

MEASUREMENT OF PHYSICAL AND OPTICAL PROPERTIES OF THIN  
FILMS WITH AN ELLIPSOMETRIC TECHNIQUE

by

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B.S., Physics, Boğaziçi University, 2008

Submitted to the Institute for Graduate Studies in  
Science and Engineering in partial fulfillment of  
the requirements for the degree of  
Master of Science

Graduate Program in Physics

Boğaziçi University

2010

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FILMS WITH AN ELLIPSOMETRIC TECHNIQUE

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DATE OF APPROVAL: 06.08.2010

## ACKNOWLEDGEMENTS

First of all, I want to thank to my thesis supervisor Prof.Mehmet Naci İnci. He is one of the most humanist people I have ever seen. Many thanks to Sabriye Açıkgöz she was always with me when I need help during my experiments. I am thankful to Tuğçe Nihal Gevrek that she prepared samples for my experiments and my dear aunt Fatma Ertuğrul who motivated me with her nice wishes everyday and helped my drawings. I especially want to thank my dear friends Elif Demirbaş, Veli Uğur Güney and İbrahim Sarpkaya. They were always with me while I was writting my thesis.

Finally, I would like to thank God, He gave me the best family members, my parents Hatice and M. Emin Ertuğrul, my sisters Aliye, Mürüvvet, Zeyneb, Meryem and my brother M. İbrahim.

## ABSTRACT

# MEASUREMENT OF PHYSICAL AND OPTICAL PROPERTIES OF THIN FILMS WITH AN ELLIPSOMETRIC TECHNIQUE

In this thesis, working mechanism of ellipsometer is studied in details. Thickness and refractive index of polyethyleneglycol(PEG), gold and silicon dioxide films are measured using an ellipsometer. PEG is attached to a gold coated surface on a BK7 glass using a chemical synthesis method. Potassiumchloride is used to control the relative humidity of the environment for insitu measurements of the humidity dependent thickness of PEG polymer film. The PEG thicknesses, which correspond to certain relative humidity levels, are measured using the ellipsometry. It is observed that the thickness increases with the increasing humidity. Apart from PEG thickness and refractive indices of  $SiO_2$  and gold films are measured.

## ÖZET

# ELİPSOMETRİK TEKNİK KULLANARAK İNCE FİMLERİN FİZİKSEL VE OPTİK ÖZELLİKLERİNİN ÖLÇÜMÜ

Bu tezde, elipsometrenin çalışma tekniği detaylı bir şekilde çalışıldı. Polietilenglikol, altın ve silikon dioksit ince filmlerinin kalınlıkları ve bazı optiksel özellikleri elipsometre ile ölçüldü. Polietilenglikol, kimyasal sentez yoluyla altın kaplı BK7 camının üzerine bağlandı. PEG in neme bağlı kalınlığı ölçülürken etrafın bağıl nem düzeyi potasyumnitrat tuzu ile kontrol edildi. Bu nem değerlerine tekabül eden polietilenglikol kalınlıkları elipsometre ile ölçüldü. Nemdeki artış ile polietilenglikolun kalınlaştığı gözlemlendi. PEG in dışında, silikon dioksit ve altın filmlerinin kalınlığı ve kırıcılık indisleri ölçüldü.

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## LIST OF SYMBOLS/ABBREVIATIONS

PEG	polyethylene glycol
AFM	Atomic force microscope
Au	Gold
BK7	Borosilicate crown glass
$CHCl_3$	Choloroform
MW	Molecular Weight
rpm	rate per minute
SEM	Scanning electron microscope
$SiO_2$	Silicon dioxide

## 1. INTRODUCTION

A thin film is a layer which has a thickness varying from sub-nanometers to a few microns. Thin films are widely used in many kinds of experimental works such as optical coatings and electronic semiconductor devices. In design of a device, optical properties of thin films are very essential. There are many ways of designating the optical properties of thin films such as thickness, index of refraction, etc.

Ellipsometry is a device which is used to determine the optical properties of thin films. Complex refractive index, dielectric function and thickness of thin films can be measured by ellipsometry. In comparison to plasmon resonance technique, ellipsometry is an easier way of measuring thickness. On the other hand, once the appropriate data about the sample of interest is entered into the software, one can get very precise information about the optical properties of the thin films instantly. That's why ellipsometric measurement is more practical than SEM(Scanning Electron Microscope) and AFM(Atomic Force Microscope) if only physical thickness and refractive index are concerned.

In situ measurements are carried out with ellipsometry. Ellipsometry takes two parameters, either angle of incidence or wavelength. It calculates the property of interest immediately by first measuring and then comparing the intensities of incoming and reflected light beams. In stead of exact measurement of the light intensity the comparison is enough to analyze the data taken.

Chapters of this thesis are ordered as follows: the second chapter is devoted to the theoretical background of the ellipsometry, which includes light and polarization, index of refraction, reflection and transmission of polarized light, ellipsometric measurements and the ambient-film-substrate system.

In the third chapter, preparation of thin films and optical setup are explained. This is followed by results on the thickness and refractive index.

In the fourth chapter, conclusion is given which includes a discussion on the results and experimental method discussed. This is followed by suggestions on future work with an ellipsometric technique.

## 2. REVIEW

### 2.1. Light and Polarization

Light is an electromagnetic(EM) wave travelling through a media. In order to understand the ellipsometry only consider the electric field( $\vec{E}$ ) component of an EM wave. The change in  $\vec{E}$  of an EM wave(or light) with respect to time is known as polarization. For the sake of generality consider that light is travelling along z axis which means that the  $\vec{E}$  have x and y components. If light has random orientation and phase , it is called as unpolarized light. In the ellipsometric measurement one should consider polarized light i.e, light wave with an  $\vec{E}$  which has a regular path.If the oscillations of  $\vec{E}$  are on a well defined line then the electromagnetic wave is said to be linearly polarized. If the  $\vec{E}$  forms a circle by rotating around the direction of

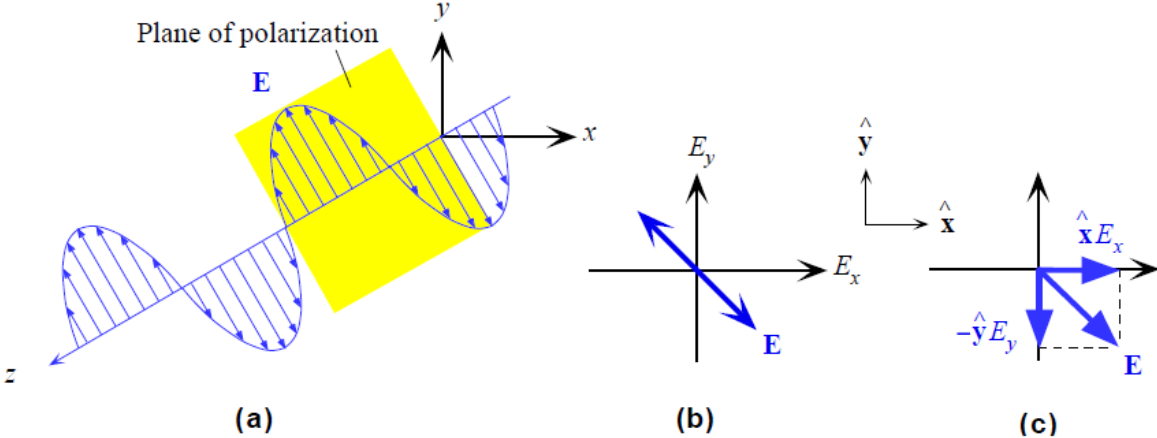


Figure 2.1. (a)A linearly polarized wave has its electric field oscillations defined along a line perpendicular to the direction of propagation, z. The field vector E and z define a plane of polarization.(b) The E-field oscillations are contained in the plane of polarization. (c)A linearly polarized light at any instant can be represented by the superposition of two fields  $E_x$  and  $E_y$  with the right magnitude and phase.

the propagation, such a wave is called circularly polarized light. As in the circular polarization if light forms an ellipse then it is called the elliptically polarized light. [1]

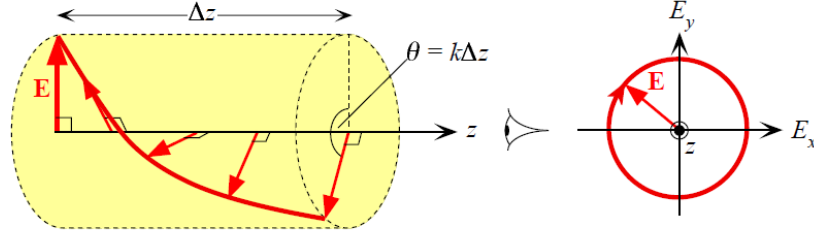


Figure 2.2. A right circularly polarized light. The field vector  $E$  is always at right angles to  $z$ , rotates clockwise around  $z$  with time, and traces out a full circle over one wavelength of distance propagated.

## 2.2. Index of Refraction

Refractive index may simply be defined as ratio of speed of light in the vacuum to the speed of light in a medium. For some materials refractive index has a complex representation:

$$N = n + jk \quad (2.1)$$

where  $n$  is called as index and  $k$  is called as extinction coefficient. Dielectric function is also useful to express optical properties of material.

$$\tilde{\epsilon} = \epsilon_1 + j\epsilon_2 \quad (2.2)$$

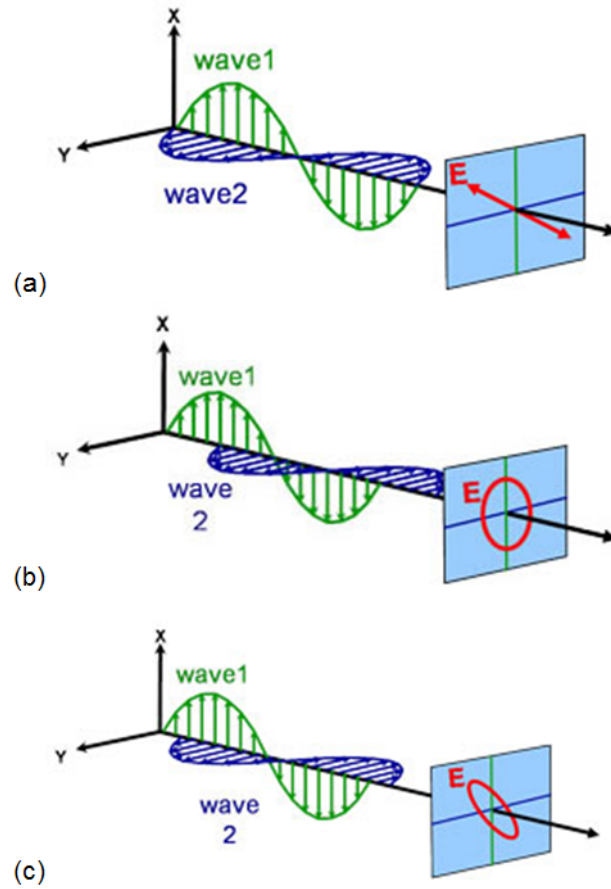


Figure 2.3. (a) Linear polarization.(b) Circular Polarization.(c) Elliptical Polarization.

The relation between index of refraction and dielectric function is

$$\tilde{\epsilon}^2 = N^2 \quad (2.3)$$

A media with high index of refraction decreases the speed of light. Extinction coefficient is used to describe the loss of wave energy which is related to absorption coefficient  $\alpha$ .

$$\alpha = 4\pi k/\lambda \quad (2.4)$$

Intensity also changes in a different medium with the the following relation [7]

$$I(z) = I(0)e^{-j\alpha z} \quad (2.5)$$

### 2.3. Reflection and Transmission of Polarized Light

The expression for the  $\vec{E}$  component of the light travelling along z direction is defined as.

$$\vec{E} = E_0 e^{j(\omega t + \delta)} e^{-j\omega N z / c} \quad (2.6)$$

where  $\delta$  is a constant phase angle,  $c$  is the free space wave velocity and  $E_0$  is the amplitude and polarization of the wave,  $c/n$  is the wave velocity and the decay rate of the amplitude is  $\omega k/c$  or  $2\pi k/\lambda$ ,  $\lambda$  is the free space wavelength.

When light travels from one medium to another,  $\vec{E}$  and  $\vec{B}$  components are reflected, refracted and transmitted at their interface.

As seen from figure 2.4,  $\vec{E}$  component of light travels along two semi-infinite media (0 and 1) with complex indices of  $N_0$  and  $N_1$ . Angle of incidence  $\Phi_0$  and reflection  $\Phi_1$  have the following relation.

$$N_0 \sin \Phi_0 = N_1 \sin \Phi_1 \quad (2.7)$$

This is known as the Snell's Law. If both media are transparent,  $N_0$  and  $N_1$  are real numbers and calculations for above picture become simple. However, when either one or both media are absorbing, angles  $\Phi_0$  and  $\Phi_1$  become complex numbers.

Let  $(E_{ip}, E_{is})$ ,  $(E_{rp}, E_{rs})$  and  $(E_{tp}, E_{ts})$  represent the complex amplitudes of  $\vec{E}$  of the incident, reflected, and transmitted waves respectively. Applying boundary conditions for tangential components of  $\vec{E}$  and  $\vec{H}$  at the interface give;

$$\frac{E_{rp}}{E_{ip}} = r_p = \frac{N_1 \cos \Phi_0 - N_0 \cos \Phi_1}{N_1 \cos \Phi_0 + N_0 \cos \Phi_1} \quad (2.8)$$

$$\frac{E_{rs}}{E_{is}} = r_s = \frac{N_0 \cos \Phi_0 - N_1 \cos \Phi_1}{N_0 \cos \Phi_0 + N_1 \cos \Phi_1} \quad (2.9)$$

$$\frac{E_{tp}}{E_{ip}} = t_p = \frac{2N_0 \cos \Phi_0}{N_1 \cos \Phi_0 + N_0 \cos \Phi_1} \quad (2.10)$$

$$\frac{E_{ts}}{E_{is}} = t_s = \frac{2N_0 \cos \Phi_0}{N_0 \cos \Phi_0 + N_1 \cos \Phi_1} \quad (2.11)$$

which are known as Fresnell reflection (r) and transmission (t) components. Using equation 2.7, one can cancel  $N_0$  and  $N_1$ :

$$r_p = \frac{\tan(\Phi_0 - \Phi_1)}{\tan(\Phi_0 + \Phi_1)} \quad (2.12)$$

$$r_s = \frac{-\sin(\Phi_0 - \Phi_1)}{\sin(\Phi_0 + \Phi_1)} \quad (2.13)$$

$$t_p = \frac{2\sin\Phi_1 \cos\Phi_0}{\sin(\Phi_0 + \Phi_1) + \cos(\Phi_0 - \Phi_1)} \quad (2.14)$$

$$t_s = \frac{2\sin\Phi_1 \cos\Phi_0}{\sin(\Phi_0 + \Phi_1)} \quad (2.15)$$

In order to make a sensible analysis, we write the Fresnel coefficients as

$$r_p = |r_p| e^{j\delta_{rp}} \quad (2.16)$$

$$r_s = |r_s| e^{j\delta_{rs}} \quad (2.17)$$

where  $|r_p|$  is the ratio of the amplitude  $E_{rp}$  to  $E_{ip}$ .  $\delta_{rp}$  is the phase shift upon reflection

[2].

## 2.4. Ellipsometric Measurement

Ellipsometric measurement is based on the ratio

$$\rho = \frac{r_p}{r_s} \quad (2.18)$$

which can be written in the form

$$\rho = \tan\Psi e^{j\Delta} \quad (2.19)$$

where

$$\tan\Psi = \frac{|r_p|}{|r_s|} \quad (2.20)$$

$$\Delta = \delta_{rp} - \delta_{rs} \quad (2.21)$$

substituting  $r_p$  and  $r_s$  and using equation 2.7 we can find the relation between two media as

$$\frac{N_1}{N_0} = \sin\Phi_0 \left[ 1 + \left( \frac{1-\rho}{1+\rho} \right)^2 \tan^2\Phi_0 \right]^{1/2} \quad (2.22)$$

. It can be seen from the above equation that  $N_1$  can be determined if  $N_0$  is known and  $\rho$  is measured at one angle of incidence [2].

### 2.4.1. Ambient-Film-Substrate System

$d_1$  is the film thickness,  $\Phi_0$  is the angle of incidence in the ambient and  $\Phi_1$ ,  $\Phi_2$  are the angles of refraction in the film and substrate, respectively. Fresnel reflection and transmission coefficients for  $0^{th}$  media are  $r_{01}$ ,  $t_{01}t_{10}r_{12}e^{-j2\beta}$ ,  $t_{01}t_{10}r_{12}^2e^{-j4\beta}$ ,

$t_{01}t_{10}r_{10}^2r_{12}^3e^{-j6\beta}$  and for the second media  $t_{01}t_{12}e^{-j\beta}$ ,  $t_{01}t_{12}r_{10}r_{12}e^{-j3\beta}$ ,  $t_{01}t_{12}r_{10}^2r_{12}^2e^{-j5\beta}$  where  $\beta$  is the phase delay the beam experiences during propagation from the top surface of the beam to the bottom surface of the film.

$$\beta = 2\pi \left( \frac{d_1}{\lambda} \right) N_1 \cos \Phi_1 = 2\pi \left( \frac{d_1}{\lambda} \right) (N_1^2 - N_0^2 \sin^2 \Phi_0)^{1/2} \quad (2.23)$$

Addition of partial waves gives the reflected and transmitted amplitude as

$$R = \frac{r_{01} + r_{12}e^{-j2\beta}}{1 + r_{01}r_{12}e^{-j2\beta}} \quad (2.24)$$

$$T = \frac{t_{01}t_{12}e^{-j\beta}}{1 + r_{01}r_{12}e^{-j2\beta}} \quad (2.25)$$

and indicating the plane of incidences as p or s;

$$R_p = \frac{r_{01p} + r_{12p}e^{-j2\beta}}{1 + r_{01p}r_{12p}e^{-j2\beta}} \quad (2.26)$$

$$R_s = \frac{r_{01s} + r_{12s}e^{-j2\beta}}{1 + r_{01s}r_{12s}e^{-j2\beta}} \quad (2.27)$$

$$T_p = \frac{t_{01p}t_{12p}e^{-j\beta}}{1 + r_{01p}r_{12p}e^{-j2\beta}} \quad (2.28)$$

$$T_s = \frac{t_{01s}t_{12s}e^{-j\beta}}{1 + r_{01s}r_{12s}e^{-j2\beta}} \quad (2.29)$$

Fresnel reflection and transmission coefficients at the 0-1 and 1-2 interfaces are more convenient to give in the form of

$$r_{01p} = \frac{N_1 \cos \Phi_0 - N_0 \cos \Phi_1}{N_1 \cos \Phi_0 + N_0 \cos \Phi_1} \quad (2.30)$$

$$r_{12p} = \frac{N_2 \cos \Phi_1 - N_1 \cos \Phi_2}{N_2 \cos \Phi_1 + N_1 \cos \Phi_2} \quad (2.31)$$

$$r_{01s} = \frac{N_0 \cos \Phi_0 - N_1 \cos \Phi_1}{N_0 \cos \Phi_0 + N_1 \cos \Phi_1} \quad (2.32)$$

$$r_{12s} = \frac{N_1 \cos \Phi_1 - N_2 \cos \Phi_2}{N_1 \cos \Phi_1 + N_2 \cos \Phi_2} \quad (2.33)$$

$$t_{01p} = \frac{2N_0 \cos \Phi_0}{N_1 \cos \Phi_0 + N_0 \cos \Phi_1} \quad (2.34)$$

$$t_{12p} = \frac{2N_1 \cos \Phi_1}{N_2 \cos \Phi_1 + N_1 \cos \Phi_2} \quad (2.35)$$

$$t_{12s} = \frac{2N_1 \cos \Phi_1}{N_1 \cos \Phi_1 + N_2 \cos \Phi_2} \quad (2.36)$$

Three angles  $\Phi_0$ ,  $\Phi_1$  and  $\Phi_2$  have the relation

$$N_0 \sin \Phi_0 = N_1 \sin \Phi_1 = N_2 \sin \Phi_2, \quad (2.37)$$

which are linked by the Snell's Law. To examine the change of amplitude and phase separately, as a plane wave is obliquely reflected from or transmitted by a film covered substrate, the overall complex amplitude and reflection ( $R_p, R_s$ ) and transmission

$(T_p, T_s)$  coefficients are written in terms of their absolute values and angles as

$$R_p = |R_p| e^{j\Delta_{rp}} \quad (2.38)$$

$$R_s = |R_s| e^{j\Delta_{rs}} \quad (2.39)$$

$$T_p = |T_p| e^{j\Delta_{tp}} \quad (2.40)$$

$$T_s = |T_s| e^{j\Delta_{ts}} \quad (2.41)$$

$|R_p|$  and  $\Delta_{rp}$  represent the amplitude attenuation and phase shift respectively for p-polarization. From measurements of the incident and reflected polarizations, the ratio

$$\rho_r = \frac{R_p}{R_s} \quad (2.42)$$

of the overall complex amplitude reflection coefficients of the ambient-film-substrate system for the p and s polarizations is determined. If we express  $\rho_r$  in terms of the ellipsometric angles  $\Psi$  and  $\Delta$  we find

$$\tan\Psi_r = \frac{|R_p|}{|R_s|} \quad (2.43)$$

$$\Delta_r = \Delta_{rp} - \Delta_{rs} \quad (2.44)$$

The relation between ellipsometric angles  $\Psi$ ,  $\Delta$  and Fresnel reflection coefficients is

given as

$$\tan \Psi e^{j\Delta} = \frac{r_{01p} + r_{12p}e^{-j2\beta}}{1 + r_{01p}r_{12p}e^{j2\beta}} \times \frac{1 + r_{01s}r_{12s}e^{-j2\beta}}{r_{01s} + r_{12s}e^{-j2\beta}} \quad (2.45)$$

From equations 2.23 and 2.37;

$$\tan \Psi e^{j\Delta} = \rho(N_0, N_1, N_2, d_1, \phi_0, \lambda) \quad (2.46)$$

breaking into two real equations gives

$$\Psi = \arctan |\rho(N_0, N_1, N_2, d_1, \phi_0, \lambda)| \quad (2.47)$$

$$\Delta = \arg [\rho(N_0, N_1, N_2, d_1, \phi_0, \lambda)] \quad (2.48)$$

where  $|\rho|$  and  $[\rho]$  are the absolute value and angle of the complex function  $\rho$ , respectively. Equations 2.47 and 2.48 which are not easy to handle with nine parameters (six from  $N$  and three from other values) can be solved by a software program included in the ellipsometry [2].

The thickness and refractive index can be extracted from the experimentally measured  $\Psi$  and  $\Delta$  pairs by fitting to a model. The best fit to experimental data is determined by minimizing the MSE (mean square error) which is equal to;

$$MSE = \frac{1}{2N - M_i} \sum \left[ \left( \frac{\Psi_i^{mod} - \Psi_i^{exp}}{\sigma_{\Psi,i}^{exp}} \right)^2 + \left( \frac{\Delta_i^{mod} - \Delta_i^{exp}}{\sigma_{\Delta,i}^{exp}} \right)^2 \right] \quad (2.49)$$

where  $N$  is the number of  $(\Psi, \Delta)$  pairs,  $M$  is the number of variable parameters,  $\sigma$  are the standard deviations of the experimental data points, and the superscripts mod and experimental values respectively. [3]

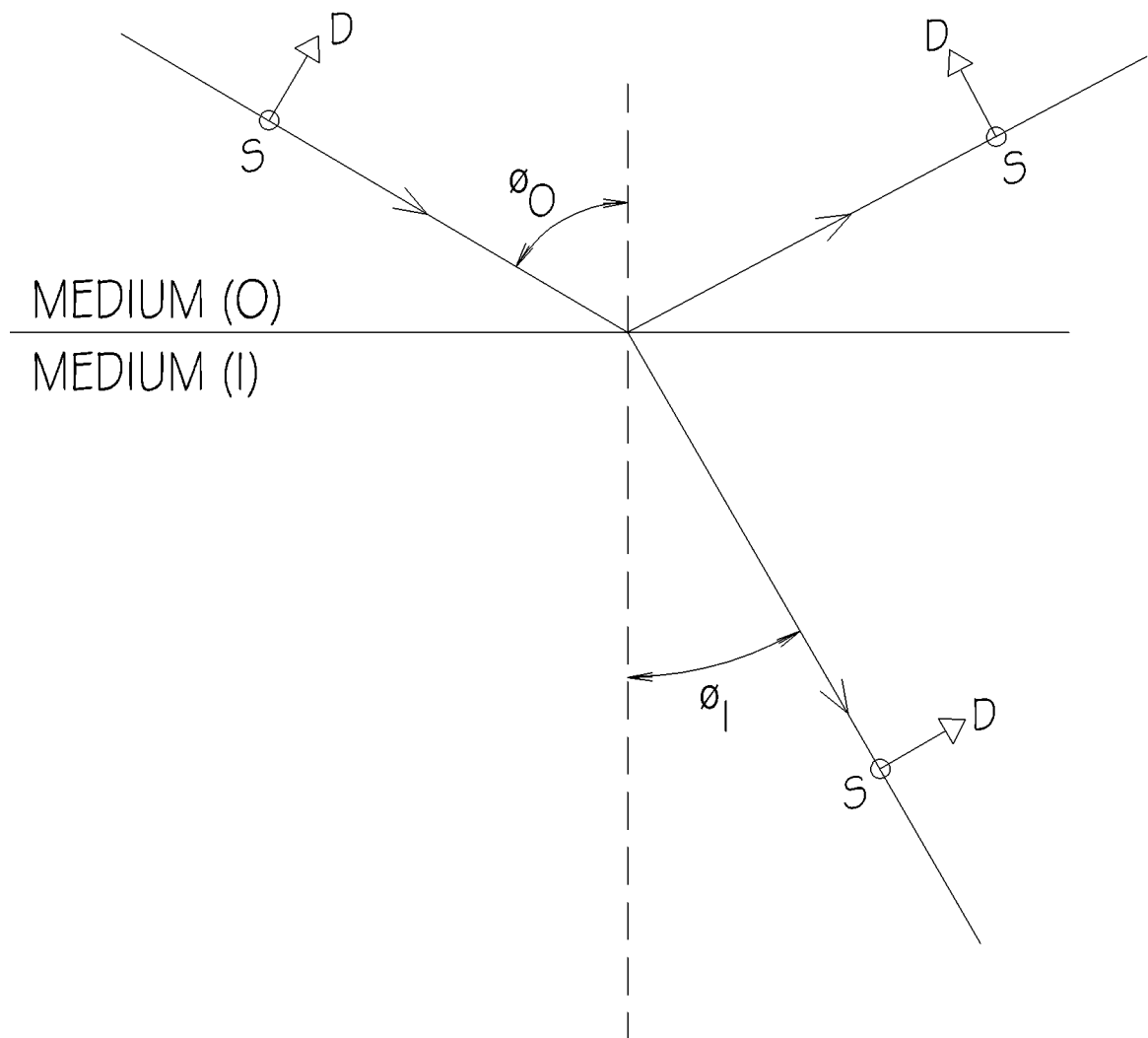


Figure 2.4. Reflection and transmission of a plane wave at the planar interface between two semi-infinite media.  $p \times s$  gives the direction of propagation.

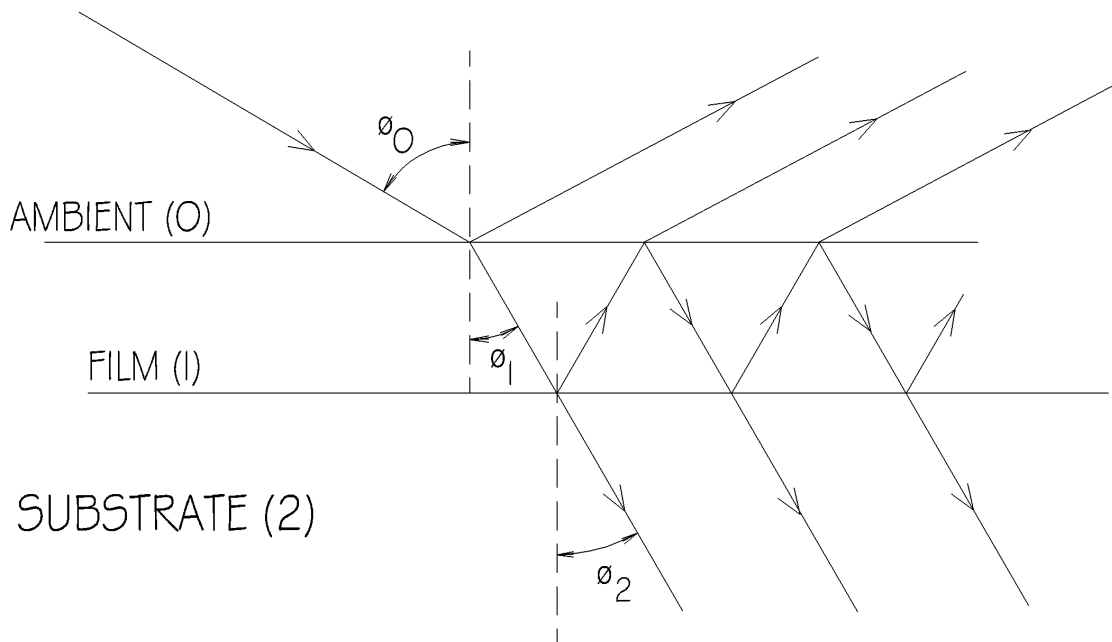


Figure 2.5. Reflection and transmission of a plane wave by an ambient(0)-film(1)-substrate(2).

### 3. EXPERIMENT AND RESULTS

#### 3.1. Gold Film Preparation

31 nm thick (measured by ellipsometry) Au layer is evaporated onto BK7 glass by vacuum evaporation technique using an Edwards Coating System (E306A). BK7 glasses are cleaned with acetone and methanol, then they are put into the evaporation machine that includes a holder. Very tiny gold particles cut from a gold plate. Gold is placed into the tungsten boat and is evaporated by the resistive heating [5].

#### 3.2. Peg Film Preparation

1000mg/ml PEG with MW=1000 is dissolved in Chloroform  $CHCl_3$  at room temperature.  $CHCl_3$  solvent with high volatility is used to decrease the non-homogeneity of the PEG film. The gold coated glass surface was blown with nitrogen in order to get rid of small dust particles and placed on a spincoater. The polymer solution was flowed through a GPC filter and dropped on the surface. The substrates were then spin-coated at 500 rpm for 10 seconds and 600 rpm for 30 seconds, respectively. 534 nm films were obtained [4].

#### 3.3. $SiO_2$ Film

Silicon dioxide film naturally grows on the Si surface due to oxidation as a result of interaction of the Si with atmosphere.

#### 3.4. Optical Setup(Ellipsometry)

Ellipsometry is used for the determination of the refractive index and thickness of PEG,  $SiO_2$ , Au layers.

Ellipsometry is a device with two arms, one is sending light the other is detecting

light with a certain wavelength; 632.8 nm He-Ne Laser onto the sample of interest.

Angle of incidence can change from 20 to 90 degrees, which can be increased 5 degrees in each step. 90 degree is not used for measurement but for adjustment.

As seen in figure 3.1 laser light source in the first arm follows the optical path and passes through a polarizer prism. Circularly polarized light is then converted into a linearly polarized form. A constant intensity linearly polarized beam is obtained depending upon the presence of a quarter wave compensator. The usage of this quarter wave compensator is determined by the computer. In the presence of this quarter wave compensator, a circularly polarized beam is obtained. The beam is projected onto the sample of interest. Depending upon the optical properties of the sample, the intensity of the incoming light beam is changed and reflected exactly onto the second arm because angle of incidence and reflectance are adjusted to the same value.

Reflected light passes through rotating analyzer prism and is then sensed by a photodetector. Photodetector converts light energy into the electric current. Before the photodetector, a filter is used to eliminate the light, which has a wavelength other than the incident wavelength. A picture of the ellipsometer device we used in our experiments is given in figure 3.2.

### 3.5. Results

The  $SiO_2$  film used in this work is naturally grown on Si substrate and is measured to have a thickness of 76.63 nm. The value of index of refraction is found to be between 1.458410 and 1.456710.(see figures 3.3 and 3.4). Gold film, coated on BK7 glass is measured to have a thickness of 31.80 nm. Refractive index  $n$  has a value of 0.166, where  $k$  equals to 3.150(see figures 3.5 and 3.6). Lastly, PEG film on top of BK7+Gold Film system is in situ studied in a relative humidity environment to have thicknesses of 534.34 nm, 538.85 nm, 660.70 nm at relative humidities 37% , 50% and 80% respectively(see figures 3.7, 3.8 and 3.9).

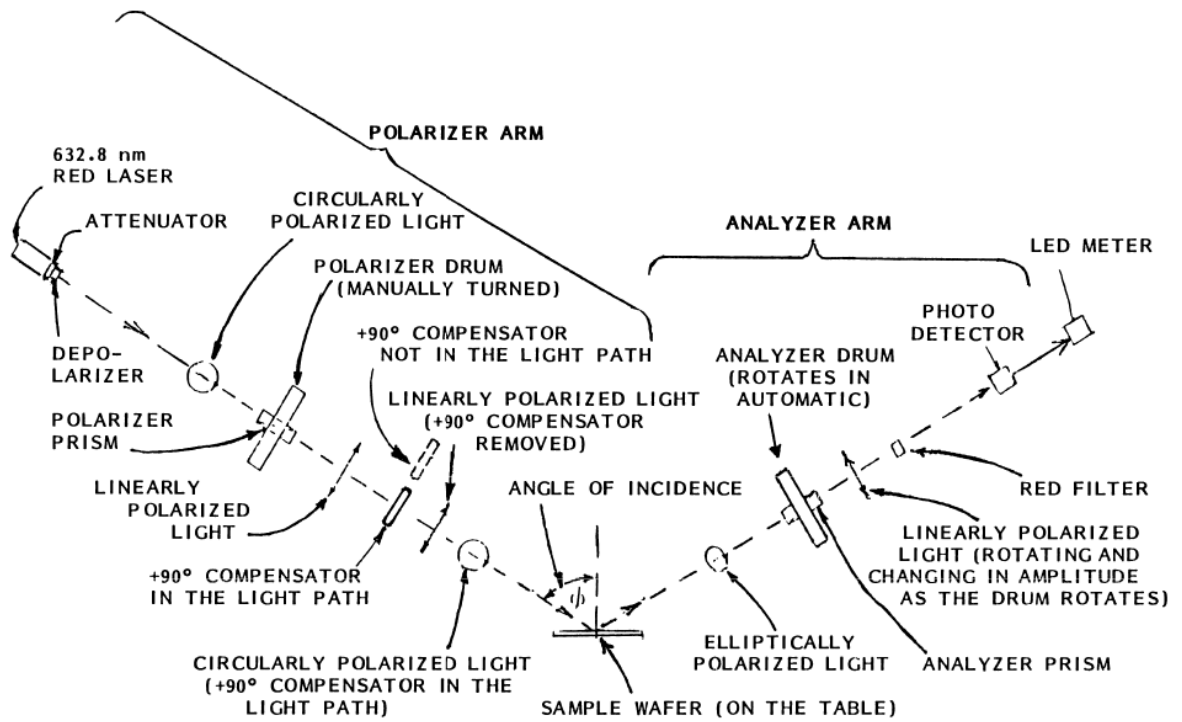


Figure 3.1. Optical setup under the ellipsometry



Figure 3.2. Picture of the ellipsometry used in our experiments

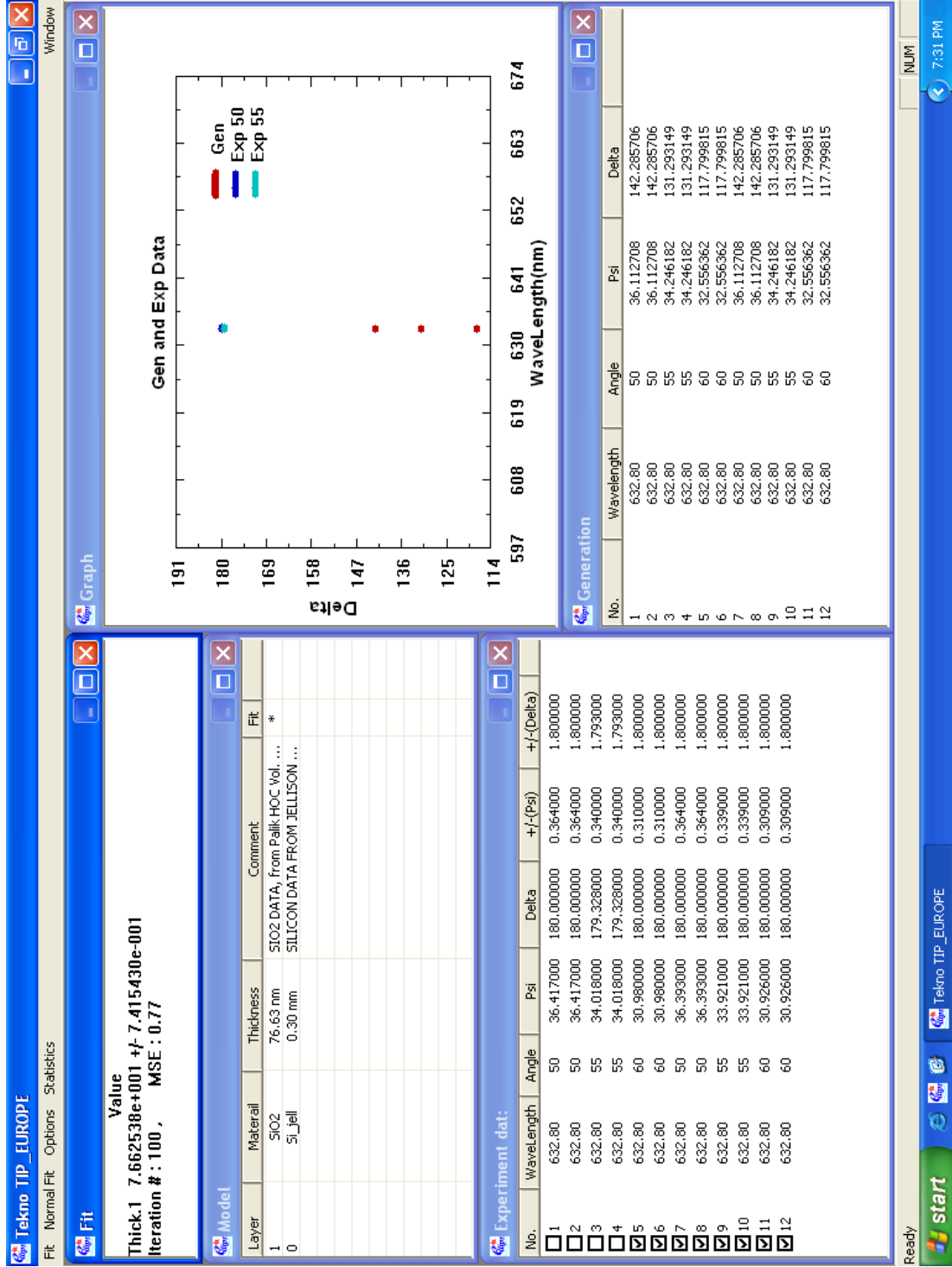


Figure 3.3. Thickness calculation of  $SiO_2$  film

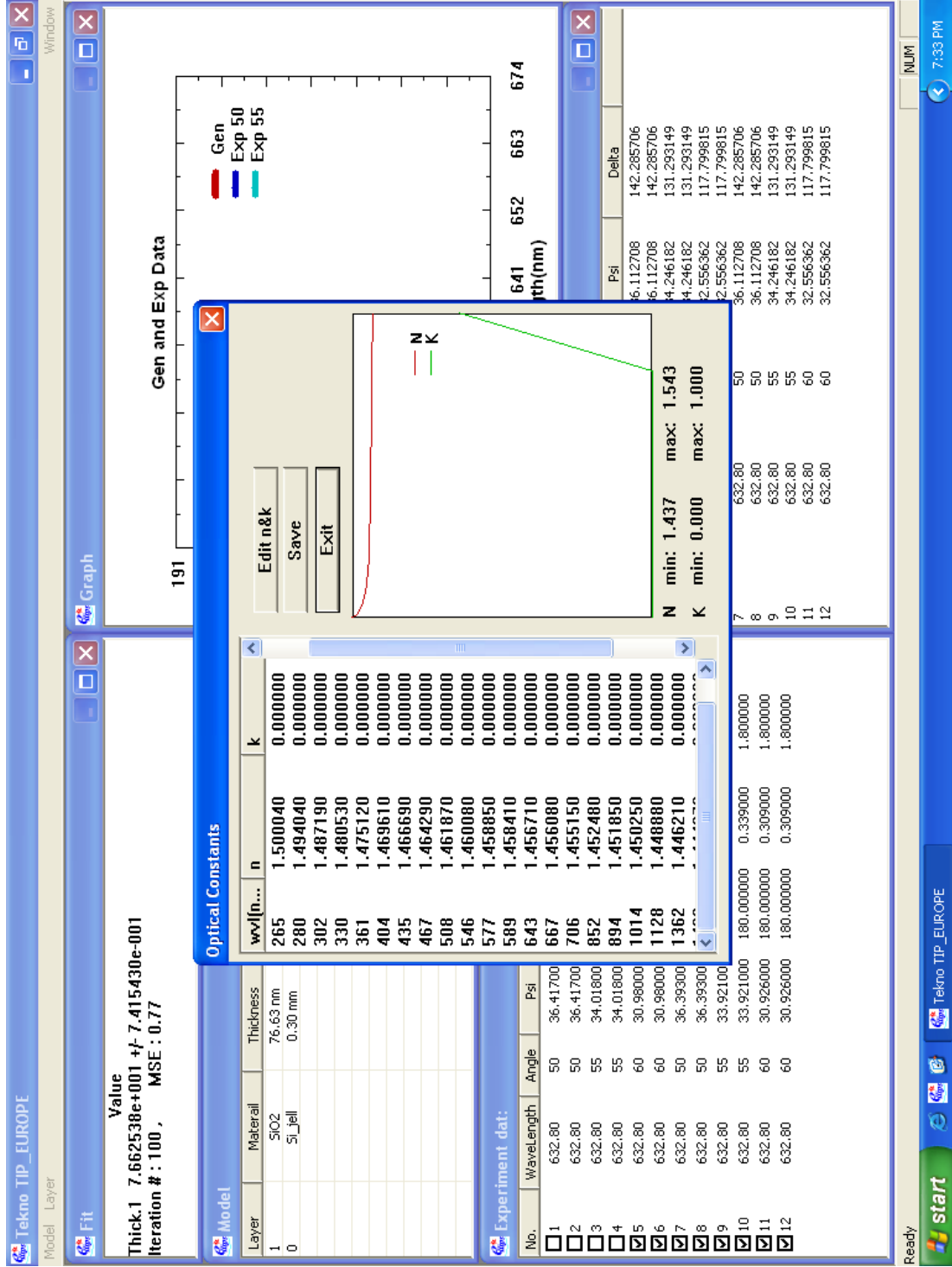


Figure 3.4. n calculation of SiO<sub>2</sub> film

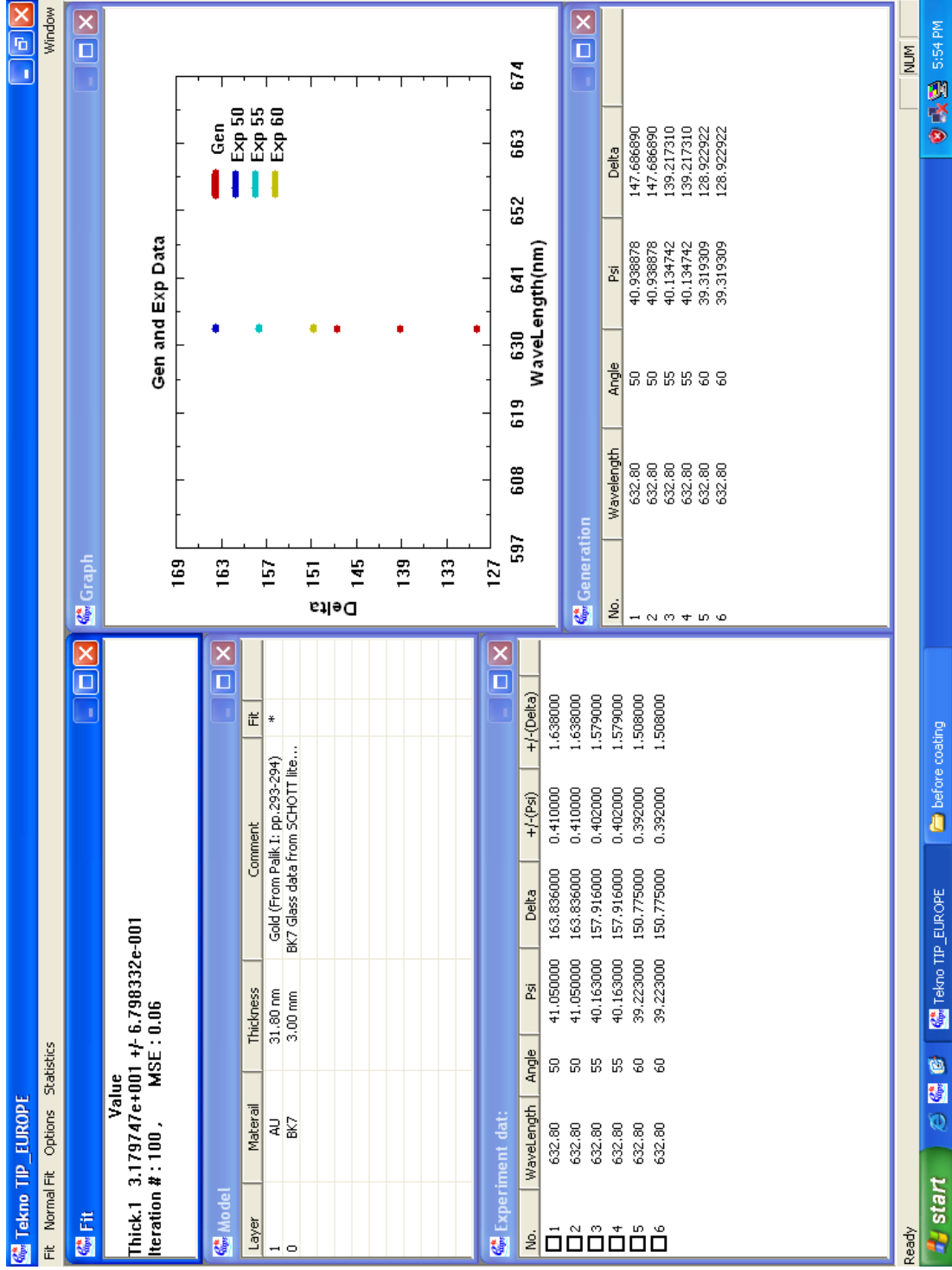


Figure 3.5. Thickness calculation of Au film

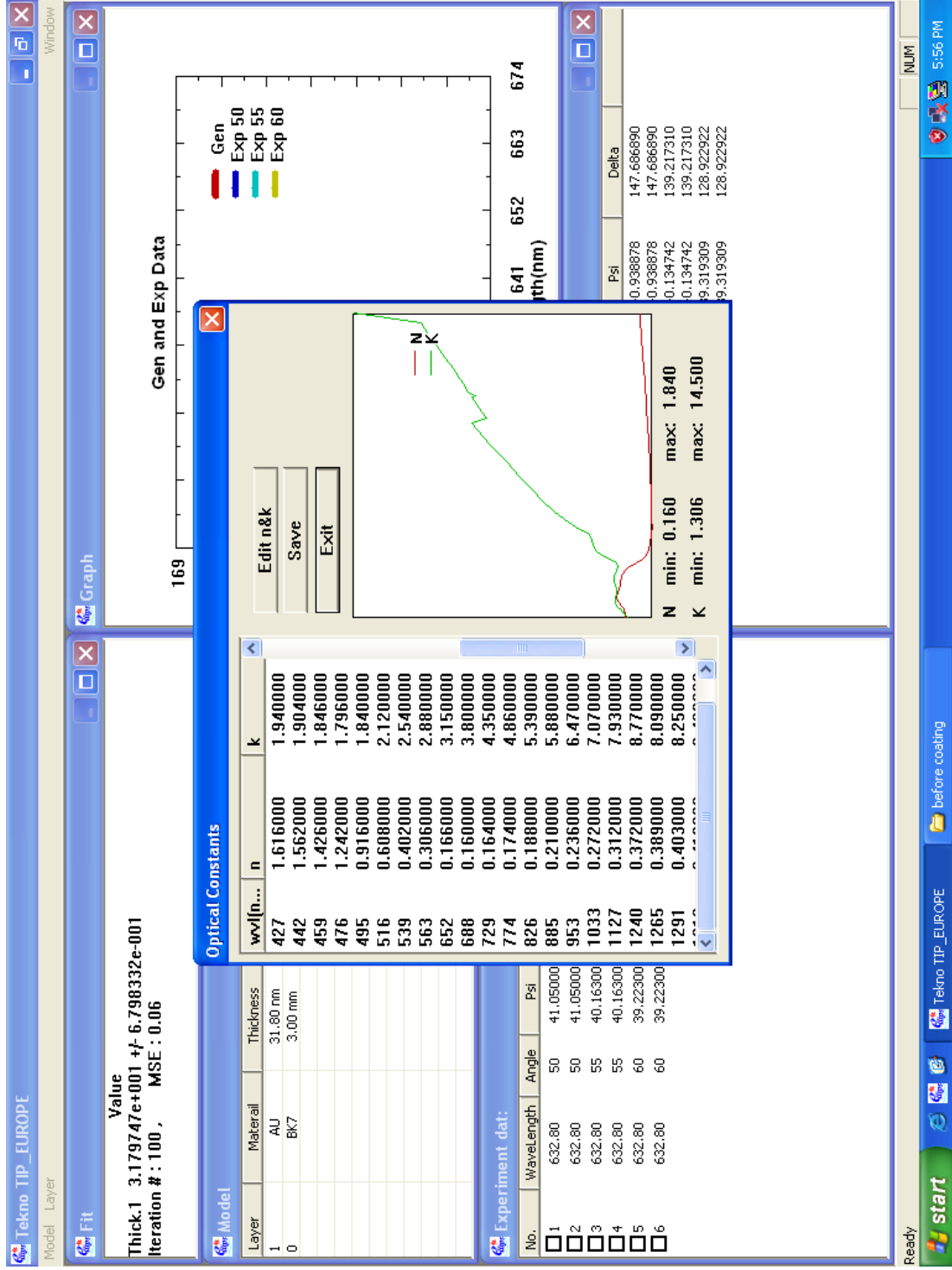


Figure 3.6. n and k calculation of Au film

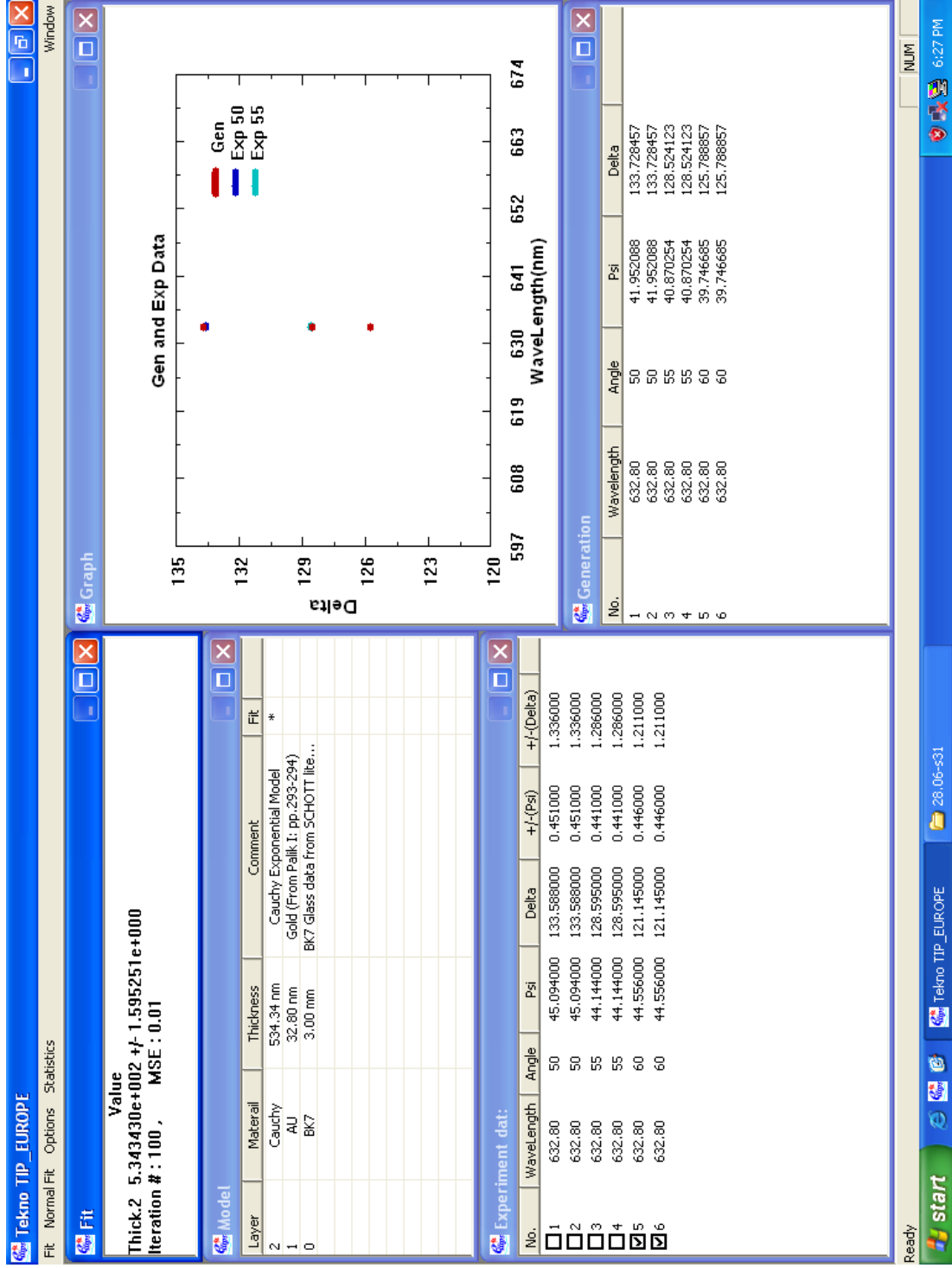


Figure 3.7. Thickness calculation of PEG film at 37% humidity

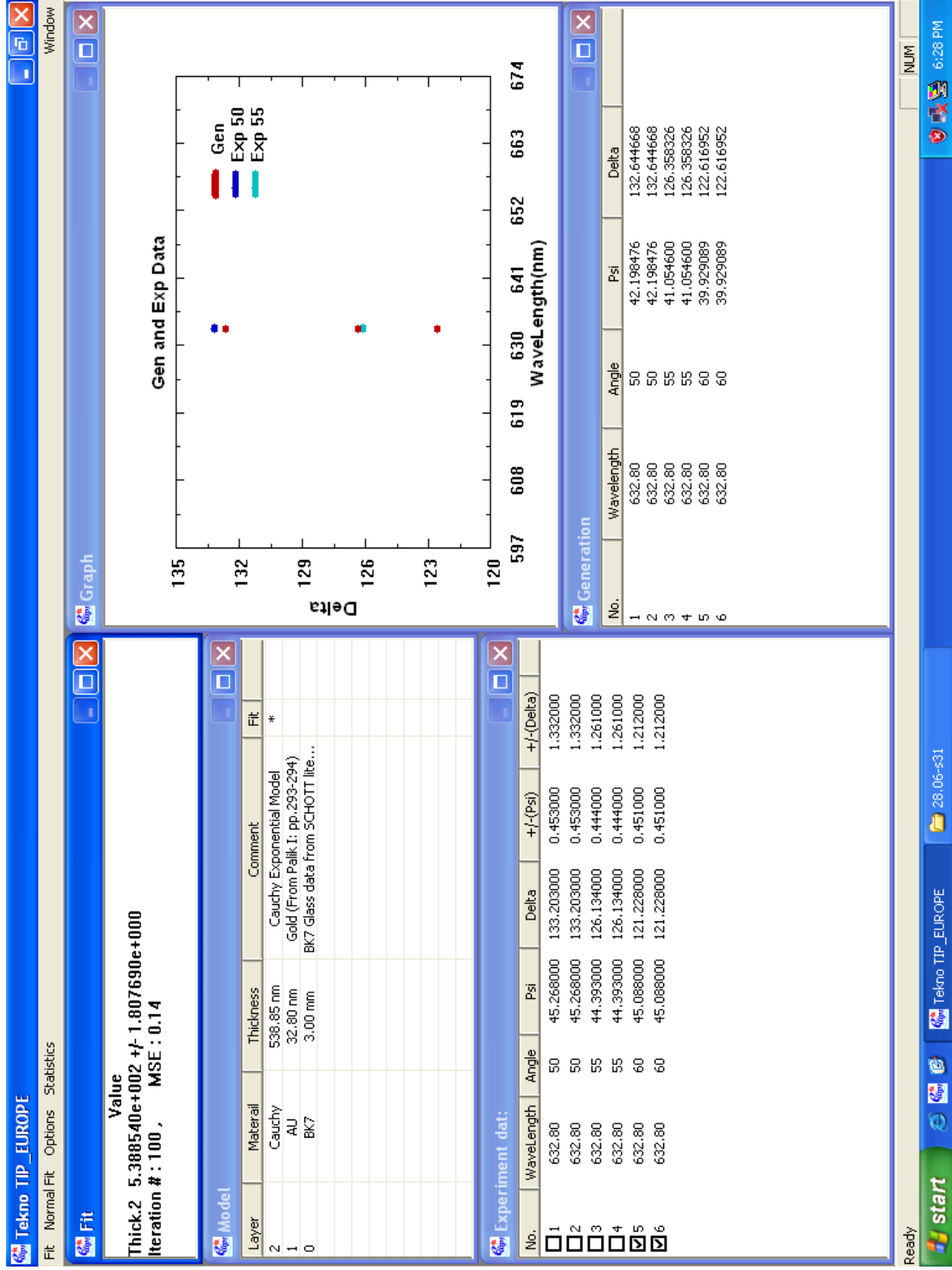


Figure 3.8. Thickness calculation of PEG film at 50% humidity

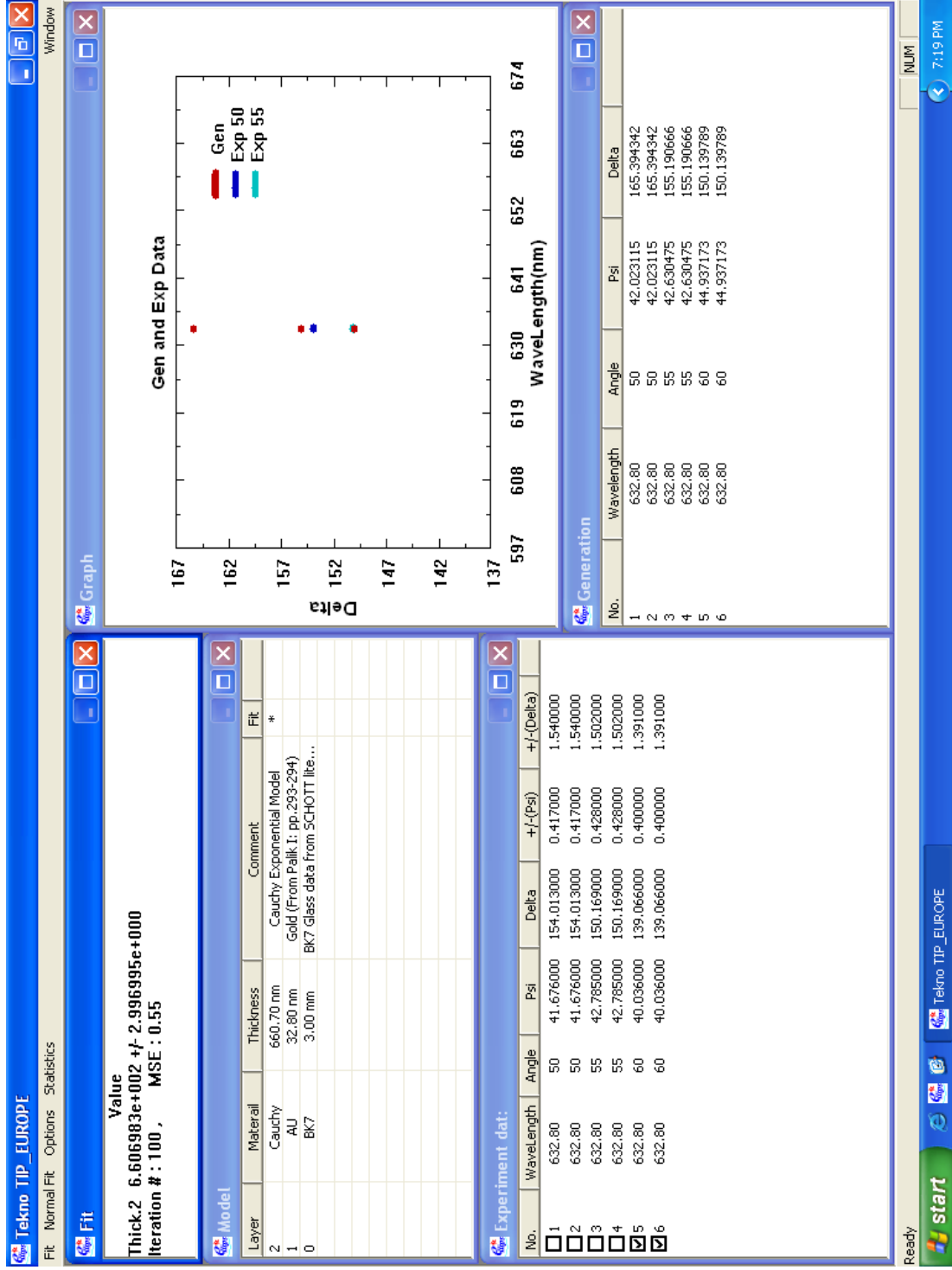


Figure 3.9. Thickness calculation of PEG film at 80% humidity

## 4. CONCLUSION

In this study, physical and optical properties of thin films are measured. Thickness of PEG film is measured at three different relative humidity levels. In many experimental work, film thickness is an important quantity. For some experiments, determination of thickness is the aim of the experiment while an intermediate step for some others. Ellipsometry provides quick, non-damaging, sensitive measurements.

Ellipsometry provides a straightforward and practical advantage for in situ measurements of  $n$  and  $d$ , which can be very useful for a future work. In this work, thickness of a PEG film is in situ measured at only three different values. However, this range can be extended further and as a future work, different kinds of polymer films can be set into a special chamber the humidity level may be extended from 10% to 100% to compare films and their values with each other. This technique can be interrogated in conjunction with other optical measurements such as optical sensors where changes of refractive index and thickness are desirable.

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