



**UNIVERSIDAD DE INVESTIGACIÓN DE  
TECNOLOGÍA EXPERIMENTAL  
YACHAY**

**ESCUELA DE CIENCIAS ESCUELA DE CIENCIAS  
FÍSICAS Y NANOTECNOLOGÍA**

**TÍTULO: STUDY OF THE TECHNIQUE OF  
MEASURING SILICA OXIDE FILMS WITH THE  
OPTICAL REFLECTION METHOD, FOR ITS  
POSSIBLE IMPLEMENTATION USING LOW-  
COST MATERIALS.**

Trabajo de integración curricular presentado como  
requisito para la obtención  
del título de Ingeniero en nanotecnología.

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Urcuquí, diciembre 2020

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## **Dedicatoria**

Quiero dedicar este trabajo a mi madre por apoyarme en todo momento y enseñarme que no importa cuán difícil sea el problema o lo cansado que me encuentre, siempre se puede dar un poquito más.

Fabian Patricio Tinoco Mosquera

## **Resumen**

En el siguiente trabajo vamos a describir el funcionamiento de un equipo capaz de medir el espesor de película de una muestra de óxido de silicio sobre un sustrato de silicio, este equipo utiliza una técnica de caracterización óptica llamada reflectancia espectral.

Para lograr eso, primero abordamos la teoría necesaria para comprender el funcionamiento del equipo, esto lo hacemos para cada parte del equipo de medición, las cuales son: fuente de luz, divisor de fibra óptica, espectrómetro y muestra. A continuación, presentamos una propuesta de diseño para un dispositivo con las mismas características y que utiliza la misma técnica de caracterización. Para la elección de los componentes de nuestra propuesta, se toma en cuenta diferentes parámetros técnicos y de costos.

Se espera que en el futuro esta propuesta de diseño se pueda utilizar para construir e implementar este equipo en los laboratorios de la Universidad Yachay Tech. Esta herramienta proporcionará a los estudiantes y profesores la capacidad de caracterizar deposiciones de película delgada de óxido de silicio para sus actividades de aprendizaje o investigación. De esta manera, se creará un impacto positivo para la Universidad Yachay Tech.

### **Palabras Clave:**

Espesor, Reflectancia espectral, Interferencia, Equipamiento, Fibra óptica

## **Abstract**

In the following work we are going to describe the operation of an equipment capable of measuring the film thickness of a silicon oxide sample placed on a silicon substrate, this equipment uses an optical characterization technique called spectral reflectance.

In order to achieve that, first we address the theory necessary to understand the operation of the equipment, we do this for each part of the measurement equipment such as: light source, fiber optics splitter, spectrometer, and sample. Later we present a design proposal for a device with the same characteristics and that uses the same characterization technique. The choice of components for our proposal is taken taking into account different technical and cost parameters.

It is expected that in the future this design proposal can be used to build and implement this equipment in the laboratories of Yachay Tech University. This tool will provide students and teachers with the capacity to characterize silicon oxide thin film depositions for their learning or research activities. In this way, a positive impact will be created for Yachay Tech University.

### **Key Words:**

Thickness, Spectral reflectance, Interference, Equipment, Fiber optic

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# Chapter 1

## Introduction

Nowadays, the manufacture and use of films of dielectric materials in various areas of science and technology is very wide. Therefore, it is common that in laboratories of different kinds where dielectric films are used or developed, there is one or more equipment to measure the thickness of the films. Within the range of thickness gauges are ellipsometers, interferometers, and refractometers. This titulation work deals with refractometers from a theoretical and practical point of view. Then, we propose the construction of one of these equipment, taking into account technical and economic parameters.

A thin-film is a layer of material ranging from a few nanometers to several micrometers in thickness. Thin-film can be produced through a variety of processes, for example: chemical vapor deposition (CVD), physical vapor deposition (PVD), evaporation through vacuum sublimation, spin coating and thermal oxidation process can be used to create oxide thin films. These examples are just a few ways to create a thin-film .

Some applications for thin films are electronic semiconductor devices; it includes integrated circuit chips, micro-fabricated mechanisms, micro-electromechanical systems (MEMS), micro-electronic optical systems, as well as light-emitting diodes (LEDs). Other applications include optical coatings, photovoltaic solar cells, communications, energy generation, and energy conservation, and thin-film batteries<sup>9 10</sup>. The thin-film industry is growing as quickly as scientists and engineers can find applications for them, this field will become even more relevant as they try to make appliances and systems smaller and thinner<sup>10</sup>.

During a thin film's manufacturing process, the thin film must go through thorough quality control of its

characteristics, such as thickness, roughness, and optical constants. Quality control ensures that the thin film has the correct characteristics for the application where it will be used<sup>11</sup>, these characteristics must be measured during and after thin-film fabrication.

Optical techniques are the best way to determine the characteristics of a film, these techniques measure how light interacts with the thin film and can determine the thickness, roughness, and optical constants of the film. This kind of techniques are the preferred method for measure thin-film because they are accurate, nondestructive and require little or no sample preparation.

The two most common types of optical measurement are spectral reflectance and ellipsometry. Spectral reflectance: It is the method on which we rely to carry out this work, this consists of measuring the amount of light reflected from a thin film over a range of wavelengths, with the incident light perpendicular to the sample. Where the amplitude and periodicity of a thin film's reflected light (reflectance) are determined by the film thickness, optical constants  $n$ , and  $k$ , where  $n$  is the refractive index, and  $k$  is the extinction coefficient of material and other properties such as interface roughness.

In cases where there is more than one interface, it is not possible to solve from film properties in closed form, nevertheless, is it possible to solve for  $n$  and  $k$  at each wavelength individually. The film's properties are determined by calculating reflectance spectra based on trial values of thickness and the  $n$  and  $k$  model parameters, then adjusting these values until the calculated reflectance matches the measures. In practice, mathematical models describe  $n$  and  $k$  over a range of wavelengths using only a few adjustable parameters.<sup>12</sup>.

Ellipsometry : On the other hand ellipsometry is almost the same, but in this case, the light reflected is measured at non-normal incidence and at two different polarizations.

Even when Spectral reflectance can measure the thickness of a broad range of thin films, this technique has limitations on the minimum thickness it can measure. Whenever there is less than one reflectance oscillation like occurs in a 1 to 30 nm thickness range films, less information is available to determine the adjustable model parameters. Therefore, the number of film properties that may be determined decreases for very thin films. If we tried to solve for too many parameters, a unique solution could not be found; more than one possible combination of parameter values may result in a calculated reflectance that matches the measured reflectance<sup>12</sup>.

As we can notice, spectral reflectance is much simpler and less expensive than ellipsometry. For these reasons, we chose spectral reflectance as the technique that we will use to measure thin film's of silicon oxide placed on a silicon substrate.

In the following chapters, we will talk about the theory necessary to understand the measurement equipment's working principle. We will also talk about each of the components that this type of measurement equipment has and its function. Subsequently, based on the components of measurement equipment available on the market, we will propose alternative components with lower cost that fulfill the same function as the available equipment.

## **1.1 Problem Statement**

Given the high importance of dielectric films, it is firstly required to know their thickness by using a measurement method such as refraction. For which its operation will be detailed in this thesis in the first place.

## **1.2 General Objective**

This work aims to propose the design of a film thickness measuring equipment capable of measuring silicon oxide films on a silicon substrate using low-cost components by using spectral reflectance .

## **1.3 Specific Objectives**

1. To describe the theoretical bases, working principle, and components of commercial measuring equipment capable of measuring a silicon oxide film's thickness on a silicon substrate.
2. To design a equipment that uses the same working principle and fulfills the same measurement function according to commercial available equipment.
3. To explain the software required for measurements.



## Chapter 2

# Methodology

### 2.1 Thin film measurement equipment working principle

In this section, we will briefly describe the components and the working principle of a device that uses spectral reflectance to measure the film thickness of silicon oxide on top of silicon substrate.

The equipment components are the following: light source, fiber optic lines, fiber optic splitter, spectrometer, and computer as is shown in Figure (2.1). First of all, the light source produces a light beam. Next, an optical fiber conducts its light to a fiber optic splitter.

The fiber optic splitter used by this type of equipment consists of two fiber optic lines, one input, and one output; these lines joined to form a tip placed perpendicular to the sample Figure (2.2).

Then this light beam lies down on the surface of a thin-film sample, and a phenomenon called Thin-film interference occurs. Thus, part of the light is reflected from the upper interface, and part is transmitted to a new medium with a different index of refraction. In this new medium, light again is reflected and transmitted when it meets another interface.

The light reflected from the upper and lower interface will interfere either constructively or destructively. The degree of interference depends on the phase difference that exists between the reflected wave from the upper interface concerning the lower one. Subsequently, the fiber optic output line collects the light reflected by the sample, and conducts it towards the spectrometer by another optical fiber. Afterwards, the spectrometer measures the reflectance

data obtained.

Finally, a computer with appropriate software connected to the spectrometer analyzes the reflectance data and determines the film thickness of the silicon oxide sample Figure (2.1).

In the following sections, the equipment components and its working principle will be described more extensively.

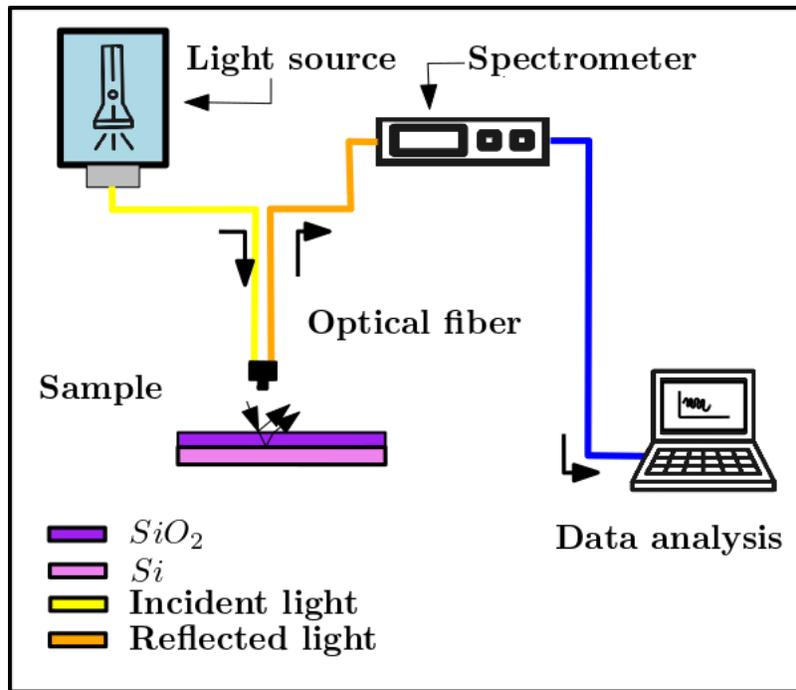


Figure 2.1: Schematic representation of the operation of a device that uses spectral reflectance to measure the film thickness of silicon oxide on top of silicon substrate.

## 2.2 Light source

Since we are going to use an optical method to measure the thin film thickness of a sample, we first need a light source (electromagnetic waves). This light source produces a light beam which interacts with the sample and produces reflection both in the upper and lower interface of the sample, a wavelength range commonly used in this type of measurement equipment is between (400 nm – 700 nm).

A wave is a disturbance of one or more fields, therefore field values oscillate about a stable equilibrium value<sup>13</sup>. Traveling sinusoidal waves are represented in terms of its velocity  $v$ , frequency  $f$  and wavelength  $\lambda$ , in the  $x$ -direction as:

$$Y(x, t) = A \cos(kx - \omega t) = A \cos(k(x - vt)) . \quad (2.1)$$

Where  $A$  is the amplitude of the wave,  $x$  is the position,  $t$  is time,  $k$  is wave number and  $\omega$  angular frequency, these terms are related to each other in the following way, Equation (2.2, 2.3) :

$$k = \frac{2\pi}{\lambda} = \frac{2\pi f}{v} = \frac{\omega}{v} , \quad (2.2)$$

$$\lambda = \frac{2\pi}{k} = \frac{2\pi v}{\omega} = \frac{v}{f} , \quad (2.3)$$

$$Y(x, t) = A \cos\left(2\pi\left(\frac{x}{\lambda} - ft\right)\right) = A \cos\left(\frac{2\pi}{\lambda}(x - vt)\right) . \quad (2.4)$$

According to the previous definition, an electromagnetic wave is an electromagnetic field that propagates through space, it consists in oscillations of an electric  $\mathbf{E}$  and magnetic  $\mathbf{B}$  fields. In a homogeneous isotropic media, these fields are mutually perpendicular and also are perpendicular to the wave propagation direction, so it is a tridimensional wave. In vacuum, such waves travel at the speed of light<sup>14</sup> ( $c = 299792458m/s$ ) .

## 2.3 Optical Fiber

Optical fiber is used to carry light from the light source to the sample and also to recollect the reflected light from the sample and lead this light to the spectrophotometer, where it will be analyzed.

Optical fiber is a transparent fiber made of silica or plastic<sup>15</sup>; it consists of a core surrounded by a cladding material with a lower index of refraction than core. The working principle of optical fibers is "the total internal reflection"; its effect is used to confine the light inside the core of the fiber, hence light is bouncing back and forth of the core-cladding boundary of the optical fiber<sup>16</sup>.

Total internal reflection occurs when light hits a boundary between core and cladding at angles greater than critical angle so light is completely reflected, its critical angle is the smallest angle of incidence at total internal reflection occurs<sup>17</sup>, and is defined by:

$$\theta_c = \arcsin(n_2/n_1) . \quad (2.5)$$

Where  $\theta_c$  is the critical angle, and  $n_1, n_2$  are the refractive indices of each medium, respectively.

In order to accomplish the task of irradiating the sample with light and collect the reflected light from a sample, an arrangement with two optical fibers is used, one for the input light and one for the output light, these two fibers are arranged together one next to the another. Apart from that, they are placed on a base which fixes these fibers in a normal position to the measured substrate.

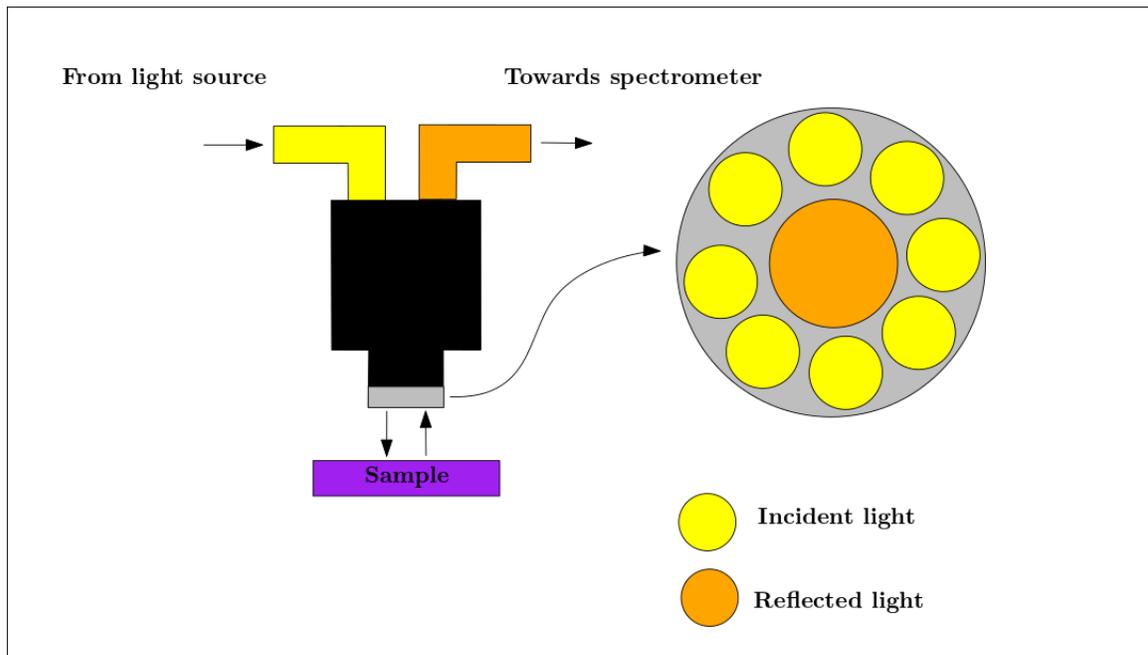


Figure 2.2: This figure shows a schematic representation of the two optical fibers used to irradiate and capture the reflected light from the sample. The yellow part represents the optical fiber through which light travels from the source to the sample, and orange represents the optic fiber that collects the light reflected by the sample and leads it to the spectrophotometer.

The arrangement of the two fibers together is of the coaxial type, as shown in Figure (2.2), where the optical fiber in the center carries the light reflected by the sample towards the spectrometer, where it will be analyzed. While the fibers that surround it, transports light from the light source to the sample. This distribution of the fibers is beneficial for the acquisition of information since it promotes a higher reception of reflected light<sup>12</sup>, also, it is important to mention that this type of optical fiber arrangement corresponds to the one found in the Filmetrics F20 thin-film

measure equipment.<sup>12</sup>.

Note that when we talk about two optical fibers, they are not just two. In really it is one fiber surrounded by many others, however we talk about two fibers to explain the concept of an input and output channel of light .

## 2.4 Interaction of light with the sample

In this section, we are going to talk about how the electromagnetic waves originated from the light source interact with the silicon oxide sample and provide the necessary information to determine the thickness of the sample.

### 2.4.1 Incidence of light on the sample

First, we have the electromagnetic waves that directly touch the sample, when this happens the waves experience a change in the displacement medium, in this case, they change of propagating in air, to propagating in a thin layer of silicon oxide. Electromagnetic waves are highly influenced by the properties of the medium in which they travel.

Taking this into account, one of the most relevant properties of a medium is its refractive index (or refraction index), which describes how fast light travels through certain material. It is a dimensionless number and is defined as the ratio of the speed of light in vacuum "c", and the phase velocity "v" of light in the medium<sup>18</sup>, the phase velocity is the speed at which the crests of the wave moves. From equation (2.6), we can appreciate that as the refractive index increase the speed of light in the material decreases.

$$n = \frac{c}{v} . \quad (2.6)$$

Refractive index varies depending on the wavelength; this causes white light to split into constituent colors when refracted, this is called dispersion, as discussed below. When light propagates in a real medium, part of it is attenuated due to the medium. This attenuation factor is taken into a count by using the complex form of the refractive index. It is defined as follows

$$n = n + i\kappa , \quad (2.7)$$

where "n" indicates the phase velocity, and " $\kappa$ " the extinction coefficient. The extinction coefficient expresses

the amount of attenuation that the electromagnetic waves suffer when it propagates in a particular material.

The attenuation of an electromagnetic wave can be appreciated by placing the complex refractive index in a wave number " $\kappa$ " for the electric field of electromagnetic wave<sup>18</sup>

$$k = \frac{2\pi n}{\lambda_0} ; n = n + i\kappa , \quad (2.8)$$

$$E(z, t) = \text{Re}[E_0 e^{i(kz - \omega t)}] , \quad (2.9)$$

$$E(z, t) = \text{Re}[E_0 e^{i(\frac{2\pi(n+i\kappa)z}{\lambda_0} - \omega t)}] , \quad (2.10)$$

$$E(z, t) = e^{-\frac{2\pi\kappa z}{\lambda_0}} \text{Re}[E_0 e^{i(kz - \omega t)}] . \quad (2.11)$$

Where  $E(z, t)$  is the electric field of an electromagnetic wave that moves in the z-direction,  $E_0$  is a vector in the x-y plane; with the units of an electric, Re indicates real part, and  $\lambda_0$  is the vacuum wavelength.

From equation (2.11), we can appreciate that the extinction coefficient gives an exponential decay or, in other words, an attenuation of the electromagnetic wave. Moreover, dielectric materials, like silicon oxide, have low DC conductivity and dielectric loss at low frequencies, which means almost no absorption. Even so, at higher frequencies, like visible light, dielectric losses increase. On the other hand metals have high conductivity and dielectric losses.

Besides, the refractive index determines how light refract when it passes into a different refractive index material, Snell's law describes this phenomenon by equation (2.12) as discussed below. Additionally, this is related to the reflection effect since it determines the amount of light that is reflected from an interface, as well as, its intensity due to the Fresnel's equations.

### 2.4.2 Reflection and refraction in the sample

In the following section, we are going to describe the phenomena of reflection and refraction that occurs in the sample. Reflection occur at the upper and lower interface of the sample, while refraction occurs within the silicon oxide layer, as we will see below<sup>12</sup>.

Reflection and refraction are two phenomena of waves that occur together. These occur when a wave is propagating through one medium and touches the interface with another medium<sup>19</sup>, therefore, part of the wave is reflected in the same medium, and another part is refracted in a new medium. We are going to study refraction and reflection from a geometric point of view.

### 2.4.3 Reflection:

Reflection is the change in the direction of the wave; it change takes place in the same medium in which propagates and stays after an impact on the surface of another medium<sup>20</sup>. It is governed by two laws: First, the incident ray, the reflected ray and the normal to the surface at the point of incidence, lay the same plane ; Second, the angles of the incident  $\alpha$  and reflection  $\beta$  are equal  $\alpha = \beta$  . Reflection does not change the wave speed, frequency, or wavelength.

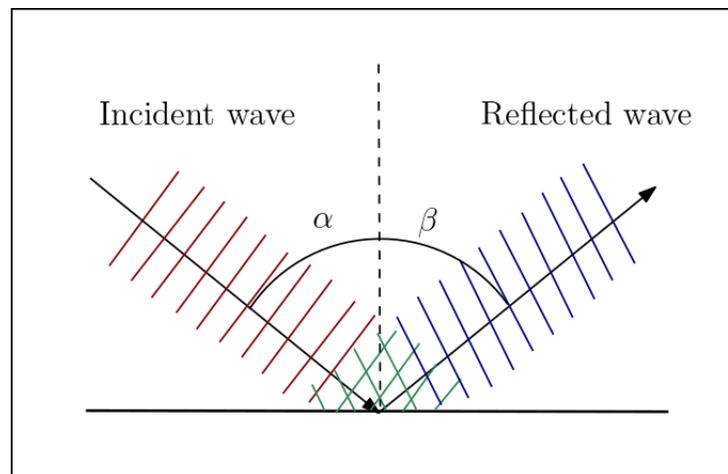


Figure 2.3: Reflection on a surface at certain angle.

### Geometric deduction:

To carry out a geometric verification of these phenomena, we first have to mention the Huygens-Fresnel Principle, this principle allows us to analyze wave propagation problems, with which we can explain phenomena such as reflection, refraction and diffraction. The statement says the following:

"Every point on an initial wavefront can be considered as a source of secondary spherical waves that extend in

all directions with the same speed, frequency, and wavelength as the wavefront from which they" originate<sup>21</sup>.

If we establish that the source from which our wave comes is far enough from the point of incidence on the reflecting surface, this wave can be considered as a plane wave.

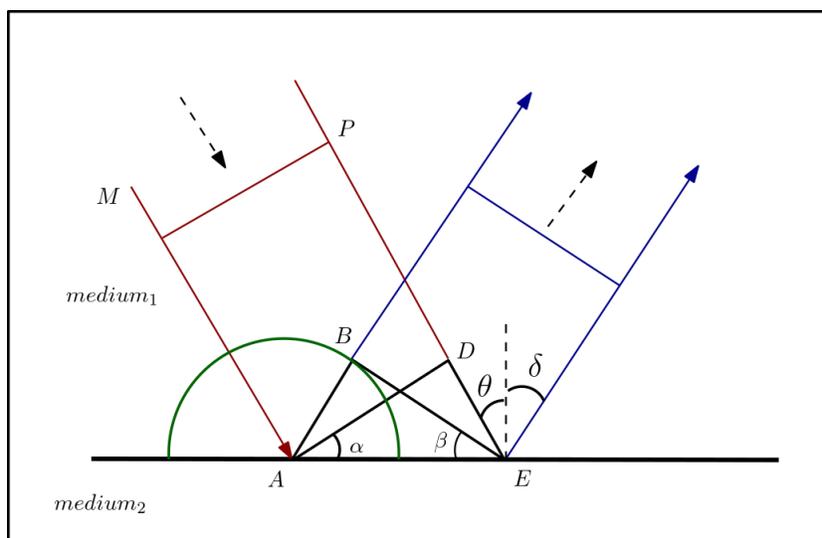


Figure 2.4: Graphic representation of reflection based on the Huygens principle

As we can see in Figure (2.4), the M point will be the first to collide with the surface, this will occur at the incidence point A, where according to the Huygens-Fresnel principle, it will become a wave emitter; this new wave will propagate at the same speed of the incident wave. On the other hand, when D of the incident wave touches the point E on the surface (travel the distance DE) then the point E will also emit a secondary wave.

Given the above scenario, we can form two triangles, ADE and ABE. These triangles are equal by having their hypotenuse and sides DE and AB equal. Therefore, the angles  $\alpha$  and  $\beta$  are also equal, so also the angles  $\theta$  and  $\delta$  are also equal since they correspond to perpendicular sides of the angles  $\alpha$  and  $\beta$ . Thus the phenomenon of reflection is explained.

#### 2.4.4 Refraction:

Refraction is the change in direction of wave motion. This occurs after passing from one medium to another in which it propagates at different speeds, in our case it would be the change from air to a silicon oxide medium.<sup>22</sup>. This is

governed by two laws:

First, the incident ray, the refracted ray and the normal to the surface at the point of incidence are in the same plane. Second, the Snell's law for refraction that establishes relationship between the angle of incidence  $\theta$ , the angle of refraction  $\delta$ , and the wave velocities in media 1 and 2,  $v_1$  and  $v_2$  respectively, according to the following formula<sup>23</sup>.

$$\frac{\sin(\theta)}{\sin(\delta)} = \frac{v_1}{v_2} = \frac{n_2}{n_1} = n_{2,1} . \quad (2.12)$$

Where  $n_{2,1}$  is the ratio of the refractive index of the second medium to the first.

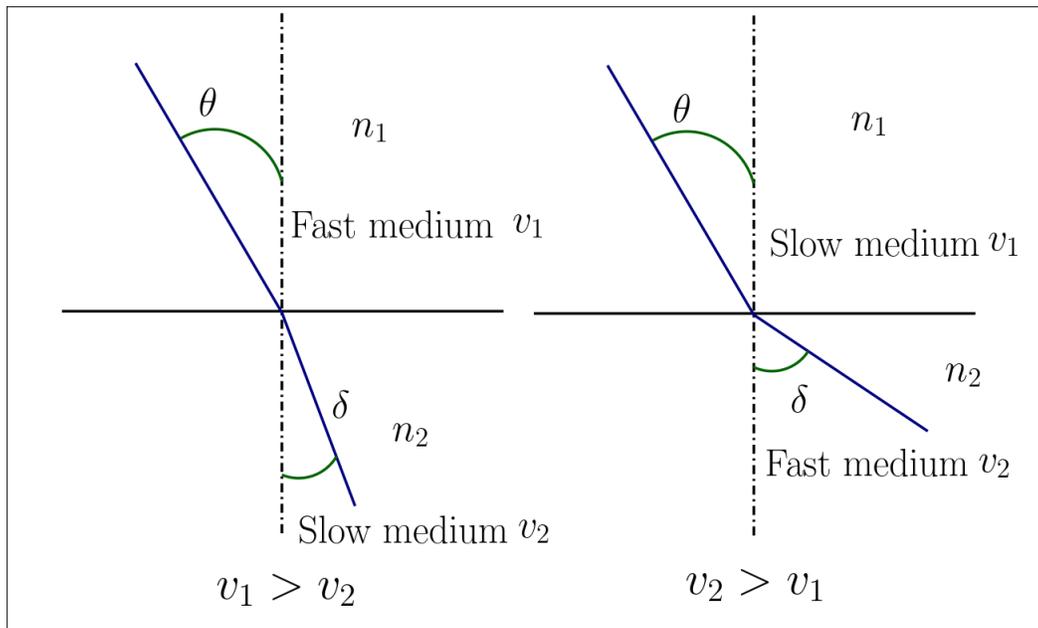


Figure 2.5: Geometric representation of refraction.

To be a little more specific, refraction occurs when a wave traveling at a certain speed in one medium passes to another medium where its speed is different.

As can be seen in Figure (2.5), when light passes from a medium where it propagates fast  $v_1$  to another where it propagates slower  $v_2$ . In the case ( $v_1 > v_2$ ), we see how the refracted ray approaches to the normal of the incidence

point, while in the other case  $v_1 < v_2$ , we see how the refracted ray moves away from the normal.

### Geometric deduction:

In order to explain the refraction phenomenon, we first have to talk about the Snell's law, which, as its name implies, was discovered experimentally by the Dutch scientist Willebrord Snell van Royen (1580 - 1626). On the other hand, the scientist Huygens was the one who gave a mathematical explanation to this experimental law, this he did using the principle of secondary waves:

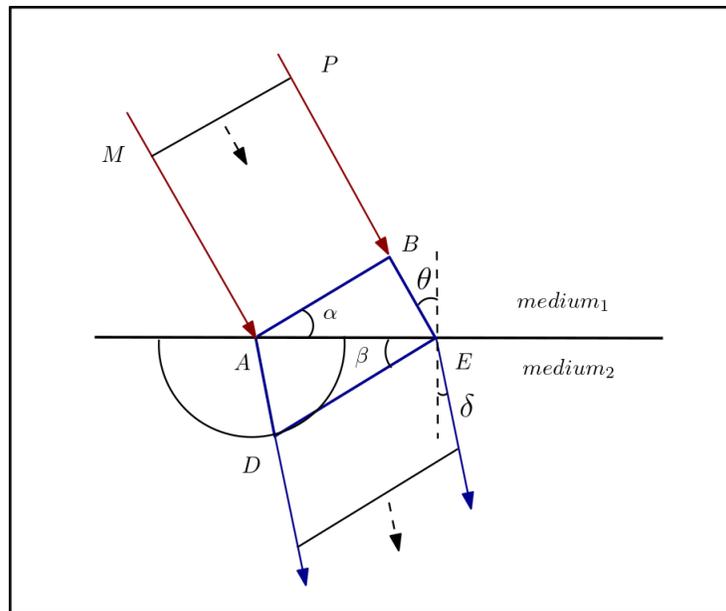


Figure 2.6: Geometric representation of refraction based on the Huygens principle.

Let us suppose that a plane wave impinges the interface between two media with speed  $v_1$  and  $v_2$ , respectively. So in the wavefront MP, as we can see in Figure (2.6), the point M is the first to reach the surface of the interface of these two media at point A, which through the Huygens principle, becomes the emitting point of new secondary waves. As a result of it, secondary waves enter in medium two and travel distance AD at the same time as point B of incident wave AB travel distance BE, thus we form two equal triangles BAE and AED. Since the distance BE and AD are traveled at the same time (t), we can equalize these expressions and obtain the relationship between speeds and traveled distances.

$$v_1 \cdot t = BE , \quad (2.13)$$

$$v_2 \cdot t = AD , \quad (2.14)$$

$$\frac{v_1}{v_2} = \frac{BE}{AD} , \quad (2.15)$$

Applying the definition of sine in triangles BAE and AED and since the angles  $\alpha$  and  $\beta$  are equal to the angles  $\theta$  and  $\delta$ , we obtain the following relationships:

$$\sin(\alpha) = \frac{BE}{AE} , \quad (2.16)$$

$$\sin(\beta) = \frac{AD}{AE} , \quad (2.17)$$

$$\frac{\sin(\alpha)}{\sin(\beta)} = \frac{BE}{AD} . \quad (2.18)$$

Equating equation (2.16) and (2.17) we obtain the Snell's law:

$$\frac{\sin(\alpha)}{\sin(\beta)} = \frac{v_1}{v_2} = \frac{n_2}{n_1} = n_{2,1} , \quad (2.19)$$

where  $n_{2,1}$  is the index of refraction of medium 1 with respect to medium 2.

In the refraction phenomenon, the wave frequency does not change, but when its propagation speed changes, it alters the wavelength, as we will see next. The speed of a wave "v" (phase velocity) depends on the medium in which it propagates. For example, the speed of light in a medium is less than in a vacuum, this implies that for the same frequency, it corresponds to a shorter wavelength in a medium than in the vacuum.

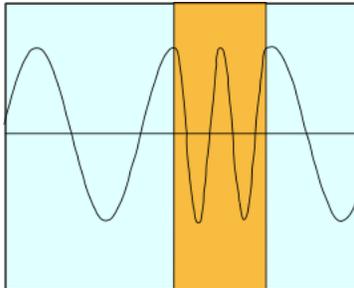


Figure 2.7: Contraction of wavelength by changing the medium in which it propagates.

The wave speed can change from one medium to another, as the speed varies depending on the wavelength. For electromagnetic waves, the wave speed in a medium is governed by its refractive index  $n$  according to:

$$v = \frac{c}{n(\lambda_0)} . \quad (2.20)$$

The equation (2.20) gives us information on how much the speed of light decreased when passing through a medium with the refractive index  $n$ . Where  $v$  is the speed of the wave,  $c$  is the speed of light in a vacuum, and  $n(\lambda_0)$  is the refractive index of the medium at wavelength  $\lambda_0$ , this wavelength  $\lambda_0$  measured in a vacuum.

The corresponding wavelength in the medium  $\lambda$  is:

$$\lambda = \frac{\lambda_0}{n(\lambda_0)} , \quad (2.21)$$

equation (2.21) tells us how much  $\lambda_0$  contracts or expands, due to refractive index  $n(\lambda_0)$  of the medium. Normally wavelengths reference measurements are in vacuum unless propagation medium is specified.

## 2.5 Wave interference

In this section, we are going to describe the interference phenomenon. First, we will approach the phenomenon in a general way, and then we will end describing the thin-film interference.

Interference phenomenon describes two waves that can come together or overlap to form another wave, this is called the superposition principle, the resultant wave may be of greater, less than or equal to the amplitude of the initial waves.

When the interference phenomenon occurs, it can be constructive or destructive. Constructive interference occurs when the phase difference between the waves is an even multiple of  $\pi$ . On the other hand, destructive interference occurs when the difference is an odd multiple of  $\pi$ .

The interference effects can be appreciated in all kinds of waves, such as acoustic waves, radio, and light. Nevertheless, we will focus on electromagnetic waves (light) with which we will explain later the phenomenon of thin layer interference.

### 2.5.1 Mathematical formulation of two waves interference

Next, we are going to analyze the sum of two waves  $Y_1$  and  $Y_2$  that travel to the right along the X-axis with the same amplitude, where  $\delta$  is the phase difference between the waves; this measured in radians.

$$Y_1(x, t) = A \cos(kx - \omega t) , \quad (2.22)$$

$$Y_2(x, t) = A \cos(kx - \omega t + \delta) , \quad (2.23)$$

Applying the trigonometric identity of the sum of cosines (2.25) and taking into account that we designate the letter a (2.26) and b for equation (2.27) for convenience to avoid negative angles:

$$Y_1 + Y_2 = A[\cos(kx - \omega t) + \cos(kx - \omega t + \delta)] , \quad (2.24)$$

$$\cos(a) + \cos(b) = \cos\left(\frac{a-b}{2}\right) \cos\left(\frac{a+b}{2}\right) , \quad (2.25)$$

$$a = (kx - \omega t + \delta) , \quad (2.26)$$

$$b = (kx - \omega t) . \quad (2.27)$$

we obtain the sum of two waves:

$$Y_1 + Y_2 = 2A \left[ \cos\left(\frac{\delta}{2}\right) \cos\left(kx - \omega t + \frac{\delta}{2}\right) \right] , \quad (2.28)$$

from equation (2.28) we can notice that (2.22) the amplitude of the sum of waves is proportional to  $\frac{\delta}{2}$ . Taking this into account, we can analyze the cases where there will be constructive or destructive interference, and the intermediate cases between them<sup>13</sup>.

### 2.5.2 Constructive interference:

Occurs if the phase difference  $\delta$  of the initial waves is an even multiple of  $\pi$

$$\delta = -2\pi, 0, 2\pi ; \left| \cos\left(\frac{\delta}{2}\right) \right| = 1 , \quad (2.29)$$

so the sum of the two waves is a wave with twice the amplitude

$$Y_1 + Y_2 = 2A \cos(kx - \omega t) , \quad (2.30)$$

### 2.5.3 Destructive interference:

This occurs if the phase difference  $\delta$  is an odd multiple of  $\pi$

$$\delta = -3\pi, -\pi, \pi, 3\pi ; \cos\left(\frac{\delta}{2}\right) = 0 , \quad (2.31)$$

here we see that the sum of two waves is zero, so the two initial waves mutually cancel each other <sup>13</sup>

$$Y_1 + Y_2 = 0 . \quad (2.32)$$

### 2.5.4 Intermediate Case

This case occurs if the difference between phases is between these two extremes (even and odd integer of  $\pi$ .), then the magnitude of the displacement of the summed waves is between the minimum and maximum values. (zero and twice the amplitude).

$$Y_1 + Y_2 = 2A \cos\left(\frac{\delta}{2}\right) \cos\left(kx - \omega t + \frac{\delta}{2}\right) . \quad (2.33)$$

Now we have a clear idea of wave interference phenomenon. Therefore, we can focus on a particular case, which is the thin film interference.

## 2.6 Thin-film interference

A thin-film is a layer of a material whose thickness is in the range of microns to nanometers, ( $10^{-6}m - 10^{-9}m$ ). Thin-film interference occurs when light lies down on the surface of a thin-film. Consequently, part of the light is

reflected, and some part is transmitted to a new medium with a different index of refraction ( $n_2$ ). In this new medium, light again reflects and transmits when it meets another interface. Using the Fresnel equations, we can quantitatively know how much light is reflected or transmitted, both at the upper and lower interfaces of our thin film.

The light reflected from the upper and lower interface will interfere either constructively or destructively. The degree of interference depends on the phase difference that exists between the reflected wave from the upper interface concerning the lower one. Furthermore, the phase difference depends on three parameters: the thickness of the layer, the incident angle of light, and the index of refraction of the thin film material.

Depending on the refractive index of the materials that are above and below the thin film, the incident wave will or will not undergo a phase change of  $\pi$  radians, the phase change takes place at the interface between two media. This phase change occurs when the incident wave changes its propagation medium, from a medium with a lower refractive index to a medium with a higher refractive index; on the contrary, this phase change would not occur.

A fascinating example in which we observe thin-film interference is in soap bubbles Figure (2.8,A), where the colors are due to interference between light rays reflected from the thin-film of soap's front and back surfaces, making up the bubble. The color of a particular region of the bubble depends on the film's thickness, ranging from black where the film is thinnest to magenta where it is thickest.

Also we can find another excellent example of thin-film interference in a thin film of oil floating on the water; it shows a color pattern when white light is incident on the film Figure (2.8,B). Likewise, variations in film thickness produce an exciting color pattern<sup>24, 25</sup>

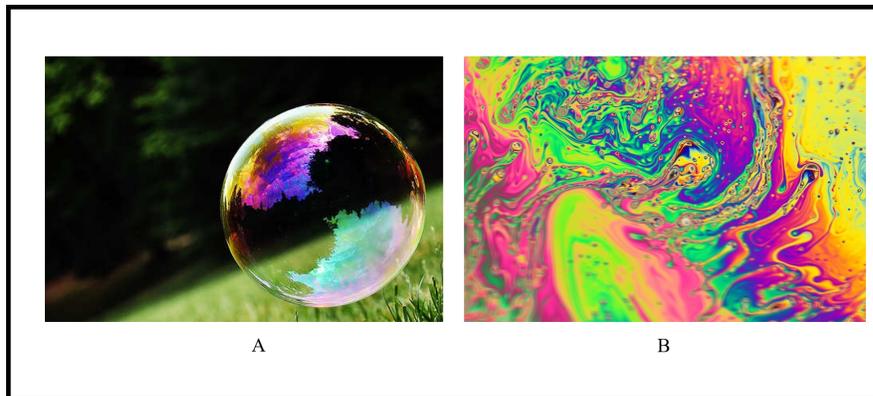


Figure 2.8: (A) Image of a soap bubble, (B) image of a thin film of oil floating on the water. Sources: Reprinted from Thin-film interference Optics Photonics News<sup>1</sup>, and LAWS OF THE UNIVERSE (3) HILOBROW<sup>2</sup>

**Mathematical deduction:** We consider the incidence of light on a thin film. Then it will be reflected at both the upper and lower limits of the film. To establish the interference condition, we have to calculate the optical path difference OPD; this is the subtraction of the path traveled by the light reflected on the upper surface minus the path traveled by the light reflected from the bottom of the thin film.

We take Figure (2.9) as a reference .

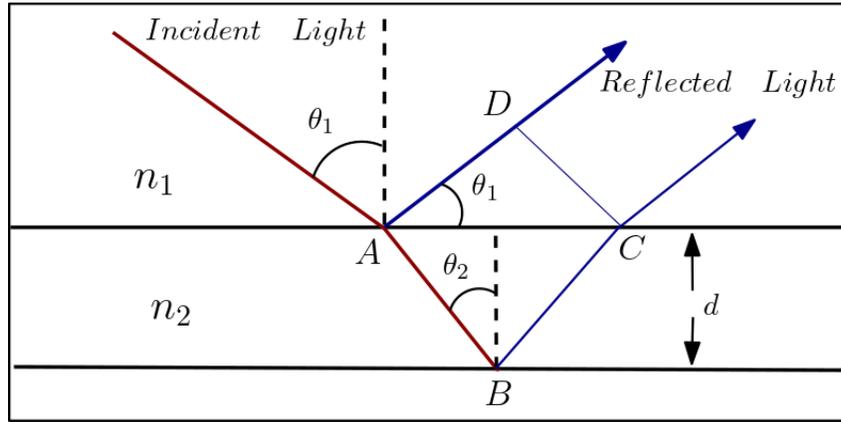


Figure 2.9: Geometric representation of thin film interference.

As we can see, the path that the reflected light travels from the top of the thin-film corresponds to  $AD$  in the medium with refractive index  $n_1$ . In contrast, the distance traveled by the light that is transmitted and reflected from below the thin layer corresponds to  $AB + BC$ , which takes place in a medium with refractive index  $n_2$ .

$$OPD = n_2(\overline{AB} + \overline{BC}) - n_1(\overline{AD}) . \quad (2.34)$$

Where:

$$\overline{AB} = \overline{BC} = \frac{d}{\cos(\theta_2)} , \quad (2.35)$$

$$\overline{AD} = 2d \tan(\theta_2) \sin(\theta_1) , \quad (2.36)$$

using the Snell law,  $n_1 \sin(\theta_1) = n_2 \sin(\theta_2)$  ,

$$OPD = n_2 \left( \frac{2d}{\cos(\theta_2)} \right) - 2d \tan(\theta_2) n_2 \sin(\theta_2) , \quad (2.37)$$

$$OPD = 2n_2d \left( \frac{1 - \sin^2(\theta_2)}{\cos(\theta_2)} \right), \quad (2.38)$$

$$OPD = 2n_2d \cos(\theta_2), \quad (2.39)$$

applying the trigonometric relationships to the triangles formed in Figure (2.9) and using the Snell's law, we obtain the OPD expression:

$$2n_2d \cos(\theta_2) = m\lambda, \quad (2.40)$$

this formula relates the OPD to the wavelength in each medium. For that reason, the expression is written for both  $n_1$  and  $n_2$ . Where "m" tells us how many times that wavelength is present on that optical path, then we will have constructive interference if the optical path difference is an integer multiple of the wavelength in that medium. On the other hand, if the difference is not an integer, then we will have destructive interference<sup>26</sup>.

It is worth emphasizing that the interference condition will depend on the refractive indices of the materials involved, there will be a phase change of 180 degrees if it comes from a medium with a refractive index less than the refractive index of the medium where it is reflected.

#### **White light:**

White light like that from the sun is a type of broadband type which creates interference patterns that appear as bands of different colors. It occurs because the wavelengths that make up white light interact, creating constructive interference for the different thicknesses of the film (if it is not homogeneous), where the spot color of a region of the film would depend on its thickness<sup>25</sup>.

Note: if the refractive index  $n$  increases, the wavelength of the transmitted part of the incident wave will decrease since it propagates at a slower rate in this new medium.

#### **Phase relationship:**

We analyze the relationship of incident rays on a thin film and how they interact with each other, the reflection of ray A from the lower thin-film surface, and the reflection of ray B from the upper surface of the thin film. These two reflections will be combined if they are in phase with each other (offset 0 degrees) or what is the same, if they share the same offset, then the resulting wave will be of higher amplitude (constructive interference).

On the other hand, these two reflections will cancel each other out if they are out of phase concerning the other is 180 degrees (destructive interference). Depending on the degree of phase shift between A and B, wave C will gradually attenuate or magnify.

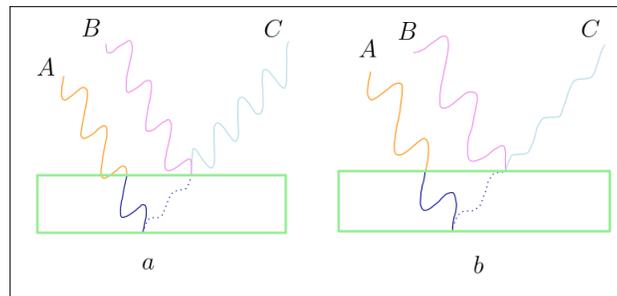


Figure 2.10: Thin film interference where: (a) constructive interference and (b) Destructive interference.

The phase relationship between these two rays will depend on the wavelength of ray A in the medium (thin film) and the film thickness. So then if the optical distance OPD traveled by ray A in the medium (remember the wavelength narrowing) is an integer multiple of the wavelength of ray A, the reflected rays A and B will be in phase and will produce constructive interference. If it is a fractional multiple of the wavelength of ray A, it will produce destructive interference.

## 2.7 Examples

In these examples, we will see two scenarios, the first in which the interference depends more on how we choose the refractive indices of the materials involved and the second where a specific thickness is chosen to provide certain properties to the thin film.

### Soap bubble:

In this case, we will analyze a thin-film of soap, which is between two layers of air (external, internal). The index of refraction of the air is 1, and for simplicity, we will assume the index of refraction of the soap film as higher than 1.

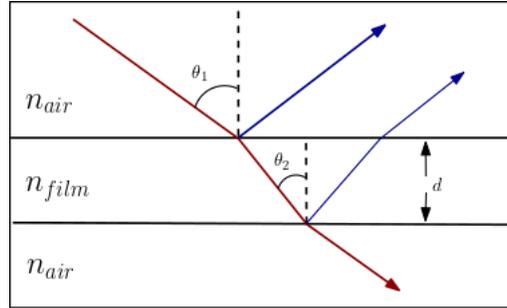


Figure 2.11: Schematic representation of thin film interference for a soap bubble.

The reflection at the upper boundary (air-soap film) introduces a phase change of 180 degrees, because the refractive index of the soap film is higher than the air refractive index, while the reflection that occurs at the lower boundary (soap film- air) will not produce any phase change because the refractive index of the soap film is higher than that of the air inside the bubble.

In this way, we have that the conditions for constructive and destructive interference are the following:

For constructive interference of reflected light:

$$2n_2d\cos(\theta_2) = (m - \frac{1}{2})\lambda , \quad (2.41)$$

For Destructive interference of reflected light:

$$2n_2d\cos(\theta_2) = m\lambda , \quad (2.42)$$

The condition for interference for a soap bubble is the following: to obtain constructive interference, this gap must be "fixed" since the refractive indices induced a phase change. This is accomplished by offsetting 180 degrees the waves, by placing the following condition on the OPD<sup>26</sup>.

$$OPD = (m - \frac{1}{2})\lambda . \quad (2.43)$$

### 2.7.1 Anti-reflection coating

In this type of coating, it reduces reflection and improves light transmission. For this, the film is designed a quarter (1/4) wavelength of the incident light. The refractive index of the coat is greater than that of air and less than the refractive index of the glass  $n_{air} < n_{coating} < n_{glass}$ . In this example, we can see how by controlling the thickness of the thin layer, we can add certain properties to the film.

$$d = \frac{\lambda}{4n_{coating}} , \quad (2.44)$$

a phase change of 180 degrees occurs in both the upper and lower reflection due to the refractive indices of the chosen materials  $n_{air} < n_{coating}$  and  $n_{coating} < n_{glass}$ .

So the equations that describe the interference in this example are as follows:

Constructive interference of light:

$$2n_2d\cos(\theta_2) = m\lambda , \quad (2.45)$$

Destructive interference of light:

$$2n_2d\cos(\theta_2) = (m - \frac{1}{2})\lambda . \quad (2.46)$$

Continue with the analysis; we are going to establish conditions: Firstly, the coating thickness equal to quarter-wavelength of the incident light, and secondly light hits normal to the surface. Thus the reflected waves will be out of phase and will always interfere destructively, which is ideal for an anti-reflective coating<sup>27</sup>.

$$dn_{coating} = \frac{\lambda}{4} \quad \text{and} \quad \theta_2 = 0 , \quad (2.47)$$

So using the above formulas we have that the conditions for constructive and destructive interference are as follows:

For constructive interference:

$$2n_c d \cos(\theta_2) = m\lambda , \quad (2.48)$$

$$2\frac{\lambda}{4}\cos(\theta_2) = m\lambda , \quad (2.49)$$

$$m_1 = \frac{1}{2} , \quad (2.50)$$

For destructive interference:

$$2n_c d \cos(\theta_2) = (m - \frac{1}{2})\lambda , \quad (2.51)$$

$$2\frac{\lambda}{4}\cos(\theta_2) = (m - \frac{1}{2})\lambda , \quad (2.52)$$

$$\frac{1}{2} = (m - \frac{1}{2}) , \quad (2.53)$$

$$m_2 = 1 , \quad (2.54)$$

so 180° offset between the two so at a normal angle of incidence there will only be destructive interference

$$m_1 = \frac{1}{2} ; m_2 = 1 . \quad (2.55)$$

## 2.8 Spectrometer

A spectrometer is an instrument that detects the characteristics of light scattered, emitted or absorbed by atoms and molecules of a sample. A spectrometer uses radiation from the UV-Vis light source that is directed toward a sample, once light interacts with the sample, it can be transmitted, emitted, or scattered by the sample. Then it is collected by mirrors or lenses that lead it towards a dispersing element where separates incoming radiation into different frequencies, and finally a suitable detector analyzes the intensity of light at each frequency<sup>28</sup>.

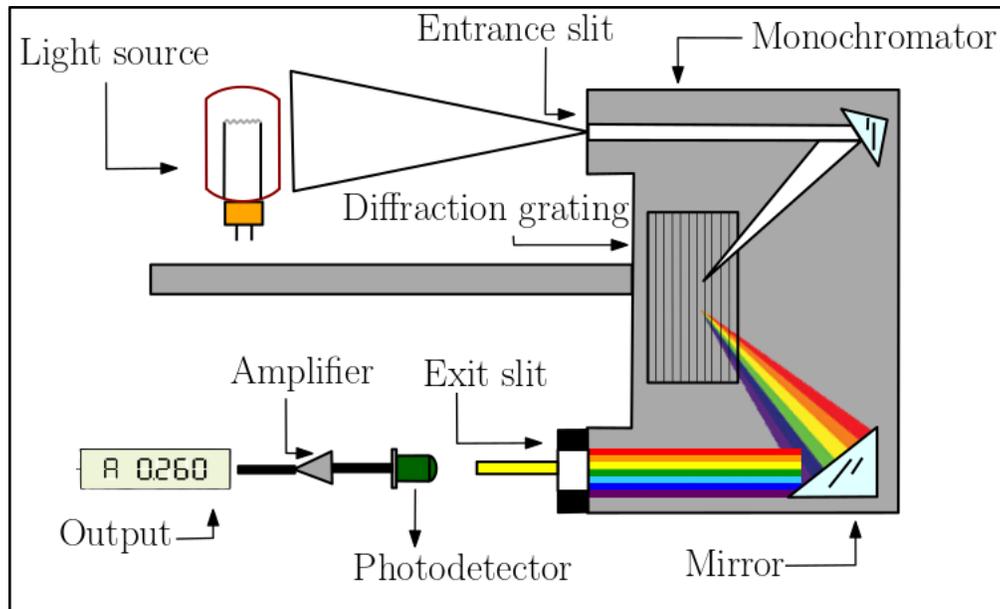


Figure 2.12: Schematic representation of a spectrometer .

The necessary components of a spectrometer are shown in Figure (2.12). They are: light source, monochromator; with its respective dispersive element and its lenses, aperture, photo detector and amplifier, output, and a readout system.

### 2.8.1 Light source:

Since this spectrometer will only carry out measurements in the visible spectrum, it uses a tungsten lamp as a light source.

The tungsten lamp is an excellent and cheapest source in visible and infrared spectroscopy, this lamp works when a current source heats the tungsten filament until it reaches approximately 2900 °C. Thus the tungsten filaments emit continuous radiation from 350 to 2500 nm<sup>29</sup>.

### **2.8.2 Monochromator:**

The monochromator is mainly composed of four components. The first is an entrance slit, through which the light coming from the light source will enter, second is a set of lenses or mirrors, which will direct the light beam towards the dispersive element and then towards the slit.

The third is a dispersive element, which splits polychromatic radiation into its individual wavelengths and isolates these wavelengths into very narrow bands. Two main types of dispersive element are widely used, a prism and a diffraction grating<sup>30</sup>. For example, a prism disperses polychromatic light from the source into its constituent wavelengths due to its ability to reflect different wavelengths to a different extent.

Finally, the fourth component of the monochromator is a wavelength selector or exit slit which is a small hole or slit on a plate that creates a light beam. This plate is connected to a mechanism that allows it to move longitudinally. Through this movement select a range of wavelength with which the equipment will work. The size of the hole or slit can be adjusted larger or smaller.

### **2.8.3 Photodetector/amplifier**

A photodetector is a device that converts light into an electrical signal using the photoelectric effect. The current signal that is measure is proportional to the light intensity. The amplifier boosts the signal to increase sensitivity.

### **2.8.4 Output:**

Spectrophotometers have a numerical readout to quantify the amount of light that comes from the sample. and thus, present it to the user<sup>31</sup>.

## **2.9 Reflectance spectroscopy**

The reflectance spectroscopy technique is used to measure the thin film thickness. This technique can quantitatively measure the intensity of reflected light (reflectance spectroscopy reference). This type of measure is achieved with a suitable setup and a spectrophotometer. The set up is based on the disposition of the sample and a reflected light collection system, as seen in Figure (2.1). Where the sample is arranged perpendicular to the incident light beam,

besides, the optical fiber that will collect its reflected light is also placed in a normal position to the sample<sup>32</sup>.

## 2.10 Data analysis

Numerous mathematical approximations are used to calculate the film thickness from the reflectance spectra of thin films. Nevertheless, we will focus on the following equation (2.56). Its equation allows us to measure the thickness of a single layer film, based on the fact that thin films in the thickness range of about 1 nm to 30 nm as minimum and a maximum of 60 microns, exhibit a constructive-destructive interference pattern as a function of wavelength. Besides, this is the approximate range in which we can make a thickness measurement using the spectral reflectance technique.<sup>33</sup>

$$d = \frac{M(\lambda_1 \lambda_2)}{2 \left[ (\lambda_1 - \lambda_2) (\sqrt{n^2 - \sin^2 \theta}) \right]}, \quad (2.56)$$

where  $d$  is the film thickness,  $M$  is the number of peaks found in the wavelength range,  $n$  is the refractive film index,  $\theta$  is the angle of incidence with respect to the normal of the sample, and finally,  $\lambda_1$  and  $\lambda_2$  are the maximum and minimum wavelengths respectively of the wavelength range.

Light measure by a spectrophotometer is expressed in a graph reflectance vs. wavelength, by a specialized software. Thus counting the number of peaks in the interference spectrum in a particular wavelength range and using equation (2.56), it is possible to determine the thickness of a thin film ( $d$ )<sup>34</sup>.

We discuss equation (2.56) more in-depth in appendix A of this work.

## 2.11 Silicon oxide sample

Oxidation refers to the conversion of the silicon wafer to silicon oxide (SiO<sub>2</sub> or, more generally, SiO<sub>x</sub>). The ability of Si to form an oxide layer is essential since this is one of the reasons for choosing Si for the microelectromechanical (systems) industry. Because this native oxide coating is a high-quality electrical insulator with high chemical stability making it very beneficial for microelectronics<sup>3</sup>. Si exposed to ambient conditions produces a native oxide layer on its surface. Its layer of oxide is approximately 1.5 -3 nm thick at room temperature; also, it stops to grow at 2 or 3 days<sup>35</sup>.

However, this is too thin for the most microelectronics applications, and hence a thicker oxide needs to be grown. In order to obtain a thicker layer of oxide, a silicon oxidation process called thermal oxidation is used.

### 2.11.1 Thermal oxidation:

Silicon dioxide ( $SiO_2$ ) is grown on a pure crystalline silicon wafer in a diffusion furnace using high temperatures (900 to 1200° C). A diffusion furnace consists of a quartz tube large enough to hold several boats of wafers and able to heat to at least 1200° C, using electric heating elements and temperature control systems.

Then the wafers are placed in quartz boats<sup>36</sup> and these boats are placed on a loading dock, which transports the boats into the furnace.

The thermal oxidation process includes three necessary steps:

- The silicon wafers are placed in a heated furnace tube (typically 900 – 1200 degrees C)<sup>36</sup>.
- A source of oxygen (gas or vapor) is pumped into the chamber. This source is either  $O_2$  or  $H_2O$ , respectively.
- The oxygen molecules react with the silicon to form a silicon dioxide ( $SiO_2$ ) layer in and on the substrate.

### 2.11.2 Comparison of dry and wet oxidation:

Next, the two methods of thermal oxidation, dry oxidation, and wet oxidation, are compared. Dry oxidation ( $Si + O_2 \rightarrow SiO_2$ )<sup>3</sup>:

- Oxygen comes from gas source.
- Silicon comes from substrate.
- Oxygen diffuse cross the existing silicon dioxide layer and react with silicon.
- The thicker of the film, the lower the growth rate.

Wet oxidation ( $Si + 2H_2O \rightarrow SiO_2 + 2H_2$ )<sup>3</sup> :

- At high-temperature,  $H_2$  is dissociated to H and H-O.

- H-O diffuses faster in  $SiO_2$  than  $O_2$ .
- Wet oxidation has a higher growth rate than dry oxidation.

### 2.11.3 Formation of oxide:

The rate of formation is dependent on the environment, including the presence or absence of water ( $H_2O$ ) and the temperature. The longer the metal is exposed to the oxygen source ( $H_2O$  or  $O_2$ ), the thicker the oxide layer. The higher the temperature, the faster the reaction rate, and the thicker the oxide.

The oxide layer consumes a portion of the silicon just as rust consumes a portion of the metal. Initially, the growth of silicon dioxide is a surface reaction only. However, after the  $SiO_2$  begins to grow on the silicon surface, new arriving oxygen molecules must diffuse through the  $SiO_2$  layer to get to silicon atoms below the surface<sup>36</sup>.

### 2.11.4 Temperature:

The rate of oxide growth is highly dependent upon temperature. The following graph shows the relationship between temperature, time and oxide thickness Figure (2.13), for both dry oxidation and wet oxidation.<sup>36</sup>

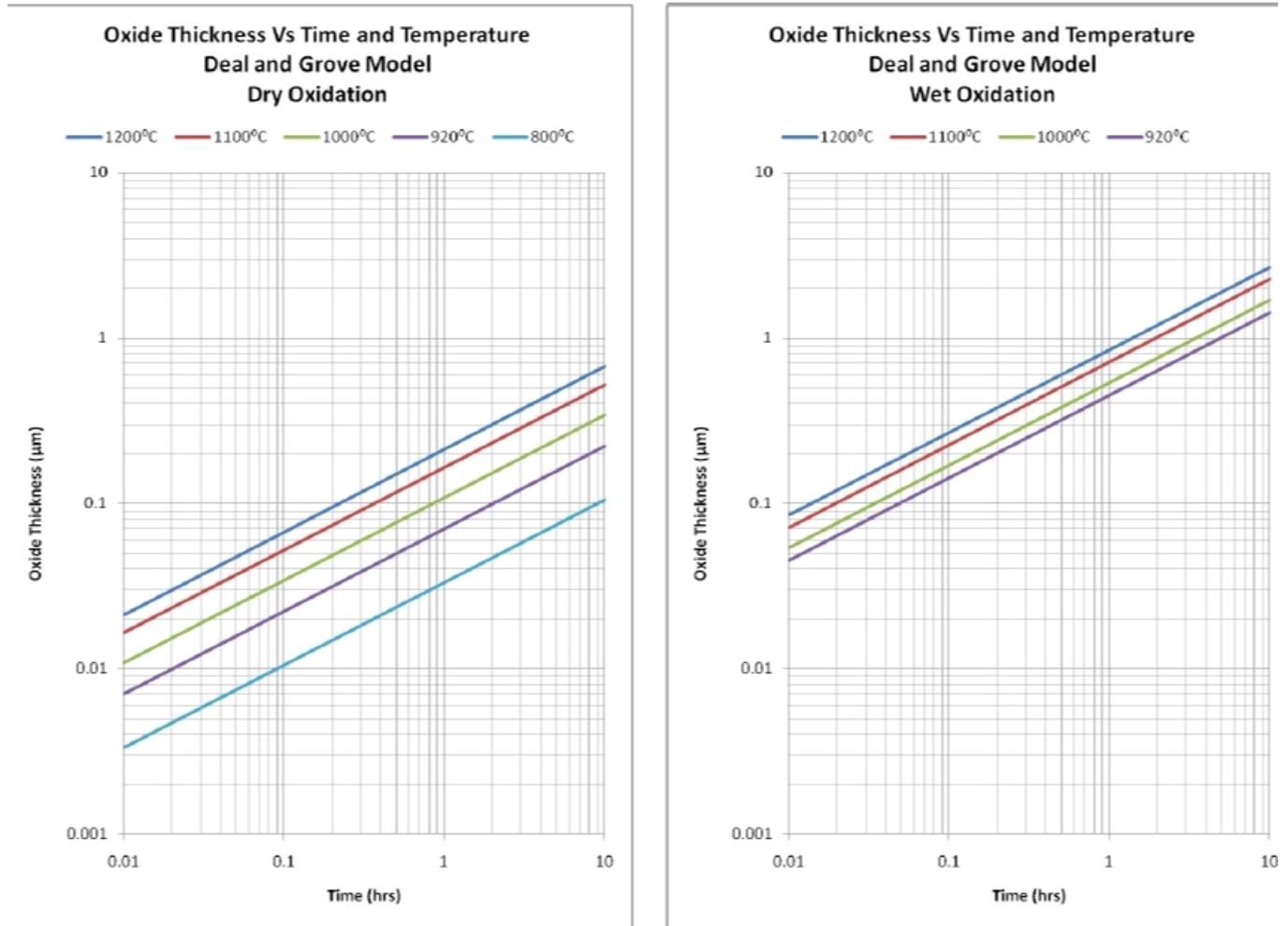


Figure 2.13: These graphs show the growth rate of oxide relative to temperature in a dry oxidation process (left graph) and a wet oxidation process (right graph). Source: Reprinted from Southwest Center for Microsystems Education<sup>3</sup>

### 2.11.5 Oxide's Color:

The color of the oxide coated wafer is caused by the interference of light reflecting off the silicon (below the oxide) and the light reflecting off the top of the oxide surface, as we see before.

As the oxide thickness changes, so do the interference and the oxide's seen color. Color charts have been developed that state the oxide's thickness based on its seen color Figure (2.14).

**Oxide Thickness vs. Color Chart**

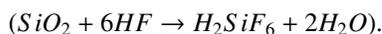
Oxide Thickness [Å]	COLOR	Color and Comments
500		Tan
750		Brown
1000		Dark Violet to red violet
1250		Royal blue
1500		Light blue to metallic blue
1750		Metallic to very light yellow-green
2000		Light gold or yellow slightly metallic
2250		Gold with slight yellow-orange
2500		Orange to Melon
2750		Red-Violet
3000		Blue to violet-blue
3100		Blue
3250		Blue to blue-green
3450		Light green
3500		Green to yellow-green
3650		Yellow-green
3750		Green-yellow
3900		Yellow.
4120		Light orange
4260		Carnation pink
4430		Violet-red
4650		Red-violet
4760		Violet
4800		Blue Violet
4930		Blue
5020		Blue-green
5200		Green (Broad)
5400		Yellow-green
5600		Green-yellow
5740		Yellow to Yellowish (May appear to be light creamy gray or metallic)
5850		Light orange or yellow to pink borderline
6000		Carnation pink

Figure 2.14: In this Oxide Thickness vs. Color Chart figure we can see how each thickness of silicon oxide has a color associated with it. Source: Reprinted from Southwest Center for Microsystems Education<sup>3</sup>

The color we see comes down to the thickness of the film that the light travels through before reaching our eyes; If we look at the film at a non-normal angle, so the light have traveled through the sample more than twice the thickness of the film; the light has therefore traveled through a longer optical path length so, the sample looks a different color.

### 2.11.6 Etching Silicon Dioxide:

Silicon dioxide is readily etched using hydrofluoric acid (HF) according to the following reaction:



HF is a weak acid, for that reason, it has a low value of hydrogen ion concentration  $[H^+]$ . In weak acids, the pH is quite vulnerable to change and these changes in pH result in changes in the etch rate, so we would obtain an irregular and little controlled etching.

This problem can be solved using a buffering solution. The customary buffer for HF is ammonium fluoride ( $NH_4F$ ). Ammonium fluoride is a salt that dissociates to form fluoride and ammonium ions. A typical volume ratio is 20 parts  $NH_4F$  to one-part HF. This stabilizes the acid and allows us to obtain a more controlled etching of the silicon oxide layer<sup>36</sup>.

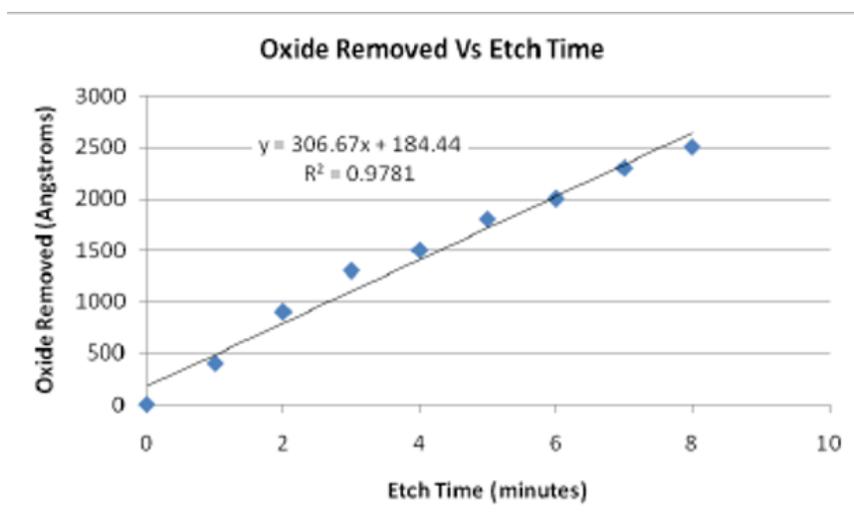


Figure 2.15: Oxide thickness Vs Etch time graph. Source: Reprinted from Southwest Center for Microsystems Education<sup>3</sup>

In this graph Figure (2.15), oxide removed vs.etch time; we can see how approximately for every minute that the sample is submerged in acid, 50 nm of silicon oxide is removed<sup>3</sup>. Using etching, we can start from a silicon oxide sample of a certain thickness and obtain samples of different thicknesses depending on the etching time. These samples of different thicknesses can be used to calibrate our measuring equipment.



## **Chapter 3**

# **Results and discussion**

The following chapter will detail the components required for our thin-film thickness measurement equipment proposal, which perform a SiO<sub>2</sub> film thickness measurement Figure (3.1). These are a light source, fiber optics, spectrophotometer, and software necessary to interpret the measured reflectance data.

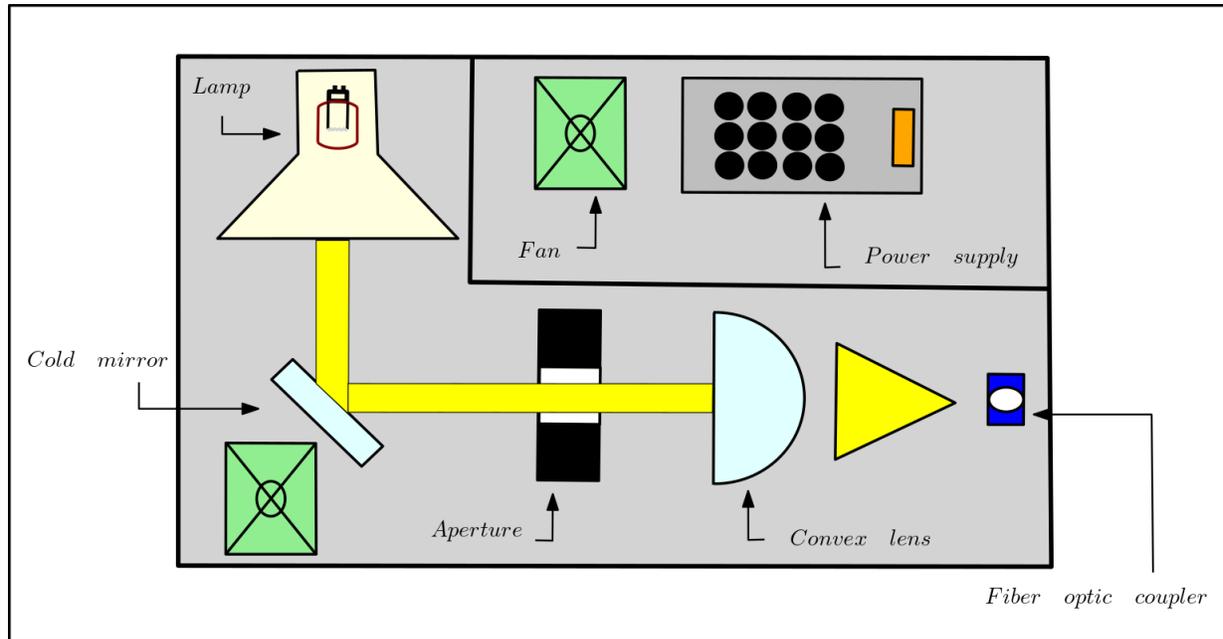


Figure 3.1: Schematic representation of a light source.

## 3.1 Light source

Next, we will describe the components necessary to build our proposal for a light source and its proper justification for the choice. We will use its source to irradiate the sample we want to measure.

### 3.1.1 Lamp

To choose our lamp, we take into account many parameters such as spectral content, typical output levels, bulb lifetime, source extent. Additionally, other parameters we took into account were - long bulb lifetime (> 2000 hours) - low IR output (heat) - low wattage - good beam directionality<sup>37</sup>. In particular, having a low IR output (heat) is essential for the correct operation of the light source. If this were not so, the heat generated would be able to melt the tip of the optical fiber, which compromises the entire experiment, since the amount of light supplied drops dramatically.

Additionally, having a low IR value increases the life of the lamp and reduces the costs of cooling equipment for the light source. Low wattage is also beneficial because it requires low-cost electrical connections, which in turn reduce the total cost of the light source.

Taking into account the above considerations, we decided to use a 60 W tungsten halogen lamp at 12V, 5A, where life span for tungsten halogen lamps ranges from about 50 to 8000 hours Figure (3.2)<sup>438</sup>. Nevertheless, one disadvantage of the halogen lamp is that they typically have high output in the IR region of the spectrum, ultimately leading to unwanted heat<sup>37</sup>. This problem can be solved using a cold mirror; this mirror has the function of removing IR frequencies from the beam; it will be placed between the lamp and convex lens. The use of this mirror will extend the useful life of optical fiber that we use to direct the light towards the sample. In case of not having a cold mirror, it is advisable not to use the equipment for long periods.

In terms of coherence, the tungsten halogen lamp like other incandescent lamps, has low coherence, both temporal and spatial. The temporal coherence of the tungsten halogen light source is low as a consequence of its broad optical bandwidth, the broader bandwidth the less coherent the lamp is. Also due to the broad bandwidth, this lamp has a low coherence length. On the other hand, the low spatial coherence is because the area of light emission is large, this makes it difficult to focus the light.

To solve this problem, we propose to incorporate a focusing reflector to the tungsten halogen lamp<sup>3940</sup>. The focusing reflector concentrate the light on a small spot in the central optical axis at a defined distance from the reflector. This type of reflector is designed with an elliptical geometry, which requires that the lamp filament be placed in the first focal point of the ellipsoid so that the projected light spot is concentrated at the second focal point<sup>29</sup>.

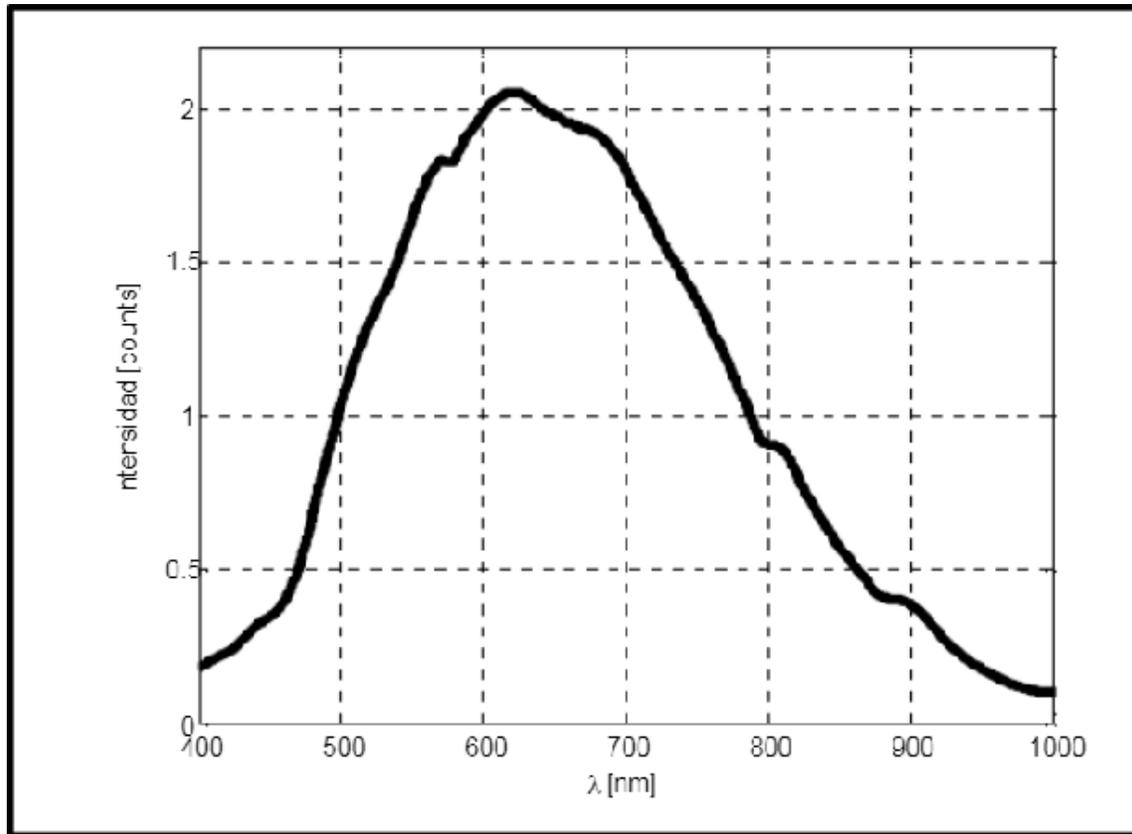


Figure 3.2: Emission spectrum of tungsten-halogen lamp Source: Reprinted from Guevara, E. *Técnica Conjunta* 2015<sup>4</sup>

### 3.1.2 Power supply

The selection of a power supply is based on some parameters; the voltage ranges available, the current supplied, and package size<sup>37</sup>. For this reason the Power Source selected for our application is a 110 v To 12 V, 10 A Cctv Transformer Power Source, the dimensions of this source are 10 x 16 x 4.3 cm.

### 3.1.3 Lenses

To focus the light from the lamp, we will use a convex lens. These have the characteristic that its focal length is positive; it is the distance at which a beam of collimated light will be focused to a single spot. In our case, its single spot will be focused on the optical fiber coupler. To Determine the focal length of a convex lens; we can use the lens equation:

$$\frac{1}{f} = \frac{1}{u} + \frac{1}{v} , \quad (3.1)$$

where  $u$  is the distance from the object to the lens,  $v$  is the distance from the lens to the image, and  $f$  is the focal length. Another way to determine the focal length of a thin convex lens is by placing a lamp at a fixed distance from the lens and placing a screen in front of the lens; we proceed to move the screen until we obtain the image of a single spot, that will be the focal length of the lens. Knowing this distance allows us to obtain a beam of light focused on the fiber optic input.

An important consideration that we propose is to use lenses made with Low-dispersion glasses, for the light source lenses, because the use of this material reduces chromatic aberration<sup>41 42</sup>. Chromatic aberration, is a deficiency of a lens to focus all colors to the same point<sup>43</sup>. This deficiency to focus occurs due to the dispersion that light undergoes when passing through a lens Figure (3.3).

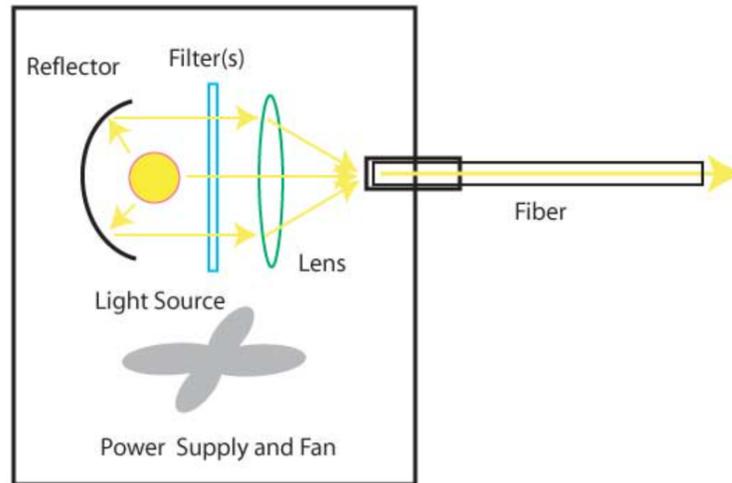


Figure 3.3: Schematic representation of how to focus the light coming from the light source in to fiber optic coupler. Source: Reprinted from FOA: Fiber Optic Lighting<sup>5</sup>.

### 3.1.4 Fiber optic coupler

A female to male Singlemode fiber optic coupler is used to, on the one hand, couple the light beam from the lamp and, on the other hand, as a port to connect a fiber optic to carry light from the source to the sample.

## 3.2 Packaging

The criteria for packaging are relatively simple, the packaging of our light source has to allow the air circulation inside the box to cool the components of the light source, it also has to be resistant to light shocks and occupy the minimum space possible.

Taking into account the above considerations, we decided to use an aluminum foil package folded in the shape of a box. Packaging consists of two parts Figure (3.4), one where the components such as the lamp, lenses, cold mirror, and fans are located, and another part acts as a box cover, this also has slots for cooling.

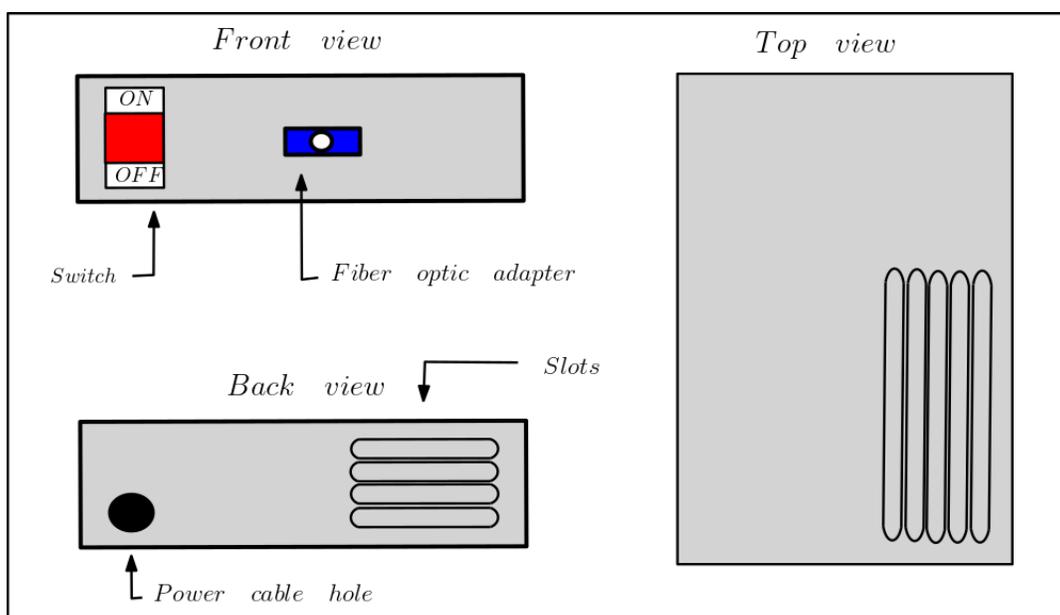


Figure 3.4: Representation of the front,back and top views of our case proposal for the light source.

### 3.3 Cooling system

The main components of our light source proposal (lamp and power source) emit much heat during operation which shortens its useful life. To solve this problem and extend the useful life of these components and, therefore, our light source, a cooling system is necessary.

For our specific light source, we will use an air-cooling system that consists of two fans connected to the power source. These fans are placed one just above the power source and the other behind the cold mirror, which will evacuate the heat from the light source.

### 3.4 Fiber optics

Fiber optic cables or lines allow us to carry light from the light source to the samples and also carry the reflected light from the sample to the spectrophotometer.

Using a fiber optics splitter, we can direct the light from the source so that it falls perpendicular to the sample and also capture the light reflected by the sample. In particular, we will use the TOS fiber optics splitter. This splitter has the particularity of having two optical fibers in each of its inputs; later, these fibers meet at the tip of the splitter where we find four optical fibers Figure (3.5).

We will use this device in the following way: two of the input optical signals will be used to irradiate the sample with light, and the other two will be used to capture the reflected light and direct it towards the spectrometer where it will be analyzed. Alternatively, we can interpret it as: Two of the four optical fibers of the tip, are used to irradiate the sample, and the other two are used to collect the reflected light from the sample Figure (3.6).

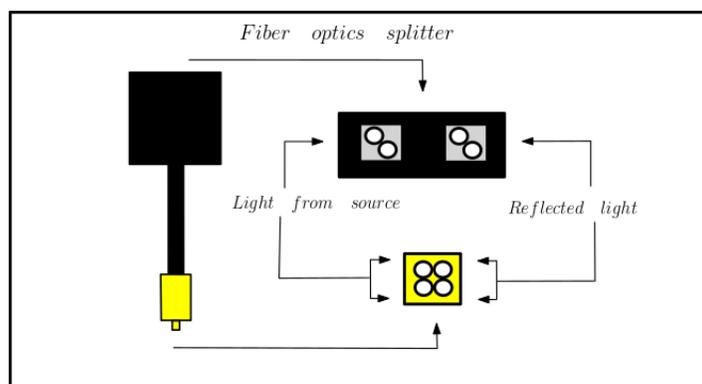


Figure 3.5: Schematic representation of a TOS fiber optics splitter .

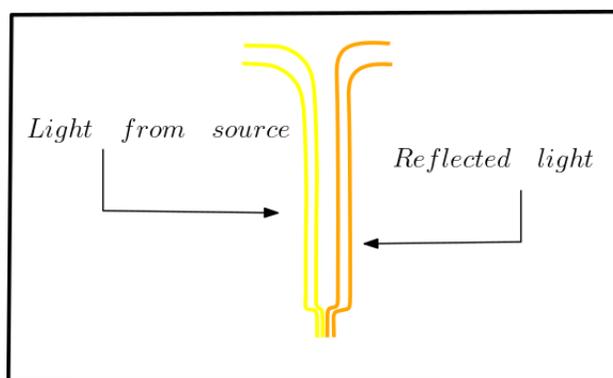


Figure 3.6: Schematic representation of the interior of a TOS fiber optics splitter.

### 3.5 Spectrometer

To choose a spectrometer for our equipment, we take into account parameters such as cost, the spectrum region measures in which it can, and the dimensions of the spectrometer. Given these considerations, we propose to use the SMA Mini spectrometer manufactured by Thunder<sup>6</sup> optics Figure (3.7), which can measure in the 350-1000 wavelength range.

Another advantage that this spectrometer provides is that it can be powered directly by a computer USB port, furthermore, this USB connection provides us high-speed data transfer. Also this spectrometer has a low cost of around 170 USD, the following technical table shows the specifications of SMA Mini spectrometer Table (3.1).



Figure 3.7: SMA Mini spectrometer manufactured by Thunder optics. Source: Reprinted from SMA Mini Spectrometer » Thunder Optics<sup>6</sup>

Wavelength Range nm	350-1000
Chip Type	CMOS
Chip Dim (pixels)	1800x1200
Chip Dim (")	1/3
Diffraction Grating( Lines/mm)	1000
Slit Width ( $\mu\text{m}$ )	120
Optical Conector- Optical Fiber	SMA 905
Power Supply	USB 2.0
Resolution	<3 nm
Software	Free
Operating Systems	Windows, Mac(1)
Dimension (mm)	111x38x14.8
Weight (gr)	300

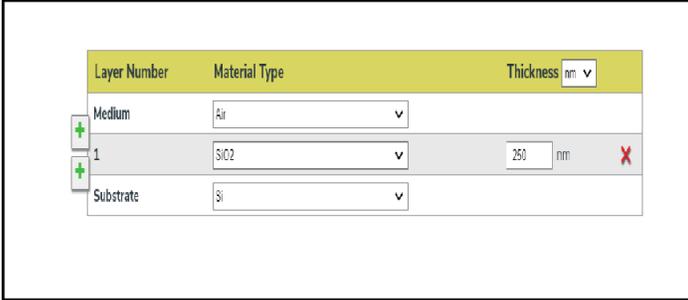
Table 3.1: Technical specifications of SMA Mini spectrometer

### 3.6 Software

The software has the function of analyzing and displaying the data that was obtained in the spectrometer. The parameters for choosing a software are firstly that it has a film thickness measurement function based on reflectance data and, on the other hand, that it is a low-cost software.

For these reasons, we decided to use Spectragryph software<sup>8</sup>, which is a free optical spectroscopy software for education, this software allows us to graph the reflectance data obtained and also has a function called film thickness, which precisely provides us a film thickness measurement tool.

Another software tool that we consider essential is a reflectance calculator; this tool provides us with a reflectance vs. wavelength graph given specific inputs such as the different sample media, in our case, the first medium is air, the second medium is silicon oxide, and the third medium is the silicon substrate Figure (3.8). Another input of this calculator is the angle of incidence of the light in the sample, the wavelength range in which we make the measurement, and the thickness of the medium that we want to measure; in our case, it would be the thickness of the silicon oxide.



Layer Number	Material Type	Thickness nm
Medium	Air	
1	SiO2	250 nm
Substrate	Si	

Figure 3.8: Filmetrics reflectance calculator: Choice of media, air, silicon oxide, and silicon substrate, also thickness of the silicon oxide layer choose. Source: Reprinted from Spectral Reflectance Calculator for Thin-Film Stacks<sup>7</sup>.

This reflectance calculator is of great help in obtaining the reflectance spectrum of the sample that we want to measure Figure (3.9). Because given the color of the silicon oxide layer, we can have an idea of its thickness, we can enter this information into the reflectance calculator and thus give us an idea of the characteristics of the spectrum that we want to measure. In this way, we fine-tune the acquisition of reflectance data.

We suggest using the Filmetrics<sup>7</sup> reflectance calculator, which is a free online tool that will provide us with valuable information about the sample.

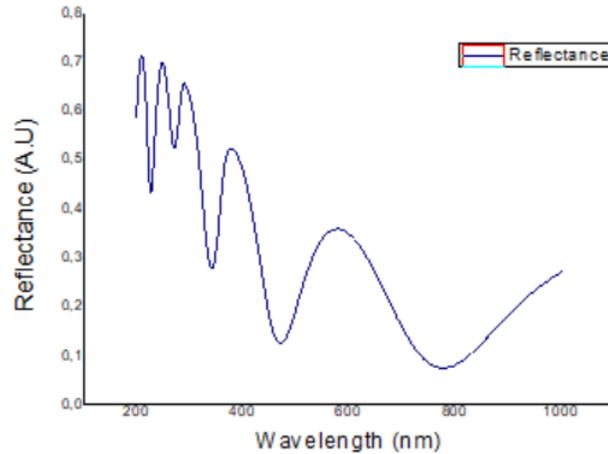


Figure 3.9: Reflectance vs wavelength plot of a 400 nm thick silicon oxide sample on a silicon substrate, in a range of 200 to 1000 nm wavelength. Obtained using the Filmetrics reflectance calculator, taken from Filmetrics Thin-Film Measurement. Source: Reprinted from Spectral Reflectance Calculator for Thin-Film Stacks<sup>7</sup>.

### 3.7 Silicon oxide sample

We propose to use the tube furnace that we have at Yachay Tech University to carry out the Thermal oxidation of silicon. Once we obtain an initial layer of silicon oxide, we etch the sample to obtain different thicknesses of silicon oxide from the same initial sample.

Due to the relationship between the color and thickness of the silicon oxide layer that we mentioned before, we can have an approximation of the thickness of the oxide layer. So, we can use this approximate thickness in the Filmetrics reflectance calculator, which will give us a reflectance spectrum that will give us an idea of what we hope to obtain experimentally in our set up.

### 3.8 Measurement procedure

In this chapter, we will detail the steps necessary to obtain the measurement of a film using the setup proposed in the previous section.

- First, we arrange the light source, TOS fiber optic splitter, spectrometer, and computer on a firm table.
- Then we turn on the light source and connect the spectrometer to a computer that has the Spectragryph software installed on it.
- Then we proceed to open this software on the computer, and we go to the Acquire section, then in the device type option, and choose USB webcam, then we click on the connect button Figure (3.10).

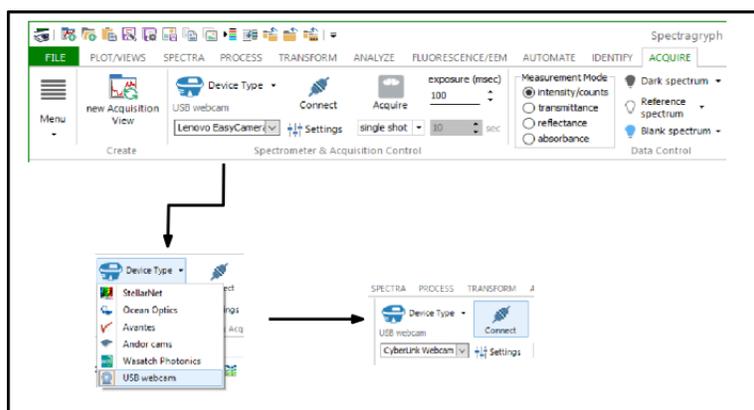


Figure 3.10: Spectragryph software:Acquire section . Sources: Reprinted from F. Menges, Spectragryph - optical spectroscopy software<sup>8</sup>.

- Now we proceed to take the characteristic reference spectrum of the halogen tungsten lamp; we achieve this by directly connecting the light source with the spectrometer through an optical fiber. We will use this spectrum to calibrate our spectrometer.

To do this, we choose the measurement mode in intensity/counts, and we click on the acquire button. Once the spectrum is obtained, we go to the x-axis calibration section; here, we will find a table with three columns: found peak desired position and use. Found peak: refers to the position of the peaks of the spectrum of the halogen lamp that we have just measured.

Desired position: it refers to the position where these peaks should be located; for this, we use the spectrum reported in the literature of the halogen tungsten lamp; from this spectrum, we will locate the most representative peaks and note their position in the desired position column. Use: refers to whether or not we want to use that peak for calibration Figure (3.11). Then we proceed to click on apply, and we will have our spectrometer calibrated.

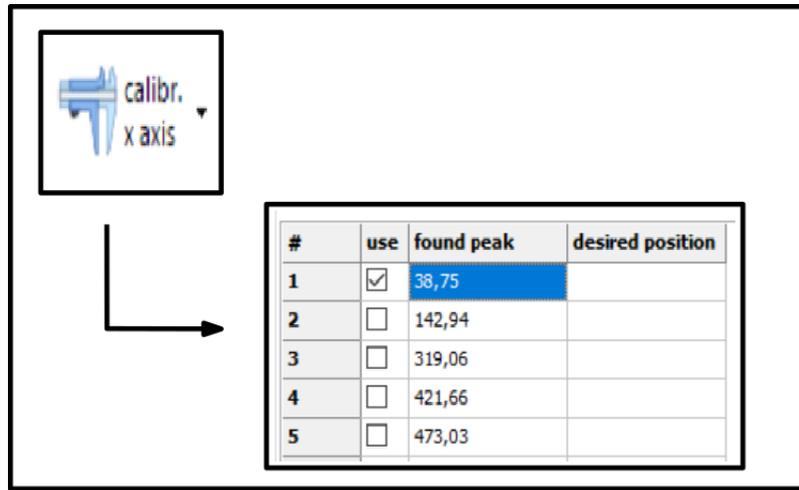


Figure 3.11: Spectragryph software: X-axis calibration section . Sources: Reprinted from F. Menges, Spectragryph - optical spectroscopy software<sup>8</sup>.

- The spectrum of a tungsten lamp is set as a reference spectrum, so it is automatically eliminated from our measurements.
- Connect the optical fiber that goes from the light source to an input of the TOS fiber optics splitter; then we connect the optical fiber that goes from the second input of the TOS splitter to the spectrometer.
- Afterward, taking into account that the color of a silicon oxide layer is related to its approximate thickness, we can have an idea of the thickness of the layer that we want to measure. This approximation of the thickness is entered in the Filmetrics reflectance calculator to obtain the reflectance spectrum of this approximate layer thickness, which will give us an idea of what we hope to obtain experimentally.
- Now, we have the spectrometer properly calibrated; we proceed to place the measurement mode in reflectance.
- We proceed to place our silicon oxide on silicon sample perpendicular to the TOS fiber optics splitter.
- We carry out the acquisition of reflectance data of the sample by clicking on the acquire button.
- Later, we go to the “analyze” section and select the option “film measure” Figure (3.12), it will ask us for the angle at which the measurement was made, which in our case is zero degrees.

It also asks us for the index of refraction of the film (silicon oxide)  $n = 1.45704$  at  $632.8 \text{ nm}^{44}$  and the wavelength range at which we want to measure (400 -1000 nm) Figure (3.13).

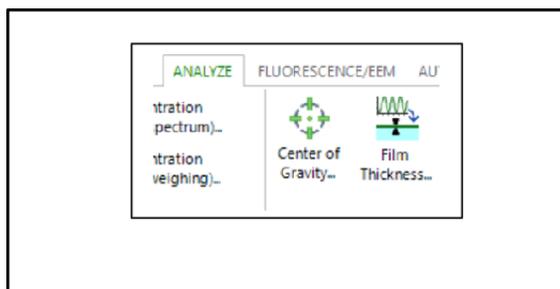


Figure 3.12: Spectragryph software: Analyze section and film measure option. Sources: Reprinted from F. Menges, Spectragryph - optical spectroscopy software<sup>8</sup>.

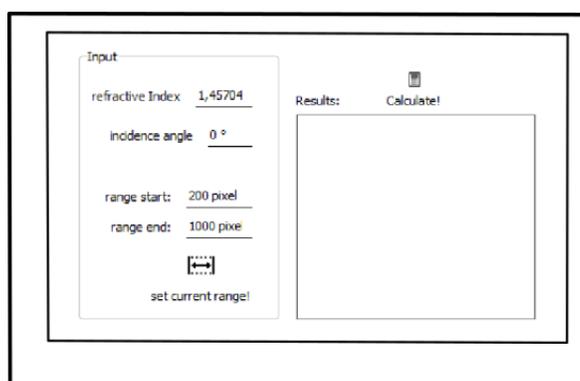


Figure 3.13: Spectragryph software: Film measure option. Sources: Reprinted from F. Menges, Spectragryph - optical spectroscopy software<sup>8</sup>.

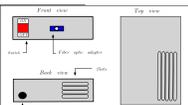
- Then we click on the calculate button, and we will obtain the value of the thickness of that silicon oxide film.

### **3.9 Recommended components for the construction of our thin-film measuring equipment proposal**

This section will list some components that we recommend for the construction of our thin-film measuring equipment proposal Table (3.2). Where the total cost of the equipment if we use convex lenses without a low dispersion treatment is 304 USD, and if we use lenses with a low dispersion treatment, the price is 402 USD.

As we can see, the total cost of the equipment (304 USD) is relatively low if we compare it with measurement equipment of these characteristics available in the market, for example, the F20 model by Filmetrics company that in its most basic version costs around 12000 USD<sup>45</sup>. With the future implementation of this equipment, we hope to improve, diversify, and facilitate the research and teaching activities at Yachay Tech University.

Table 3.2: Table of components that we recommend to use to construct the measuring equipment and its price.

Component	Producer	Price (USD)	Figure
<b>Halogen tungsten lamp</b>		15	
<b>Cold mirror</b>	Knight Optical	47	
<b>Convex lenses</b>	Edmund optics	22- 120 (low dispersion)	
<b>TOS fiber optic splinter</b>		15	
<b>Fiber optics</b>		10	
<b>SMA Mini spectrometer</b>	Thunder Optics	170	
<b>Software Spectra Gryph</b>	Dr. Friedrich Menges Software	Free	
<b>Packaging</b>	CNC laser cut producer	15	
<b>Fans</b>		15	
<b>Power supply</b>		10	



## Chapter 4

# Conclusion and outlook

In conclusion, in this work, we detail the theoretical bases necessary to understand the working principle of equipment capable of measuring film thickness based on the spectral reflectance technique. Besides, the components that these type of measuring device were named and described.

Secondly, based on the components of a commercial film thickness measuring equipment. We develop an alternative design of film thickness measuring equipment. In this design, we consider theoretical, technical, and economic parameters. In this way, for each component of the commercial measurement equipment, we proposed a variant component that performs the same function, with similar technical parameters, but that adapts to a lower financial budget than the measurement equipment that we can find already assembled in the market, also an essential factor that we consider for selecting the variant component is its availability in the local market.

Finally, we detail how to use an optical spectroscopy software called SpectraGryph, which has the crucial task of interpreting the experimentally obtained data and providing a sample thickness measure.

In the future, we hope to use the design proposed in this work to build equipment with these characteristics, the implementation of this equipment will contribute a new characterization tool to the Yachay tech university's nanotechnology laboratory. Thus this equipment will benefit and facilitate both research and academic activities for teachers and students of The school of physics and nanotechnology.



## Appendix A

### Deduction of equation (2.56)

From a film over a substrate with thickness “d”

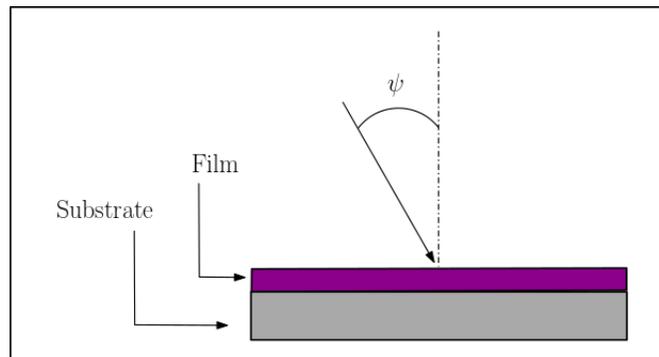


Figure A.1: The angle of incidence  $\psi$  of light on the silicon oxide sample on a silicon substrate

The reflected light from both the film and the substrate create constructive or destructive interference.

As Figure (A.2) shows, the spectrum of interference as a function of the wavelength.

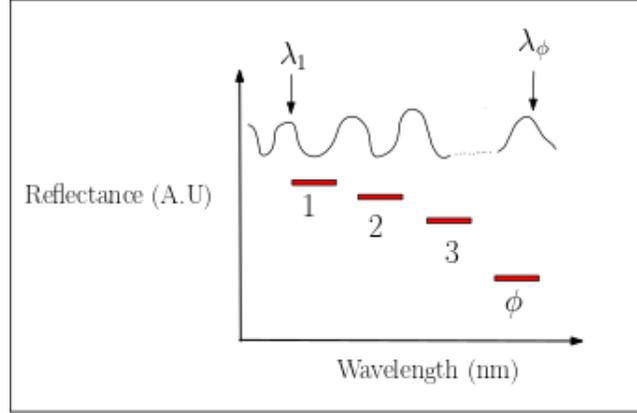


Figure A.2: Number of oscillations between  $\lambda_1$  and  $\lambda_\phi$

Let us see how to obtain the relationship between  $d$  and parameter available to obtain by measuring. First let us now assume that we have chosen a particular film thickness so that the diffracted waves interfere constructively when having performed one loop in the film.

The phase gain while propagating through the film has been calculated previously, we have :<sup>46</sup>

$$2\delta = \frac{4\pi}{\lambda_\phi} n_\phi d \cos(\psi) , \quad (\text{A.1})$$

Where  $\delta$  is the phase ,  $\lambda_\phi$  is the wavelength,  $n_\phi$  is the refractive index of the medium for  $\lambda_\phi$  ,  $d$  is the film thickness and  $\psi$  is the incident angle of light Figure (A.1).

Then applying Snell's law, we obtain.

$$2\delta = \frac{4\pi n_\phi d}{\lambda_\phi} \frac{1}{n_\phi} \sqrt{n_\phi^2 - n_v^2 \sin^2(\phi)} , \quad (\text{A.2})$$

Where  $n_v = 1$  is the vacuum refractive index , so

$$2\delta = \frac{4\pi d}{\lambda_\phi} \sqrt{n_\phi^2 - \sin^2(\phi)} , \quad (\text{A.3})$$

Since the phase don't change<sup>47</sup>

$$\frac{4\pi d n_1}{\lambda_1} = \frac{4\pi d}{\lambda_\phi} \sqrt{n_\phi^2 - \sin^2(\phi)} , \quad (\text{A.4})$$

Or

$$n_1 = \frac{\lambda_1}{\lambda_\phi} \sqrt{n_\phi^2 - \sin^2(\phi)} , \quad (\text{A.5})$$

The thickness is obtained from<sup>46</sup>

$$d = \frac{\lambda_1}{4n_1} j_1 , \quad (\text{A.6})$$

Where  $j_1$  is the order of  $\lambda_1$  and is approximate

$$j_1 \cong 2P \frac{\lambda_1}{\lambda_1 - \lambda_\phi} , \quad (\text{A.7})$$

Here P is the number of oscillations between  $\lambda_1$  and  $\lambda_\phi$  , see Figure (A.2).

Equating equation A.6 and A.7 give us  $n_1$  as

$$n_1 = \frac{\lambda_1}{2d} \frac{P\lambda_1}{\lambda_1 - \lambda_\phi} , \quad (\text{A.8})$$

Replacing in equation A.5

$$\frac{\lambda_1}{2d} \frac{P\lambda_1}{\lambda_1 - \lambda_\phi} = \frac{\lambda_1}{\lambda_\phi} \sqrt{n_\phi^2 - \sin^2(\phi)} , \quad (\text{A.9})$$

Then we obtain the equation (2.56).

$$d = \frac{P\lambda_1\lambda_\phi}{(\lambda_1 - \lambda_\phi) \sqrt{n_\phi^2 - \sin^2(\phi)}} , \quad (\text{A.10})$$



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