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Development of a Flexible Load-Based Micro-Indentation System

Chia-Nung Chou

**Thesis submitted to the
College of Engineering and Mineral Resources
at West Virginia University
in partial fulfillment of the requirements
for the degree of**

**Master of Science
in
Mechanical Engineering**

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2011

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Modulus; Material Characterization; Flexible; Geometry Independent.**

Abstract

Development of a Flexible Load Based Micro Instrumented Indentation System

Chia-Nung Chou

Various types of indentation methods have been used for the determination of mechanical properties of material; nevertheless, conventional indentation test units are restricted by the geometry of the samples. It is difficult to obtain the mechanical properties from non-flat surface objects. In this research, based on an in-house developed load-based micro-indentation test method, a suitable flexible micro-indentation system was developed to determine material mechanical properties such as hardness and elastic modulus of test samples with arbitrary surface geometry.

The focus is in developing a flexible micro-indentation unit. Due to nonlinearity of the system compliance and possible change of indentation direction during testing, an in-situ calibration methodology is developed to obtain the correct mechanical properties of the testing material. To validate the capability of the flexible micro-indentation instrument, aluminum 6061, bronze 932 and H13 tool steel were tested with various indentation arm lengths and angles such as 0° , 30° , 45° , 60° and 90° for Young's modulus measurement. In each case, the data were reprocessed with the in-situ calibration method and accurate results were obtained.

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Chapter 1: Introduction

For the past two decades, extensive nano and micro-indentation technologies have been developed. Instrumented Indentation is capable of providing material mechanical properties such as hardness, Young's modulus, fracture toughness, etc., and it has become a standard testing method for thin-film and small-size bulk material mechanical properties measurement. These advanced material characterization techniques have demonstrated measurement reliability and accuracy and has replaced some existing material characterization devices in many applications.

Recently, a load based multiple-partial unloading micro-indentation technique was developed in WVU. Comparing to conventional nano/micro-indentation methods, which require precise measurements of indentation depth and contact load; multiple-partial unloading technique simplifies the measurement that such technique only measures the total displacement and contact load, along with an in-house loading frame design, the load-depth indentation system is capable of determining Young's modulus of metallic alloys samples accurately.

In order to achieve the goal of performing in-situ and/or on-site indentation testing on structural components, in this thesis research, the same methodology is applied to a flexible indentation design. In the early days of designing and testing flexible indentation system, the test results were not satisfactory; however, the data obtained were consistent. A discovery of the impression shape indented by the flexible indentation system shows that indentation is not solely in the direction normal to the sample surface, but also exhibits lateral movements. Therefore, an assumption was made that the total indentation displacement (measured by the PZT actuator) is split into X, Y and Z directions, where X and Y are lateral directions and Z is the normal direction of the specimen's surface. The movement of Z direction is yet proportional to the total displacement. In other words, in order to get the correct elastic modulus, a calibration factor must be applied. The calibration methodology developed in this research shows that after applying such method, despite the arm length and arm angle, the test results were

accurate and consistent when performing indentation tests on standard alloys such as aluminum, bronze and steel. The technique has led the development of an adjustable flexible load-depth sensing indentation system, which has the capability of in-situ material mechanical property measurement.

1.1 Problem Statement

Existing nano and micro-indentation systems are bulky and lack portability, more importantly, these indentation testing systems require bringing small test samples onto the test stand and are incapable of performing in-situ, on-site indentation tests on structural components. This thesis research is focused on implementing a recently in-house developed multiple partial unloading technique in combination with an in-situ calibration method for the development of a flexible micro-indentation system.

1.2 Research Objectives

The objective of this research is to develop a flexible load-based micro-indentation system for material mechanical property measurement on structural components with either flat or curve surface geometry. The testing methodology is based on the previously in-house developed multiple loading/unloading indentation procedure at WVU with the incorporation of a flexible design concept of the indenter such that indentation testing on non-flat surface geometry or in-situ indentation testing on bulky structural components can be realized.

Chapter 2: Contact Mechanics

Contact mechanics is a knowledge of deformation of solids that making contact with each other. The subject is also a foundational to the field of mechanical engineering, which provides essential information for quality control of the products or even predicting system failure. The original work in this study can be chased back to 1882, a publication “On the contact of elastic solid” by Heinrich Hertz [1]. In this work, Hertz claims that the least amount of force applied to a conical indenter and produce a permanent impression on a material is the absolute value of hardness of the material. This postulate gave the influence of development for Brinell, Rockwell, Vickers, and Knoop hardness tests, as well as modern day indentation techniques.

2.1 Elastic Contact

Found by Hertz, elastic contact between two a rigid spheres can be described using Equation (1), where a is the contact area between the two indenters and P is the load applied to the indenters with diameter of d_1 , d_2 and E_R is the reduce modulus between those two spherical objects. However, in order to describe the phenomena of indenting on a flat surface, the formula was re-written by changing d_2 to infinity, shown in Equation (2). Through the discovery of this relationship between contact radius and indentation load, Hertz gained much acclaim [1].

$$a^3 = \frac{3P}{8E_R \left(\frac{1}{d_1} + \frac{1}{d_2} \right)} \quad (1)$$

$$a^3 = \frac{4}{3} \frac{kPR}{E} \quad (2)$$

The variable k accounts for the elastic mismatch between the indenter and the specimen, as seen in Equation (2), where E , ν and E' , ν' are the elastic modules and Poisson's ratios for the specimen and the indenter, respectively [2]

$$k = \frac{9}{16} \left[(1 - \nu^2) + \frac{E}{E'} (1 - \nu'^2) \right] \quad (3)$$

The maximum tensile stress of the material was also found by Hertz in which it occurred at the edge of the contact surface, is a function of the contact area and is found using Equation (4), where a is again the contact area, ν is Poisson's ratio and P is the load.

$$\sigma_{\max} = (1 - 2\nu) \frac{P}{2\pi a^2} \quad (4)$$

This stress, acting in a radial direction on the surface around the indentation, is usually a factor that triggers cracking on the sample surfaces. The mean contact pressure is defined as the indentation load divided by the contact area between indenter and sample, allowing this to become a useful normalizing parameter. Substituting the load into the mean pressure as of it shown in Equation (2), a result of Equation (5) is rendered. The mean pressure is generally represented in these terms for matters concerning indentation [2].

$$P_m = \left(\frac{3E}{4\pi k} \right) \frac{a}{R} \quad (5)$$

The mean contact pressure is referred to as the indentation stress and the quantity a/R as the indentation strain or a factor describes the degree of penetration. When those two parameters of a material plotted against each other, the result is similar to the material's tension or compression test.

2.2 Indentation Response

Studied by Hertz, found the hardness value is intimately related to the mean contact pressure P_m (Defined as Pressure over the contact area) beneath the indenter at a limited or known condition of compression. Once the mean contact pressure P_m is plotted against the degree of penetration a/R , where a is the indented radius and R is the indenter radius, the information about plastic and elastic properties of a material could be extracted. This stress strain response of an elastic plastic solid can be divided into three main categories which are dependent on the yield strength Y of the material [3]. When mean contact pressure values are less than $1.1Y$ are fully elastic, in such state, there would be no permanent or residual

impression left on the test sample after loading is removed. However, with mean pressure of around $1.1Y$, would result in plastic deformation beneath the surface of the indenter, surrounded by elastic region where it located around the contact surface. With a higher value than $1.1Y$, would cause extending of the plastic region beneath the indenter.

2.3 Piling Up and Sinking In

When an indentation test begins, during the elastic state of indentation, the surface of the specimen is drawn downward, hence sink-in effect occurred, however, as the state switched from elastic to plastic deformation, the material may either be affected by pile-up or sink-in at the crater of the indentation. As it shown in Figure 1, when indentation reaches fully plastic zone, pile-up or sink-in may occurred, such behavior is not only found to be dependent upon the ratio of elastic modulus E to the yield stress Y of the specimen, but also it is related to the work or strain hardening of the testing material [4].

Figure 1: Pile-Up and Sink-In Effects

In cases that strain hardening is not considered, materials with large E/Y ratios, pile-up is expected to be observed in those material, and similarly, materials with low or smaller E/Y ratios, sink-in is likely to occur during indentation tests [5] [14]. In the other hands, material exhibits strain hardening, the yield strength increases significantly as the strain increases. When indentation is performed on such specimen, as the indenter driven deeper into the sample, the material within the plastic zone becomes harder in which the location that contact between the indenter tip and the sample, however, the crater or the surrounding of the indentation remains elastic, therefore sink-in effect taking place.

Pile-ups and sink-ins influence the measurement of the contact area, which is undesirable for hardness and elastic measurement; particularly, the effect become significant when the indentation size is relatively small or shallow, which reflects the importance of constructing stabled loading frame to create low system compliance testing environment.

Chapter 3: Indentation Testing

Indentation testing is a mechanical testing method which was designed to determine material's mechanical properties; hardness and elastic modulus can be calculated by the characteristics of the impression of the sample, such as the diameter, length of the diagonal, indentation depth and indentation load etc.

Even though indentation techniques was first developed for use on metals, it was found to be applicable for many different types of materials in the industries; factories could use indentation techniques to verify the qualities of products, including glass productions, ceramics, and even composite materials.

Indentation testing had been used for hardness testing dating back to the 1800's, nevertheless; these techniques had tremendous improvements in the past few decades, which these techniques had gone far beyond the capabilities of these original testing procedures and techniques. For modern day's applications, indentation techniques had been able to perform indentation testing on delicate samples, such as thin-film mechanism in micro or nano scales and determine materials' hardness as well as elastic modulus of the material.

3.1 Indentation hardness

Indentation hardness tests typically involve the size measurement of the permanent impression done by the indenter on the test sample such as the indentation depth, the diameter or the diagonal length of the dent, which the measurement is a function of the indentation load [6].

Different types of materials often use different geometries of indenters. As it mention before, In order to prevent cracking on the test specimens it is suggested to use blunt or spherical indenters when it comes to ductile material testing, and using sharp indenters for brittle material testing.

3.1.1 Brinell Indentation Hardness

The Brinell indentation hardness which characterizes the degree or the scale of penetration done by the indenter into the sample's surface, was proposed by a Swedish engineer, Johan August Brinell in the early 20th century; due to its simplicity and inexpensive design, Brinell indentation hardness testing is not only the first widely accepted but also the most popular, reliable and standardized hardness test [7].

Brinell's instrument could be used for a variety of material, such as metals, alloy and even some wood. As it's shown in the following schematic, Figure 2, a small ball which is the indenter is pressed into the surface of the specimen. It's not necessary to know the material of the indenter as long as its hardness is way above the testing specimen. It depends on the testing specimen; the full load is normally applied for 10 to 15 seconds for iron and steel and for other material, the load could be hold for up to 20 to 30 seconds, then the applied load is removed. After the plastic deformation, a spherical indentation will be left onto the specimen; the specimen is taken to a low powered microscope, and measure the diameter of the indentation caused by the load and indenter.

Figure 2: Brinell Hardness Test

The diameter which is measured by the microscope along with the applied load and the diameter of the indenter are needed in order to calculate the specimen's hardness in Brinell

hardness scale. Equation (6), is the formula to calculate Brinell hardness, where D_B is the diameter of the indenter and d_B is the diameter of the dent caused by force. Although this small indentation is permanent, Brinell hardness test is usually considered to be non-destructive due to its indentation depth being relatively shallow when it's compared to other hardness tests that are using sharp indenters.

$$H_B = \frac{2F}{\pi D_B \left(D_B - \sqrt{D_B^2 - d_B^2} \right)} \quad (6)$$

3.1.2 Vickers Hardness

The Vickers hardness test was developed in 1924 by British engineers, Smith and Sandland at Vickers Ltd. Vickers hardness test is an alternative hardness testing method to Brinell approach. Instead of using spherical indenter, Vickers hardness testing unit uses pyramid shaped indenter with an apical angle of 136 degree shown in Figure 3; the material of the indenter often to be diamond, therefore, Vickers hardness test is also known as the diamond pyramid test [8].

Figure 3: Vickers Hardness Test

With a known load, the diamond indenter will be pushing against the specimen. After the load causes a permanent impression on the sample's surface, then the indenter is withdrawn. Again, a microscope is needed in order to determine the diagonal length of the impression. The measurement of the indentation diagonal is substituted into the Equation (7)

to calculate the hardness base on the Vickers scale; where H_V is the Vickers hardness value, F is the applied force, and d_V is the length of the diagonal measured by the microscope.

$$H_V = \frac{1.854F}{d_V^2} \quad (7)$$

The Vickers testing method is very reliable and time-efficient, as only one scale covers the entire hardness range. Although typically it used for metals, Vickers is also helpful when it comes to brittle material tastings.

3.1.3 Rockwell Hardness

The Rockwell hardness testing was developed in 1920, by two American metallurgists, Hugh M. Rockwell and Stanley P. Rockwell. It was invented as a hardness testing tool, which compares the original indentation depth under a preload to a deeper indentation depth done by a larger applied force [9], which is shown in the Figure 4.

Figure 4: Rockwell Hardness Test

By creating a difference in penetration depth d_R between the preload (F_1) and the second applied force (F_2), Rockwell hardness can then be calculated by applying Equation (8), where E_R , is a function of the indenter and d_R is the difference in depth caused by two loads.

The calculation of Rockwell's hardness scale is depending upon the applied force and the indenter used during the test that different indenters have their own function E_R , which makes Rockwell hardness value so unique, and there is no alternative testing method to obtain Rockwell hardness value.

$$H_R = E_R - d_R \quad (8)$$

3.1.4 Micro Hardness

The term, Micro-hardness testing actually has no true definition. When it comes to hardness testing on a small, thin or fragile material, macro indentation is no longer valid. Similar to macro indentation, micro indentation employed with much lower indentation load, which produces smaller and much shallow indentation, yet it would not damage the test sample.

Due to its nature to be non-destructive, micro indentation has become popular for material characterization. Although, much type of indenters could be used for micro indentation hardness test, Knoop and Vickers are the most common seem testing methods.

Knoop hardness test, which was developed in 1937, by Frederick Knoop and his colleagues at the National Bureau of Standards, today's NIST [10]. As it shown in the Figure 5, a pyramid-shaped indenter is pressed into a polished surface; the geometry of Knoop indenter is an extended pyramid with the length to width ratio being 7:1 and respective face angles are 172 degrees for the long edge and 130 degrees for the short edge.

Figure 5: Knoop Hardness Test

With a known force, Knoop hardness value then could be calculated by using Equation (9), where H_K is the Knoop hardness value, F is the applied force, C_K is correction factor related to the shape of the indenter, ideally 0.070279, and L_K is the indentation depth.

$$H_K = \frac{F}{C_K L_K^2} \quad (9)$$

A few drawbacks on Knoop hardness test are that indentation depth is hard to be determined; unlike other hardness tests, Knoop testing requires optical and high priced depth sensing equipments. In the other hand, a fine surface polishing is required for this test, which sample polishing can be time consuming or even destructive to the sample.

3.1.5 Hardness Value Conversion

There are several hardness techniques existing, yet they cannot be compared to each other for which is better or not. Even though there are hardness charts available, but the charts can only give an approximation of hardness from one scale to another. The reason of conversion being inaccurate is that each technique measures the resistance to deformation in slightly a different way; there are too many parameters that were not taking consideration in hardness testing, such as Poisson's Ratio of the sample, indentation sunk-in and pile-up effects, and indenter types used in the experiment. In short that there's no direct conversion for hardness test from one scale to others, yet it is not intended to replace existing testing techniques [11].

3.2 Micro and Nano Instrumented Indentation

Employed with high resolution instrumentations which continuously monitors and control the displacement of the indenter; Instrumented indentation systems are able to perform indentation testing in micro or nano scale and these techniques have become reliable tools for collecting and evaluating elastic response of the testing samples. The data of load/displacement curve recorded on unloading which provides valuable information of the elastic response [12].

Chapter 4: Instrumented Indentation Data Analysis

Unlike conventional hardness techniques, instrumented indentation ingeniously uses the geometry of the indenter along with the load cell reading and the traveled length of the PZT actuator to acquire the information of contact area, indentation load and depth. Therefore, the elastic modulus and hardness value of the test sample may both be obtained by using instrumented indentation. Even though indentation testing could be performed with various geometries of indenters, the research is focus on micro indentation with spherical-geometry indenter.

4.1 Spherical Indenters

Stresses and deflections arise upon the contact between two elastic objects. Even though there is an infinite amount of geometries, yet the most common and most interesting case is the contact between a rigid sphere and a flat surface, which is shown in the Figure 6, where h_p is the length between the contact circle and the tip of the indenter along the normal direction, h_a is the depth from the free surface of the sample to the contact circle, h_t is the total penetration depth, in the other words, h_t is the sum of h_p and h_a ; R is the radius of the indenter, and a is the radius of the contact circle [3].

Figure 6: Spherical Indentation

As it mentioned in the previous chapter, the radius of the contact circle is dependent upon the applied load and elastic modulus of those two contact bodies, and such relationship was found by Hertz [1]. Shown in the Equation (10), where P is the applied load, essentially, the

indentation load, and the quantity E^* is also known as the reduce modulus or the combined modulus of the two bodies.

$$a^3 = \frac{3}{4} \frac{PR}{E^*} \quad (10)$$

The relationship, seen in Equation (11) [13], that E , ν and E' , ν' are the elastic modulus and the passion ratio of the two contact objects, or the sample and the indenter, respectively.

$$\frac{1}{E^*} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu'^2)}{E'} \quad (11)$$

Base on the assumption seen in Equation (12) by Oliver and Pharr [5] [14], where h is the indentation depth, a is the radius of the contact circle, R is the indenter radius, the Equation (16) now can be modified to form Equation (13) [3].

$$h = \frac{a^2}{R} \quad (12)$$

$$h^3 = \left(\frac{3}{4E^*} \right)^2 \frac{P^2}{R} \quad (13)$$

With known combined elastic modulus between the sample and indenter, now the indentation depth can be determined by applied load and the indenter radius. Equation (13) could also be rearranged, seen in Equation (14), which showed the relationships among the contact load, elastic modulus, indenter radius and the indentation depth.

$$P = \frac{4}{3} E^* R^{1/2} h^{3/2} \quad (14)$$

4.2 Load Displacement Curves

Unlike conventional Indentation hardness test, after decades of developments, instrumented indentation has achieved way beyond old fashion indentation tests' capabilities; not only hardness value, but also elastic modulus could be determine by one single test, performed by instrumented indentation system. Typically, load and indentation depth information are recorded throughout the entire test, which the load or the depth would be incrementally predefined by the user. Due to the fact of producing shallow impression,

instrumented indentation does not rely on optical techniques, ingeniously it calculate the contact area base on the indentation depth and the indenter geometry, along with the load information, the hardness value is determined. After indentation load is removed, the test specimen's tendency of regaining its original shape provides the information that leads the calculation for material's elastic modulus; shown in Figure 7.

Figure 7: Single Unloading Indentation Test

Originally, there was a dent caused by the applied load; after removing the force completely, the impression recovered due to the elastic response of the material, and a permanent impression left onto the test specimen. In order to determine the elastic modulus of the test specimen, analyzing the indentation load-displacement curve was found to be very useful, not only calculating the hardness value and modulus, but also the load-displacement curve could be used to indentify abnormal response of the test specimen as well as the loading frame, such as popping, cracking or other environment related issue, for instance, vibration [3].

4.3 Single Point Unloading Indentation Analysis

Single point unloading analysis is the most commonly seen indentation technique, which utilizes one unloading point within the load displacement curve, seen in Figure 8, in which the reduced modulus is calculated; alone with the equation shown in the previous page, Equation (15), with known elastic modulus and Poisson's ratio of the indenter, the specimen's elastic modulus can be easily estimated.

Figure 8: Single Point Unloading Analysis

Due to the simplicity and high accuracy of this analysis technique, single point unloading analysis is employed by the majority of instrumented indentation system. In order to determine the slope of elastic recovery, Equation (15) is now taking derivative with respect to the height, seen in the Figure 11, where $\frac{dh}{dP}$ represents the slope of elastic unloading and h_e is the elastic recovery displacement [15].

$$\frac{dP}{dh} = 2E^* R^{1/2} h_e^{1/2} \quad (15)$$

For an elastic displacement caused by a spherical indenter, the formula is given by Equation (16) where h_e is the length of elastic recovery, and it is equal to the radius of the contact circle a squared divided by the radius of the indenter R [3].

$$h_e = \frac{a^2}{R} \quad (16)$$

Substituting Equation (20) back to Equation (19), Equation (17) is formed; this equation shows the slope of unloading is a function of combined modulus E^* and the radius of contact circle.

$$\frac{dP}{dh} = 2E^* a \quad (17)$$

Thus, rearranging Equation (21) would lead the result of Equation (18), which is used to determine the combined modulus or reduce modulus between the test specimen and the indenter. Where E^* is the combined modulus and A is the contact area.

$$E^* = \frac{1}{2} \frac{dP}{dh} \frac{\sqrt{\pi}}{\sqrt{A}} \quad (18)$$

Lastly, with known parameters, the elastic modulus would be obtained by Equation (11), where E' , ν' and ν are elastic modulus of the indenter and the indenter's Poisson's ratio, and the Poisson's ratio of the sample, respectively.

4.4 Multiple Partial Loading/Unloading Indentation Analysis

In order to determine the indentation depth, high precision depth sensors are usually required, thus the contact area can be calculated base on the known geometry of the indenter, and otherwise optical measuring techniques would be applied. To simplify the indentation test, typical indentation techniques require the measurements of indentation depth, contact area and contact load; nevertheless load-based multiple partial loading/unloading method which was developed at WVU [16] [17] is capable of determining elastic modulus of a specimen with the measurements of overall indentation depth and contact load.

Figure 9: Multiple Unloading Indentation Test

As shown in Figure 9, a schematic load-displacement curve in which multiple partial unloading during a single indentation test was introduced.

Due to the system compliance, a portion of the overall displacement which is the total indentation depth causes the system to deflect, and the rest of the displacement would induce permanent impression onto the specimen's surface [16] [17]. Seen in the Equation (19), where h_t is the indentation depth, h_s is the system deflection, and the sum of both is the result of the total deflection, h_T , caused by the indentation applied load.

$$h_T = h_t + h_s \quad (19)$$

Taking derivative on both sides with respect to the indentation load in Equation (19) leads to Equation (20). Where $\frac{dh_T}{dP}$ can be easily determine by unloading curve, $\frac{dh_s}{dP}$ is the system compliance, which is also assumed to be a constant value within a load range, and $\frac{dh_t}{dP}$ is the compliance slope of the specimen.

$$\frac{dh_T}{dP} = \frac{dh_t}{dP} + \frac{dh_s}{dP} \quad (20)$$

Solving Equation (20) for the indentation depth and deriving with respect to the applied load, $\frac{dh_t}{dP}$ is found, seen in Equation (21), where the constants R and E^* are the indenter radius and reduced modulus, respectively.

$$\frac{dh_t}{dP} = \left[6R(E^*)^2 \right]^{-1/3} P^{-1/3} \quad (21)$$

By substitution, a relationship between the total and system deflections with respect to the load, radius, and reduced modulus are created, seen in Equation (22), where P and $\frac{dh_s}{dP}$ are the maximum load and inverse slope corresponding to each individual unloading.

$$\frac{dh_T}{dP} = \left[6R(E^*)^2 \right]^{-1/3} P^{-1/3} + \frac{dh_s}{dP} \quad (22)$$

That P and $\frac{dh_T}{dP}$ represent the maximum contact load and inversed slope corresponding to each unloading point. Due to the fact of linear relationship found between $\frac{dh_T}{dP}$ and $P^{-1/3}$ in the governing equation of multiple unloading method, fundamental algebra equation $y=mx+b$ could be applied, furthermore, the slope m is rearranged in order to find the reduce modulus E^* , shown in the Equation (23) [18].

$$E^* = \sqrt{\frac{1}{6R(m)^3}} \quad (23)$$

For rigid indentation system, the system compliance can be assumed to be constant when the system is subjected to a certain load range; however, for flexible indentation system, the system compliance continuously changes upon the contact load.

Chapter 5: Load-based Micro-indentation System-Components and Construction Considerations

Due to the fact of the system being sensitive, in order to extract the correct hardness value and elastic modulus from the testing specimen, the testing environment is recommended to be vibration free and its temperature cannot be changed dramatically, the thermal expansion effect may affect the testing accuracy. The electronic equipments should be wired correctly and wires should have enough space to each other so that electronic currents would not affect each other, by doing so, the electronic error and noise could be reduced. System errors or electronic noise could also be introduced by using equipments that are not compatible to the others. For instance, an indentation load of 50 Newton, using a 1000 pound load cell would not be sensitive enough which would cause losing information of load response, yet if a 15 pound load cell were applied to the system; it would pick up a lot of noise due to its high sensitivity.

5.1 Instrument Construction

Despite the testing scale of the indentation test or the loading frame design, a typical instrumented indentation system contents with an actuator, load cell and indenter, which shown in Figure 10.

Figure 10: Indentation Chain Components

Loading frame is required to create the boundary, the platform for the indentation test, the actuator that is used to provide displacement of the indenter into the testing specimen.

Alike the actuator, the load cell is monitored throughout the entire test, and the acquired data is used for load response, along with the known displacement then elastic modulus could be calculated; Detailed information of indentation chain components could be found in Appendix A.

5.1.1 Actuator

PZT is the short term for piezoelectricity, originated from Greek piezo or piezein, which means to squeeze or to press. The piezoelectric effect is a phenomenon of expansion and contraction of certain material when electric field changes. Inside the PZT actuators, there are stacks of disks separated by thin metallic electrodes [3]; those high stiffness disks not only provide desired stability while testing, but also exhibit a linear electromechanical interaction between applied electrical field and its displacement; Therefore, PZT actuators are the most common used for instrumented indentation applications for its high displacement resolution, low compliance, and the ability of changing the displacement not only reversibly but also linearly when electric field or voltage applied.

5.1.2 Load cell

Just like other applications, load cell is used as a load transducer in the instrumented indentation system, which converts contact force into electrical signals. Although there are other different types of load cells that are available, such as hydraulic or even piezoelectric load cells, strain gauge load cell yet tend to be the most popular ones due to the reason that such load cell is the most accessible and least cost compare to other different kinds of load cells. A strain gauge load cell usually consists of four strain gauges that form a configuration of Wheatstone bridge; the contact force will be sensed by measuring the changing in electrical resistance within strain gauges when the load cell is being contacted or compressed [3]. Lastly, the output of the load cell signals are typically in the order of few mili-volts, hence, an amplifier is a requirement to acquire the signal.

5.1.3 Indenter

Before an indentation test even started, it is very important to choose the right indenter. In order to decide which indenter tip is going to be used throughout the test, not only the material but also the geometry of the indenter must be taken as consideration. There are several different materials that can be made into indenters; however, the material must need to have a high elastic modulus. Among those high elastic materials, sapphire or tungsten indenters are most common used; diamond indenters are also available yet due to the reason that diamond is brittle which can be easily crack or chipped, and also hard to be manufactured, it is still desired to use a tungsten indenter [3]. There are two main categories of indenter geometries, which are sharp and blunt or spherical indenters. Sharp indenters are very popular for thin film analysis; even though elastic would occur once the contact is made, yet the state would transit to plastic deformation immediately. Unlike sharp indenters, as the spherical indenter is driven into the sample's surface, a transition would first occurred from elastic until indenter reaches deeper indentation depth, plastic deformation occurs, and the elastic state last longer when spherical indenter is employed in the system. Despite the material or the geometry of the indenter, mounting the indenter onto the system is important as well; the indenter has to be firmly attached to the indentation shaft, and have a perfect alignment along the direction of indentation, so that the system compliance can be minimized.

5.1.4 Loading Frame

Loading frame is the most important part in the entire indentation system; it provides a stabilized platform for indentation testing, which minimized the effect of system compliance. Therefore, the majority of indentation loading frames were heavily constructed in symmetric geometries [19]. Once the decision of making a flexible indentation system has been made, the research will run into a give or take dilemma. In order to reach any degree of freedom, the arm-linked system is adjustable for sample of different sizes, geometries or even the sample been placed at different locations. It could be a quite achievement yet higher mobility leads to lower rigidity of the system, the principle could apply reversibly, higher rigidity would cause lower mobility. However, there's a balance point that with minimum rigidity of the system, it could

yet acquire correct load and unloading data, then with or without system calibration to get elastic modulus of the testing sample.

5.2 Specimen Mounting

It is ideal to place the testing specimen, and adjust the sample's surface to be parallel to the loading plane of the indenter, in the other words; the specimen shall be placed in the normal direction that's aligned with the indentation axis. In the case of miss alignment, it is possible to get incorrect results due to the load is applied in an angle. The data would not reflect the perpendicular reaction of the sample, and other issues, such as friction, would be introduced and affects the result as well [19]. Typically, the specimen is mounted on a large, vibration-free base. Specimen base or sample holder is also desired to be rigid, which the indentation load could also cause deflection of the holder; in the other words, despite the testing angles, the sample must be mounted on a rigid sample holder. Clamps and magnets could be applied to secure the specimen onto the sample holder, for the reason of reducing system compliance.

Tightening the clamps with an exclusive amount of force would result in sample deformation thus induce residual stress into the specimen thus affecting the elastic modulus; this thought process is found to be untrue for micro scaled indentation tests. In the recent study by Oliver and Pharr, the affects of residual stress caused by clamping is found to be minor, therefore could be neglected.

5.3 Working Distance

An instrumented indentation test has a limited travel range of displacement over which the indentation depth may be measured. Therefore, it is necessary to ensure the actuator after full expansion, the indenter could be driven, and create enough penetration into the specimen. Rather than use actuator to bring down the indenter to the sample, some adjustment must be made in order to drive the indentation chain closer to the surface of the sample. Once the working distance has been defined, the actuator will expand to make the initial contact between the indenter and the surface of the specimen, with a minimum amount of force. Even

though the contact force is minimal, yet a shallow indentation would have been made, which is essentially the reference position and the initial contact load and depth must be accounted in the data analysis [19].

5.4 Mismatch of PZT actuator and PI Controller

Due to the fact that each controller is built for each one spectacular PZT actuator, it's better to purchase them as a package from the manufacture. However, using an actuator which did not built for a particular PI controller would have some miner affects on the response, the slope of $\frac{dh}{dP}$ vs. $P^{-1/3}$ may not be correct but the trend line shall still be linear; therefore, it could be calibrated by applying a correction factor, which is the theoretical slope that divides the experimental slope. While load cell is collecting correct load response from the system, the displacement of the PZT actuator is incorrect, in which the problem arises. As an example, if the reading of strain gauge, transducer in the PI controller was from zero to 10 volt, which is correspond to the displacement of zero micrometer and the maximum displacement of the actuator.

5.5 Factors Effecting Flexible Indentation System

Although Instrument indentation is simple in theory, there are yet various of errors may happened during the test that are associated with the system, the environment or even material related problems. There errors are unavoidable during the indentation test, yet they could be minimized. In the following, these errors and alternative correcting methods will be discussed.

5.5.1 Drift

There are two common drift behaviors that can be found during an indentation tests, creep and thermal drift. Start with creep, such a behavior describes the tendency of a solid material to slowly move or deform permanently under the influence of stress. Unlike cracking or fracture, creep deformation does not occur suddenly upon the applied load, but such

deformation is time dependent; creep effects would be even more severe when the material is exposing in high temperature environment [21].

Not only the creep effect, but also thermal expansion of the testing material would also affect and induce error to result that the indentation depth would be altered due to the dimension of the specimen is changing when the environmental temperature changes; however, thermal and creep effects are highly dependent on the material types; ductile materials tend to creep yet brittle materials would be cracked and fractured before creep even occurred.

In order to minimize these effects, a temperature stabled environment must be established, room temperature is more desired. In this research aluminum, bronze, and stainless steel would be analyzed under room temperature, those materials would not be affected by creep in room temperature condition; however, there are yet several material that would be affected even when the temperature is low, such as solders and lead. To combat this problem, adding a step of withhold, staying at the maximum contact load for roughly thirty seconds, which again, the withholding time can be varied with different test material. The withhold time would be used to settle down the material, allowing the material to reach its equilibrium state.

5.5.2 Initial Penetration Depth

Ideally, the indentation depth is supposed to be measured from the free surface of the sample. However, in practice, an initial contact between the indenter and the sample must be first established; using the initial contact as the reference, then load and indentation depth measurement could start. Even though the initial contact could be made by a very small load, yet it would still cause some kind of penetration in the specimen; such information acquired from the initial contact is undesirable and should be neglected that it does not reflect the true material response. Surface roughness of the sample, for instance, when the initial contact is established, a very shallow indentation is made as well. As it's shown in the left hand side of Figure 11, it is hard to analyze the contact area or depth of the shallow indentation; the data

collected from shallow indentation depth is dominant by surface effect and it does not reflect the true material response. Displayed in the right hand side of Figure 11, as the indenter driven further into the specimen, surface effect is no longer governing the data, thus sufficient amount of indentation depth would extract much more accurate material response than shallow indentation [20].

Figure 11: Schematic Drawing of Surface Effect and Shallow Indentation

To overcome this dilemma, multiple partial unloading techniques are applied; when it comes to data analysis, simply remove the first few unloading points, which would cause non-linearity of the slope. By doing so, true stiffness response of the material is ensured.

5.5.3 Instrument Compliance

Unfortunately the displacement of the PZT actuator does not fully reflect the true indentation depth that would be made onto the specimen. Even though PZT actuator presents the true profile of total displace, yet the totally displacement is combined by the length of the indentation made on the specimen and the deflection length of the system due to the contact load.

Compliance is a term that could be used in various kinds of subjects. As of it in the indentation testing, system compliance is referring to the tendency of the system to move or deflect upon the contact load [3]. Referred to the deflection, it is acting in a proportional manner to the load applied; therefore, a larger applied load will result in a large deflection, a smaller load would produce a smaller deflection. The angle of indentation is also a concern; however the effect of angled indentation had been studied by an Australian group, which showed there was no significant change in elastic modulus until the indenter tilted more than 30 degree [21].

The deflection angle of these soft systems are functions of the indentation loads, which constantly change upon the contact force; shown in Figure 12, the problems and errors arise when indentation testing is performed by a system with large system compliance and asymmetric deflection.

Figure 12: Lateral Indentation

Due to the majority of movement that would be used to deflect the system, efficiency of penetrating test sample would reduce significantly, even more severely when bi-directional deflection or multi-directional deflection are induced in the system, which would result in shallow indentation, providing a response combined with system and the sample surface; And yet it has no efficient method to distinct surface response from scattered data. As the result, indentation test shall be performed under a load frame with sufficient amount of restrictions; in order to minimize system compliance, shaft collars and clamps could also be applied.

Deflection is not only exhibit within the flexible indentation system but also the sample holder; therefore, take derivative with respect to the contact load, would lead the conclusion

that system compliance is the combination of compliance from the flexible indentation system and the sample holder compliance. It is possible to find the correlation between structure simulation results and system behaviors of the flexible indentation system; however, sample holders are not fixed models for structure simulation, and compliances of sample holders varies by their geometry; it would be time consuming and prolonging work to determine overall system compliance by simulation.

5.5.5 Indentation Size Effect

Indentation size effect is also known as ISE, such phenomena often observed on testing homogeneous materials; theoretically, material's mechanical response of a homogeneous material shall not vary as the indentation size and depth changes; however, the discovery of Indentation size effect states the different. As an example, hardness tends to increase as the indentation size decreases.

Indentation size effect is actually a combination of several artifact issues. A thin oxide layer on the top of the specimen would result in two sets of responses, the stiffness response of the oxide layer and the substrate; surface roughness of the sample could also induce error that at small contact load or the earlier stage of the indentation test, the contact area cannot be calculated correctly by the indenter geometry. Furthermore, residual stress could also be built up at the surface of the specimen done by the polishing or even manufacturing. As the indenter is driven deeper into the sample, higher load may cause cracking in the sample, which would not reflect the true stiffness response from the sample due to the reason that cracking releases stress and the contact load would reduce instantly as the cracking occurred.

Chapter 6: Flexible Micro-Indentation System Development

After decades of development, instrumented indentation testing had been not only a reliable method to determine material hardness and elastic modulus, but also a cost effective tool which does not require high priced depth sensing equipments nor optical measuring techniques. Despite the benefit that provided by conventional indentation systems, there are still limitations of those systems. Therefore, the objective of the thesis is to expand the research from tabletop testing units to a flexible indentation system therefore indentation testing will no longer be restricted by the size or the geometry of the test specimen.

Although the loading frame design is structurally simplistic yet it does require considerable experimental experience, CAD drawing or even FEA structural analysis techniques. In order to acquire hardness or elastic modulus from a sample of any size and shape, the flexible indentation system must first be adjustable. The adjustments need to be performed quickly, effectively, precisely but most importantly; the entire system is secured after locking all the joints and collars. Lastly, eliminate environmental related issues, such as dramatic temperature changing and vibrations, which would affect the test result critically.

6.1 System One Development

The first system, seen in Figure 13, was a combination of a flexible indentation arm and a tabletop loading frame. The loading frame is composed of two 1.5 inches in diameter stainless steel rods and four stainless steel plates. The top and bottom plates were placed to provide stable boundary condition while those two stainless steel plates in the center of the frame are used to adjust the height and the angle of the indentation arm holder; the arm holder is also constructed with stainless steel with bronze bearing, thus with threaded mechanism, the indentation shaft could reach desired location smoothly; Similarly, the height of the entire system could be adjusted with the thread mechanism which located at the top plate. Collars were welded onto the plates that after the indenter is placed at the desired location, locking all the collars would provide higher stability for the system.

Figure 13: General View of System One

The indentation shaft is consisted with an one inch in diameter stainless steel rod and the indentation chain; the load cell used in system one has the capacity of withstanding and acquiring loads up to 100 pounds; the PZT actuator has high accuracy which is capable of nano scale positioning, and at the end of the indentation chain, a tungsten carbide indenter is attached. Detailed drawings and dimensions could be found in Appendix A.

6.1.1 Results and Discussion

At the time, a larger loading frame would benefit the indentation system to have smaller system compliance, thus producing not only consisting but also accurate indentation results; unfortunately, this thought process is found to be incorrect. The size of the system showed no direct correlation to the accuracy of indentation tests, furthermore, due to the scale of the indentation system and its weight, the system is difficult to operate.

Multiple partial unloading technique is a self calibrated system that despite the magnitude of the system compliance, as long as the compliance is behaving in a linear manner in terms of direction and magnitude, such method would acquire the correct hardness and elastic modulus. With an asymmetry design, System One suffered from bending which induces multi-directional deflection to the system. Therefore, not all but a portion of PZT actuator movement is used to indent the test specimen. The direction of indentation would continuously change upon the indentation load; result in scattered data points and inaccurate unloading

slope. Seen in the Figure 14, indentation testing involved with bending, which would cause dragging and shallow indentation, extracts untrue information that combined with indentation and friction.

Figure 14: Pro Mechanica Structure Simulation and Optical microscope Image

Unluckily, there is no efficient method to distinct elastic response from scattered data, but increase rigidity of the system would help reducing data scattering; with consisting and linear data points, correction factor could be applied by using a calibration method. With enough rigidity sometimes the system even does not need calibration. The elastic modulus results obtained using System One at the setup shown in Figure 15, could be found in

Table 1, in which displays a reliable and correct result without correction factor.

Figure 15: System One Setup at Zero Degree and 7.5 Inches in Height

Table 1: System Two Elastic Modulus Results

Test	Aluminum	Bronze	Steel
Number	Modulus (GPa)	Modulus (GPa)	Modulus (GPa)
One	85.43	148.24	230.61
Two	67.71	127.02	183.34
Three	76.81	126.55	199.46
Four	67.87	110.40	205.83
Five	76.21	124.02	228.68
Six	69.22	116.70	211.14
Seven	68.82	117.11	224.65
Eight	69.99	126.93	229.50
Nine	78.35	128.97	204.70
Ten	74.55	141.01	203.15
Average Value	73.50	126.70	212.11
Standard Deviation (Percentage)	7.89	8.88	7.44

The elastic modulus for those testing materials, Aluminum which is ranged from 68 to 71 GPa, Bronze is from 103 to 124 GPa, and Steel is ranged from 190 to 210 GPa [22] and the error percentage of each result was calculated with the mean value of these materials, thus Aluminum is assumed to be 70 GPa, Bronze is 113 GPa and Steel is set to be 200 GPa ; The ten test results were deviate from the mean value of the elastic modulus for each material, yet most of the results fell within the range of known values. Found in Table 2, the differences in percentage associate with each indentation tests are shown. Although there is no true value for elastic modulus of a material but a range of modulus value is expectable for each material, therefore, the comparison was made by the mean value of each material's elastic modules range.

Table 2: System One Elastic Modulus Percent Difference

Test	Aluminum	Bronze	Steel
Number	Percent Difference	Percent Difference	Percent Difference
One	+22.92	+30.61	+15.30
Two	-2.58	+11.91	-8.33
Three	+10.52	+11.50	-0.27
Four	+9.65	-2.73	+2.92
Five	-0.40	+9.27	+14.34
Six	-0.98	+2.82	+5.57
Seven	+0.71	+3.18	+12.33
Eight	+12.73	+11.83	+14.75
Nine	+7.27	+13.63	+2.23
Ten	+5.76	+24.24	+1.58

Surface conditions of the sample may cause fluctuation of test result; performing indentation on spots, in which close to the edges or cracks could alter the result as well as thin layers of oxidation or rough testing surfaces. Hence, surface polishing prior to indentation testing would benefit the system to have consistent data, thus more accurate result.

Derivation of total PZT displacement with respect to the contact load is known as the total compliance which is a combination of system compliance and the specimen compliance; and it's assumed to remain constant throughout the indentation process within a proper loading range as it is a function of the testing conditions as well as the specimen.

Within the ten tests of each material, the first test seems to have a much higher modulus than its book value, and the system compliance of the first test is also distinct from other tests; such phenomena could be explained that the system was not fully settled after locking all the joints and collars; the contact load caused minor popping thus small movement in the system resulted in larger system compliance.

System One was rearranged to the setup shown in Figure 16; unlike the first setup, at this time, all the parts within the system are subjected to torque; the values of those unloading

slope at this setup was incorrect, however, the unlading data points were considered to be linear and consistent slopes, therefore a correction factor was applied for this particular setup.

Figure 16: System One at 90 Degree with 10 Inches in Height

The elastic modulus results obtained using System One at the setup shown in Figure 16, could be found in Table 3, and material compliances is displayed in

Table 4.

Table 3: System Two Elastic Modulus Percent Difference

Test	Aluminum	Bronze	Steel
Number	Modulus (GPa)	Modulus (GPa)	Modulus (GPa)
One	29.87	37.84	50.92
Two	30.21	36.46	48.47
Three	30.57	35.00	52.88

Table 4: Experimental Compliances of Materials

Test	Aluminum	Bronze	Steel
Number	Compliance	Compliance	Compliance
One	0.6149	0.5328	0.4473
Two	0.6106	0.5448	0.4603
Three	0.6062	0.5583	0.4377

Due to the fact of having high system compliance at this setup, the slope of $\frac{dh}{dP}$ Vs. $P^{-1/3}$ may not be correct which leads incorrect elastic modulus.

In order to establish the calibration algorism, the assumptions had been made that deflections in X, Y and Z direction are proportional to the expanded length of PZT actuator and

follows Pythagoras Theorem which shown in Equation (23), where h is the total Deflection and X_d, Y_d, Z_d are the magnitude of deflection in X, Y, Z directions respectively.

$$h = \sqrt{X_d^2 + Y_d^2 + Z_d^2} \quad (24)$$

To verify the assumption, Pro Mechanical structural analysis was also used, shown in Figure 15; at this particular setup, the system was subjected to 10 lbf of force at the end of the shaft, with an increment of 10 lbf each time to 50 lbf;

Figure 17: Pro Mechanical Structure Analysis

the results are displayed in Figure 15 and Table 3; and the structure study illustrates not only the total deflection exhibits linear behavior upon applied load, but also the deflection in Z direction which is normal to the surface of the specimen.

Figure 18: Plots of Applied Force and Deflections

Table 5: System Two Elastic Modulus Percent Difference

Applied Load(lbm/s2)	Total Deflection (micro-Inch)	Deflection in X(micro-Inch)	Deflection in Y(micro-Inch)	Deflection in Z(micro-Inch)
10	7.128	6.79	1.668	1.373
20	14.26	13.58	3.336	2.746
30	21.38	20.37	5.003	4.12

40	28.51	27.16	6.671	5.492
50	35.64	33.95	8.339	6.865

thus, by using standard linear regression between theoretical material compliances and experimental material, the correction factor could be determined. With known compliance values of Aluminum, Bronze and Steel, the correction factor C is defined as the slope of theoretical compliance values of these three materials versus the experimental materials' compliances. Plotting the experimental results into the equation shown in Figure 19, the corrected material compliances were conducted [23].

Figure 19: Theoretical Material Compliances Value Versus Experimental Values

Found in Table 6, in which the elastic modules were calibrated with the correction factor.

Table 6: System Two Elastic Modulus Percent Difference

Test	Aluminum		Bronze		Steel	
	Modulus (GPa)	Error %	Modulus (GPa)	Error %	Modulus (GPa)	Error %
One	72.58	3.69	113.39	0.35	233.41	16.71
Two	74.05	5.79	101.94	-9.79	201.92	0.96
Three	75.61	8.01	108.84	-3.68	258.25	29.13

Lastly, System One was rearranged again to the setup shown in Figure 20. The stability of the system at this setup is considered to be poor; the two stainless steel plates which suppose to serve the purpose of providing ridge boundary to the indentation arm holder were not working as expected.

Figure 20: System One at 90 Degree

The crucial bending moment which conducted by the arm length and indentation load pushed these steel plates, thus created undesirable movements and led to data scattering. In order to combat this situation, external forces were required to stabilize the system; hence two C-clamps were applied to restrict the movement of the plates, and then locked all the shaft collars, prior to the indentation tests. The experimental and calibrated material compliances are found in Table 7; in which the calibration equation is defined in Figure 21 and the calibrated elastic modulus are shown in Table 8.

Table 7: Experimental Results and Calibrated Results with the Correction Factor

Test	Aluminum		Bronze		Steel	
Number	Experimental	Calibrated	Experimental	Calibrated	Experimental	Calibrated
One	0.4803	0.370771	0.393	0.305942	0.2826	0.223959
Two	0.4699	0.363048	0.3683	0.2876	0.2757	0.218835
Three	0.4762	0.367726	0.3676	0.28708	0.2707	0.215122
Four	0.4977	0.383692	0.3537	0.276758	0.2557	0.203983
Five	0.4723	0.36483	0.3597	0.281213	0.2823	0.223736

Figure 21: Plot of Theoretical Material Compliances Versus Experimental Material Compliances

Table 8: Elastic Modulus of Testing Materials after Calibration

Test	Aluminum		Bronze		Steel	
Number	Modulus (GPa)	Error %	Modulus (GPa)	Error %	Modulus (GPa)	Error %
One	71.00	1.43	101.97	-9.76	199.11	-0.45
Two	73.78	5.40	115.31	2.04	210.59	5.30
Three	72.08	2.97	115.73	2.42	219.69	9.85
Four	66.73	-4.67	124.71	10.36	251.60	25.80
Five	73.12	4.46	120.69	6.81	199.59	-0.21

The value of system compliance provides the means of system movement during indentation testing. Theoretically, multiple unloading technique would work properly despite the appearance of large system compliance, yet the deflection or the movement must be normal to the indentation surface. Unfortunately, the direction of indentation force changes continuously throughout the entire indentation test when it was performed on a flexible system, and result in dragging and shallow indentation depth; data extracted from flexible system is incorrect that recorded displacement of the PZT actuator does not reflect the true indentation depth. Even though the calibration algorithm can be used to cope with the situation of continuously changing in indentation direction due to the contact load, yet such method required high linearity and consistency of the compliance slope. Results shown in the previous, the calibration method seems to worked well for Aluminum and Bronze but steel, furthermore, the data points gathered from steel specimen had lower Coefficient of determination value, that indicates System One was not structurally stable, and it did not work well as expected.

6.2 System Two Development

Base on the design principles of the first system, the structure of the second system, shown in Figure 22, is a lot smaller than System One; conspicuously, the major difference between System and previous system is that System Two uses one 1.5 inches stainless steel shaft as its support, which differ from System One that used two shafts and a flanged shaft collar is applied, which forms the bonding between the shaft and the base plate thus provides much more stable platform for indentation testing .

Figure 22: General view of System Two

The reduction in scale provides the benefits of low cost and since the weight of the system dropped significantly, System Two is easier to operate as well. The indentation arm holder is now directly locked on the base-shaft instead of using two steel plates to compress and restrict its movements, such minor change seems to be negligible yet this new holder design reduces significant amount of system compliance. Prior to the indentation test, arm holder is first place to certain height which depends on the eight of the testing sample as well, followed with locking all the shaft collars on the arm holder; after the course adjustment had been made, the indenter is placed at the proper working distance by turning the loading thread, then lock the half inch collars to fix the movement of indentation chain. Dislike the loading thread mechanism used in System One which was attached to the system, the load thread is build-in into the system, which increases the mobility of the indenter arm, and it's served as a

position regulator that adjusts the distance between indentation chain and test specimens fast and effectively. The load cell used in system one has the capacity of withstanding and acquire loads up to 50 pounds, which has higher sensitivity compare to the one used in System One; the PZT actuator has high accuracy which is capable of nano scale positioning, and at the end of the indentation chain, a tungsten carbide indenter is attached. Found in Appendix B, the dimensions of these parts using in System Two are found to be much smaller than ones used in System One.

In order to perform indentation testing on various angles and height, two sample holders were made; shown in Figure 23A and Figure 23B.

Figure 23A: Sample Holder for Testing 90 Degree and 45 Degree Angles.

Figure 23B: Sample Holder for Testing 30 and 60 Degree Angles.

Both sample holders were heavily constructed with steel; all parts within these two sample holders were welded onto each other, thus created ridged and stable platform for indentation testing in angles. The detailed dimensions of those sample holders could be found in Appendix C and D.

6.2.1 Results and Discussion

Even though the design principles were similar to that of the first system, System Two was machined with care and higher precision, thus produces far more reliable result in terms of linearity and consistency. The size reduction benefits the system to be user friendly that with lighter setup, it allows easier and faster adjustment prior to indentation testing. The system had

been tested in several of angles, and the results produced from zero degree setup with 4.5 inches in height from indenter tip to the bottom which can be seen in Figure 24, are found in Table 9.

Figure 24: System Two at Zero Degree with Height of 4.5 Inches

Table 9: Experimental and Calibrated Material Compliances

Test	Aluminum		Bronze		Steel	
Number	Experimental	Calibrated	Experimental	Calibrated	Experimental	Calibrated
One	0.1726	0.371044	0.1439	0.297873	0.1082	0.206856
Two	0.1696	0.363395	0.1456	0.302207	0.1165	0.228017
Three	0.1727	0.371299	0.143	0.295579	0.1136	0.220623
Four	0.1754	0.378182	0.1483	0.309091	0.1121	0.216799
Five	0.1665	0.355492	0.1448	0.300168	0.1087	0.208131

Once again, the material compliances were not correct, due to the fact of mismatching of PZT actuator, PI controller and system bending. However, those factors cause untrue material compliances exhibit linear relationship to the correct material compliance. Therefore, the previous calibration algorithm would work on the condition that the system is getting linear response from the specimen, despite of using different controllers, different load cells and even different PZT actuators. Displayed in Table 10, in which the corrected modulus of testing materials are found.

Table 10: Elastic Modulus that Calibrated with Correction Factor

Test Number	Aluminum Modulus (GPa)	Error %	Bronze Modulus (GPa)	Error %	Steel Modulus (GPa)	Error %
One	69.43	-0.81	105.26	-6.85	242.63	21.32
Two	72.12	3.03	102.29	-9.48	190.80	-4.60
Three	69.35	-0.93	106.89	-5.41	206.46	3.23
Four	67.08	-4.17	97.88	-13.38	215.48	7.74
Five	75.10	7.29	103.67	-8.26	238.82	19.41

The samples were later on placed at 8.5 and 10.5 inches tested by the flexible indentation system at zero degree angles. The setups and tests results are shown in the following figures and tables, as seen in Figure 25, the system was placed at the height of 8.5 inches and the results are displayed in Table 11 and Table 12.

Figure 25: System Two at Zero Degree with Height of 8.5 Inches**Table 11: Experimental and Calibrated Material Compliances**

Test Number	Aluminum		Bronze		Steel	
	Experimental	Calibrated	Experimental	Calibrated	Experimental	Calibrated
One	0.1695	0.379743	0.14	0.301904	0.1155	0.237258
Two	0.1627	0.3618	0.1354	0.289766	0.1055	0.210872
Three	0.16	0.354676	0.1364	0.292405	0.1143	0.234092
Four	0.1711	0.383964	0.1382	0.297155	0.1034	0.205331
Five	0.1649	0.367605	0.1396	0.300849	0.1044	0.20797

With the calibrated material compliances, the corrected elastic modules of these materials are found in Table 12, which shows high consistency at this particular setup.

Table 12: Elastic Modulus of Testing Materials after Calibration

Test	Aluminum		Bronze		Steel	
Number	Modulus (GPa)	Error %	Modulus (GPa)	Error %	Modulus (GPa)	Error %
One	66.58	-4.89	102.50	-9.29	174.01	-13.00
Two	72.71	3.87	111.22	-1.58	230.96	15.48
Three	75.42	7.74	109.21	-3.35	179.45	-10.28
Four	65.26	-6.77	105.76	-6.41	247.35	23.68
Five	70.62	0.89	103.21	-8.66	239.29	19.65

After running indentation tests at height of 8.5 inches, the system was moved again to 10.5 inches, seen in Figure 26.

Figure 26: System Two at Zero Degree with Height of 10.5 Inches

Rearranging the system leads the changing of boundary condition, thus the calibration equation that was obtained from previous setup is no longer valid; a new calibration equation is expected.

Table 13: Experimental and Calibrated Materials' Compliances

Test	Aluminum		Bronze		Steel	
Number	Experimental	Calibrated	Experimental	Calibrated	Experimental	Calibrated
One	0.1815	0.378321	0.1481	0.297513	0.1125	0.211383
Two	0.1766	0.366466	0.149	0.299691	0.122	0.234367
Three	0.1755	0.363805	0.1417	0.282029	0.1105	0.206544
Four	0.1768	0.36695	0.1566	0.318078	0.1194	0.228076
Five	0.1691	0.348321	0.1562	0.31711	0.1175	0.223480

Shown in the Table 13, the results indicate the system remains high consistency despite of changing the height from 4.5 inches to 8.5 inches; and in Table 14 is where the calibrated results are shown.

Table 14: Elastic Modulus of Testing Materials after Calibration

Test	Aluminum		Bronze		Steel	
Number	Modulus (GPa)	Error %	Modulus (GPa)	Error %	Modulus (GPa)	Error %
One	67.03	-4.24	105.51	-6.63	229.55	14.78
Two	71.02	1.46	104.00	-7.96	178.97	-10.52
Three	71.97	2.81	117.48	3.96	243.59	21.80
Four	70.85	1.21	92.59	-18.06	190.68	-4.66
Five	77.99	11.41	93.14	-17.58	200.14	0.07

As the height of the indentation unit increases, the system compliance increases, and the Coefficient of determination drops, thus the deflection of the system is the key factor that affects the linearity and consistency of indentation testing; this though process found to be correct by Pro-Mechanica simulation and proved experimentally. Seen in Figure 28 and Table 13, the deflection of the indentation units exhibits linear relationship upon arm length and indentation load. However, it is impractical to determine the correlation between Pro-Mechanica simulation and experimental results that system setup may remain the same, yet the system compliance could vary upon different sample holder, and even different sample mounting methods.

The system is later subjected to 30 degree angle to evaluate Elastic modulus of Aluminum, Bronze and steel, with the setup shown in Figure 27. The elastic modulus for those testing materials, Aluminum which is ranged from 68 to 71 GPa, Bronze is from 103 to 124 GPa, and Steel is ranged from 190 to 210 GPa [22]; with the calibration method, the result displayed in Table 15 and Table 16 showed high consistency across three materials.

Figure 27: System Two at 30 Degree Setup

Table 15: Experimental and Calibrated Materials' Compliances

Test	Aluminum		Bronze		Steel	
Number	Experimental	Calibrated	Experimental	Calibrated	Experimental	Calibrated
One	0.2002	0.373937	0.1478	0.285721	0.1058	0.215014
Two	0.2017	0.376462	0.1448	0.280671	0.1124	0.226125
Three	0.1926	0.361142	0.1472	0.284711	0.1096	0.221412
Four	0.1952	0.365519	0.1453	0.281513	0.1	0.20525
Five	0.2069	0.385216	0.1481	0.286226	0.112	0.225452

Table 16: Elastic Modulus of Materials after Calibration

Test	Aluminum		Bronze		Steel	
Number	Modulus (GPa)	Error %	Modulus (GPa)	Error %	Modulus (GPa)	Error %
One	69.91	-0.13	116.85	3.41	219.94	9.97
Two	69.07	-1.32	121.16	7.22	194.60	-2.70
Three	74.50	6.43	117.69	4.15	204.68	2.34
Four	72.87	4.10	120.42	6.57	247.6	23.80
Five	66.26	-5.34	116.43	3.04	195.98	-2.01

In order to determine the liability of the indentation system, the system moved again to the new setup shown in Figure 28. Aluminum, Bronze and Steel were again evaluated at 45 degree angle.

Figure 28: System Two at 45 Degree Setup

The experimental compliances and calibrated compliance can be found in Table 17, and the calibrated elastic modules of these materials are displayed in

Table 18.

Table 17: Experimental and Calibrated Materials' Compliances

Test	Aluminum		Bronze		Steel	
Number	Experimental	Calibrated	Experimental	Calibrated	Experimental	Calibrated
One	0.2347	0.37697	0.1686	0.272829	0.1315	0.214378
Two	0.2354	0.378073	0.1736	0.280707	0.1365	0.222256
Three	0.2379	0.382011	0.1689	0.273302	0.1359	0.22131
Four	0.234	0.375867	0.1686	0.272829	0.1376	0.223989
Five	0.226	0.363263	0.1784	0.288269	0.1409	0.229188

Table 18: Elastic Modulus of Testing Materials after Calibration

Test	Aluminum		Bronze		Steel	
Number	Modulus (GPa)	Error %	Modulus (GPa)	Error %	Modulus (GPa)	Error %
One	67.47	-3.61	125.78	11.31	221.57	10.79
Two	67.11	-4.13	118.62	4.97	202.80	1.40
Three	65.87	-5.90	125.33	10.91	204.90	2.45
Four	67.82	-3.11	125.78	11.31	199.05	-0.48
Five	72.17	3.10	112.38	-0.55	188.51	-5.75

The results of aluminum and bronze fell in the theoretical modulus range, results of steel in the other hand, were not as stable as the other two materials. At this particular setup, the range that bounded by maximum and minimum of steel can be easily distinguished from

the range of bronze and aluminum, thus even though the results were not as accurate yet it is acceptable.

According to previous results at different angles and setups, the system cannot determine the true elastic modulus of the samples yet shows relative elastic responses among those tested materials, thus the system requires another step of calibration. A known material is placed against the testing piece; an in-situ calibration is performed on the known material, after the correction factor obtained, the calibration piece will be removed and the real elastic modulus evaluation taking place and calibrated by the correction factor obtained from the known material piece.

To validate the calibration procedure, few tests were performed at Zero degree angle; shown in Table 19, Table 20 and

are the result of Aluminum, Bronze, and Steel substrates being evaluated and calibrated with different type of calibration pieces respectively.

Table 19: Aluminum Substrate In-Situ Calibration Result at Zero Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Aluminum Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.1720	0.375	2.180233	0.1740	0.37936	68.11GPa	-2.70
Copper Calibration Piece	0.1522	0.29	1.95388	0.1866	0.355545	76.67GPa	9.53
Steel Calibration Piece	0.1030	0.22	2.135922	0.1824	0.389592	64.93GPa	-7.24

Table 20: Bronze Substrate In-Situ Calibration Result at Zero Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Bronze Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
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Al calibration Piece	0.1755	0.375	2.136752	0.1444	0.308547	100.30GPa	-11.24
Copper Calibration Piece	0.1579	0.29	1.773274	0.1676	0.297201	107.97GPa	-4.45
Steel Calibration Piece	0.099	0.22	2.2	0.1402	0.30844	100.37GPa	-11.18

Table 21: Steel Substrate In-Situ Calibration Result at Zero Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Steel Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.1717	0.375	2.184042	0.097	0.211852	228.27GPa	14.13
Copper Calibration Piece	0.151	0.29	1.92053	0.1080	0.207417	240.94GPa	20.47
Steel Calibration Piece	0.1083	0.22	2.031394	0.1042	0.211671	228.76Pa	14.38

As the result, the in-situ calibration method does not provide absolute accuracy; however, it is a quick evaluation technique and most of the value fell into the literature range elastic modulus. Displayed in Table 19, Table 23 and Table 24 are the result for the flexible system adjusted to 30 degree angle and Table 25, Table 26 and Table 27 are the results at 45 degree setup, which followed the in-situ calibration algorithm.

Table 22: Aluminum Substrate In-Situ Calibration Result at 30 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Aluminum Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2586	0.375	1.450116	0.2739	0.397187	62.73GPa	-10.39
Copper Calibration Piece	0.1853	0.29	1.56503	0.2467	0.386093	65.99GPa	-5.73
Steel Calibration Piece	0.1340	0.22	1.641791	0.2345	0.385000	66.33GPa	-5.24

Table 23: Bronze Substrate In-Situ Calibration Result at 30 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Bronze Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2394	0.375	1.566416	0.1941	0.304041	103.23GPa	-8.65
Copper Calibration Piece	0.1985	0.29	1.460957	0.1941	0.283572	101.85GPa	5.00
Steel Calibration Piece	0.1402	0.22	1.569187	0.1932	0.303167	103.81GPa	-8.13

Table 24: Steel Substrate In-Situ Calibration Result at 30 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Steel Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2676	0.375	1.401345	0.1489	0.20866	237.26GPa	18.63
Copper Calibration Piece	0.1905	0.29	1.52231	0.1508	0.229564	187.79GPa	-6.10
Steel Calibration Piece	0.1533	0.22	1.435095	0.1511	0.216843	215.37Pa	7.69

Table 25: Aluminum Substrate In-Situ Calibration Result at 45 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Aluminum Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.3123	0.375	1.200768	0.3115	0.37404	69.88GPa	-0.17
Copper Calibration Piece	0.2204	0.29	1.315789	0.3053	0.40171	61.48GPa	-12.17
Steel Calibration Piece	0.1922	0.22	1.44641	0.3237	0.374984	69.56GPa	-0.63

Table 26: Bronze Substrate In-Situ Calibration Result at 45 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Bronze Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2992	0.375	1.253342	0.2498	0.313085	97.49GPa	-13.73
Copper	0.2513	0.29	1.153999	0.252	0.290808	112.76GPa	-0.21

Calibration Piece							
Steel Calibration Piece	0.1703	0.22	1.291838	0.2165	0.279683	122.04GPa	8.00

Table 27: Steel Substrate In-Situ Calibration Result at 45 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Steel Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2728	0.375	1.374633	0.1549	0.212931	225.36GPa	12.68
Copper Calibration Piece	0.2333	0.29	1.243035	0.1754	0.218028	212.50GPa	6.25
Steel Calibration Piece	0.1987	0.22	1.107197	0.1943	0.215128	219.65Pa	9.83

Despite of the fact that each material was evaluated only once but not the average of multiple tests, the main objective is to shorten the testing time for a rough estimation of elastic modulus. Most of the result at this setup fell in the desired range of elastic modulus, even though 225.36 GPa is slightly higher than its' theoretical value of steel, yet it could be easily distinguished from Aluminum and Bronze.

The system then adjusted again to perform indentation tests at 60 degree angle with the calibration procedure previously developed; instead of making precise evaluation on elastic modulus of material, such calibration method provide coarse elastic calculation but faster evaluation. The new setup is seen in the

Figure 29, and the results are displayed in Table 28,

Table 29 and

Table 30.

Figure 29: System Two at 60 degree setup

Table 28: Aluminum Substrate In-Situ Calibration Result at 60 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Aluminum Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2876	0.375	1.303894	0.288	0.375522	69.38GPa	-0.89
Copper Calibration Piece	0.2138	0.29	1.356408	0.277	0.375725	69.31GPa	-0.99
Steel Calibration Piece	0.1927	0.22	1.141671	0.3194	0.36465	73.19GPa	4.56

Table 29: Bronze Substrate In-Situ Calibration Result at 60 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Bronze Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2911	0.375	1.288217	0.2265	0.291781	112.01GPa	-0.88
Copper Calibration Piece	0.2329	0.29	1.24517	0.2231	0.277797	123.75GPa	9.51
Steel Calibration Piece	0.1719	0.22	1.279814	0.2252	0.288214	114.81GPa	1.60

Table 30: Steel Substrate In-Situ Calibration Result at 60 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Steel Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2913	0.375	1.287333	0.1633	0.210221	232.78GPa	16.39
Copper Calibration Piece	0.2209	0.29	1.312811	0.1693	0.222259	202.80GPa	1.40
Steel Calibration Piece	0.1704	0.22	1.29108	0.1769	0.228392	190.06Pa	-4.97

As the angle of the indentation system increases, the compliance seems to increase as well, data scattering had become even more severe; however, scattering can be improved by using different loading and unloading patterns; found for higher compliance systems, increasing the unloading length would stabilize the data thus it produces more linear and more consistent results; on the other hand, ridge systems do not require long range of unloading to extract elastic response from the specimen. The unloading length at 60 degree setup had been changed to 3 micro-meters, which is much greater than the 1.5 micro-meters unloading in 45 degree setup. The testing pattern of 7.5 micro-meters loading and 3 micro-meters unloading was experimentally tested that produced most linear and consistent results, yet there was no mathematical method applied to determine the reason of using such loading and unloading pattern. A survey indentation test had also been conducted at 60 degree setup on an aluminum specimen; its unloading length was set to be 2.7 micro-meters, yet the analysis was made by selecting different length of unloading length within a same test. Shown in Figure 30, the unloading lengths were selected as 2.7, 2.1, 1.5, 0.9 and 0.6 micrometers; the result showed that as the unloading length increases the Coefficient of Determination (R^2) value increases as well, which provides the means of higher linearity. And in the Figure 31, it shows the material compliance values would vary by changing the unloading length yet the changes are minimal.

Figure 30: Changing of Linearity by selecting different ranges of unloading length

However, selecting an unloading range that exceeded the elastic range would extract plastic information from the specimen and causes data scattering; thus, either not enough unloading length or too much unloading length should be avoided; and for acquiring the desired elastic response of the specimen, the unloading curve shall be highly linear [5] [14].

Figure 31: Material Compliances at different unloading length

System Two then arranged to 90 degree angle, shown in the Figure 32, followed the same calibration procedure and the results for different substrates are displayed in Table 31,

Table 32 and Table 33. And Plots of $\frac{dh}{dP}$ versus $P^{-\frac{1}{3}}$ for in-situ calibration can be found in Appendix F.

Figure 32: System Two at 90 degree setup

Table 31: Aluminum Substrate In-Situ Calibration Result at 90 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Aluminum Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2986	0.375	1.255867	0.3132	0.393336	63.83GPa	-8.81
Copper Calibration Piece	0.2201	0.29	1.317583	0.3001	0.395275	63.27GPa	-9.61
Steel Calibration Piece	0.1793	0.22	1.226994	0.3149	0.38638	65.90GPa	-5.86

Table 32: Bronze Substrate In-Situ Calibration Result at 90 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Bronze Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2839	0.375	1.320888	0.2213	0.292312	111.60GPa	-1.24
Copper Calibration Piece	0.2274	0.29	1.301966	0.2101	0.273543	127.75GPa	13.05

Steel Calibration Piece	0.1692	0.22	1.300236	0.2108	0.27409	127.23GPa	12.59
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Table 33: Steel Substrate In-Situ Calibration Result at 90 Degree Setup

	Experimental Slope	Theoretical Slope	Correction Factor	Steel Substrate Slope	Corrected Slope	Corrected Young's Modulus	Error %
Al calibration Piece	0.2883	0.375	1.300728	0.1639	0.212409	226.76GPa	13.38
Copper Calibration Piece	0.2249	0.28	1.289462	0.1853	0.238937	171.24GPa	-14.38
Steel Calibration Piece	0.174	0.22	1.264368	0.1733	0.219115	209.93Pa	4.97

Chapter 7: Conclusion and Recommendations

In this research, two flexible micro indentation systems were constructed, coupled with a multiple partial loading/unloading technique. In the early days of system one's development and performing indentation tests, the results were not satisfactory; however, the data obtained were consistent. Base on the observation of results from system one and Pro Mechanica structure analysis, a calibration method was developed; applying such method, a correction factor is conducted. Despite the fact that using the correction factor which would lead to an accurate result of the testing, the correction factor is highly dependent on the system configuration; thus, the calibration method is not capable of producing a universal correction factor for system one. System two was constructed with much less material than the previous system, which allows the user to adjust the system faster and efficiently, alone with a modified calibration method, the in-situ calibration provides the capability of performing indentation tests on various indentation arm angles and lengths. Base on 45 different tests that in-situ calibration method was used, at smaller arm angles, such as 0° and 30° , the in-situ calibration would produce much more accurate results than higher compliant system configuration, for instance, 45° and 60° . Due to the nature of flexible indentation system, the majority of PZT displacement causes the system to deflect and results in making insufficient amount of penetration into the specimen. Refinements could be made on increasing the rigidity of the system, thus reduce system compliance or using a smaller indenter to increase the indentation depth.

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Appendix A: Experimental Setup

- Physik Instrumente Piezoelectric Actuator
- 3.6 nm Resolution
- 120 μm Travel Range

- Honeywell Sensotec Model 31 Load Cell
- 0.15% Accuracy
- Compression and Tension
- 50 lb

- Spherical Sapphire
- 793.5 μm Radius
- High Temperature Stability
- Compression Mounted

Appendix B: System One Schematics

*Note: All dimensions are given in inches.

SCALE: 0.25X TYPE: PART NAME: HOLDER SIZE: A

SCALE: 1:1000 TYPE: PART NAME: CLAMP-CORR SIZE: A

SCALE: 3/32" TYPE: FRACTION NAME: INDOOR-HAFT SIZE: A

SCALE: 100% TYPE: PART NAME: L2V0321H01D REV: A

both ends of the rod were tapped 3/8-24 for one inch deep

1/4-20 thru holes, all those holes are one inch apart

Appendix C: System Two Schematics

*Note: All dimensions are given in inches.

SCALE: 1:100 TYPE: PART NAME: BLOCK-10 SIZE: A

SCALE: 1/8" = 1'-0" TYPE: PART NAME: BLOCKEE SIZE: A

SCALE: C:500 TYPE: PART NAME: NEWMOSE SIZE: A

Appendix D: 45 Degree Sample Holder Schematics

*Note: All dimensions are given in inches.

SCALE: C300 TYPE: PART NAME: PART-A SIZE: A

SCALE: (2X) TYPE: PART NAME: PARTED SIZE: A

Appendix E: 30 and 60 Degree Sample Holder Schematics

*Note: All dimensions are given in inches.

Appendix F: Plots of dh/dp versus $P^{-1/3}$ for In-Situ Calibration

In-Situ Calibration at Zero Degree Setup

In-Situ Calibration at 30 Degree Setup

In-Situ Calibration at 45 Degree Setup

In-Situ Calibration at 60 Degree Setup

In-Situ Calibration at 90 Degree Setup

In-Situ Calibration at 90 Degree Setup (Extended One Inch)