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Surface Analysis of Optical Flint Substrate by Multiple Angle Ellipsometric Method

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Abstract: The present research consists in characterizing the surface quality of polished optical glass (LF7) by ellipsometry and use of effective medium model. A correlation between effective roughness layer and ellipsometric measurements has been established as glass fraction f %. Ellipsometric parameters in dependence of polishing time have been measured and treated on the base of the theory of Maxwell-Garnett assuming an effective medium layer of effective thickness d_e and refractive index n_e.

Key words: Ellipsometry, effective medium, surface roughness, optical testing, polishing

INTRODUCTION

The choice of the roughness measurement method is related to the importance of the required information, the product quality and its purpose in the market, because the measuring accuracy of roughness is strongly related to the measurement method.

The contact methods for the determination of roughness are undesirable because they are destructive and let prints on tested surface. They are limited in the field of the space frequencies, i.e., they are not successful when surface has a very low roughness.

Quality control of a functional surface must be precise, in order to give to this one an optimal functional state, enough rapid and economic.

There are many methods of measuring surface roughness, such as image processing, microscopes, stylus type instruments, profile tracing instruments, etc.(Çolak *et al*., 2007).

This research consists in presenting a method without mechanical contact, based on an optical phenomenon ellipsometry which is the change of the state of the light polarization after reflection on optical surface.

The ellipsometry is an optical technique devoted to the analysis of surfaces. To define roughness parameters by ellipsometry, the top rough layers were treated as thin films according to the effective medium approximation (Mykhaylyk *et al*., 2007). Details about the Influence of lateral dimensions of the irregularities on the optical quantities of rough surfaces using effective medium approximation have been reported in reference (Franta and Ohlidal, 2006).

Theory: Nothing is richer in information than a surface, where it has a determining role on the geometrical, mechanical, thermal, physicochemical and optical properties. A surface is generally characterized by the heights H_i of its asperities compared to its mean level. The most parameter used to characterize roughness is the average quadratic height δ, also noted R_q. (Marioge, 1993; Bennet and Mattsson, 1999).

$$\delta = \sqrt{\frac{1}{N}\sum_{i=1}^{N} H_i^2} \tag{1}$$

However, within ellipsometry and roughness model such as effective medium approximation it is possible to obtain the relationship between the rms values of the heights as volume fraction and the ellipsometric parameters.

Ellipsometry is an optical technique of characterization, non destructive and without contact. It is based on exploiting the polarization transformation that occurs as a beam of polarized light when this is reflected under oblique incidence.

After reflection the vibration is elliptic; ellipsometry analyzes this ellipse by the intermediary of the ratio ρ of the coefficients of parallel and perpendicular reflection (Azzam and Bashara, 1977; Bouafia and Manalla, 2000; Vorburger and Ludema, 1988).

The ellipsometry makes it possible to determine in a precise way either permittivity or thickness of the thin layers (Perrin, 1981).

Let us consider a plane interface separating two isotropic homogeneous transparent media of complex refractive indices $N_1 = n_1 + ik_1$ and $N_2 = n_2 + ik_2$, respectively.

The plan of incidence, as shown in Fig. 1, is defined by the normal at the interface and the vector of the incidental wave; in which θ_1 is the incidence angle of the

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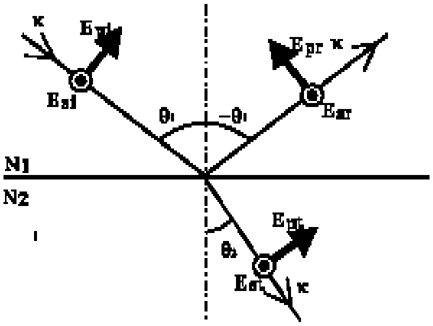

Fig. 1: Reflection and transmission with the interface between two homogeneous media of different indices

electromagnetic wave and θ_2 is the refraction angle under which the transmitted wave is propagated (Bosch, 1991; Yokota *et al.*, 1969).

E_i, E_r and E_t indicate the incident, reflected and transmitted electric fields, respectively.

The indices p and s designate that is about the parallel or perpendicular components to the plan of incidence and κ the wave vector.

The reflection coefficients have complex numbers that can be written in form:

$$r_p = \frac{E_{pr}}{E_{pi}} = |r_p| \exp(j\delta_p) \qquad (2)$$

$$r_s = \frac{E_{sr}}{E_{si}} = |r_s| \exp(j\delta_s) \qquad (3)$$

The Snell-Descartes law gives the relation between the angles θ_1 and θ_2:

$$N_1 \sin(\theta_1) = N_2 \sin(\theta_2) \qquad (4)$$

In addition, the Fresnel equations translate the continuity of the tangential component of the electric field to the interface:

$$r_p = \frac{E_{pr}}{E_{pi}} = \frac{N_2 \cos(\theta_1) - N_1 \cos(\theta_2)}{N_2 \cos(\theta_1) + N_1 \cos(\theta_2)} \qquad (5)$$

$$r_s = \frac{E_{sr}}{E_{si}} = \frac{N_1 \cos(\theta_1) - N_2 \cos(\theta_2)}{N_1 \cos(\theta_1) + N_2 \cos(\theta_2)} \qquad (6)$$

ρ is the following ratio given as:

$$\rho = \frac{r_p}{r_s} = \tan(\Psi) \cdot \exp(j\Delta) \qquad (7)$$

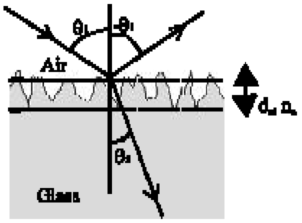

Fig. 2: Substitution of the roughened surface layer by an equivalent film with planeparallel boundaries

then

$$\tan(\Psi) = \frac{|r_p|}{|r_s|} \qquad (8)$$

and

$$\Delta = \delta_p - \delta_s \qquad (9)$$

A simplified approach to study the effect of surface roughness on ellipsometric parameters Ψ and Δ is to replace the roughened surface layer by an equivalent film with plane-parallel boundaries, Fig. 2 whose thickness is equal to a characteristic roughness height parameter (e.g., the rms value) and whose optical properties are determined from those of the substrate and ambient according to the theory of Maxwell-Garnett (1904).

This theory is based on the representation of discontinuous film (or the roughened surface layer) by a random distribution of small-diameter (compared to the wavelength of light) spherical particles of film material embedded in a dielectric ambient.

The roughened surface layer can be seen as an inhomogeneous system which is proved to be equivalent to a homogeneous one with an effective complex refractive index n_e given by:

$$\frac{n_e^2 - n_a^2}{n_e^2 + 2n_a^2} = f\,\frac{n_m^2 - n_a^2}{n_m^2 + 2n_a^2} \qquad (10)$$

Where,

n_m and n_a = The refractive indices of the substrate and ambient materials, respectively.
f = The volume fraction occupied by the spherical particles in the discontinuous film (inclusions of glass in our case).

Often the ambient is vacuum or air so that $n_a = 1$ and Eq. 10 becomes:

$$\frac{n_e^2-1}{n_e^2+2}=f\,\frac{n_m^2-1}{n_m^2+2} \tag{11}$$

from which:

$$n_e^2=\frac{1+2fq}{1-fq},$$

and

$$q=\frac{n_m^2-1}{n_m^2+2}$$

This model is valid when both of heights and the spacing of the roughness asperities are much smaller than the wavelength of light.

Aspnes *et al.* (1979) pointed out how using this model in rough surface applications. Note that many other models were developed for studying and determining surface roughness (Vorburger and Ludema, 1988; Fenstermaker and McCrackin, 1969; Ohlîdal and Lukes, 1972; Bruggeman, 1935).

MATERIALS AND METHODS

Present research consists in studying the evolution of the surface quality of polished optical glass according to the state of polarization of the light after reflection on the surface of this one and for different polishing time PT. The effective medium approximation can then be used to analyze the measured ellipsometric values assuming the effective medium layer to consist of a physical mixture interface of air and glass.

The glass sample is of Flint LF 576/424 (LF7), with index of refraction N = 1,572. First, the sample undergoes a grinding to bring back roughness to the order of micrometers. For that surface is subjected to the action of grains of abrasive with decreasing fractions of alumina powder: F30, F15, F9 and F3. A revolving disc of a speed of 88 tr/min ensures grinding.

Then the polishing operation is carried out with a polyurethane polishing brush using oxide of cerium as polishing agent whose concentration in grains is of 10 g cm^{-3} in distilled water. A polishing force of 60 N approximately is applied. The choice of grinding and polishing parameters was justified by (Bouzid *et al.*, 1997).

The measuring equipment is an ellipsometer SE 400 of Sentech Instruments GmbH, it is mounted on a support at which a telescope of autocollimation and two arms are fixed. These arms swivel around a common center of rotation. The polarizer module is placed in one limp metal fixed at the left arm. It is composed of a laser He-Ne (λ = 6328 Å), a polarizer and a compensator and the analyzer module is assembled on the right-hand arm, where all the mechanical and electronic components are placed in one limp metal.

The measured values of Ψ and Δ according to the angle of incidence on the interval of 40 to 70° are gathered in Table 1 versus polishing time from 0 min until 16 min, with steps of 2 min.

RESULTS

The corresponding curves to the measured ellipsometric angles Psi and Delta are shown in Fig. 3 and 4.

The method of modeling consists in determining the values of the index of refraction n_e, thickness d_e and glass refraction f of the roughened surface, starting from the measured values of Ψ and Δ by means of SE400 software of Sentech Instruments GmbH.

The results obtained allow drawing up the Table 2 for the values of n_e, d_e and f versus polishing time for different angle of incidence, by considering that the roughened surface be a layer made up of an effective medium of air-glass with refraction index n_e and of thickness d_e.

Note that by the relation of Maxwell-Garnett (1904) we determine the quantity f which represents the percentage of the glass as volume fraction situated between two limits of the layer with effective thickness d_e. The most striking difference in behavior of a smooth and a rough surface is the fact that the a smooth plane will

Table 1: Values of Ψ and Δ for different values of angle of incidence

	Angle 40°		Angle 45°		Angle 65°		Angle 70°	
t (min)	Ψ (°)	Δ (°)	Ψ (°)	Δ (°)	Ψ (°)	Δ (°)	Ψ (°)	Δ (°)
0	07.79	336.83	08.14	340.26	09.27	355.58	10.89	338.80
2	49.80	39.55	06.23	348.84	14.51	357.93	19.73	350.05
4	50.45	43.32	05.83	348.94	15.04	357.89	20.86	350.70
6	63.57	58.87	05.70	351.24	15.17	357.31	21.26	349.15
8	67.18	66.60	05.48	358.52	15.39	357.19	21.55	349.05
10	84.43	82.90	05.36	06.72	15.56	357.00	21.76	348.78
12	89.14	87.71	05.23	07.38	15.78	356.99	22.07	348.37
14	89.65	273.02	05.19	10.75	16.07	356.24	23.02	347.81
16	87.89	277.68	05.44	10.87	15.63	356.41	22.61	348.40

Table 2: Values of d_e, n_e and f % for different values of angle of incidence

	Angle 40°			Angle 45°			Angle 65°			Angle 70°		
t (min)	d_e (nm)	n_e	f %	d_e (nm)	n_e	f %	d_e (nm)	n_e	f %	d_e (nm)	n_e	f %
0	180	1.153	0.301	195	1.135	0.266	225	1.111	0.220	235	1.097	0.193
2	164	1.214	0.414	170	1.200	0.389	190	1.181	0.353	191	1.173	0.338
4	162	1.219	0.424	167	1.212	0.411	185	1.190	0.370	186	1.185	0.361
6	159	1.222	0.429	165	1.215	0.416	179	1.192	0.374	179	1.188	0.366
8	156	1.227	0.438	160	1.220	0.425	176	1.196	0.381	177	1.191	0.372
10	153	1.229	0.442	155	1.224	0.433	173	1.199	0.387	175	1.193	0.376
12	152	1.231	0.446	154	1.228	0.440	171	1.203	0.394	172	1.196	0.381
14	150	1.232	0.447	152	1.230	0.444	163	1.208	0.403	165	1.206	0.400
16	150	1.225	0.444	153	1.223	0.442	168	1.200	0.389	169	1.202	0.392

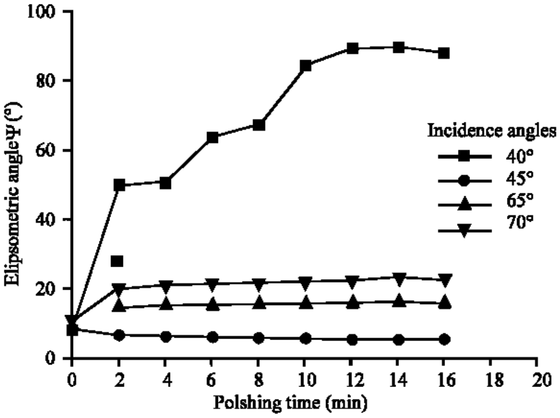

Fig. 3: Variation of Ψ according to the polishing time

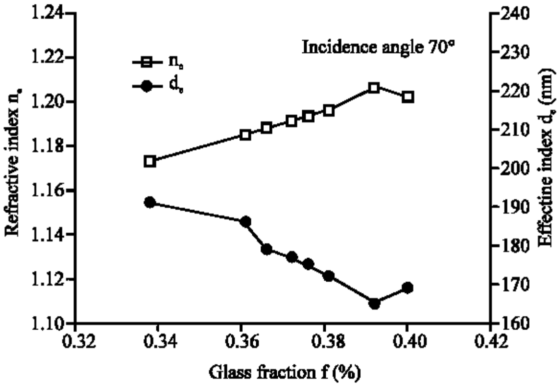

Fig. 5: Variation of refractive index n_e and effective thickness d_e versus glass fraction f %

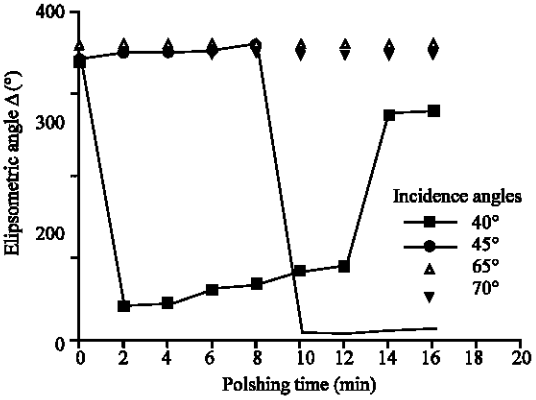

Fig. 4: Variation of Δ according to the polishing time

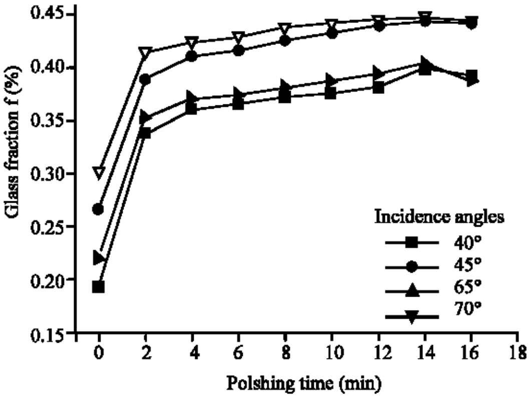

Fig. 6: Variation of the fraction of glass f % versus

reflect the incident wave specularly in a single direction, whilst a rough surface will scatter into various directions and certain privileged directions may receive more energy than others, consequently the same surface may be either rough or smooth for different angles of incidence (Beckmann and Spizzichino, 1963).

Figure 5 shows the variation of index n_e and effective thickness d_e by an incidence angle of 70° according to glass fraction f for polishing time evolution (Table 2).

The distribution of air and glass in effective layer (Fig. 6) is determined by fitting the measured values during polishing processes. The behavior of the glass fraction as function of polishing time represents the evolution of the surface quality.

We can see obviously that the glass fraction increases versus the polishing time between the two limits of the effective layer. At the optimal polishing time value, i.e. at PT = 14 min, the surface reaches the best quality, then by longer polishing time the glass fraction decreases and therefore, it is an indication that the roughness increases according to the commonly used effective medium approximation by Maxwell-Garnett (1904).

The variation of r_p and r_s according to the angle of incidence shows the favorable zones of measurement for an interface air-glass.

For an ideal interface, i.e., abrupt, between two media of indices of refraction N_1 and N_2, ρ is cancelled and changed sign to the Brewster incidence which is defined by $\tan \theta_B = N_1/N_2$. In the case of a real surface, i.e. nonnull thickness, ρ takes with θ_B a nonnull value, measurable, which depends on the index N and thickness d of the layer or the interface. For a very thin layer Ψ presents at θ_B a nonnull minimum but not easily detectable. Dephasing Δ is extremely sensitive to any variation of N and d, thus in the vicinity of the Brewster angle, witch is about 57, 54° degree for optical glass LF7, the measurement sensitivity is maximum. The judicious choice is to work with an incidence angle shifted of the Brewster angle which constitutes a compromise between a great sensitivity in Δ and a sufficiently detectable ellipticity (Gauthier, 2000).

So, the best curves (i.e., the finest values) are obtained by the incidence angles of 65 and 70°. These angles are the most used in thin films characterization using ellipsometry.

CONCLUSION

The measured ellipsometric parameters enabled us to lead to a correlation with roughness It is necessary to use a model to describe the ellipsometric response of a sample i.e., to calculate the coefficients of reflection and thus find the measured Ψ and Δ. The variation of r_p and r_s according to the angle of incidence shows the zones favorable of measurement for the interface air-glass: In the vicinity of the angle of Brewster the sensitivity of measurement is highest that we will have to avoid.

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