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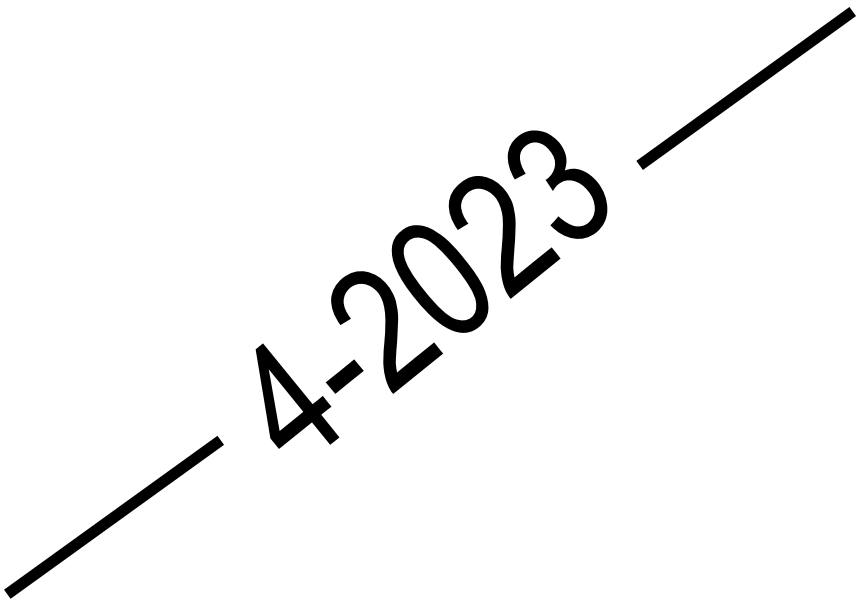

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FTIR-АНАЛИЗ ТОНКИХ ПЛЕНОК КРЕМНИЯ И ОКСИДА КРЕМНИЯ

FTIR ANALYSIS OF SILICON AND OXIDE SILICON THIN FILMS

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Annotatsiya

FT-IR spektrometrlari zamonaviy analitikaning turli muammolarini hal qilish uchun ishlatiladi. Sifatli tahlil qilish, yupqa plyonkalarning sinishi indeksini, yupqa plyonkalarning qalinligini tekshirish uchun mo'ljallangan. Biz ion-plazma usulida olingan o'rta infraqizil uzatish va yutilish spektrlarini, sindirish ko'rsatkichini, qatlam qalinligini, standart og'ishini, tushish burchagini, o'rtacha interferentsiya chekkalarini va kremniy va kislorod o'rtasidagi bog'lanishni o'rgandik.

Аннотация

ИК-Фурье-спектрометры используются для решения различных задач современной аналитики. Предназначен для качественного анализа, проверки показателя преломления тонких пленок, толщины тонких пленок. Исследованы спектры пропускания и поглощения среднего инфракрасного диапазона, показатель преломления, толщина слоя, стандартное отклонение, угол падения, средние интерференционные полосы и связь между кремнием и кислородом тонких пленок, полученных ионно-плазменным методом.

Abstract

FT-IR spectrometers are used to solve various problems of modern analytics. Designed for qualitative analysis, checking the refractive index of thin films, the thickness of thin films. We studied the mid-infrared transmission and absorption spectra, refractive index, layer thickness, standard deviation, incident angle, average interference fringes, and the coupling between silicon and oxygen of thin films obtained by the ion-plasma method.

Kalit so'zlar: *kremniy oksidi, FT-IR spektrometri, yupqa plyonkalar, yutilish spektrlari, uzatish spektrlari, "ion-plazma" usuli, sinish indeksi, ATR spektri.*

Ключевые слова: *оксид кремния, ИК-Фурье-спектрометр, тонкие пленки, спектры поглощения, спектры пропускания, «ионно-плазменный» метод, показатель преломления, спектр НПВО.*

Key words: *silicon oxide, FT-IR spectrometer, thin films, absorption spectra, transmission spectra, "ion-plasma" method, refraction index, ATR spectrum.*

INTRODUCTION

FT-IR spectrometers are used to solve various problems of modern analytics. Designed for qualitative analysis, checking the refractive index of thin films, the thickness of thin films.

We used the chamber of the magnetron device (Epos-PVD-Desk-Pro) to create silicon oxide using the Ion-Plasma method in high vacuum using a molecular turbopump (Pfeiffer Vacuum). The layer thickness, refractive index, absorption and transmission spectra of nanofilms formed on the silicon surface were measured. The experimental results were obtained on FT-IR spectrometers (IRTracer-100, Bruker-Alpha-II), which require high accuracy.

Due to the high refractive index of silicon oxide samples, there is a corresponding high reflection from the sample. If the sample is placed directly in the sample compartment, vertically to the incident beam of the source, the reflection of the source returns to the interferometer, the beam is reflected by the separator, and then illuminates the sample again. As a result, the length of the sample when measuring the sample differs from the background measurement, and even when using a cleaned device, there is an excess of noise due to water vapor in the atmosphere. Although the angle of incidence of the sample can be adjusted relative to the light source, the use of a vacuum FT-IR device allows the sample to be measured vertically until the surface is illuminated without any interference due to water vapor [1].

MATERIALS AND METHODS

When preparing the sample, A p-type silicon plate with a thickness of 500 μm is selected in the direction of 111 crystals. The size of the samples is 1.5 cm, 1.5 cm.

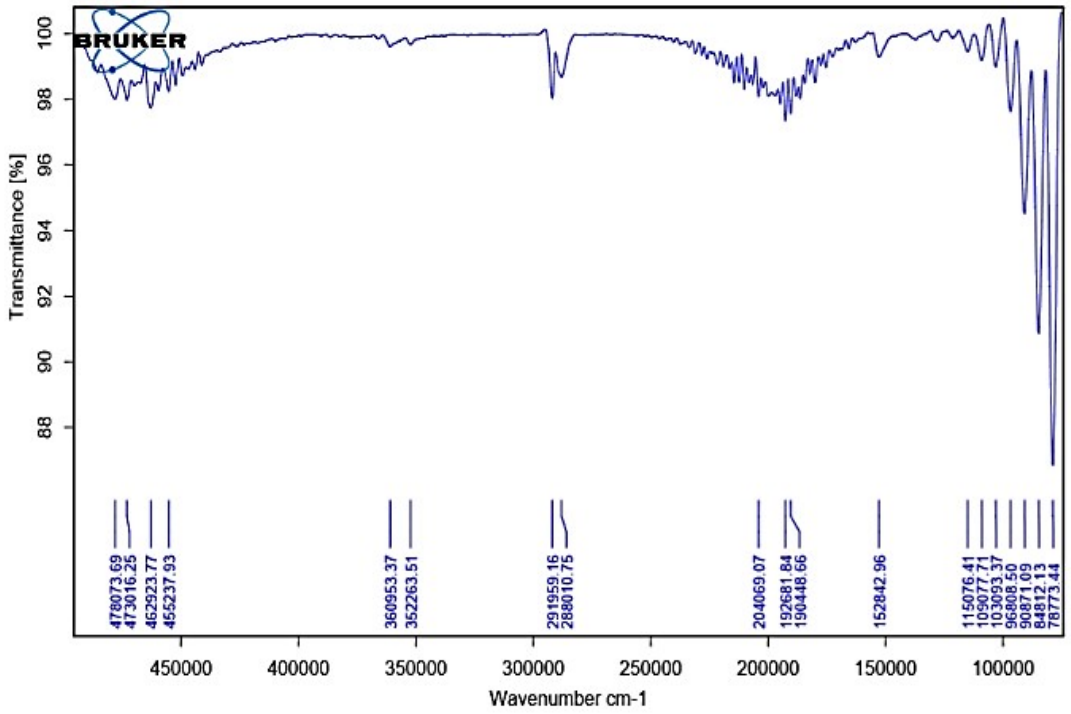
Samples of Si (111) sputtered semiconductor crystals of various thicknesses in the form of crystal plates; thin nanostructured films of silicon and oxidized silicon. Thin films obtained by the “ion-plasma” method using a magnetron device are applied to the silicon surface. There are many factors that affect the material parameters of thin films, for example, the production environment is moderate, the size of the crystallite and the adjustable orientation, the uniformity of the film, etc.

The Fourier infrared spectrophotometer is a reliable, compact and easy-to-use device that allows qualitative and quantitative analysis of a wide range of gases and solid samples, liquids [2]. The Michelson interferometer has a system of dynamic numerical alignment of elements with a signal-to-noise ratio of 30,000:1 (for KRS-5.4 cm^{-1}, 1 min, 2100 cm^{-1}). Specialized software, including the fast “Labsolutions – IR” software, is enriched with “mixture analysis” and “substance determination” systems, as well as data analysis and processing systems.

Fourier spectrophotometers “Bruker-Alpha-II” and “IRTracer-100” were used to increase the reliability of the results [3]. A graph of the dependence of the radiation intensity on the number of waves is obtained.

RESULTS AND DISCUSSION

The spectra obtained on the Bruker – Alpha-II infrared spectrophotometer were used for processing and analysis in the “OPUS” multifunctional software.



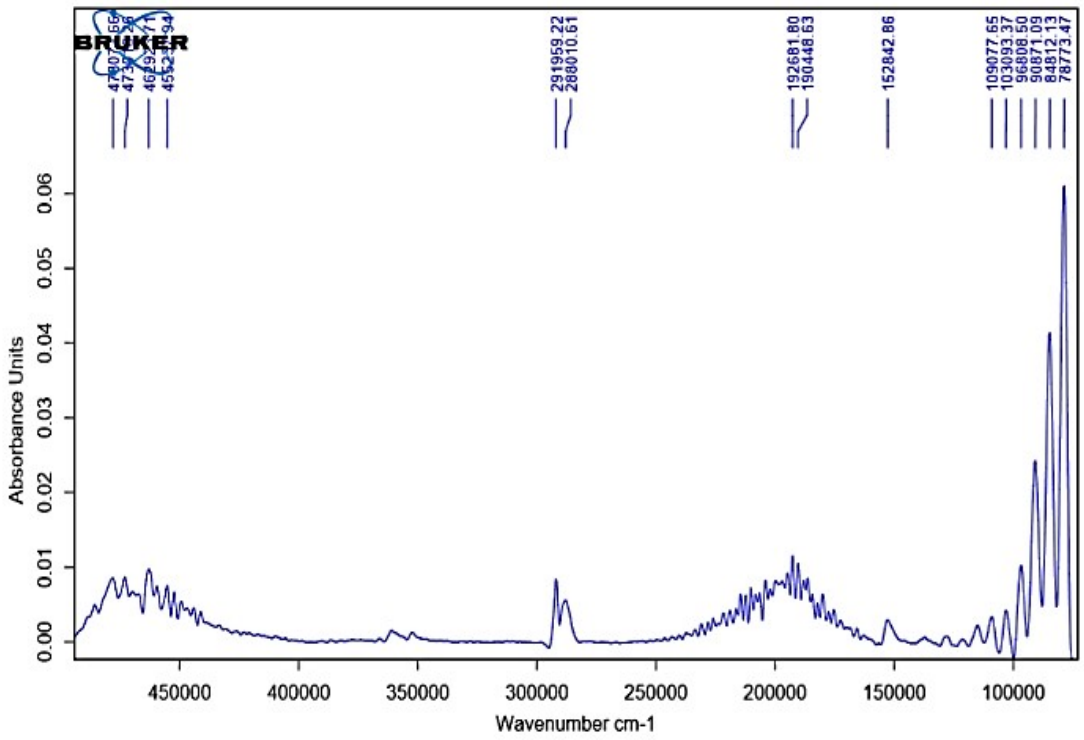


Fig.1. Transmission and absorption spectra of silicon film obtained using the Bruker Alpha-II infrared spectrophotometer

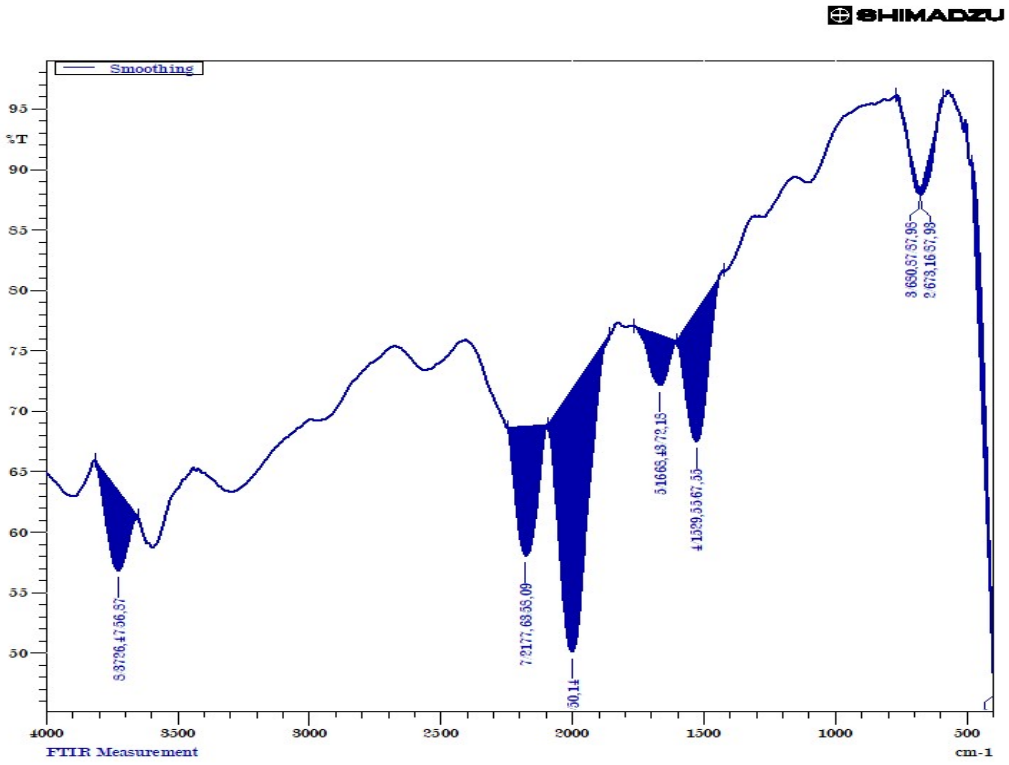


Fig.2. p-type semiconductor silicon displacement spectrum in FT-IR measuring spectroscopy

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The results of the spectrum of the silicon semiconductor p-type Si(111) obtained on the IRTracer-100 spectrophotometer in FT-IR are shown in Table-1.

Table 1

No	Peak sm^{-1}	Intensity %	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	399,26	47,65	0,00	484,13	399,26	2231,540	-388,964
2	673,16	87,93	0,58	678,94	588,29	783,087	62,069
3	680,87	87,98	0,19	767,67	678,94	715,065	12,992
4	1529,55	67,55	10,73	1604,77	1423,47	4783,971	936,783
5	1668,43	72,18	4,16	1764,87	1604,77	4098,920	331,342
6	2000,18	50,14	21,76	2094,69	1857,45	8935,176	2449,650
7	2177,63	58,09	10,70	2247,07	2094,69	5669,501	915,123
8	3726,47	56,87	6,54	3815,20	3653,18	6463,916	571,052

Triple bond fluctuations were observed in the region of 2000.18 and 2177.63 cm^{-1} in the infrared spectra of silicon "smoothing". Fluctuations of the double bond were observed in the region of 1668.43 cm^{-1}. Peaks between the regions of 673.16, 680.87 and 418.55 cm^{-1} were observed deformation vibrations of oxygen and carbon atoms in the upper field of infrared radiation on the silicon surface.

The obtained spectra included water vapor (H_2O) or carbon dioxide (CO_2) molecules subjected to multiple analysis of “atmospheric corillation" in order to reduce exposure.

Figure 3 shows the transmission and absorption spectra of the SiO_2/Si(111) film in the range 400÷4000 cm^{-1}, with water vapor (H_2O) or carbon dioxide (CO_2) in the spectra of molecules undergoing the following adjustments to reduce the effects: addition, smoothing, zero correction of the baseline, normalization, filtration and ATR correction.

When silicon is oxidized (naturally, in the atmosphere of air or under the influence of high temperatures), Si-OH hydroxyl groups are formed on its surface, and siox groups are formed in the near-surface layer. The presence of hydroxyl groups leads to adsorption of atmospheric moisture and corresponding changes in the spectra of samples[4].

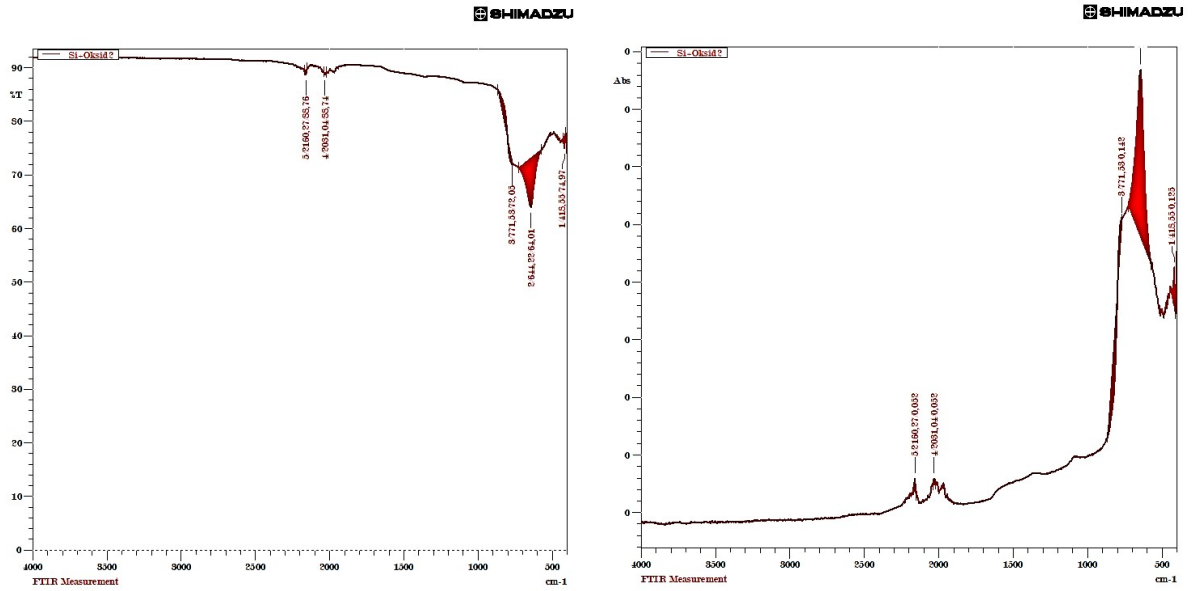

Fig.3. Smothing analysis of the transmission and absorption spectrum of SiO_2/Si(111)

Figure 3 shows the infrared absorption and transmission spectra of the SiO_2 film formed on the silicon surface. In the absorption spectrum, the peak was observed in the region of 769.60 cm^{-1}. These lines correspond, respectively, to antisymmetric stretching oscillations of Si-O-Si groups. The peak of the transmission spectrum in the region of 644.22 cm^{-1} corresponds to the “fingerprint" region of the pure silicon spectrum. Silicon dioxide layers have three absorption zones: a low-

frequency band of 418.55 cm^{-1}, a weak band of 771.53 cm^{-1} and an intense broadband band with a maximum of 644.22 cm^{-1}. These lines relate to the oscillations of the pendulum, symmetric stretching and antisymmetric stretching of Si-O-Si groups, respectively. Depending on the brittleness of the oxide, the final strip can have a half width from ≈95 cm^{-1} to ≈ 140 cm^{-1} for dense oxide. While studies of silicon oxides have shown that SiO_x (x=1÷2) is formed during deposition and annealing, with a decrease in *x*, the maximum boundary of the *n* band (SiOSi) shifts to the region of lower wave numbers (915 cm^{-1} at x=1, 980 cm^{-1} at x=2). The frequency, on the contrary, increases from 780 to 835 cm^{-1}; the frequency of oscillation of the pendulum increases with increasing x.

Table 2

No	Peak sm^{-1}	Intensity %	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area
1	418,55	0,125	0,014	428,20	410,84	2,049	0,111
2	644,22	0,194	0,058	725,23	572,86	24,347	3,597
3	771,53	0,142	0,003	866,04	767,67	9,744	-0,491
4	2031,04	0,052	0,002	2038,76	2023,33	0,786	0,019
5	2160,27	0,052	0,005	2167,99	2150,63	0,854	0,038

In the case of thin films, infrared interference spectra carry information about the anisotropy of the material and make it possible to determine the refractive index and rotation of molecules located in the IR region of the spectrum [5].

Infrared spectroscopy is known as one of the most useful methods for evaluating these types of thin films, which provides information about the molecular structure and orientation, as well as their optical thickness and electrical properties. Infrared reflection-absorption spectroscopy is useful for analyzing the molecular structure of very thin films on metal substrates tens of nanometers thick.

When light is reflected from certain materials (Diamond, ZnSe, etc.) at a critical angle, the light is completely reflected with the absorption of a small amount of light by the material (sample) in contact with the crystal surface, Fig.5.

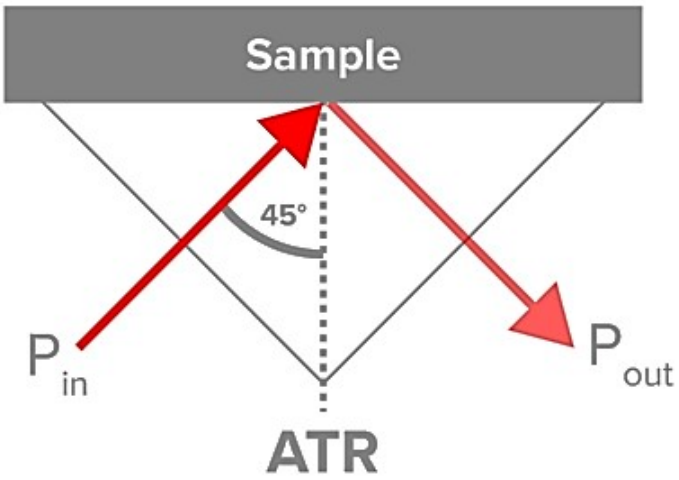

Fig.4. The path of light for the ATR experiment [6]

In the ATR reflection spectrum, the thickness of the layer increases with an increase in the angle of incidence of penetrating infrared light into the crystal. The amount of interference does not change.

Table 3

№	Semple name	Incident angle	Average interference fringes	Thickness (um)	Standard deviation (um)
1	SiO_2/Si	0.00	131	73.91	271.23
2		30.00	131	75.43	276.82
3		45.00	131	77.05	282.78
4		60.00	131	78.78	289.13
5		75.00	131	80.13	294.07
6		90.00	131	80.64	295.94

The depth of penetration of infrared light depends on the refractive index of the sample and the crystal. Since the refractive index depends on the wavelength, the spectra obtained using ATR have a slightly different intensity ratio over the entire spectrum, and an adjustment may be required to compare with the transmission spectra[7].

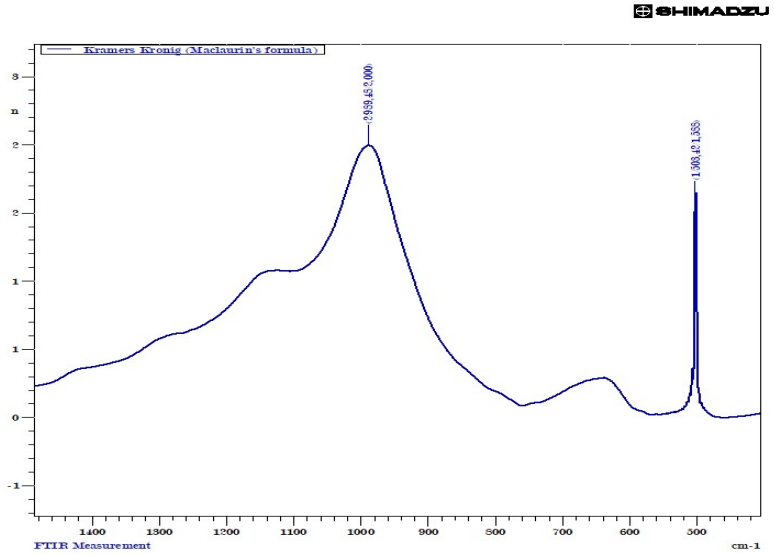

Fig.5. Si(111) silicon obtained in FT-IR measuring spectroscopy is a Kramers-Kroing spectrum by the mclarwin method

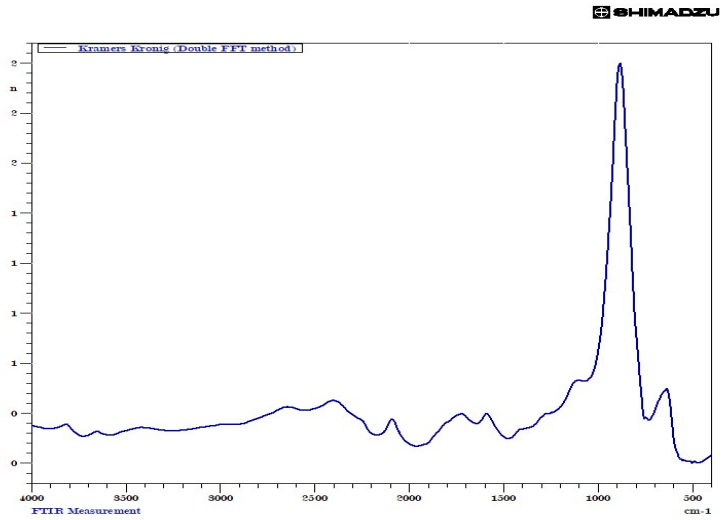

Fig.6. Kramers-kronig SiO_2/Si silicon spectrum in the "double FFT" method obtained in FT-IR measuring spectroscopy

When measuring the refractive index of thin films, various methods can be used, we used the following modified expression of Sellmeyer by Edwards and others, which is the most suitable dispersion equation for ambient temperature [8].

$$n^2 = \varepsilon + \frac{A}{\lambda^2} + \frac{B\lambda_1^2}{\left(\lambda^2 - \lambda_1^2\right)}$$

Where λ_1=1.1071 μm, ε=1.16858·10^1, A=9.39816·10^{-1} and B=8.10461·10^{-3}. The results obtained gave λ=1.99807 microns for silicon and λ=1.0111223 microns for silicon oxide.

Knowing the angle of incidence and refractive index in the “calculating film density” function in the data processing section of the IRTracer-100 spectrophotometer, it is possible to measure the thickness of the film, the average number of interference fields and the standard deviation. Table 4 below shows the measured parameters of films of different thicknesses obtained in the magnetron device.

Table 4

Sample name	Si (111)	SiO_2 /Si (111)	Literature Si (111)
Range (sm^{-1})	503	401,19 - 3974,38	489,84
Refractive index	3.45323	3,417	3,9766
Incident angle	45	90	
Average interference fringes	24	122	
Thickness (um)	12,34	51,36	
Standard deviation (um)	10,06	64,32	
Minimum peak (%)	47.6527	64.007	23.47
Maximum peak (%)	96.5350	92.099	64.35

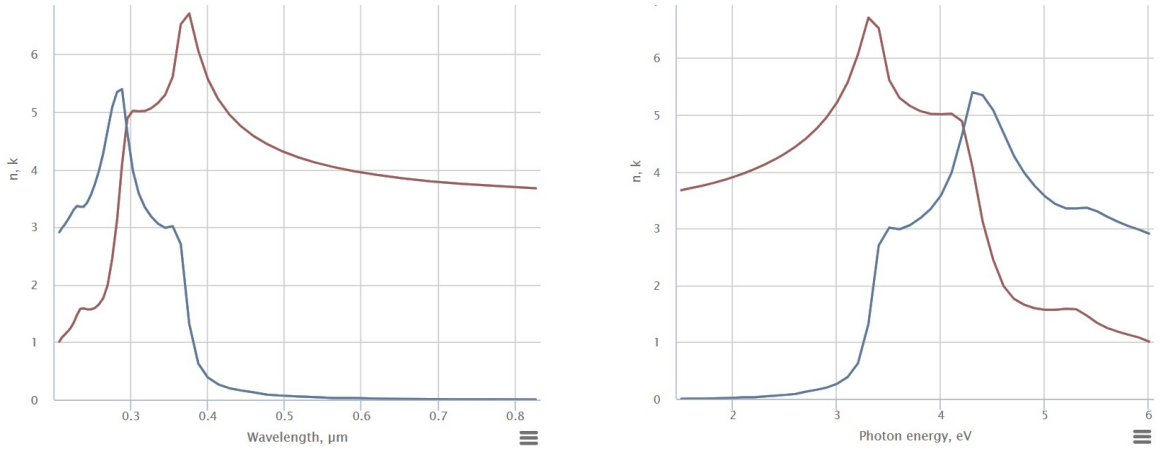

Fig.7. Sample refractive index and absorption coefficient Si(111) [9]

In the cited sources, the data are as follows [9]: wavelength 0.5876 μm, refractive index: n=3,9766, extraction coefficient k=0.030209, complex refractive index (n+ik).

CONCLUSION

In this manuscript, we conducted a qualitative and quantitative analysis of the absorption and transmission spectra of nanofilms obtained by the Ion-plasma method under high vacuum conditions. The fields of valence and deformation vibrations of atoms in the film are investigated. The thickness of the thin film and the refractive index were measured by analytical and experimental methods. the refractive index is *n*=3.45323 for silicon and *n*=3.417 for silicon oxide. For silicon oxide and pure silicon, “average interference edges” were measured. The depth of penetration of infrared light into the crystal was measured. The measured data show that the electrical and optical properties of various nanofilms obtained by “ion-plasma” and “ion-sputtering” methods play an important role in illumination.

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