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Characterization of sputtered aluminum oxide films using spectroscopic ellipsometry

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Abstract: Aluminum oxide thin films were prepared on polished silicon wafers by radio frequency sputtering. The optical constants and the thicknesses of the deposited layers were determined by spectroscopic ellipsometry in the wavelength range from 191 *nm* to 1690 *nm*. The dielectric function of the aluminum oxide thin films was modelled by the combination of the Cauchy dispersion relation and the Urbach absorption tail. The optical properties of the sputtered films were compared with those prepared by atomic layer deposition, electron beam evaporation, and pulsed magnetron sputtering in other laboratories.

Keywords: Aluminum oxide; optical properties; refractive index; sputtering; ellipsometry

1 Introduction

Aluminum oxide thin films find various important applications in technology for example in optoelectronics, tribology, sensorics, nanolithography because of their attractive properties. These films have been deposited by various techniques as magnetron sputtering [1], atomic layer deposition [2,3,4], electron beam evaporation [5,6,7], spray pyrolysis [7], oxidation of aluminum film [6]. The precise knowledge of the optical properties and the thickness of the deposited layers are important in the design of optical film systems. The aim of the present communication is to report the optical constants of aluminum oxide thin films deposited at room temperature by radio frequency sputtering.

2 Experimental

Thin aluminum oxide layers were deposited by RF (Radio Frequency) sputtering at room temperature onto single crystalline Si (100) substrates. RF sputter deposition was carried out in a Leybold Z400 apparatus evacuated at $5 * 10^{-5}$ Pa. Sputtering has been performed under a mixture of high purity argon and oxygen gases with an applied wall potential of 1500 V DC yielding a plasma pressure of 2.5 Pa. Oxygen was incorporated into the layers by flowing it continuously into the sputtering chamber at different flow rates, resulting partial pressure of oxygen up to 16% of the total one. The target was coupled to the RF generator operating at 13.56 MHz. A wafer of technical ceramic served as target for the low deposition rate process. We experienced that high purity ceramic (e.g. DUROX AL) is a dense, non-porous, vacuum tight material which makes it excellent for sputtering targets. The disadvantage of the RF sputtering method is that the deposition rate is low; it is about 4 nm/min. The Table (1) shows the preparation parameters for the different samples.

Table 1: Preparation parameters for the different samples.

Sample number	Partial pressure of oxygen [%]	Sputtering power [W]
A426-b	16.0	255
A427-b	14.8	255
A428-b	14.0	255
A429-b	8.4	255
A430-b	6.0	255
A431-b	6.0	190
A432-b	12.0	130

Characteristic optical properties of thin film structures can be derived from spectroscopic ellipsometry (SE) measurement, which is known to be a high-precision optical characterization technique [8,9,10,11].

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Ellipsometry measures the change in polarized light upon light reflection on a sample. It measures two quantities, Ψ and Δ. These are the amplitude ratio Ψ and phase difference Δ between light waves known as p- and s-polarized light waves. The method of Variable Angle Spectroscopic Ellipsometry (VASE) allows measurements at multiple angles of incidence. A new angle will change the length of the light penetrating through the materials. Multiple angles are helpful to improve the confidence limits of the results yielded by the evaluation of the measured spectra.

Ellipsometric measurements have been done using a rotating compensator spectroscopic ellipsometer (Model M-2000DI produced by J.A. Woollam Co., Inc.) in the wavelength range of 191-1690 nm with angles of incidence: of 55°, 60°, 65° and 70°; the measured data were analyzed using the WVASE32 software [12].

3 Results and discussion

The optical constants of the deposited layers have been evaluated from SE measurements using a two layer optical model consisting of a surface roughness layer, a bulk-like layer and the substrate. The bulk-like layer represents the amorphous aluminum oxide film, whereas its refractive index and extinction coefficient was modelled using the Cauchy dispersion formula together with the Urbach absorption tail [1]. The roughness layer was taken into account on the basis of effective medium approximation [13], the roughness layer consists of 50% of the underlying material and 50% of void. The wavelength dependence of the refractive index and the extinction coefficient for the single crystalline silicon substrate was taken from the database of the WVASE32 software [12]. During the evaluation of the measured SE data, seven free parameters were involved in the computation: the thickness of the surface roughness layer, the thickness of the bulk-like aluminum oxide layer, and the other five ones are the parameters belonging to the combination of the Cauchy dispersion relation and the Urbach absorption tail. The calculated spectra were fitted to the measured ones using a regression algorithm. The measure of the fit quality is the mean square error (MSE). The unknown parameters are allowed to vary until the minimum of MSE is reached. In order to avoid the local minimum in the regression algorithm, a careful global search procedure has been applied (involving a wide range of initial parameter values).

Figs. (1) and (2) illustrate the measured and fitted values of the ellipsometric parameters Ψ and Δ for sample A432-b, respectively. The agreement between the measured and generated spectra is good. Fig. (3) shows the refractive index n and extinction coefficient k as function of wavelength for the sample A432-b.

Table (2) shows the parameters of the Cauchy dispersion relation and the Urbach absorption tail yielded by the evaluation besides the data of preparation.

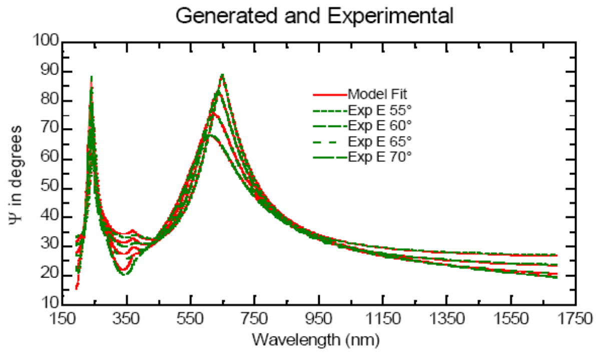

Fig. 1: Measured and fitted values of the ellipsometric parameter Ψ for sample A432-b using a two layer optical model.

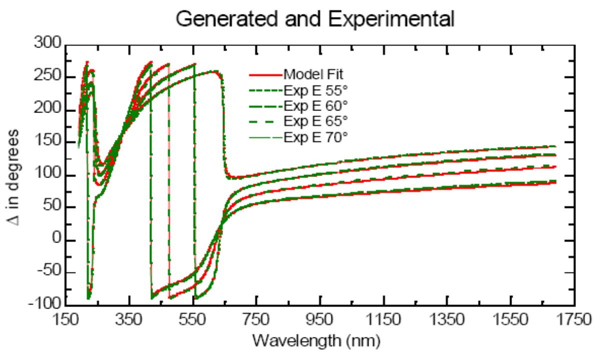

Fig. 2: Measured and fitted values of the ellipsometric parameter Δ for sample A432-b using a two layer optical model.

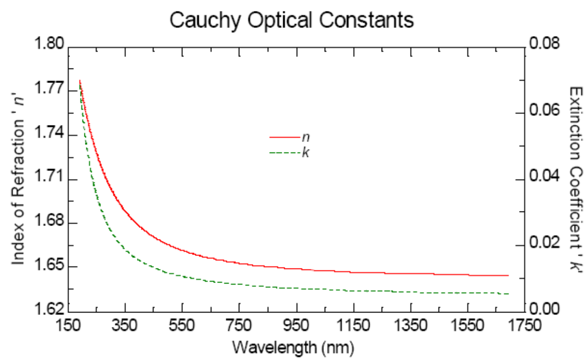

Fig. 3: Refractive index (n) and extinction coefficient (k) as a function of the wavelength for the bulk-like aluminum oxide layer of sample A432-b.

Table (3) summarizes the values of the refractive index n and extinction coefficient k at wavelength of 550 *nm* together with the layer thickness values, the preparation parameters are included as well. The total layer thickness is equal to the thickness of the bulk-like aluminum oxide layer plus the half of the thickness of the

Table 2: Parameters of the Cauchy dispersion (n = A + B/λ^2 + C/λ^4; columns 4-6) with Urbach absorption tail (k = G*$e^{H[E-F]}$ with F=6.49 eV [191 nm]; columns 7-8) together with the uncertainties yielded by the evaluation..

1	2	3	4	5	6	7	8
Sample number	Partial pressure of oxygen [%]	Sputtering power [W]	A	B	C	G	H
A426-b	16.0	255	1.6447± 0.0005	0.0070± 0.0001	-0.000053± 0.000007	0.094± 0.003	0.84 ± 0.02
A427-b	14.8	255	1.6441± 0.0005	0.0064± 0.0001	-0.000017± 0.000007	0.082± 0.003	0.90± 0.03
A428-b	14.0	255	1.6402± 0.0004	0.0071± 0.0001	-0.000044± 0.000006	0.083± 0.003	0.94± 0.03
A429-b	8.4	255	1.6026± 0.0008	0.0099± 0.0002	-0.00010± 0.00001	0.039± 0.004	1.0± 0.1
A430-b	6.0	255	1.6154± 0.0005	0.0080± 0.0002	-0.000040± 0.000008	0.051± 0.004	1.14± 0.07
A431-b	6.0	190	1.6174± 0.0004	0.0092± 0.0001	-0.00012± 0.000006	0.083± 0.003	0.91± 0.03
A432-b	12.0	130	1.6425± 0.0008	0.0059± 0.0001	-0.000036± 0.000007	0.069± 0.001	0.44± 0.01

Table 3: Columns 2-3 contain the preparation parameters, columns 4-8 contain the values of the refractive index n and extinction coefficient k at the wavelength of 550 nm together with the layer thickness values. The total layer thickness is equal to the thickness of the bulk-like aluminum oxide layer plus the half of the thickness of the surface roughness layer.

1	2	3	4	5	6	7	8
Sample number	Partial pressure of oxygen [%]	Sputtering power [W]	Refractive index n at the wavelength of 550 nm	Extinction coefficient k at the wavelength of 550 nm	Thickness of the surface roughness layer [nm]	Thickness of the bulk-like Al_2O_3 layer [nm]	Total layer thickness [nm]
A426-b	16.0	255	1.6674	0.0029	4.7± 0.2	116.4± 0.1	118.8 ± 0.2
A427-b	14.8	255	1.6651	1.6651	4.2± 0.2	120.0± 0.1	122.1± 0.2
A428-b	14.0	255	1.6632	0.0015	2.0± 0.2	131.79±0.09	132.8± 0.2
A429-b	8.4	255	1.6343	0.0005	4.4± 0.3	100.4± 0.1	102.6± 0.3
A430-b	6.0	255	1.6415	0.0004	6.4± 0.2	117.5± 0.1	120.7± 0.2
A431-b	6.0	190	1.6466	0.0018	3.1± 0.2	126.1± 0.1	127.7± 0.2
A432-b	12.0	130	1.6616	0.0107	2.4± 0.2	111.7± 0.1	112.9± 0.2

surface roughness layer. We found that the refractive index n varies in a relatively narrow range from 1.634 to 1.667 at the wavelength of 550 *nm* for different preparation parameters.

Table (4) lists the methods of preparation applied by different research groups, the substrate material, the thickness of the aluminum oxide layers and the refractive index at the wavelength of 550 *nm*. Kim et al. applied the atomic layer deposition technique and they have observed that the optical properties of the deposited aluminum oxide layers were considerably affected by the different substrates [3].

Similarly, Kumar et al. have found that the refractive index of the aluminum oxide films on silicon compared to that of films on soda lime glass is lower by about 0.03 [4]. Single crystalline silicon wafers usually have a native silicon oxide with a layer thickness of about 2-3 *nm*, this may contribute to the refractive index of the film as it is computed from the reflection spectra. The obtained difference, however, is approximately an order of magnitude larger than what may be caused by the presence of the native oxide, and it is approximately the same for the film thickness from 200 *nm* to 400 *nm*. One possible explanation may be based on the supposition that the densities of the aluminum oxide films growing on different substrates may exhibit different values.

4 Conclusion

Aluminum oxide thin films were deposited on single-crystalline silicon by radio frequency sputtering at room temperature. The as-deposited films showed refractive index at the wavelength of 550 *nm* in the range of 1.634 - 1.667. The optical properties of the sputtered films were compared with those of layers prepared by

Table 4: Method of preparation applied by different research groups for deposition of aluminum oxide layers, the substrate material, layer thicknesses and refractive indices at the wavelength of 550 nm of the aluminum oxide layers and the reference. Our results are indicated by the words of "Present work".

Method of preparation	Substrate material	Layer thickness [nm]	Refractive index n at wavelength of 550 nm	Reference
Atomic layer deposition	Silicon	10-38	1.62	[3]
Atomic layer deposition	100-nm-thick SiO2 covered Silicon	10-38	1.64	[3]
Atomic layer deposition	Silicon	200-400	1.645	[4]
Atomic layer deposition	Soda lime glass	200-400	1.675	[4]
Radio frequency sputtering	Silicon	112-132	1.634-1.667	present work
Electron beam evaporation	Quartz	80-135	1.61	[7]
Electron beam evaporation	Silicon	1262	1.753	[5]
Pulsed magnetron sputtering	Silicon	1130-1960	1.645-1.693	[1]

atomic layer deposition, electron beam evaporation, and pulsed magnetron sputtering in other laboratories.

Acknowledgments

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