IR spectra of ICPCVD SiNx thin films for MEMS structures

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Abstract. Optical properties of non-stoichiometric silicon nitride (SiNₓ) films for thermo sensitive membranes of microelectromechanical systems (MEMS) and microoptomechanical systems (MOMS) has been studied applying infrared (IR) spectroscopy. For the structures SiNx/Si and (thin metal layer)/SiNx/Si transmission and reflection spectra in the region of wave numbers of 500-7000 cm⁻¹ has been investigated. For the investigated structures analysis of optical properties observed in the IR spectra both in the form of selective absorption bands and interference modulation of a baseline was conducted.

1. Introduction
Silicon nitride is widely used in microelectronics as a material for hard mask, dielectric and passivation layers [1] formation. Silicon nitride is characterized by highly thermal stability, good dielectric and mechanical properties and chemical resistance in various aggressive liquid or gaseous environments even at high temperatures. The main advantage of silicon nitride for application in solar cells and various thermal-imaging structures lays in its quite intensive absorption band of IR spectra of about 12 um wavelength that corresponds to the IR window of the atmosphere.

Stoichiometric silicon nitride (Si3N4) films produced by high temperature CVD method are characterized by great internal stress which causes to their destruction, and can’t be applied in suspended membranes or consoled in 3D multi-element devices of MEMS or MOMS systems. In order to solve these problems, low temperature PECVD methods are applied by decomposing silane, ammonia or nitrogen at temperature lower than 300°C to form layers of a non-stoichiometric silicon nitride (SiNₓ). PECVD methods allow to generate SiNₓ films with controlled inner stresses and good mechanical properties [2,3]. For successive development of the MEMS/MOMS devices based on the SiNₓ layers, detailed studies of optical and structural properties of films produced from that material are required.

2. Experiment
2.1. Samples fabrication
SiNₓ thin films were deposited on (100) n-Si double-polished substrates and performed on an inductive coupled plasma chemical vapor deposition (ICPCVD) system (PLASMALAB 133, Oxford Instruments, Oxford, UK). The ICPCVD process ran at 250°C, under 10 mTorr pressure, 1200W RF power (13.56 MHz) and gas flow ratio (SiH₄:N₂) - 1:3. Prior to deposition the wafers was RCA
cleaned to remove organics and particles. Metal reflecting layers were magnetron sputtered. Detailed samples characteristics are listed in table 1. All films were deposited on frontal surface.

2.2. Thin film analysis

The film surfaces were examined using atomic force microscopy Smena™ VV (NT-MDT, Russia) with silicon cantilever NSG10S (NIIFP, Russia). The AFM results were obtained using tapping mode and scan speed of 4 nm/sec, which provide high resolution. In all cases, the roughness and grain size was measured for scanned area of 2x2 μm².

Transmission and reflection experiments were conducted using a Perkin-Elmer Spectrum 100 IR Fourier Spectrometer. The IR spectra were taken at wave number range of 500-7800 cm⁻¹ with 4 cm⁻¹ spectral resolution. For each sample, 16 series of the measured spectra were performed for good signal-to-noise ratio determination. The transmission spectra (T) were obtained for normal incidence of non-polarized IR radiation. The reflection spectra (R) were measured using PIKE Technologies VeeMAX™ reflector and KRS-5 polarizer attached to the IR Fourier Spectrometer. The reflection of the p-polarized IR radiation at 30° were registered both on the frontal surface of the sample (R_f) and on its back surface (R_b).

The film thickness and reflective index were measured using spectral ellipsometer Senduro (Sentech, Germany). Table 1 shows the film parameters at wavelength of 632.8 nm.

3. Results and discussion

3.1. Surface morphology

The AFM examination of the SiNx film surface revealed that the surface roughness increases by increasing thickness. Detailed surface parameters are shown in table 1. The data given in table 1 show that 50 nm of NiCr coating results in a slight change of surface roughness. However, for 200 nm Al

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Table 1. Samples information surface parameters

| No. | SiNx thickness, nm | Reflecting layer | Reflecting layer Index | Maximum change of height, nm | Roughness, nm | Average lateral grain size, nm | Average height of grain, nm |
|-----|--------------------|------------------|------------------------|-----------------------------|--------------|-------------------------------|----------------------------|
| 1   | 497                | n/a              | 2.12                   | 4.8                         | 0.6          | 73                            | 2.1                        |
| 2   | 402                | Al (200 nm)      | 2.12                   | 77                          | 10           | 98                            | 51                         |
| 3   | 280                | n/a              | 2.13                   | n/a                         | n/a          | n/a                           | n/a                        |
| 4   | 288                | Ni (5 nm)        | 2.11                   | n/a                         | n/a          | n/a                           | n/a                        |
| 5   | 236                | n/a              | 2.10                   | 3.2                         | 0.4          | 56                            | 1.5                        |
| 6   | 254                | NiCr (50 nm)     | 2.12                   | 2.4                         | 0.3          | 61                            | 1.3                        |
| 7   | 128                | n/a              | 2.10                   | 1.9                         | 0.2          | 48                            | 0.8                        |
| 8   | 128                | Al (200 nm)      | 2.11                   | 52                          | 7            | 86                            | 34                         |
| 9   | 0                  | n/a              | n/a                    | n/a                         | n/a          | n/a                           | n/a                        |
the surface roughness is higher than that of the initial surface, especially clearly seen for thin SiNₓ film.

### 3.2. Spectral analysis

Figure 1 and figure 2 show transmission IR spectra for samples with different SiNₓ thickness. As it is seen from the figures, in the wave number 700-1200 cm⁻¹ appear both selective absorption bands of the SiNₓ and modulation the baseline. This is due to the interference of the IR in the film and in fact is Fabry-Perout modes. Peaks selective absorption bands correspond to minimum value of the transmission spectra. Identification of absorption bands is based on the [5, 6].

![Figure 1](image1.png)  ![Figure 2](image2.png)

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**Figure 1.** IR transmission spectra for samples with different SiNₓ thickness for wave numbers 500-700 cm⁻¹ (a) and fragment of these spectra in the range wave numbers 500-1400 cm⁻¹ (b).

The most intensive, quite wide and complex absorption band within the range of wave numbers of 700-1020 cm⁻¹ with a minimum in the range of 820-860 cm⁻¹ corresponds to absorption bands of stretching vibration of Si-N groups. It is worth noting that this absorption band detected may cause an interest in silicon nitride as a heat-sensitive material which is suitable for application in MEMS and MOM devices due to its window of spectral transparency of the atmosphere [7].

![Figure 2](image3.png)  ![Figure 2](image4.png)

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**Figure 2.** IR transmission spectra for samples with different SiNₓ thickness for wave numbers 500-700 cm⁻¹ (a) and fragment of these spectra in the range wave numbers 500-1400 cm⁻¹ (b).

Interference extremes in the modulated spectral baseline for various samples in figure 1 and figure 2 are observed at different wave numbers associated with different SiNx layer thickness. In the transmission spectra of the most thickest SiNx film the neighboring broad interference maxima in the spectra of the modulated baseline appear at wave numbers of ~2580 cm⁻¹ and of ~7520 cm⁻¹. An
approximate estimate of intensity of selective absorption bands of stretching vibrations of Si-N at 860 cm\(^{-1}\) with a minimum transmittance \(~14\%\) and transmittance at baseline (background level) \(~49\%\) showed the value of 0.544 for layer thickness of 497 nm. Baseline level \(~49\%\) of transmittance in the IR spectrum at the wave number of \(~860\ \text{cm}^{-1}\) is determined basically by nonselective reflection and interference modulation baseline.

Figure 3 shows reflection spectra for samples with metal reflector. The spectrum of bare silicon is also given to compare. The identification method of IR reflectance spectra allows to qualitative estimate the dependence of selective absorption on the SiN\(_x\) thickness. For samples with IR semitransparent NiCr modulation baseline is clearly seen. Obviously that is due to the interference of the radiation in the SiN\(_x\) film. However, selective absorption bands SiNx practically are not visible because the angle \(30^\circ\) does not provide sufficient sensitivity method for recording reflection spectra of thin films bordered with the metal surface \([8, 9]\).

On the other hand, samples with Al and different SiNx thickness are characterized by huge differences. For 128 nm SiN\(_x\) film modulation baseline is actually absent and absorption bands stretching vibration Si-N in the range 820-860 cm\(^{-1}\) is extremely weak. But in case with 402nm SiN\(_x\) film district interference modulation of the baseline and rather intense selective absorption band Si-N in the 820-860 cm\(^{-1}\) are observed. This obvious difference in the selective absorption of the reflection spectra of SiN\(_x\) films with different thickness is due to the nonlinear dependence of the intensity of the absorption band bordering on the metal surface of a thin dielectric film on the film thickness in the optical system at low angles of incidence of IR radiation at the metal - dielectric interface \([8, 10]\).

![Figure 3. IR reflection spectra for samples with metal reflector for wave numbers 500-700 cm\(^{-1}\) (a) and fragment of these spectra in the range wave numbers 500-1400 cm\(^{-1}\) (b).](image)

### 4. Conclusion

For the non-stoichiometric silicon nitride films of thickness range 120-500 nm in the SiN\(_x\)/Si and (thin metal layer)/SiN\(_x\)/Si structures designated for using in MEMS/MOMS, IR transmission and reflection spectra were investigated for IR region. One possible way to increase the amount of light energy absorbed by the SiN\(_x\) film in the window of spectral transparency of the atmosphere is the selection of SiN\(_x\) film thickness so the interference peak occurs in the region wave numbers 700-1100 cm\(^{-1}\). Taking into consideration the analysis of SiN\(_x\) selective absorption bands and interference modulation of the base line in IR spectra, the optimal thickness of SiN\(_x\) layer was confirmed to be about of 1.0-1.5 \(\mu\text{m}\).

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