DESIGN AND CONSTRUCTION OF AN INTERFEROMETER SYSTEM FOR THE STUDY OF THIN METAL FILMS

By

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A thesis submitted in partial fulfillment of the requirements for the degree of

Bachelor of Science

Houghton College

July 2013

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Abstract

A laser interferometer system is currently under development at Houghton College for thin metal film analysis. Films are produced by thermal evaporation under high vacuum. In order to measure the curvature of the films at elevated temperatures, the laser interferometer generates a topographical map of each film using a three-inch diameter collimated beam of light and several optics components. The interferometer will be attached directly underneath the deposition chamber, allowing for the films to be measured without breaking vacuum. Using the relationship between curvature and temperature, the stresses of the films can be calculated.

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Chapter 1: HISTORY AND MOTIVATION

1.1 Thin Films

1.1.1 Introduction

Thin films are produced when a material is deposited onto a substrate, with the film ranging in thickness from less than a nanometer up to a few micrometers. Thin films are utilized in a countless number of different ways. They are used in optical devices such as mirrors, anti-reflective coatings, or simply for lustrous decoration. Thin films also are very important in electronics such as computer chips, memory disks, and fiber optic cables. This is because as time goes on, technology is getting smaller and smaller. The cell phones and tablets out now would not be available without the help of thin metal films. Furthermore these films can serve thermal, mechanical, magnetic, chemical, and biological functions; the possibilities of these films are virtually endless [1].

When a thin metal film is deposited, its molecular structure is different to that of the bulk material [2]. For this reason, thin films possess properties unattainable to both the original bulk material and the substrate on its own [3]. These properties are still to this day not completely understood, hence why the research of thin metal films is so vital.

1.1.2 Stress in Thin Films

One of these properties which are not completely understood in thin films is stress. Stress is an expression of the internal forces between neighboring particles in a body. Stresses that are exhibited in thin films fall into 2 main categories, intrinsic and extrinsic stresses. Intrinsic stresses form as a film is being grown on a substrate. These stresses are largely dependent upon deposition conditions such as the deposition materials, temperature, and growth rate. Furthermore, intrinsic stresses are usually reproducible and persist at room temperature for long periods of time. Extrinsic stresses however are induced after deposition. Some common factors which induce extrinsic stress include temperature

change, electrostatic or magnetic forces, chemical reactions, and phase transformations [4]. Both intrinsic and extrinsic stresses will be discussed further in Section 2.3.

After film deposition or deformation, the remaining stress is known as the residual stress in the film. This stress can be either tensile or compressive. When a body experiences internal 'pulling' forces, it undergoes tensile stress. Vice versa, a body undergoes compressive stress when it experiences internal 'pushing' forces. This phenomenon of tensile and compressive stress is shown in Figure 1.



Figure 1. Diagram of tensile and compressive film stress. The left side shows a film under tensile stress while the right side shows a film under compressive stress. The bottom shows the curvature a film undergoes while under tensile or compressive stress [5].

1.1.3 Previous Research

Arizona State and Harvard have contributed research in the area of silver thin films, a special topic of interest for Houghton College. Zoo and Alford of Arizona State studied the relation of substrate roughness to grain orientation and stress in silver films. Grain orientation can be represented by a Miller index, a three integer notation indicating the plane direction and angle in a crystal lattice. To determine a Miller index, a plane is made through a set of three or more atoms in the crystal orientation. The x, y, and z-intercepts of this plane are then placed in a set as integers and then inverted, leaving

 $(x_{int}^{-1}, y_{int}^{-1}, z_{int}^{-1})$. This set is known as a Miller index [6]. The two primary orientations for silver are (100) and (111).

Films were deposited by electron-beam evaporation, on either SiO_2 or PEN (polyethylene naphthalate) surfaces. The films were then investigated through XRD using a Phillips Materials Research diffractometer. In general the silver thin films showed the (111) preferred orientation; the scientists attributed the (111) planes to having the lowest surface energy. The silver films on smooth SiO_2 substrates had stronger (111) textures when compared with the films on PEN. Furthermore, the PEN films had comparatively higher (200) textures, which theoretically evolved to minimize the strain energy arising from the intrinsic stress [7].

Del Vecchio and Spaepen from Harvard measured the effect of deposition rate on intrinsic stress in copper and silver thin films. The formation of these stresses will be discussed further in Section 2.3.1. A high-vacuum chamber was used for vapor deposition, while scanning laser system was used to measure curvature. From this, stress could be calculated using the Stoney formula, which is explained in Section 2.4. The researchers found a decrease in postcoalescence compressive stress with increasing deposition rate, although it was more pronounced in silver films. For both silver and copper films it was found that the average stress at the tensile maximum decreased as the thickness at the tensile maximum increased [8].

Past research in thin film stress have brought about theories, but further development is needed. There is not a complete working model explaining stress in thin films, which is why it is important that research be continued.

1.1.4 Houghton College Research

The Houghton College Physics Department is currently in the process of growing their own thin metal films. Silver will be evaporated in a low pressure deposition chamber and adhere onto a silicon substrate. These thin films can then be analyzed through a number of available techniques including laser interferometry, x-ray diffraction, atomic force microscopy, and scanning electron microscopy.

Houghton is interested in is measuring stresses in thin films induced both thermally and during deposition. These measurements can be performed in situ using a laser interferometer.

1.2 Interferometry

1.2.1 Introduction

Interferometry is a technique which uses the interference between multiple waveforms to make a measurement. The interfering waveforms can originate from different sources or the same source. Optical interferometry has been in use for over a hundred years starting with Albert Michelson and is still used today. Interferometers have a wide range of applications such as precise measurements of distances and vibrations; optical tests; topography and spectroscopy studies; measurements of temperature, pressure, and electrical and magnetic fields [9].

1.2.2 Young's Double Slit Experiment

Thomas Young is often regarded as first discovering interference of light waves with his double slit experiment. A diagram of Young's experiment is shown in Figure 2. Young shined coherent light from a single source onto two thin slits. After the light had travelled through the slits, it was projected onto a screen. On the screen, light and dark fringes were visible. These were caused by the interference between the light coming from the two separate slits [10] [11]. This phenomenon will be explained in Section 2.2.



Figure 2. Diagram of Young's double slit experiment. The light starts at S1, travelling through the slit at *a*. This ensures the light is coherent. Then at S2, the light travels through the two slits at *b* and *c*. The two separated light waves then interfere creating an interference pattern on the screen at F. This pattern is shown on the far right by the light and dark linear fringes [12].

1.2.3 Michelson Interferometer

In 1881, Albert Michelson designed and constructed the first interferometer [13]. A sketch of his original apparatus is shown in Figure 3. His interferometer has been since known as the Michelson interferometer, and is the most widely known interferometer to date. Michelson used the interferometer to perform an experiment along with Edward Morley. The purpose of the experiment was to observe the ether drift proposed by Maxwell in 1878. Since it was accepted at the time that light acted like a wave while in a luminiferous ether, Michelson believed that his interferometer could be used to measure the relative motion between the moving earth and the stationary ether [14]. While Michelson and Morley were unable to measure the supposed ether drift, they had assembled an instrument that became fundamental for the progression of modern physics.



Figure 3. Drawing of Michelson's original interferometer. Light starts at the light source at *a*. The light then passes through the half-silvered mirror at *b*, splitting the light along two paths. The two paths of light then reflect off the mirrors at *c* and *d*. Finally the two paths of light pass back through the silvered mirror at *b*, and recombine. The recombination of light can then be observed by the telescope at *e* [15].

1.2.4 Twyman-Green Interferometer

In 1916, Frank Twyman and Alfred Green patented a modified Michelson interferometer, which is known as the Twyman-Green interferometer. The main difference between this and the Michelson interferometer was that the Twyman-Green interferometer incorporated lenses, and therefore has a larger diameter collimated beam. The interferometer was originally used to test optical components, such as lenses, mirrors and prisms, for defects [16]. This made the instrument applicable to a number of fields and was used in the testing of devices ranging from microscope equipment to camera lenses [17] [18].

Like the Michelson interferometer, the Twyman-Green interferometer uses a beam of monochromatic light which gets separated by a beam splitter along two paths. These beams are then reflected back into the beam splitter and recombine. The recombination of beams forms a series of bright and dark fringes, called an interference pattern or an interferogram. In the Twyman-Green interferometer, optical components can be placed in the path of the testing beam and analyzed by seeing how they affect the interferogram. Some sample interferograms can be seen in Figure 4.



Figure 4. Drawing of interferograms from a Twyman-Green interferometer. The different interferograms were caused by different defects in the lenses that were being tested. The top row shows the interferograms produced by the interferometer, the middle row shows the topography of the lenses being used, and the bottom row names the different defects present in the lenses [19].

1.2.5 Phase Shifting Interferometry

Phase shifting interferometry (PSI) is a process that uses the measurement of multiple interferograms taken at different reference phases. This is done by moving one of the two mirrors each at the end of a beam path, such as mirrors c and d in Figure 3. The moving mirror is known as the reference mirror. The variations in intensity of the interferograms are then fit to a cosine squared function to calculate the phase shift. PSI uses complex data evaluation and was therefore not possible until the recent development of computers [20]. Phase shifting interferometry also goes by many other names such as phase stepping interferometry, phase sampling interferometry, phase measuring interferometry, fringe scanning interferometry, real-time interferometry, AC interferometry and heterodyne interferometry; However, all of these refer to the same basic technique [21] [22].

The team of Bruning, Herriott, Gallagher, Rosenfeld, White, and Brangaccio are attributed to first using PSI in 1974. Their interferometer can be seen in Figure 5. It was their intention to use PSI to achieve higher accuracy measurements than the $\lambda/2$ contour interval in the fringe pattern from a Twyman-Green interferometer. For their system they used a modified Twyman-Green interferometer with a piezoelectrically driven reference mirror. Therefore they were able to take repeated samples at different set phases. An Analog to Digital converter was used to change diode signals into digital information for the computer while a digital to analog converter was used to change computer signals to analog voltages, driving the reference mirror to the desired position [23]. PSI continues to be used today to achieve measurements from 0.1 λ to 0.001 λ resolution [24].



Figure 5. Diagram of original phase shifting interferometer. The interferometer is a modified Twyman-Green interferometer. It uses a He-Ne laser as its light source. The phase shifting is performed by the piezoelectrically driven reference mirror, which is controlled by the computer. The interferograms are measured using a 32 x 32 diode array camera which outputs to the computer where they are processed [25].

More specifically, PSI has been used to measure thin films [26] [27]. Lee, Tien, and Hsu deposited Hb_2O_5 (niobium pentoxide) thin films via ion-beam sputtering onto glass substrates. PSI was used with a

Twyman-Green interferometer to measure the curvature of the films. Then the stress of the films was calculated using the Stoney formula, which explained in Section 2.4. Lin, Tong, Cheng, Chung, and Hsu used PSI to measure the residual stress in silver, gold, and copper thin films deposited on a paddle cantilever beam. The silver films were found to have tensile residual stress while the gold and copper films were found to have compressive residual stress. A diagram displaying tensile and compressive film stresses is shown in Figure 1.

Chapter 2: THEORY

2.1 Waves

2.1.1 Wave-Particle Duality

It is a well-known concept of modern quantum mechanics that all particles exhibit both wave and particle properties. This concept is known as duality. It is because of this that light is able to be modeled using wave equations.

2.1.2 Sinusoidal Wave Form

The general form of a sinusoidal wave function is

$$A(x,t) = A_o \cos(kx - \omega t + \varphi). \tag{2.1}$$

From this equation, A(x, t) is the magnitude of the wave at a given point along the x-axis, x, and a given point in time, t. A_o is the amplitude of the wave. k is the wave's wave number where

$$k = 2\pi/\lambda \tag{2.2}$$

and where λ is the wavelength of the wave. ω is the wave's angular frequency where

$$\omega = 2\pi/T \tag{2.3}$$

and where T is the period of a wave. Lastly, φ is the phase shift of a wave.

2.1.3 Complex Exponential Wave Form

Eq. 2.1 can be rewritten as

$$U(\mathbf{r},t) = A_o e^{i(\mathbf{k}\cdot\mathbf{r}-\omega t+\varphi)}$$
(2.4)

where k is the wave vector, r is the position vector and where A is the real part of U(r, t) This form utilizes the complex number plane rather than the Cartesian plane. Eq. 2.4 can be further simplified using a complex valued amplitude

$$U_o = A_o e^{i\varphi} \tag{2.5}$$

instead of the real valued amplitude A_o . This gives

$$U(\mathbf{r},t) = U_o e^{i(\mathbf{k}\cdot\mathbf{r}-\omega t)}$$
(2.6)

for the complex exponential form of the wave equation.

2.2 Interference

2.2.1 Interference of Two Waves

When two waves interact, they interfere with one another. With light, this interference can be observed through the intensity of an optical interference pattern. The complex amplitudes of two waves at a point \boldsymbol{r} are given by

$$U_1(\mathbf{r}) = A_1(\mathbf{r})e^{i\varphi_1},$$
 (2.7)

$$U_2(\mathbf{r}) = A_2(\mathbf{r})e^{i\varphi_2}.$$
 (2.8)

The intensity of the interference between these two waves is then given by

$$I(\mathbf{r}) = |U_1(\mathbf{r}) + U_2(\mathbf{r})|^2$$
(2.9)

which expands to

$$I(\mathbf{r}) = (A_1(\mathbf{r})e^{i\varphi_1} + A_2(\mathbf{r})e^{i\varphi_2})(A_1(\mathbf{r})e^{-i\varphi_1} + A_2(\mathbf{r})e^{-i\varphi_2})$$
(2.10)

$$= A_1(\mathbf{r})^2 + A_2(\mathbf{r})^2 + A_1(\mathbf{r})A_2(\mathbf{r}) \left[e^{i(\varphi_1 - \varphi_2)} + e^{-i(\varphi_1 - \varphi_2)} \right]$$
(2.11)

Assuming both waves are propagated from the same source,

$$A_1(r) = A_2(r) = A(r)$$
(2.12)

which gives

$$I(\mathbf{r}) = 2A(\mathbf{r})^{2} + A(\mathbf{r})^{2} \left[e^{i(\varphi_{1} - \varphi_{2})} + e^{-i(\varphi_{1} - \varphi_{2})} \right].$$
(2.13)

The offset or phase shift between the two waves is defined as

$$\delta = \varphi_2 - \varphi_1 \tag{2.14}$$

which gives

$$I(\mathbf{r}) = 2A(\mathbf{r})^{2} + A(\mathbf{r})^{2} [e^{-i\delta} + e^{i\delta}]$$
(2.15)

Then using the trigonometric identity

$$\cos\theta = \frac{e^{i\theta} + e^{-i\theta}}{2} \tag{2.16}$$

 $l(\mathbf{r})$ can be reduced to the trigonometric form

$$I(\mathbf{r}) = 2A(\mathbf{r})^{2} + A(\mathbf{r})^{2} [2\cos(\delta)]$$
(2.17)

$$= 2A(r)^{2}[1 + \cos(\delta)]$$
(2.18)

$$= 2A(\mathbf{r})^2 \left[2\cos^2\left(\frac{\delta}{2}\right) \right]$$
(2.19)

$$= 4 \left[A(\boldsymbol{r})^2 \cos^2\left(\frac{\delta}{2}\right) \right]. \tag{2.20}$$

From this it can be seen that the interference pattern caused by the interference of two waves has an intensity proportional to that of a cosine squared function, and is dependent on the phase shift, δ .

2.2.2 Constructive and Destructive Interference

When two waves are in phase

$$\delta = m\lambda \quad (m = 0, \pm 1, \pm 2, \cdots). \tag{2.21}$$

This means that there is no offset between the waves, resulting in a phase shift of 0°. Since the waves are in phase, the resulting intensity is

$$I(\mathbf{r}) = 4A(\mathbf{r})^2 \left[\cos^2\left(\frac{m\lambda}{2}\right)\right] \quad (m = 0, \pm 1, \pm 2, \cdots)$$
(2.22)

$$=4A(\mathbf{r})^2\tag{2.23}$$

which is the maximum interference intensity. This is called constructive interference. When two wave are out of phase

$$\delta = \left(m + \frac{1}{2}\right)\lambda \quad (m = 0, \pm 1, \pm 2, \cdots). \tag{2.24}$$

In this case the wave phases are completely offset, resulting in a phase shift of 180°. This then yields an intensity of

$$I(\mathbf{r}) = 4A(\mathbf{r})^{2} \left[\cos^{2} \left(\frac{(m+1/2)\lambda}{2} \right) \right] \quad (m = 0, \pm 1, \pm 2, \cdots)$$
(2.25)

$$= 0$$
 (2.26)

which is the minimum interference intensity. This is called destructive interference. Interference patterns consist of both constructive and destructive interference, and depend on the phases of the waves, as shown.

2.2.3 Michelson Interferometer

Michelson applied this idea of interference when constructing the Michelson interferometer. Light from a coherent light source is shined into a half-silvered mirror or beam splitter. This reflects half of the light and allows half of the light to pass through the mirror. The two beams of light then follow their respective paths and are each reflected by a mirror back into the beam splitter. Half of the beam that was originally reflected passes through the beam splitter, while half of the beam that originally passed though is reflected. These two new beams recombine and can be viewed at the detector [28]. A diagram of the instrument is shown in Figure 6.



Figure 6. Diagram of Michelson interferometer. Light starts at the coherent light source on the left. Then it either is reflected by or passes through the half-silvered mirror. The two separate beams are then reflected by separate mirrors, back into the half-silvered mirror. Finally the beams recombine and hit the detector [29].

The two beams create an interference pattern, which depends on the length of the paths that each of the beams travelled. Relating this to the equations in Section 2.2.1, let us define the distance from beam splitter to one of the mirrors as d_1 and the distance to the other mirror as

$$d_2 = d_1 + \Delta d. \tag{2.27}$$

This means the distance traveled by the light along each separate path is $2d_1$ for the first mirror and

$$2d_2 = 2d_1 + 2\Delta d \tag{2.28}$$

for the second. The path length difference between the two beams of light as they reenter the beam splitter is then

$$(2d_1 + 2\Delta d) - 2d_1 = 2\Delta d \tag{2.29}$$

or twice the physical difference in distance between each of the mirrors and the beam splitter. The phase shift δ of the two waves can then be defined as

$$\delta = 2\Delta d \tag{2.30}$$

giving us for our interference equation,

$$I(\mathbf{r}) = 2[A(\mathbf{r})^2 \cos^2(\Delta d)].$$
(2.31)

2.2.4 Twyman-Green Interferometer

The Twyman-Green interferometer behaves very similarly to the Michelson interferometer. The only difference between the two instruments is that the Twyman-Green interferometer uses lenses to collimate a larger beam that travels through the apparatus. Therefore, intensity of an interferogram depends on the coordinates perpendicular to the beam axis. This is why curved mirrors or lenses produce patterns like the ones shown in Figure 4. A diagram of the Twyman-Green interferometer is shown in Figure 7.



Figure 7. Diagram of Twyman-Green interferometer. This interferometer is a modified Michelson interferometer incorporating two lenses L_1 and L_2 . L_1 collimates the light source while L_2 decollimates the final recombined beam. [30].

2.2.5 Phase Stepping Interferometry

PSI is a process that can be performed using a number of different interferometers, including a Twyman-Green interferometer. PSI takes several interferograms, which have been recorded at different reference phases, and fits their variations in intensity to the cosine squared function in Eq. 2.20. One of the ways to fit the data is by using the method of least squares. This method minimizes the sum of the squares of the error made for all the measured intensities. This sum can be defined as chi squared with the equation

$$\chi^{2} = \sum_{i}^{N} (F(\delta) - y_{i})^{2}$$
(2.32)

where N is the total number interferograms, $F(\delta)$ is the best fit cosine squared function, and y_i is the measured intensity of a particular interferogram. An example of a least squares fit is shown in Figure 8. After minimizing χ^2 , δ can be calculated for each individual pixel.



Figure 8. Graph of fitting PSI intensities. The intensities of a point on the interferogram are measured at different reference phases. Reference phase is shown in arbitrary units by the x-axis while intensity is shown by the y-axis. Then a cosine squared curve is fit to the points, as shown. From there δ can be calculated by comparing this curve to the curves of other nearby points on the interferogram, and determining their phase shift [31].

Consider the Twyman-Green interferometer in Figure 7. If this interferometer was being used for PSI, let us call M_1 the testing mirror and M_2 the reference mirror. The reference mirror is moved forwards and backwards on the nanometer scale, allowing for the measurement of interferograms at different reference phases. Meanwhile, the testing mirror remains still. What each interferogram is really displaying in its fringes is the path length difference between the light reflecting off the 2 separate mirrors, M_1 and M_2 . If it was then assumed that the reference mirror M_2 was perfectly flat, the interferogram would really just be the topography of M_1 .

Applying this concept, PSI can be used to measure the topography of a reflective object such as a thin metal film. First measurements are taken using a silicon substrate for M_1 for a reference. Then the film is deposited on the substrate and measurements are taken again. Since the reference mirror and substrate are not perfectly flat, the first measurements can be subtracted from the measurements of the film, giving a more accurate topography.

2.3 Causes of Stress in Thin Films

2.3.1 Growth Stresses in Thin Films

When thin films are deposited, they follow certain growth modes. For physical vapor deposition of silver onto a silicon substrate, the Volmer-Weber growth mode is favored. In this growth mode, three-dimensional islands form and eventually coalesce to form a continuous film [32]. In situ measurements have found that films of this growth mode follow a specific stress behavior. This behavior is characteristic of an initial compressive stress, which is followed by a tensile stress, and then returns back to a compressive stress [33]. This pattern is known as CTC (compressive-tensile-compressive) and follows the formation, growth, and coalescence of islands during film deposition [34]. Figure 9 shows graphs of average film stress vs. mean film thickness which display CTC.



Figure 9. Graphs of average film stress vs. mean film thickness during deposition. The graph on the right is for the deposition of silver onto a silicon substrate. Note the x-axis is given in units of Ångströms. The graph on the left shows the general deposition curve followed by most materials (including silver). The y-axes of both graphs show average film stress where positive stress is tensile and negative stress is compressive. It can be seen that both curves follow the CTC behavior common in Volmer-Weber growth [35].

In the earliest stage of film growth, crystallites forming on the growth surface become firmly attached to the substrate. However as these crystallite islands grow, they are constrained by their initial attachment, and therefore undergo a compressive stress. Once the islands have grown large enough, the film switches to tensile stress. This is because the gaps between the islands, also known as grain boundaries, are so small that adjacent islands pull together to try and 'zip up' the gaps. It has been calculated that the maximum gap that can be closed in this way is 0.17 nm. The point in time of the highest tensile stress, shown by the peaks in Figure 9, is known as the tensile maximum. Finally as more atoms are deposited, they fill the grain boundaries, causing the film to once again revert back compressive stress. This is due to an excess number of atoms comprising the film. These three stages of film growth are shown in Figure 10 [36].



Figure 10. Diagram of stress stages during film growth. The first stage shows the formation of islands, when the film is under compressive stress. The second stage shows the islands growing larger, when the film is under tensile stress. The third stage shows the islands coalescing, when the film is under compressive stress once again.

2.3.2 Thermal Expansion in Solids

When a solid is heated, it expands according to the thermal expansion coefficient for that particular material. Assuming the effects for pressure to be negligible, the relationship for linear expansion of a solid is

$$\alpha_L = \frac{1}{L} \frac{dL}{dT} \tag{2.33}$$

where α_L is the linear expansion coefficient, *L* is the length of the solid, and dL/dT is the rate of change of *L* per unit change in temperature. Therefore a small change in length of a solid ΔL is given as

$$\frac{\Delta L}{L} = \alpha_L \Delta T \tag{2.34}$$

for a change in temperature ΔT . The same relationship is also applicable for area and volumetric thermal expansion. For area expansion of a solid,

$$\alpha_A = \frac{1}{A} \frac{dA}{dT} \tag{2.35}$$

where α_A is the area expansion coefficient, A is the area of the solid, and dA/dT is the rate of change of that area with temperature. This then yields

$$\frac{\Delta A}{A} = \alpha_A \Delta T \tag{2.36}$$

for a small area change in a solid ΔA with a change in temperature ΔT .

For isotropic materials it is known that the area thermal expansion coefficient is two times the linear expansion coefficient, or

$$\alpha_A = 2\alpha_L. \tag{2.37}$$

This is because an area expansion is simply a linear expansion in two mutually orthogonal directions. For an isotropic area expansion ΔA along two dimensions l and w, it is given that:

$$A_i + \Delta A = (l + \Delta l)(w + \Delta w)$$
(2.38)

$$= (l + \alpha_L l\Delta T)(w + \alpha_L w\Delta T)$$
(2.39)

$$= lw(1 + \alpha_L \Delta T)^2 \tag{2.40}$$

$$= A_i [1 + 2\alpha_L \Delta T + (\alpha_L \Delta T)^2].$$
(2.41)

Dividing both sides by A_i then gives

$$1 + \frac{\Delta A}{A_i} = 1 + 2\alpha_L \Delta T + (\alpha_L \Delta T)^2$$
(2.42)

$$\frac{\Delta A}{A_i} = 2\alpha_L \Delta T + (\alpha_L \Delta T)^2.$$
(2.43)

Since $\alpha_L \Delta T \ll 1$ for typical values of ΔT , the $(\alpha_L \Delta T)^2$ term can be neglected, which leaves

$$\frac{\Delta A}{A_i} = 2\alpha_L \Delta T. \tag{2.44}$$

Dividing by Equation 2.36 gives

$$1 = \frac{2\alpha_L}{\alpha_A} \tag{2.45}$$

which is equivalent to Equation 2.37.

2.3.3 Curvature due to Thermal Expansion

Consider a silver thin film deposited on a silicon substrate. When the film and the substrate are heated, they will expand at different rates according to their coefficients of thermal expansion. For silver, the linear expansion coefficient is $\alpha_L = 18 (\times 10^{-6}/^{\circ}C)$, while the area expansion coefficient is $\alpha_A = 36$. Likewise for silicon, the linear expansion coefficient is $\alpha_L = 3$, while the area expansion coefficient is $\alpha_A = 6$. It is a concern that the thermal expansion coefficient for a thin film may be different than that of the bulk material. Fortunately the coefficient for silver, depending on substrate material [37]. Since the expansion for silver is greater than the expansion coefficient for silicon, the film will expand more than the substrate. However, because the two are rigidly adhered to one another, they will exert opposing stresses, shown in Figure 11. These stresses will then cause the film and substrate to bend together until they have reached equilibrium. This is demonstrated in Figure 12.



Figure 11. Diagram of thermal expansion of thin film and substrate. When heated, the thin film expands linearly ΔL_f while the substrate expands linearly ΔL_s . Since the thin film and substrate are rigidly adhered to one another, their differences in expansion manifest as opposing stresses, shown by the arrows above. As a result of these stresses, the film and substrate bend, as shown in Figure 12.



Figure 12. Diagram of two-dimensional curvature of thin film and substrate. As a result of the stresses described in Figure 8, the thin film and substrate curve to reach a state of equilibrium. It can be seen that the thin film has expanded more than the substrate as $\Delta L_f > \Delta L_s$. The thicknesses of the film and substrate are h_f and h_s respectively, where $h_s \gg h_f$. The bending of the film and substrate can be fit to a circle of radius R where the curvature of the film and substrate $\kappa = R^{-1}$.

2.4 Relating Curvature to Stress

2.4.1 Deriving the Stoney Formula

Stoney derived a relation between the curvature κ of a substrate and film and the stress of the film σ_f [38]. First, stress is defined as

$$\sigma = \frac{F}{A} \tag{2.46}$$

or force divided by area. Another similar concept is strain, which is

$$\varepsilon = \frac{\Delta L}{L} \tag{2.47}$$

or change in length divided by the original length. Stress and strain on a substrate can be related using Hooke's Law, given by

$$\sigma_s = M_s \varepsilon_s \tag{2.48}$$

where the biaxial modulus of the substrate material M_s is further defined as

$$M_s = \frac{E_s}{(1 - \nu_s)} \tag{2.49}$$

where E_s and v_s are the Young's modulus and Poisson's ratio for the substrate, respectively. Assuming uniform curvature of a thin film/substrate system where $h_s \gg h_f$, the system's curvature is defined at the midpoint of the substrate. The strain on the substrate is then given by a relation of similar triangles, shown in Figure 13.



Figure 13. Diagram showing relation of substrate strain. It can be seen that if *a* is the original length *L* of the substrate, δ is the change in length ΔL . It can also be seen that triangle *aR* is similar to triangle δz . This then yields the proportionality $\delta/a = z/R$.

Using the relation in Figure 13, the strain for the substrate is given as

$$\varepsilon_s = \frac{\Delta L_s}{L_s} = \frac{\delta}{a} = \frac{z}{R}.$$
(2.50)

The strain in the substrate is then found using Equation 2.48, giving us

$$\sigma_s = \frac{M_s z}{R}.$$
(2.51)

For a thin film/substrate system is static equilibrium, the thin film and substrate must have moments such that the sum of the two is zero. Therefore

$$\psi_f = -\psi_s \tag{2.52}$$

where ψ is the moment per length along the radius in the plane of the substrate or film. Torque is defined as

$$\tau = F \times r = \sigma A r = \sigma A z \tag{2.53}$$

making the moment

$$\psi = \frac{\tau}{l} = \sigma h z. \tag{2.54}$$

For the thin film $z = \frac{h_s}{2}$, assuming the film has negligible thickness. Therefore, the moment about the film is

$$\psi_f = \frac{\sigma_f h_f h_s}{2}.\tag{2.55}$$

The moment about the substrate however will need to be integrated across its thickness such that

$$\psi_{s} = \int_{-h_{s}/2}^{h_{s}/2} \sigma_{s} z dz.$$
(2.56)

Using σ_s found in Equation 2.51, the moment is found to be

$$\psi_s = \int_{-h_s/2}^{h_s/2} \left(\frac{M_s z}{R}\right) z dz = \frac{M_s}{R} \int_{-h_s/2}^{h_s/2} z^2 dz$$
(2.57)

$$= \frac{M_s}{3R} z^3 \left| \frac{h_s/2}{-h_s/2} = \frac{M_s}{3R} \left(\frac{h_s^3}{8} - \frac{-h_s^3}{8} \right) = \frac{M_s h_s^3}{12R} \right|$$
(2.58)

Equation 2.52 can then be used to relate ψ_f and ψ_s such that

$$\frac{\sigma_f h_f h_s}{2} = \frac{-M_s {h_s}^3}{12R}.$$
 (2.59)

Solving for film stress then yields

$$\sigma_f = \frac{-M_s h_s^2}{6h_f R} \tag{2.60}$$

so that when Equation 2.49 and $R = \kappa^{-1}$ are substituted in, Stoney's formula is found to be

$$\sigma_f = \frac{-E_s h_s^2 \kappa}{6h_f (1 - \nu_s)} \tag{2.61}$$

This equation shows film stress to be directly proportional to curvature. It also shows that film stress increases as film thickness increases, but decreases as substrate thickness increases.

2.4.2 Applying the Stoney Formula

Using PSI with a Twyman-Green interferometer, the topography of a thin metal film on a substrate can be measured. Whether then thin film and substrate bend due to deposition or to heating, this curvature can be seen through the measured topography. Finally, the Stoney formula can relate this curvature to the stress in the film. Thus, the measured stress in the thin metal films can be related to the deposition period or heating which caused the stress to occur.

Chapter 3: EXPERIMENT

3.1 Interferometer Apparatus

3.1.1 Overview

Houghton College has designed and built a laser interferometer system for measuring the topography of thin metal films. The interferometer is a phase shifting, Twyman-Green interferometer which has been constructed on a small movable optical table. A diagram of the interferometer is shown in Figure 14 and a picture in Figure 15. The interferometer will be attached directly underneath a vacuum deposition chamber and have the ability to measure the thin films in situ. This is shown in Figure 16.



Figure 14. Diagram of Houghton College interferometer. This diagram labels the important parts of the interferometer such and the laser, beam splitter, optical table, and various lenses and mirrors. The beam path is also shown in red. This diagram was made using Autodesk Inventor.



Figure 15. Picture of Houghton College interferometer. The components of the interferometer are labeled as they are in Figure 14. The beam path is shown in red. Above the upper converging lens a hole can be seen where the beam travels into the deposition chamber and will reflect off a thin film. Also, after the beams recombine through the beam splitter, the new beam reflects off the image mirror. This beam then projects an interferogram on the screen, where a camera is able to capture the image.



Figure 16. Diagram of interferometer and deposition chamber assembly. The interferometer and deposition chamber are attached to the inner frame. The inner frame is then hung on springs, from the outer frame. The springs are used to prevent background noise and vibrations. This diagram was made using Autodesk Inventor.

3.1.2 Laser

The light source for the Houghton College interferometer is a 25mW helium neon laser. The laser is red and its light has a wavelength of 633 nm. The laser is screwed down to a ¹/4" thick aluminum plate. The plate is then attached to the optical table with three stainless steel optical mounting posts. These ensure the laser does not move and stays aligned.

3.1.3 Lenses

The interferometer includes one small diverging lens and 2 large converging lenses. The diverging lens is a 6 mm diameter, 6 mm focal length, plano/concave lens After the laser beam leaves the laser, the first thing it passes through is the diverging lens, and then the beam splitter. The lens can either be placed on its own stand, or screwed directly into the side of the beam splitter facing the laser. Having it screwed directly into the beam splitter means there is one less optical component that needs aligning.

After travelling through both the diverging lens and beam splitter, the beam separates and each new beam passes through a converging lens. The converging lenses are 90 mm diameter, 500 mm focal point, plano/convex lenses. These lenses collimate the beams at 82.5 mm in diameter. The lenses need to be placed at a precise distance from the beam splitter in order for the beam to be properly collimated. To check this, the beam width was measured up to 8 meters beyond the lens and checked to be 82.5 mm. Furthermore, each lens is attached to the optical table with lens holders and two stainless steel optical mounting posts, ensuring they stay in correct position and alignment.

3.1.4 Mirrors

The interferometer has three flat mirrors. Two of the mirrors, the reference and temporary mirror, reflect the separated beams back into the beam splitter. These mirrors are three inch diameter polished silicon wafers. The third mirror, the image mirror, reflects the recombined beam onto the screen which displays the interferogram. This mirror is made of Pyrex with a metallic coating. All of the mirrors are connected to the optical table with holders and stainless steel optical mounting posts.

The temporary mirror will be removed when the interferometer is attached to the deposition chamber as shown in Figure 16. Instead of reflecting off the temporary mirror the beam will travel through the hole in the interferometer box (which can be seen in Figure 15), through the window in the bottom of the deposition chamber, and reflect off the deposited thin metal film. The temporary mirror is used only for testing purposes of the interferometer, not for measuring thin films.

The reference mirror reflects the second beam, also known as the reference beam. The reference mirror moves forward and backward several hundred nanometers, following a triangle wave. This is done through the use of piezoelectric ceramics, which expand as a voltage is put across them. The larger the voltage, the more the piezo expands. There are 3 piezos on the reference mirror, assuring movement orthogonal to the face of the mirror. Also, springs are used to apply a pre-load to the piezos which is

necessary to optimize their performance. The reference mirror and a piezoelectric ceramic are shown in Figure 17.





Figure 17. Pictures of reference mirror and piezoelectric ceramic. The picture on the left shows the reference mirror, along with the converging lens, pre-loading springs, and wires connecting the piezos to the electronics. The picture on the right shows a piezo next to a dime, for scale. Each piezo is connected to two wires as shown in order to induce a voltage drop across it.

3.1.5 Electronics

The electronics controlling the interferometer are shown in Figure 18. First the camera, a Microsoft LifeCam Studio webcam, takes pictures which are sent to the computer via USB. These pictures are processed by Yamcam and LabView which will be discussed later in Section 3.2.



Figure 18. Diagram of interferometer electronics. Each of the electronic components are labeled, along with their various inputs and outputs. The red wires represent positive voltage, the black negative voltage, the green ground, the purple triangle waves, and the blue USB connections. The inner workings of the circuit box are shown in Figure 19.

The piezoelectric ceramics are powered by a triangle wave that originates at the computer. LabView controls NI-DAQmx through USB. The NI-DAQmx creates the wave, with a voltage range from -2.16V to +2.16V and a period of 10 seconds. For testing purposes, the computer and NI-DAQmx can be replaced with a function generator. This wave is then amplified using a simple non-inverting operation amplifier circuit. A diagram of this circuit is shown in Figure 19. The circuit was soldered to a circuit board and attached in a box with banana plug sockets. This allows for wires from other electrical components to be easily attached to the circuit. A picture of this circuit and its box is shown in Figure 20.



Figure 19. Diagram of op-amp circuit. The circuit is a simple noninverting operation amplifier circuit. The various inputs and outputs are labeled with their corresponding banana socket size and color. A picture of the circuit is shown in Figure 20.



Figure 20. Picture of op-amp circuit. The op-amp circuit from Figure 19 is shown soldered to a circuit board. The circuit it connected to banana plug sockets, as seen at the top of the box. The connections to these banana plugs is shown in Figure 18.

The op-amp circuit amplifies the wave to a voltage range from -30V to +30V. The circuit output is connected to one side of each of the three piezos while -30V is connected to the other. This creates a voltage drop ranging from 0V to +60V across the piezos, which is enough to make them expand to a point where the reference mirror moves at least one half wavelength of light (317 nm).

3.2 Image Processing

3.2.1 Yawcam

Yamcam (which stands for Yet Another WebCAM software) is a free webcam software for Microsoft Windows. It has a variety of different features but is used with the interferometer for taking a series of pictures automatically. With Yamcam, a picture can be taken every second, and saved in sequence. This is convenient as these pictures can then be read directly into LabView. Furthermore, Yamcam can control settings on the camera such as focus and brightness to ensure clear images of the interferograms. A picture of an interferogram taken using Yawcam is shown in Figure 21.



Figure 21. Picture of interferogram from Houghton College interferometer. This picture was taken using a test mirror. Each pair of bright and dark fringes represents a path length difference of 317 nm, half of the light source wavelength.

3.2.2 LabView

LabView is a graphical programming platform from National Instruments. This program analyzes the images from the camera to create a topography, and also drives the piezos in the reference mirror using the NI-DAQmx. To drive the piezos, the program simply communicates with the NI-DAQmx to output a triangle wave. This occurs while Yawcam is taking pictures.

The analysis of the pictures is much more complex. There are two separate LabView programs that are used to do this. The first takes multiple pictures saved by Yawcam and outputs them as a 3D intensity array. The second takes one of these arrays and fits each of the pixels to a cosine squared function (like in Figure 8) and outputs a topographical map. Important parts of these programs are shown and discussed in following pages. Figure 22 shows the input of pictures into a 3D array for LabView to process.



Figure 22. LabView program part 1. This is the main component of the first LabView program. At A, a compound string is being constructed in order to open the correct jpeg file that was saved by Yawcam. At B, the opened jpeg is converted into a 2D intensity array. At C, multiple 2D intensity arrays collected over time are placed into a 3D intensity array. At D, the 3D intensity array is being output to the second LabView program.

Figure 23 shows the initial fit for the center pixel of the interferograms. This initial fit is a least squares fit that is solved by iterating the constants of the function. Since it is a premade program that does not use the properties of the data to predict values for the next iteration, the fit is slow. The fact that it finds all constants, not just phase shift, adds to its slowness. For these reasons, a separate least squares fit has been written and used to fit the remaining pixels.



Figure 23. LabView program part 2. This is the first main component of the second LabView program. At A, the 3D intensity array from Figure 22 is inputted into the second program. At B, the pixel height and width of the interferograms are divided by two in order to find the center pixel. At C, the 1D intensity array of the center pixel over time is fit to a sine squared function. This yields the amplitude and radians/frame of the center pixel. Since this fit is slow, it is only used for the center pixel.

The least squares method of fitting has been previously mentioned in Section 2.2.5. For this fit, chi squared is given as

$$\chi^2 = \sum_i^N (F(\delta) - y_i)^2 \tag{3.1}$$

for pictures taken every second for N seconds. This parameter compares the measured intensity of a pixel to a squared sinusoidal function dependent on phi. Error is minimized when

$$\frac{d\chi^2}{d\delta} \cong 0, \tag{3.2}$$

and at a minimum. The derivative of chi squared is calculated for two separate phi values and the slope between them is found. If the initial guess is close to the minimum of chi squared, it can be assumed that the derivative of chi squared is roughly linear in this region. The line can then be followed to where derivative of chi squared is zero. This then yields the best value of phi for the pixel. These steps are shown in Figure 24. This process is a significantly faster way of determining phi than LabView's premade fit (used for the center pixel), thus making PSI feasible. The special fit also checks its guess before moving on, ensuring fit accuracy.



Figure 24. LabView program part 3. This is the second main component of the second LabView program. The 3D array of pixels over time is subdivided into 2D and then 1D arrays of pixels over time at A and B respectively. At C, the derivative of chi squared is calculated. At D phi is altered and the derivative of chi squared is again calculated (for the new phi) at E. At F, the slope of the derivative of chi squared is calculated and then phi is found where the derivative equals zero.

Then the pixels must be compared to their surrounding neighbor pixels so phi can be adjusted to the correct factor of π . This is shown in Figure 25. The LabView program also has an option to subtract out a previously saved background topography from the measured topography, resulting in a more accurate measurement.



Figure 25. LabView program part 4. This loop is the third and final main component of the second LabView program. The loop goes through each pixel in a quadrant of the image, looping through different rows at A, and looping through the pixels in a given row at B. At C, a pixel is compared to an adjacent pixel. Multiples of π are subtracted from the pixel's phi value until its difference from the adjacent pixel is less than $\pi/2$. There are 3 nearly identical loops to the one shown for the other 3 quadrants of the interferogram. Each quadrant's comparisons start with the middle pixel and move outward.

3.2.3 Resolution

As discussed previously, PSI yields resolutions unmatched by standard interferometry techniques. This resolution is known as the out-of-plane or vertical resolution. Most interferometers are limited by the wavelength of their light source. PSI however uses a fit of multiple interferograms, which means the vertical resolution is determined by how accurately the data can be fit to a function.

The vertical resolution of the Houghton College interferometer will be found by taking two images of an identical sample and subtracting them from one another. Any resulting height differences are therefore error in the interferometer system. The standard deviation of the height would then be the maximum vertical resolution. This being said, the Houghton College interferometer system surely has a vertical resolution that is a small fraction of the 633 nm wavelength laser being used.

Another form of resolution in the interferometer is the in-plane or lateral resolution. The main limiting factors here are optical magnification and detector pixel size [39]. Currently the Houghton College interferometer system takes images with an 800 x 600 pixel size, thus achieving an interferogram resolution of 200 pixels per inch. However, the Microsoft LifeCam Studio webcam is capable of taking images up to a size of 1920 x 1080 pixels, an interferogram resolution of 360 pixels per inch.

3.3 Error

3.3.1 Sources of Error

While there are many different error sources associated with PSI, they tend to fall into three categories:

(1) errors associated with the data acquisition process,

- (2) errors associated with environmental effects,
- (3) and errors associated with defects in optical and mechanical design and fabrication [40].

Errors related to data acquisition include phase shifting errors, detector nonlinearities, and quantization errors. Phase shifting errors occur when interferometer measurements do not match up the expected sinusoidal function. Detector nonlinearities refer to when the measured intensity of pixel on an interferogram is different from the pixels actual intensity. Quantization errors occur when converting analog measurements to digital signals in the computer.

Environmental effects comprise both vibration errors and air turbulence. Vibrations disturbing the mechanical stability of the interferometer, and thus affecting measurements, are vibration errors. Air currents and turbulence can also affect high accuracy measurements.

Lastly, source stability, extraneous fringes, and interferometer optical errors are all due to defects in the physical apparatus of the interferometer. Source stability refers to a light sources ability to keep a consistent frequency and amplitude. Extraneous fringes are unexpected features in an interferogram caused by background light or unwanted reflections. Improper alignment or defects in the optical equipment cause interferometer optical errors [41].

3.3.2 Error Prevention

While some of these errors are statistically insignificant, others can have noticeable effects on measurements. Therefore, precautions have been made to minimize these effects. The interferometer is tested on a floating optics table and will eventually be hung on springs to limit vibrations. A box was built around the interferometer to minimize background light and air currents. The interferometer was carefully aligned and collimated to reduce optical errors. Finally, the ability of Houghton's interferometer system to subtract out a background image allows for the elimination of extraneous fringes.

Chapter 4: FUTURE CONSIDERATIONS

4.1 Current Status

Currently the Houghton College interferometer is built and aligned. Interferograms can be seen using a test mirror and pictures of these interferograms can be taken with the camera. The electronics have also been built and are functioning properly, outputting a -30V to +30V triangle wave. The LabView program has been written and tested with sample data that was taken by hand. The topography produced by the LabView program is shown in Figure 26.



Figure 26. Topography output from LabView program. The left image is one of the interferograms that were taken and inputted into LabView. The right image is the topography outputted by LabView.

The deposition chamber has also been constructed and researchers are in the process of trying to make a thin film. The inner and outer frames from Figure 16 have been built as well. While the deposition chamber has been attached to the inner frame, the interferometer has not. It will be attached as soon as preliminary testing is complete.

4.2 Future Goals

The main test still needed to be completed is a live test. This will include use a testing mirror, Yawcam, and LabView all together. The test will be live and fully automated by the computer. In addition, two consecutive tests should be run and the two measured topographies subtracted from one another. This should result in a blank image, verifying the interferometer's reproducibility. From there, the interferometer will be mounted and aligned to the deposition chamber. Then the only thing left before the interferometer and deposition system is fully functional is the production of a thin film.

Once the system has been completed, more improvements are still planned to be made. One of these involves the heating of the films. Currently when a film is heated, the entire deposition chamber is heated, causing it to expand. This is a concern with using the interferometer because if the chamber window is heated, it will curve, thus affecting the measured interferograms. A heat shielding system would need to be designed and installed; a heat mirror has already been acquired for this purpose.

The interferometer system could also be used for measuring the thickness of films. The deposition chamber is already equipped with a deposition rate monitor that predicts approximate film thickness, but the interferometer could measure this at a much higher resolution. This would be especially useful for stepped films, which the deposition chamber is capable of producing.

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