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## Study of optical properties and surface structure of thin films of non-stoichiometric silicon nitride formed by means of low-temperature plasma-chemical deposition for an implementation in MEMS-structures.

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**ABSTRACT**

In the presented paper optical properties and surface morphology of non-stoichiometric silicon nitride $SiN_x$ layers used for manufacturing of heat-sensitive membranes in microelectromechanical systems (MEMS) and microoptomechanical systems (MOMS) are studied using infrared (IR) spectroscopy and atomic-force microscopy (AFM) methods. For model structures "silicon wafer – film of $SiN_x$" and "silicon wafer – film of $SiN_x$– thin metal layer" transmission and reflection spectra in a region of wave numbers of 500-7000 $cm^{-1}$ are investigated. Aluminum, nickel and nickel-chromium alloy were used for formation of metal coatings. For the studied thin film structures analysis of optical properties appearing in IR spectrum both in a form of selective absorption bands and interference modulations of a base line was carried out. Analysis of AFM images allowed to define parameters of surface roughness of studied samples.

**Keywords:** non-stoichiometric silicon nitride $SiN_x$, microoptoelectronic systems, MOMS, microelectromechanical systems, MEMS, heat-sensitive elements, infrared spectroscopy, atomic force microscopy, AFM.

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## INTRODUCTION

Silicon nitride films posses a large number of unique physical and chemical properties, which allow their wide application in silicon technology for a manufacturing of microchips with a large density of integration, solar cells and other semiconductor based microelectronic devices [1,2,3]. Main advantages of silicon nitride as a material for an implementation in microelectronics in a form of lithographic layers, dielectric insulating and passivating layers are as follows: compatibility and chemical inertness relatively to semiconductors and many other construction materials, high thermal stability, good dielectric and insulating properties, increased chemical stability in various deleterious liquid and gaseous media even in cases of comparatively high temperatures. In a case of implementation in solar cells and various thermal imaging structures, one of advantages of silicon nitride is quite intensive absorption band for infrared radiation with wavelength around 12 µm, which corresponds to low-loss transmission window of earth atmosphere.

Films of stoichiometric silicon nitride $Si_3N_4$ are usually produced by means of chemical vapor deposition (CVD) method through synthesis from dichlorsilane $SiH_2Cl_2$ and ammonia $NH_3$ with temperatures of 700-900 $^oC$ and pressure of 30-50 Pa [4,5,6]. However, films of stoichiometric silicon nitride produced by means of that method can't be implemented for suspended membranes or consoles in 3D multi element devices of microelectromechanical (MEMS) and microoptomechanical (MOMS) systems due to large internal stress in $Si_3N_4$ films and their damage in a case of thicknesses more than several hundreds of nanometers and incompatibility of temperature budget of a process with previously formed metal elements. In order to solve those problems low temperature plasma-enhanced chemical vapor deposition (PECVD) methods are applied, which comprise decomposition of ammonia, silane derivatives and other precursors for temperatures lower than 300 $^oC$ for a formation of layers of non-stoichiometric silicon nitride $SiN_x$ [7,8]. Used types of PECVD method allow to form $SiN_x$ films with controlled internal stresses and good mechanical properties. It allows to create MEMS and MOMS devices with structural elements on a basis of $SiN_x$ membranes, which are freely positioned on some distance from a substrate. Intensive new studies on a development of various structures of microelectronic devices on a basis of layers of non-stoichiometric silicon nitride require further studies of optical and structural properties of films produced from that material.

The presented study contains an investigation of spectral property in infrared (IR) wavelength region and profile of surfaces of films of non-stoichiometric silicon nitride of various thickness on a surface of polished wafers of monocrystalline silicon. For a characterization of samples we used IR transmission and reflection spectroscopy, AFM and ellipsometry. An analysis was carried out, which comprised study of selective absorption bands and interferential phenomena in IR spectrum during measurements with various optical configurations of ray course. By means of AFM method we obtained data on a structure of surfaces of films' of $SiN_x$ and metals of various thicknesses.

## METHODOLOGY

### Sample preparation

Deposition of films of non-stoichiometric silicon nitride was carried out using PlasmaLab100 ICPCVD (product of Oxford Instruments company) equipment for plasma-chemical deposition in high-frequency inductively coupled plasma of high density [9]. Synthesis was carried out with the following technological parameters: temperature of substrates holder was held at 250°C, pressure in working chamber – 10 mTorr, high frequency power – 1200 W, ratio of flows of silane and nitrogen ($SiH_4$:$N_2$) is 1:3. 100 mm diameter wafers made from monocrystalline silicon doped with phosphorus, with surface orientation (100), double-sided polishing and 4.5 Ω/cm resistivity were used as substrates. Immediately before the deposition process substrates were washed in peroxide-ammonium solution in order to remove organic contaminants and particles from a surface. $SiN_x$ film was formed on one of surfaces of a silicon wafer, which in further will be called "frontal" surface.

**Table 1. List of studied samples: layers of non-stoichiometric silicon nitride and metals, which were deposited on silicon substrates (d – thickness of a deposited layer, n – value of refractive index for $SiN_x$ film for radiation with 632.8 nm wavelength).**

| No. of sample | Deposited layers | n ($SiN_x$) |
|---|---|---|
| 1 | Layer of $SiN_x$ (d = 497 nm) | 2.12 |
| 2 | Layer of $SiN_x$ (d = 402 nm) – layer of Al (d = 200 nm) | 2.12 |
| 3 | Layer of $SiN_x$ (d = 280 nm) | 2.13 |
| 4 | Layer of $SiN_x$ (d = 288 nm) – layer of Ni (d = 5 nm) | 2.11 |
| 5 | Layer of $SiN_x$ (d = 236 nm) | 2.10 |
| 6 | Layer of $SiN_x$ (d = 254 nm) – layer of NiCr (d = 50 nm) | 2.12 |
| 7 | Layer of $SiN_x$ (d = 128 nm) | 2.10 |
| 8 | Layer of $SiN_x$ (d = 128 nm) – layer of Al (d = 200 nm) | 2.11 |
| 9 | Si wafer without deposited layers | - |

List of the samples prepared for study is presented in table 1. Samples 1, 3, 5 and 7 are $SiN_x$ films of 497 nm, 280 nm, 236 nm and 128 nm thickness, respectively, which were deposited on surfaces of silicon wafers. For samples 2 and 8, an aluminum layer of 200 nm thickness was deposited over $SiN_x$ layers of 402 nm and 128 nm thickness respectively. For sample 4, a nickel layer of 5 nm effective thickness was deposited over a $SiN_x$ layer of 288 nm thickness. For sample 6, a layer of NiCr alloy of 50 nm thickness was deposited over an initial layer of silicon nitride of 254 nm thickness.

Samples 2, 6 and 8, which had layers of Al or NiCr system on top of a layer of non-stoichiometric silicon nitride, were used for study of selective IR absorption in a layer of silicon nitride during registration of IR radiation reflected from a back surface of a silicon substrate. Thin layers of Al, NiCr alloy or Ni (sample 4) were deposited by means of magnetron sputtering method using MSx100 equipment (product of FHR company).

## RESEARCH METHODS

Study of the samples' surface profile was carried out by means of AFM method. Measurements were carried out using scanning probe microscope Smena$^{TM}$ VV (product of NT-MDT company, Russia) in intermittent contact mode. During studies we used silicon microcantilever NSG10S (product of NIIFP company, Russia). Speed of scanning was 0.5 raster line per second and scanning pitch by X and Y was approximately equal to 7,6 nm. Sizes of presented scans are 2x2 µm and number of pixels in image is 256x256. On a basis of obtained scans roughness parameters of silicon nitride films of various thicknesses were defined; they are presented in table 2.

Measurement of IR spectra of transmission and reflection was carried out using Perkin-Elmer Spectrum 100 IR Fourier spectrometer in a region of wavenumbers of 500-7800 $cm^{-1}$ and 4 $cm^{-1}$ spectral resolution. Comparatively good signal/noise ratio in resultant spectra was obtained by means of averaging of 16 sequentially measured spectra. Transmission spectra (T) were measured for normal incidence of nonpolarized IR radiation on a sample surface. Reflection spectra (R) were measured using PIKE Technologies VeeMAX$^{TM}$ specular reflectance accessory and a lattice polarizer based on a plate from KRS-5 during incidence of p-polarized IR radiation on a surface of a sample at $30^o$ angle. Reflection spectra were registered during incidence of probing IR radiation both on a frontal surface of Si-wafer with deposited layers ($R_f$) and on a back surface of Si-wafer without deposited layers ($R_b$).

Measurements of thickness and refractive index of obtained films of $SiN_x$ were carried out using Sentech Senduro spectral ellipsometer. Table 1 presents values of refractive index for $SiN_x$ films for radiation of 632.8 nm wavelength.

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## RESULTS AND DISCUSSION

### Surface morphology

Figure 1, 2 and 3 present AFM images of frontal surface of samples 1, 7 and 8 with deposited layers, respectively. Table 2 presents results of statistical analysis of AFM images of surfaces of samples 1, 2, 5-8. Statistics was gathered on a surface of 2x2 μm$^2$ and number of analyzed pixes is 65536.

**Table 2. Roughness parameters and grain sizes for surfaces of samples 1, 2, 5-8 defined by means of AFM images.**

| Sample | 1 | 2 | 5 | 6 | 7 | 8 |
|---|---|---|---|---|---|---|
| Maximum change of height, nm | 4.811 | 77 | 3.151 | 2.4 | 1.944 | 52 |
| Roughness, nm | 0.611 | 10 | 0.363 | 0.3 | 0.201 | 7 |
| Average lateral grain size, nm | 73 | 98 | 56 | 61 | 48 | 86 |
| Average height of grain, nm | 2.1 | 51 | 1.5 | 1.3 | 0.8 | 34 |

As it can be seen from figure 1, 2 and table 2, in case of samples without metal layers, values of all surface roughness parameters monotonously increasing with transferring from thinner to thicker films of $SiN_x$. For example, for samples 7, 5 and 1 with thicknesses of $SiN_x$ layers equal to 128 nm, 236 nm and 497 nm, respectively, an average lateral size of a grain is 48 nm, 56 nm and 73 nm, respectively, and an average height of grain is 0,8 nm, 1,5 nm and 2,1 nm respectively.

On a basis of comparison of results of AFM testing for samples 5 and 6 (see table 2), it can be noted that a deposition of NiCr layer of 50 nm thickness on top of $SiN_x$ layer's surface resulted in a comparatively small change of a surface roughness parameters. An average lateral size of grain increased, approximately, by 10% and an average height of grain even decreased, approximately, by 20%. However, for Al layer deposited on top of $SiN_x$ layer roughness parameters are significantly bigger then for $SiN_x$ layer of the same thickness (see figure 2, 3 and table 2). For example, for sample 8 with Al layer of 200 nm thickness, which was deposited on top of $SiN_x$ layer of 128 nm thickness, an average lateral grain size and an average grain height were equal to 86 nm and 34 nm, respectively, which significantly exceeds values of those parameters (48 nm and 0.8 nm, respectively) for sample 7 with $SiN_x$ layer of the same thickness.

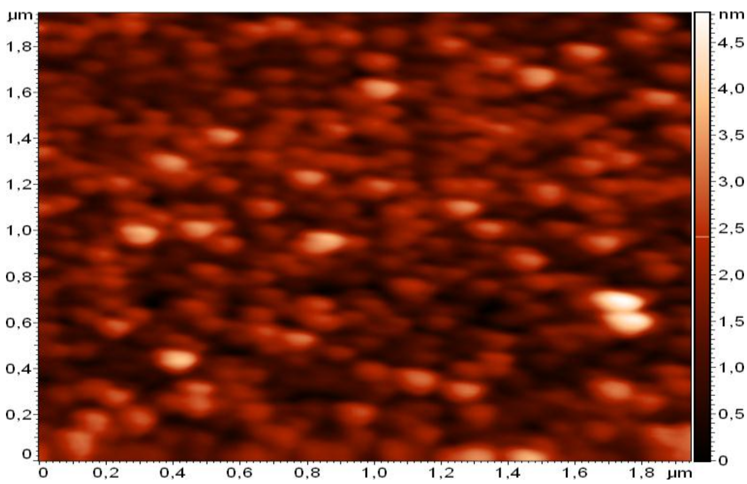


**Fig.1. AFM image of a surface of sample 1 (Si wafer –$SiN_x$ layer of 497 nm thickness).**

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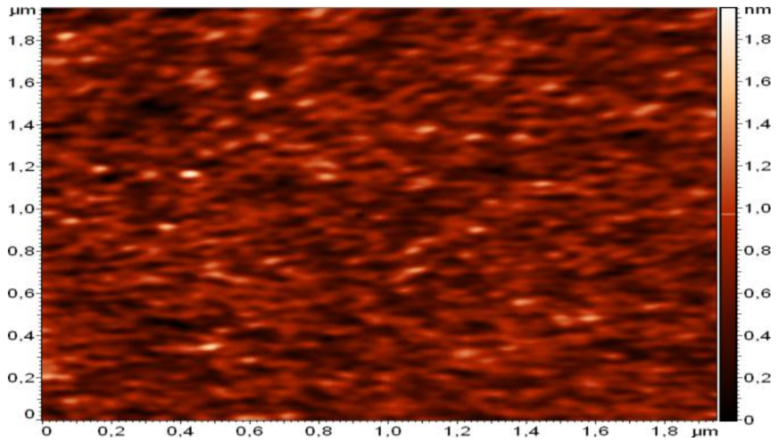



**Fig.2. AFM image of a surface of sample 7 (Si wafer –$SiN_x$ layer of 128 nm thickness).**

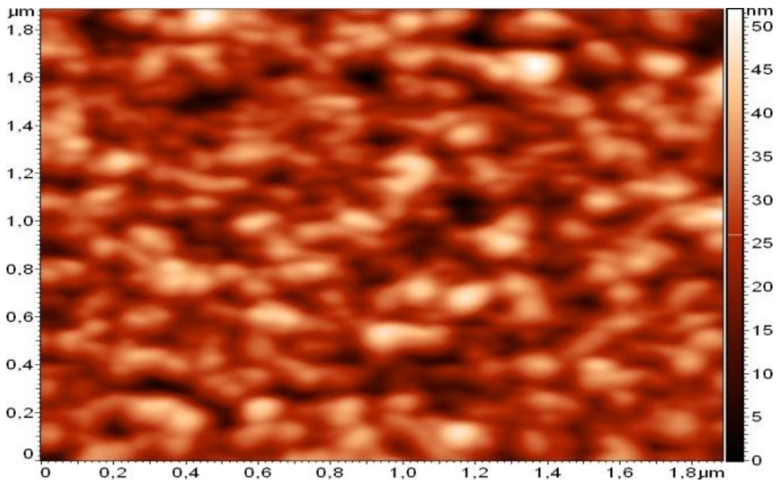



**Fig.3. AFM image of a surface of sample 8 (Si wafer – $SiN_x$ layer of 128 nm thickness – Al layer of 200 nm thickness).**

**Spectral properties**

Figures 4 and 5 demonstrate transmission spectra for samples 1, 3, 4, and 5, 7, 9, respectively. For all figures with IR spectra (figures 4-7), part (a) shows broadband IR spectra in a wavenumber (ν) region from 500 $cm^{-1}$ to 7000 $cm^{-1}$ and part (b) shows fragments of those spectra in narrower range of ν from 500 $cm^{-1}$ to 1400 $cm^{-1}$, which comprises intensive absorption bands of silicon nitride and silicon oxide. As it can be seen from figures 4 and 5, IR transmission spectra of samples graphically demonstrate both characteristic selective absorption bands of silicon nitride, silicon oxide and silicon in a region of wavenumbers of 700-1200 $cm^{-1}$ and significant modulations of base lines resulting from interference of IR radiation in silicon nitride films, which are essentially Fabry-Pérot modes in thin films [10]. Peaks of selective absorption bands of the samples correspond to minimums of the transmission spectra. Identification of absorption bands in transmission spectra of the studied samples is based on the previous studies [11,12,13,14] and is presented in table 3. The most intensive and quite wide and complex absorption band in the region of 700-1020 $cm^{1}$ with minimum in a region of 820-860 $cm^{-1}$ corresponds to absorption bands of stretching vibrations of Si-N groups in $SiN_x$ films. It worth mentioning that exactly a presence of that absorption band, belonging to spectral low-loss transmission window of atmosphere, is a reason for interest for silicon nitride as heat-sensitive material, which can be applied in solar cells and various devices of MEMS and MOMS type [15,16,17,18].

**Table 3. Attribution of the most intensive absorption bands in IR transmission spectra of samples 1-9 (table 1) according to [11,12,13,14].**

| Position of peak of absorption band, $cm^{-1}$ | Type of vibrations |
|---|---|
| 620 | phonon vibrations of silicon |
| 820-860 | stretching vibrations of Si-N |
| 1120 | stretching vibrations of Si-O |
| 2300 | stretching vibrations of Si-H |

Interference extremums in spectral dependencies of modulated baselines for various samples in figures 4 and 5 were observed for various values of wavenumbers related with various thicknesses of $SiN_x$ layers of various samples. In a transmission spectrum of sample 1 with the thickest layer of $SiN_x$ (figure 4) the adjacent broad interference maximums in modulated form of the baseline spectral dependence appear at, approximately, $\nu_1 \approx 2580$ $cm^{-1}$ and $\nu_2 \approx 7520$ $cm^{-1}$. It worth noting, that a region of spectral measurements of 7800-7000 $cm^{-1}$, which is available for used IR Fourier spectrometer is not presented in figure 4. By knowing the geometry thickness of $SiN_x$ layer for sample 1 (d = 497 nm), which was defined by means of ellipsometer measurements in visible region (see table 1 and section 2.2) and the difference in wavenumbers $(\nu_2 - \nu_1) \approx$ 4940 $cm^{-1}$, it is possible to estimate refractive index (n) of the studied non-stoichiometric $SiN_x$ in middle IR region by means of the equation:

$$2nd(\nu_2 - \nu_1) = 1.$$

Obtained value of n ≈ 2.02 for middle IR region is quite close to a value of refractive index 2,12, which was found for red region of visual spectra for $SiN_x$ layer in sample 1 (see table 1 and section 2.2).

For an approximate estimation of intensity of selective absorption band (ΔA) of stretching Si-N vibrations at ν ≈860 $cm^{-1}$ with minimal transmission $T_1 \approx$ 14% and transmission close to base line level (background level) $T_2 \approx$ 49% we used the equation:

$$\Delta A = -\log\left(\frac{T_1}{T_2}\right).$$

Found value of ΔA = 0.544 corresponds to absorption in $SiN_x$ layer of 497 nm thickness. Level of base line $T_0 \approx$ 49% in IR transmission spectra of sample 1 at ν≈ 860 $cm^{-1}$ is mainly defined by means of non selective reflection and interference modulation of base line for a silicon wafer with dielectric film having refractory index of 2.02 in a given region of spectrum. For an approximate estimation of absorption efficiency in a layer of silicon nitride, it can be stated that selective absorption in $SiN_x$ layer for sample 1 was a reason of a decrease of transmission of a sample for, approximately, 35%: from 49% level for base line to 14% level for peak of the discussed absorption band. In this connection, it can be noted that one of possible ways for an increase of total amount of light energy absorbed by $SiN_x$ layer in spectral region of low-loss transmission window of atmosphere is a selection of thickness of $SiN_x$ film in a way that interference maximum in transmission spectra of a sample was in 700-1100 $cm^{-1}$ region. It can be achieved, for instance, by an implementation of silicon nitride film with 1-1.5 µm thickness. As it can be seen from figure 5, for thinner $SiN_x$ film of 128 nm thickness for sample 7, values of $T_1$ and $T_2$ for wavenumber ν ≈ 860 $cm^{-1}$ are approximately equal to 33% and 50% respectively. It allows to obtain an approximate estimation of ΔA = 0.18 for intensity of band at ν ≈ 860 $cm^{-1}$ for sample 7.

Figure 6 shows reflection spectra for sample 3 (Si wafer – $SiN_x$ layer (d = 280 nm)) and sample 4 (Si wafer – $SiN_x$ layer (d = 288 nm) – Ni layer (d = 5 nm)), which were measured for two different orientations of a sample in specular reflectance accessory using two optical systems. Reflection spectra $R_f$ were measured for incidence of IR radiation beam on a frontal surface of Si wafer, where there is a layer of silicon nitride (sample 3) or structure consisting from a layer of silicon nitride and an ultrathin Ni layer (sample 4). Reflection spectra $R_b$ were measured for incidence of IR radiation beam on a clean back surface of Si wafer, which didn't have deposited films.

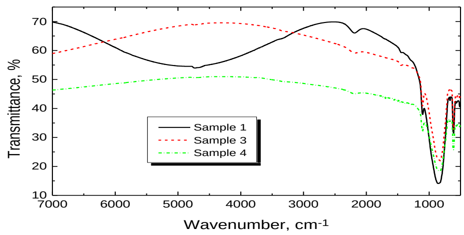
For sample 3 with $SiN_x$ layer (see figure 6-a) in a region of wavenumbers of 1300-7000 $cm^{-1}$, where there are no strong selective absorption bands, the reflection spectra $R_f$ and $R_b$ for those two types of incidence of IR radiation beam on a surface of a sample are very similar and modulation amplitudes for the base lines are almost equal. However, for sample 4, which contains not only $SiN_x$ layer, but also Ni layer, the reflection spectra $R_f$ and $R_b$ in spectral range of 1300-7000 $cm^{-1}$ differ significantly. In $R_b$ spectrum there is a noticeable decrease of modulation amplitude in spectral dependence of base line, also, an average value of reflection coefficient increases as compared to spectrum of reflection from a frontal surface of a sample $R_f$. In terms of quality it is in agreement with relative differences in transmission spectra for the same samples 3 and 4 presented in figure 4. Thin layer of Ni in sample 4 leads both to a total decrease of transmission and a decrease of modulation amplitude in base line spectrum as compared to transmission spectrum of sample 3. It is obvious that observed changes in transmission and reflection spectra are related to an effect that deposition of partly transparent in IR region ultrathin metallic coatings on a surface of heat-sensitive materials like silicon nitride can influence their structure, as well as dielectric parameters of a surface of films made from those materials. It is necessary to take into account that fact during design of solar cells and MOMS devices, e.g. during a formation of antireflective coating of various nature.

It is worth noting that in a region of wave number of 700-1020 $cm^{-1}$, where there are intensive absorption bands of silicon nitride (see figure 6-b), reflection spectra $R_f$ and $R_b$ for samples 3 and 4 significantly differ. Character of $R_b$ spectrum for a certain sample to a certain degree is similar to transmission spectra T for that sample, while $R_f$ spectrum significantly differ from $R_b$ spectra. Observed differences of $R_f$ и $R_b$ spectra for the studied samples are related to well known differences in dispersion of real and imaginary parts of complex dielectric permittivity of material of absorbing film in laminar structures for two discussed orientations of a sample's surface relatively to IR radiation beam.

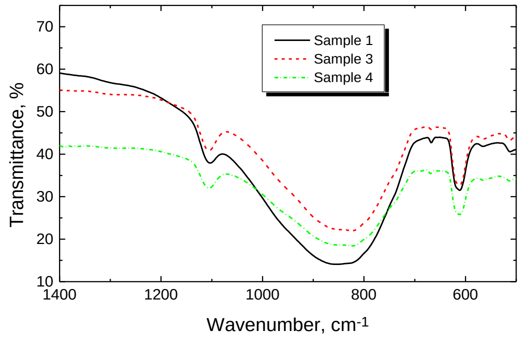
Figure 7 shows reflection spectra $R_b$ for samples 2, 6 and 8 (see table 1), which structure has layers of Al or NiCr. For a comparison that figure also shows reflection spectra for a clean silicon wafer. Measurement scheme for IR reflection from a back surface of a sample $R_b$ used in the presented paper allows to obtain a quality estimation of a relationship between a value of selective absorption of silicon nitride layer situated between Si substrate and metal layer and thickness of silicon nitride layer. Such evaluations can be useful, for example, during design of MOMS elements with non-transparent or semi-transparent reflective metal coatings on a surface of mobile heat-sensitive membranes.

In reflection spectrum $R_b$ for sample 6 with a semi-transparent in IR region of frequencies NiCr layer on top of $SiN_x$ film of 254 nm thickness (figure 7) there is a clear modulation of spectrum of base line appearing due to interference of IR radiation in $SiN_x$ film. However, selective absorption bands of $SiN_x$ are almost invisible, because incidence angle of $30^o$ doesn't allow to achieve a necessary sensitivity of the reflection method for a registration of spectrum of thin films adjacent to a surface of a metal [19,20].

It's worth mentioning that significant differences in reflection spectra $R_b$ for samples 2 and 8 (figure 7) with covering layer of Al of 200 nm thickness and layers of $SiN_x$ of different thicknessed. In $R_b$ spectrum for sample 8 with $SiN_x$ layer of smaller thickness (d = 128 nm) there is almost no modulation of base line and very weak absorption band of stretching vibrations of Si-N groups in a region of 820-860 $cm^{-1}$ (figure 7-b). However, in $R_b$ spectrum for sample 2 with $SiN_x$ layer of bigger thickness (d = 402 nm) there are both clear interference modulation base line and quite strong selective absorption band of stretching vibrations of Si-N groups in 820-860 $cm^{-1}$ region. It is necessary to note that relative intensity of selective absorption of silicon nitride in a region of wave number of 820-860 $cm^{-1}$ in reflection spectrum for sample 2 are significantly lower than in transmission spectrum for sample 1 with $SiN_x$ layer of comparable thickness. However, that ratio for the reflection method can be significantly improved during testing of reflection spectra for studied structures with thicker films of silicon nitride. That kind of clear difference in selective absorption of $SiN_x$ films of various thickness in $R_b$ reflection spectra of samples 2 and 8 can be explained by a significant non-linear relationship between intensity of absorption band of thin dielectric film adjacent to a surface of metal and thickness of a film in optical scheme of reflection-absorption spectroscopy for small incidence angles of IR radiation on metal-dielectric film interface [20,21].

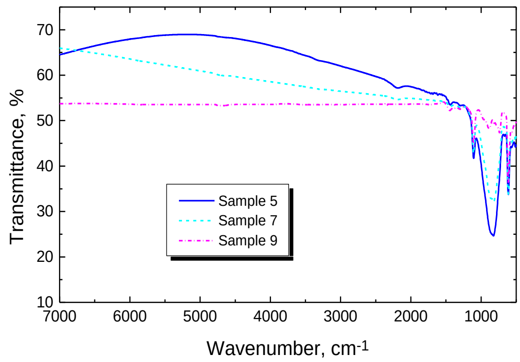



a)

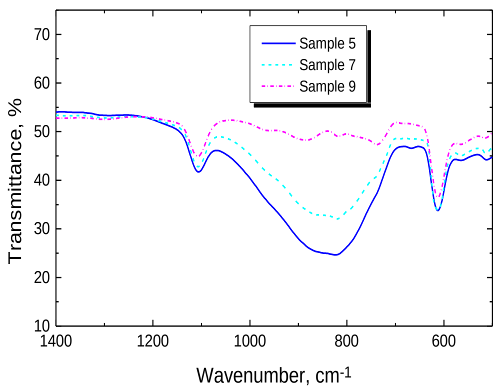



b)

**Fig.4. IR transmission spectra for samples 1, 3 and 4 (description of samples is presented in table 1) for normal incidence of IR radiation on a surface of silicon substrate for a wavenumber range of 500-7000 cm$^{-1}$ (a) and fragments of those spectra in a range of 500-1400 cm$^{-1}$ (b), where there are intensive selective absorption bands of a sample.**

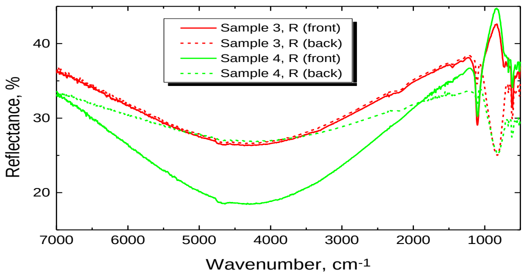



a)

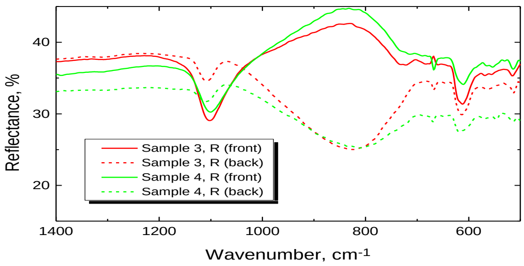



b)

**Fig.5. IR transmission spectra for samples 5, 7 and 9 (description of samples is presented in table 1) for normal incidence of IR radiation on a surface of silicon substrate for a wavenumber range of 500-7000 cm$^{-1}$ (a) and fragments of those spectra in a range of 500-1400 cm$^{-1}$ (b).**

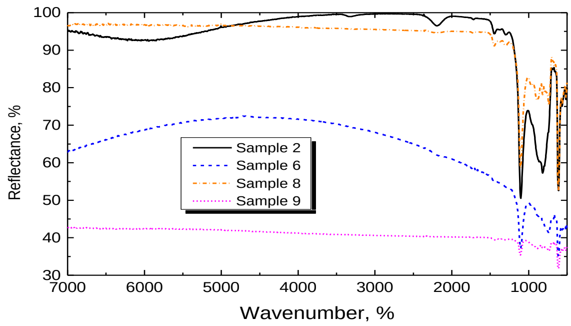



**a)**

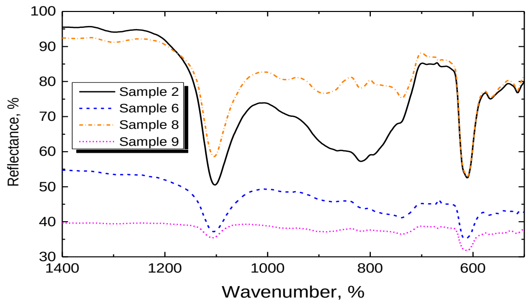



**b)**

**Fig.6. IR reflection spectra $R_f$ and $R_b$ for samples 3 and 4 (description of samples is presented in table 1) for an incidence of p-polarized IR radiation on a surface of silicon substrate at 30° angle for a wavenumber range of 500-7000 cm$^{-1}$ (a) and fragments of those spectra in a range of 500-1400 cm$^{-1}$ (b).**




**a)**




**b)**

**Fig. 7. IR reflection spectra $R_b$ for samples 2, 6, 8 and 9 (description of samples is presented in table 1) for an incidence of p-polarized IR radiation on a surface of silicon substrate at 30° angle for a wavenumber range of 500-7000 cm$^{-1}$ (a) and fragments of those spectra in a range of 500-1400 cm$^{-1}$ (b).**

## CONCLUSION

For films of non-stoichiometric silicon nitride with thickness in a range of 120 nm – 500 nm in model structures of "silicon wafer – $SiN_x$ film" and "silicon wafer –$SiN_x$ film – thin metal layer" designated for an implementation in devices based on MOMS and MEMS we studied surface morphology and IR transmission and reflection spectra in middle IR region. On a basis of analysis of selective absorption bands of $SiN_x$ and interference modulation of base line in IR spectra we made a conclusion about an implementation of optimal thickness of $SiN_x$ layer equal to 1-1.5 µm. It is planned to continue a study of efficiency, optical and electro-physical properties of multielement MOMS and MEMS devices with heat-sensitive elements, which structure comprises suspended membranes and consoles based on non-stoichiometric silicon nitride and a number of other materials.

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