glass and a 6 mm thick plate of perspex.

The results from these two latter experiments show that it is not favourable to employ these materials for the subsequent experiment. The silicone monolayer experimental results in phase unwrapping errors with a non-linear relationship between the depth and the load. Perspex was found to be very rigid material and was
not sensitive in detecting low load. The background noise was also found to be high with rms values ranging from 2-5 nm.

The experiment using the double layer substrate was repeated up to five times. This was analysed by means of plotting the calibration curve of the depth against the force. The result however shows inconsistencies of up to 10-15%. This was then further investigated in order to find the reasons for this inconsistency. This includes several measures such as repeating the experiment in a 'clean' environment so as to remove the effect of dirt and dust as well as spraying the sample with PTFE spray in order to mitigate the effect of the tangential forces. The results however still generate this inconsistency which is still under investigation. Nevertheless these results were employed as the calibration curve in order to analyse the results from the layered and the sandpile experiments.

The experimental data were then fitted with a mathematical equation in order to quantify the value of the depression for each successive weight of the ball bearing. From extensive analysis, it was found that the Lorentzian equation provides a good fit with 2-4% differences.

The sandpile experiments were achieved by pouring 200g of sand in 50g interval at a point source. This results in the formation of arches which was measured by the experiment. The acquired image showed only a small region within the whole pile as the interferometer was static. To achieve a complete measurement, the interferometer needs to be on a translation stage which is part of the future work of the project. The result shows that the force is not distributed evenly between the balls and increases as the load increases.

The layered experimental results were analysed by comparing the measured forces with both the calculated mean force and with a statistical approach known as the 'q' model. The experiments involved placing five layers of ball bearings in a face-centred cubic pattern. At each layer measurements were made and the force values were measured. The sum of the forces at each layer was compared with the relevant fraction of the total force. The results show good agreement between the two values suggesting the experiment provides a good estimate of the forces.
Histograms were plotted in order to statistically analyse the force distribution at each layer. The probability functions were calculated and plotted against that expected from the theoretical ‘q’ function. Ideally the experiments should be repeated many times and include many more measurement areas. This again will involve mounting the interferometer onto a translation stage. The results however show that the method is suitable for testing the ‘q’ model theories.

8.1 Future Work

The techniques developed in this project have been aimed in developing a feasible technique that is able to measure the contact forces in a granular bed. The interferometer technique is based on using a frequency-scanning interferometer as the source of illumination as well as to shift the phase by means of tuning the wavelength. There are however, several issues that would benefit from further work.

The current technique, involved mounting the interferometer statically on an optical table. This render difficulties in trying to acquire the whole image of the sample as it is restricted to only one field of view. To overcome this, it is proposed that the interferometer is attached to a translation stage. The translation stage can be controlled to move laterally which will enable the experiment to acquire more images of the sample. This will increase the number of areas for the measurement, which allows more indentation points, and provide a more complete experimental result.

Once the interferometer is on a translation stage, the sandpile experiment can also be repeated. This will ensure the whole area of the sample can be measured and this will gives the force distribution under the pile. In this experiment, the sandpile was constructed by means of a point source. This can be extended by including the distributed method which then forms wedges rather than arches. Both results can be compared to yield the differences between the two.

The current experiment uses 4-mm diameter ball bearings. This can be extended to include various sizes of ball bearings. By repeating the mentioned experiments for these various sizes of ball bearing would provide more experimental data.
In Chapter 5, the fabrication of the sample was explained in details. The method employed here in fabricating the silicone is developed from [3]. The use of a spin-coater in manufacturing the silicone has mitigated most of the problems encountered especially the damage towards the silicone and the glass itself. The fabrication of the top layer of the sample needs further investigations. It was found to be difficult to achieve a uniform layer of epoxy without bubbles. The PTFE layer also left a slight surface texture. Other materials may possibly replace the epoxy PC10 and hence the method to fabricate the top layer will change based on the required manufacturing procedure. This however will need the FE analysis to be repeated in order to achieve the optimised combination and thicknesses.

The results from the double layer elastic substrate experiments yields 10-15% inconsistencies. Further investigation is required to ensure that the cause of these inconsistencies can be identified and hence rectified.

The laser used in the experiment is the New Focus Vortex Tunable Laser, which was found to be among the main problems as it gives mode hops. This raises difficulties in completing the experiment. The laser needs to be repaired in order to achieve working conditions as it shows mode hops behaviour even though all the required conditions of the laser have been put in place. The repair was undertaken by the manufacturer during the project which the researcher found to be very costly (in terms of time). The laser still shows same signs of mode hop behaviour despite the repair.

The project presented in this thesis is a new method in trying to measure the grain-wall contact force. The method employs an interferometry method using a wavelength tunable laser. The results from the experiments show that the method proposed has significant potential in measuring the force distribution which will provide an insight into the behaviour of granular media.
**REFERENCES**

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Graphs for PL3 – Silicone

PL3-Silicone at $\alpha = 0.1$

PL3-Silicone at $\alpha = 0.3$
Graphs for PL3 - Glass

PL3-Glass at $\alpha = 0.3$

PL3-Glass at $\alpha = 0.5$
Graphs for PL3 – PC10
Graphs for Glass – Silicone

Glass-Silicone at $\alpha = 0.05$

Glass-Silicone at $\alpha = 0.10$
Graphs for Glass – PL3

Glass-PL3 at $\alpha = 0.1$

Glass-PL3 at $\alpha = 0.3$
Glass-PL3 at $\alpha = 2.0$

-2.62 \times 10^{-6}

\begin{align*}
-2.64 & \quad \text{Interface} \\
-2.66 & \quad \text{Surface}
\end{align*}

$y_{77,3}$ (mm)

$x$ (mm)

$0 \quad 0.2 \quad 0.4 \quad 0.6 \quad 0.8 \quad 1 \quad 1.2 \quad 1.4 \quad 1.6 \quad 1.8 \quad 2$
Graphs for Glass – PC10

Glass-PC10 at $\alpha = 0.05$

Glass-PC10 at $\alpha = 0.1$
Glass-PC10 at $\alpha = 0.3$

Glass-PC10 at $\alpha = 0.5$
Graphs for PC10 – Silicone

PC10-Silicone at $\alpha = 0.05$

PC10-Silicone at $\alpha = 0.3$
Graphs of PC10 – Glass

PC10-Glass at $\alpha = 0.3$

PC10-Glass at $\alpha = 0.5$
Graphs of PC10 – PL3

PC10-PL3 at $\alpha = 0.05$

PC10-PL3 at $\alpha = 0.1$
PC10-PL3 at $\alpha = 2.0$

$y$ (mm)

$x$ (mm)

$10^{-3}$
Graphs of Silicone – Silicone

Silicone-Silicone at $\alpha = 0.1$

Silicone-Silicone at $\alpha = 0.3$
APPENDIX B
Graphs showing the relationship between $\alpha$ and $p$ for Glass - PL3 and Glass - PC10.