Deep-ultraviolet Raman investigation of silicon oxide: thin film on silicon substrate versus bulk material

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Abstract
Raman spectroscopy is a powerful experimental technique for structural investigation of silicon based electronic devices such as metal–oxide–semiconductor-type structures. It is widely used for characterization of mechanical stress distribution in silicon substrate. However, in the case of Raman measurements of oxide layer on silicon substrate visible excitation makes this technique almost useless. The reason for this difficulty is two-phonon scattering from silicon substrate which masks the signal from oxide layer. Application of deep-ultraviolet (deep-UV) excitation reduces the penetration depth of the radiation into silicon substrate about 30 times. As a result, the simultaneous measurement of one-phonon scattering from silicon substrate and the Raman spectrum of the oxide layer become possible. This work presents the study of thin silicon oxide film on silicon substrate with application of deep-UV Raman scattering. The spectra measured for thin film are compared with reference spectra obtained for bulk material.

Keywords: Raman scattering, MOS structure, oxide film, mechanical stress

Classification numbers: 4.10, 5.01

1. Introduction

The Raman spectra of silicon-based systems show relatively strong two-phonon scattering from silicon substrate [1]. This background makes Raman scattering in the standard version almost useless for structural investigation of the dielectric layer in silicon–based metal–oxide–semiconductor (MOS)-type structures [2]. Raman micro-spectroscopy has so far been used for determination of mechanical stress in semiconductor substrate [3]. Attempts made to calculate the stress with Stoney formula [4] did not give satisfying results. The main reasons are: non-crystalline structure of the oxide layer [5] and existence of densified domains in dielectric layer [6]. The structure of thin SiO\textsubscript{2} film differs from the structure of bulk material. The main difference between the dielectric layer and bulk silica is the existence of pseudo-crystalline structures of cristobalite, tridymite or coesite [5]. Domains with densified silica can be found in thin film whereas in amorphous bulk material they are not present [5]. The structure of the SiO\textsubscript{2} film also depends on the procedure of its manufacturing (surface oxidation or deposition) and the crystallographic orientation of the substrate [5].

An important factor determining electric properties of MOS structures is non-uniform distribution of the mechanical stress in MOS structures [7]. However, the relation between stress and Raman spectra of silica was obtained for bulk material [6, 8–10] and not for the oxide film. Because of structural differences between bulk silica and silicon oxide thin film, the conclusions driven for bulk material can be imprecise in the case of thin film. That is why the application of the relation between stress and Raman spectra obtained for bulk silica in the interpretation of the data obtained for thin dielectric film introduces a low level of confidence in the conclusions. To sum up: direct structural studies of dielectric film are necessary for understanding phenomena determining electric properties of MOS structures.

The aim of this work is to present the application of deep-ultraviolet (deep-UV) Raman scattering in the study of thin oxide film on silicon substrate. The majority of
strong lines coming from crystalline forms of silica are observed for Raman shift below 520 cm\(^{-1}\). That is why the identification of expected pseudo-crystalline domains like cristobalite, tridymite, coesite or \(\alpha\)-quartz [5] requires the analysis of Raman scattering from oxide layer in the range of Raman shift below the position of the silicon line. In the case of standard excitation, which means usually with Ar\(^+\) laser lines 488 or 514 nm, the range of Raman shift from \(\sim 220\) to \(\sim 500\) cm\(^{-1}\) is dominated by two-phonon scattering from silicon substrate. The application of deep-UV light for excitation strongly reduces the light penetration depth into silicon substrate: from \(\sim 3\) \(\mu\)m for 488 nm to \(\sim 100\) nm for 266 nm (about 30 times). This reduction of penetration depth should result in strong reduction of two-phonon intensity in comparison with the Raman scattering from the oxide layer. This work presents not only the study of the thin oxide film on the silicon substrate, but also compares the Raman spectra measured for the silicon-oxide film with the data obtained for bulk material with deep-UV excitation.

2. Experimental

Prior to Raman study, dielectric layers were characterized with transmission electron microscopy (TEM), spectroscopic ellipsometry and ultra violet–visible–near infrared (UV–Vis–NIR) transmission/reflection spectroscopy.

Transmission electron microscopy (TEM) specimens were prepared by the conventional cross-section method using a low-temperature argon ion milling process. The SiO\(_2\) layer microstructure was observed by conventional TEM and high resolution TEM (HRTEM) techniques in the JEM-2100 JEOL microscope.

Variable angle spectroscopic ellipsometer (VASE, J.A. Woollam Company Inc., USA) was used for characterization of thin dielectric films. Measurements were performed for four incident angles: \(65^\circ\), \(70^\circ\), \(75^\circ\) and \(80^\circ\) (near Brewster angle of the SiO\(_2\)/Si system). The spectral range of measurements started at 250 nm and ended at 1000 nm. The distance between two consecutive measurements was equal to 5 nm. The measurements were repeated three times (at three different places) for each wafer: once at the middle and twice near the edge (about 15 nm from the edge of the wafer).

Spectroscopic ellipsometry characterizes the sample by two parameters: (i) ratio of amplitudes of reflected light observed for polarizations \(p\) and \(s\): \(\Psi\); (ii) phase change of reflected light observed for polarizations \(p\) and \(s\): \(\Delta\).

To extract quantitative information about a sample, it is necessary to construct the model including a suitable number of species characterized by parameters like thicknesses and complex refractive index. Then the functions \(\Psi\) and \(\Delta\) are fitted to experimental data. The Cauchy dispersive model is usually used to fit ellipsometric data for dielectrics and semiconductors in spectral regions if the dielectric layer is transparent. Transparent means the extinction coefficient is equal to zero. This transparency reduces the complex refractive index to its real part. The Cauchy model allows determining of refractive index and thickness of dielectric layer. The relationship between wavelength and refractive index is given by Cauchy’s dispersion formula

\[
n(\lambda) = A + \frac{B}{\lambda^2} + \frac{C}{\lambda^4} + \cdots,
\]

where \(A\), \(B\), \(C\) are fitted coefficients, and \(\lambda\) stands for wavelength in \(\mu\)m.

The optical model used for ellipsometric data analysis consisted of a silicon dioxide layer as a Cauchy layer and a silicon substrate. The parameters describing silicon substrate were fixed. The values were taken from Ellipsometer data table (http://eceee.colorado.edu/hart/book/ellips.htm). In the applied model, the two parameters \(A\) and \(B\) from equation (1) and SiO\(_2\) layer thickness were fitted. The third component of equation (1) was neglected. In opposition to the approach presented in [11–13] the interface between Si substrate and SiO\(_2\) was not included in the model.

For transmission and reflection measurements UV–3600 UV–Vis–NIR absorption spectrometer was used (Shimadzu, Japan). The measurements were performed for three configurations of the spectrometer: transmission, diffuse reflectance and relative specular reflectance.

Two kinds of samples were investigated with Raman spectroscopy: three silicon wafers covered with thin silicon-oxide film and silica plate. The dielectric film was prepared by surface oxidation at a temperature equal to 1000°C on three silicon wafers of \([111]\) crystallographic orientation. The diameter of the wafer was equal to 76 mm and its thickness equal to 0.7 mm. The thickness of the oxide film was equal to 60 nm. As a sample of bulk material the plane-parallel plate made from Suprasil I was used.

Raman scattering was measured with a MonoVista 2750i micro-Raman spectrometer based on the Olympus BX51 microscope. As an ultraviolet excitation source, the laser line \(\lambda = 266\) nm (FQCW 266–10, CryLas GmbH, Germany) was used. Reference visible Raman spectra were excited with Ar\(^+\) laser line \(\lambda = 488\) nm (543-Ap-A01, CVI-Melles-Griot, USA). UV Raman spectra were collected with a UVB objective lens (magnification 40×). Spectra were analyzed with a SectraPro 2750i (Princeton Instruments, USA) spectrograph equipped with ultraviolet grating (3600 lines mm\(^{-1}\), blazing wavelength 250 nm). Measured spectra were recorded with a nitrogen-cooled CCD camera with maximum efficiency at 250 nm (LN/2048×512B/UVAR, Spec-10 System, Princeton Instruments, USA). To obtain visible spectra, the sample was illuminated through a visible objective lens (magnification 100×). The grating used for analysis of visible Raman spectra had 1800 lines mm\(^{-1}\) and was blazed for visible spectral range. The spectral CCD camera applied to record visible Raman spectra (LN/1340×100B EXCELON, Spec-10 System, Princeton Instruments, USA) had the maximum efficiency in visible spectral range between 500 and 700 nm. Images were recorded with a TM 2040GE camera (JAI, Japan).

Mathematical procedure including offset correction, smoothing, baseline correction and normalization was applied to measured spectra prior to analysis. The mathematical treatment was performed with the program Grams Suite 8 (Thermo Scientific, USA).

Since the main aim of this work was to show the utility of deep-UV Raman spectroscopy in the investigation of thin dielectric film on silicon substrate, results obtained from TEM, ellipsometry and transmission/reflection will be presented in the discussion of Raman spectra.
3. Sample characterization

3.1. Samples preparation

Silicon oxide films were manufactured in the Division of Silicon Microsystem and Nanostructure Technology (Institute of Electron Technology, Warsaw, Poland). Three-inch diameter Czochralski-grown, p-type [111]-oriented silicon wafers were used as substrates. After an initial hydrogen-peroxide-based cleaning sequence, the wafers were subjected to thermal oxidation process at 1000 °C in order to grow silicon-dioxide layers of thicknesses of approximately 60 nm. Reference samples were prepared from Suprasil I with standard procedure used to manufacture plane-parallel optical plates.

3.2. Transmission electron microscopy

TEM and HRTEM cross-sections of the SiO$_2$/Si structure are shown in figure 1. The top surface of the SiO$_2$ layer (protected by an epoxy layer during the preparation of the sample for TEM (figure 1(a)) and the SiO$_2$/Si interface (figure 1(b)) was uniform and smooth. The thickness of the SiO$_2$ layer was 60 nm (marked in figure 1(a)). No crystallites were formed and visible within the SiO$_2$ layer, and the layer was fully amorphous (figure 1(c)).

3.3. Spectroscopic ellipsometry

An example of the relation between the refractive index and the irradiation wavelength is presented in figure 2. The refractive index measurements on other samples show deviations from these values not larger than 0.018. Measured values of refractive index were similar to the refractive indices reported for Suprasil I [14]. The thickness of the oxide layer calculated from ellipsometry measurements was equal to 64 nm. The dispersion of the values obtained form different measurements was not larger than 2 nm.

3.4. Transmission/reflection spectroscopy

Figure 3 presents data obtained from transmission and reflection measurements. The black line shows the data obtained for diffuse reflectance, red for specular reflectance and green for transmission. The decrease of transmittance in the range 1000–1100 nm corresponds to a band gap equal to about 1.1 eV, which is in agreement with Si band gap [15]. Signal significantly larger for specular than for diffuse reflectance confirms the smoothness of SiO$_2$/Si interface showed by TEM.

4. Results and discussion

Figure 4 shows the contribution of silicon line ‘520 cm$^{-1}$’ to the measured spectrum in the range of Raman shift from 150 to 1200 cm$^{-1}$. Figure 4(a) presents the measured spectrum (black solid line) and the reconstruction of silicon line with single Lorentzian profile (dashed red line). It is clearly visible that the influence of the line ‘520 cm$^{-1}$’ is important within the range about ±20 cm$^{-1}$ around the maximum. Outside this range the contribution to the spectrum is negligible. Figure 4 compares two methods of extraction of silicon line from the spectrum. The black line represents the measured spectrum after removing the range corresponding with the line ‘520 cm$^{-1}$’ by means of zap function implemented in Grams Suite. The red line represents measured spectrum after subtraction of fitted Lorentzian profile shown in figure 4(a). The gray semitransparent rectangle marks the range of Raman shift dominated by silicon line. Outside this area, spectra obtained with both procedures can be treated as almost identical. Negligible differences in both spectra presented in figure 4(b) show that both procedures applied to remove silicon line: zapping of spectral range with dominant intensity of silicon line and subtracting Lorentzian profile fitted to the line ‘520 cm$^{-1}$’ give the same results. Because of this the easier procedure, namely zap function, was used to remove silicon line from spectra, presented in a further part of this work.

Figure 5 presents the contribution of two-phonon scattering from silicon substrate to the spectrum measured with UV excitation. Figure 5(a) shows spectra of Si/SiO$_2$ wafer measured with different excitation wavelengths 266 and 488 nm. In both spectra the silicon line was removed...
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Figure 2. Example of the relation between the refractive index of silicon oxide layer and the irradiation wavelength.

![Refractive index of SiO\textsubscript{2} dielectric film on Si wafer](image)

Figure 3. Example of the results obtained for transmission / reflection measurements of the SiO\textsubscript{2}/Si sample.

![Transmission / Reflection measurements of Si / SiO\textsubscript{2} sample](image)

Figure 4. Contribution of silicon line (‘520 cm\textsuperscript{-1}’) to the Raman spectrum of Si/SiO\textsubscript{2} sample. (a) Presents measured spectrum (black solid line) and Lorentzian profile (red dashed line) fitted to the silicon line. (b) Compares two methods of removing silicon line: application of zap function in Grams Suite 8 (black line) and subtraction of fitted Lorentzian profile from measured spectrum (red line).

![Raman scattering from SiO\textsubscript{2} / Si plate, \(\lambda_{\text{ex}} = 266\) nm](image)

![Raman scattering from silicon oxide thin film, \(\lambda_{\text{ex}} = 266\) nm](image)

The range of Raman shift with the important contribution of line ‘520 cm\textsuperscript{-1}’ is marked with the semitransparent gray rectangle. This range of Raman shift is larger than in figure 4 because the line ‘520 cm\textsuperscript{-1}’ is much stronger in the case of visible excitation than for ultraviolet excitation light. Both spectra were normalized in order to compare their shapes. The intensities in the band placed between 930 and 1030 cm\textsuperscript{-1} were assumed to be equal for visible and ultraviolet Raman spectra. This band is assigned to multi-phonon scattering coming from silicon substrate [1] and is used here as standard for comparison of Raman spectra measured with different excitation wavelengths. Let us assume that in the case of visible spectra the contribution of the scattering from the oxide layer is negligible. The spectrum obtained by subtraction of the Raman spectrum measured with visible excitation from data obtained with ultraviolet excitation should contain the signal coming from the oxide layer with negligible contribution of two-phonon scattering from Si substrate. Presented in figure 5(b), the subtracted spectrum in comparison with data obtained directly from
Figure 5. Comparison of multi-phonon Raman scattering in silicon substrate with Raman spectra measured with UV excitation.
(a) Normalized in the range 930–1030 cm\(^{-1}\) spectra measured with ultraviolet excitation (black line) and visible excitation (red line).
(b) The spectrum obtained by subtraction of visible Raman signal from ultraviolet one.

Figure 6. Comparison of silicon oxide Raman spectra obtained for thin film on silicon substrate (black line) and bulk material (red line). The positions of maxima of the most important bands are also presented in the plot.

measurement with UV excitation shows a very similar shape. The shape of the subtracted spectrum confirms the hypothesis that strong reduction of light penetration depth into silicon substrate results in negligible two-phonon scattering from the substrate. The exception is the two-phonon silicon band placed in range of Raman shift between 930 and 1030 cm\(^{-1}\) used here as a normalization standard for spectra measured with different excitation wavelengths.

Figure 6 presents the comparison of the Raman spectra measured for thin silicon oxide film (black line) and bulk material Suprasil I (red line). The positions of the main bands (maxima) are also presented. Font colors are the same as in the case of presented spectra. The maximum of silicon line equal to 519.7 cm\(^{-1}\) is indicated in the plot as well. Since the spectral resolution of MonoVista 2750i in ultraviolet is equal to about 1 cm\(^{-1}\), the position of maximum of silicon line can be treated as in the case of non-stressed silicon wafer.

The shape of the spectrum measured for bulk material is similar to the silica Raman spectrum from literature [16]. The differences between measured and reported spectra are observed for Raman shift below 300 cm\(^{-1}\). The so-called boson peak with maximum at about 60 cm\(^{-1}\) [17] and the band with maximum at about 245 cm\(^{-1}\) contributing to the main Raman band of bulk material and assigned to scissoring in tetrahedron [SiO\(_2\)] [18] cannot be found in the ultraviolet spectrum. The differences in Raman spectra obtained for the same sample with different excitation wavelengths were already observed for materials without long range order. An example of such a phenomenon is delivered by Raman spectroscopy of different forms of carbon. Depending on excitation wavelength, a pair of bands \(D\) and \(G\) \([19, 20]\) or \(T\) and \(G\) are observed \([21]\), respectively. In the case of visible excitation vibrations of sp\(^2\)-phase are excited. These vibrations are observed as \(D\) and \(G\) bands \([21]\). \(D\) band is dispersive: the linear relationship between maximum position and excitation energy has the slope in the range 43–61 cm\(^{-1}\)eV\(^{-1}\) \([19, 20]\). In the case of hydrogenated amorphous carbon (a-C:H) and B ion implanted glassy carbon (B-II-GC) films, \(D\) and \(G\) bands show depressive behavior for visible excitation light \([22]\). The linear relationship between maxima positions and excitation energy is interpreted in terms of \(\pi - \pi^*\) resonant Raman scattering from various size sp\(^2\) clusters \([22]\). The maxima positions of \(T\) and \(G\) bands measured for nitrogenated...
tetrahedral amorphous carbon (ta-C:N) films show the linear dependence on the concentration of nitrogen used in the deposition process [21]. There are two mechanisms that can be responsible for dispersive behavior of G band in UV-Raman spectrum: size-dependent resonant scattering from sp²-clusters or bending of sp³ bonds due to large internal stress in carbon clusters [21]. The scale of the internal stress depends on the nitrogen content. The variation of T band maximum position is correlated with the content of sp³ phase. The content of sp³ phase in carbon film decreases with the increase of nitrogen content used in the deposition process. Higher nitrogen concentration results in larger sp²-phase content and the proportion sp³/sp²/sp³−sp² is shifted towards sp²−sp³ bonds. As a result, the T band is shifted towards larger values of Raman shift and for low concentration of sp³ phase, the position of T band is close to the position of D band observed in visible Raman spectra [21]. To sum-up: the behavior of Raman spectra of carbonic sample is an example of the relationship between excitation wavelength and the shape of observed spectra. The differences between spectra measured with visible and ultraviolet excitations are significantly large in the case of amorphous materials due to dispersed values of atomic structure parameters, mainly bond angles.

The main band in the spectrum obtained for bulk material consists of two parts: the broad one with maximum at about 434 cm⁻¹ and a relatively narrow line with maximum at about 486 cm⁻¹. The maximum position of the narrow line corresponds to D₁ line reported in literature [17]. This line is assigned to vibration of four-member rings—defects in the structure of bulk silica. The asymmetric shape of the broad part of the main band suggests its complex character. Firstly, there is observed a relatively steep increase of the intensity in the range of Raman shift 280–300 cm⁻¹. In the range 300–434 cm⁻¹, the changes of intensity are gentle. For values of Raman shift between 434 and ∼520 cm⁻¹, the intensity of the main band decreases. The shape of the main band described above suggests that it should consist of at least three components. Two of them should have maxima placed between 300 and 400 cm⁻¹. The maximum of the third component should be placed between 434 and 470 cm⁻¹. The main band of bulk silica was described in literature with application of three components with maximum placed between 300 and 400 cm⁻¹; D₁ ≈ 295 cm⁻¹, D₂ ≈ 380 and ∼465 cm⁻¹ [18]. Reported in literature, data were assigned to the following vibrations: D₁—scissoring in extended tetrahedron [SiO₄]/2−[Si₄/4], D₄ and ∼465 cm⁻¹—bending in 5+ rings (five-members rings or larger) [18]. The line 601 cm⁻¹ corresponds to the D₂ line reported in literature as a trace of vibrations in three-member rings [17]. The position of the maximum of the next band 792 cm⁻¹ and its complex shape corresponds to the band denoted in literature as ‘800 cm⁻¹’ [23]. The last two bands recorded for bulk material: 1063 and 1200 cm⁻¹ correspond to bands 1065 and 1200 cm⁻¹ reported in literature [23].

Denoted with a black line, the spectrum measured for thin film differs from data measured for bulk material. A small band with maximum at 233 cm⁻¹ can be correlated with a strong line of cristobalite 230 cm⁻¹ [24]. Correlation with the band assigned to scissoring in tetrahedron [SiO₄]/2−∼245 cm⁻¹ [18] is much worse. The main band consists of two parts: the broad band and relatively narrow line. The maximum of the narrow line is placed at 488 cm⁻¹ and can be correlated with D₁ line observed for bulk material. Since the line is placed near the boundary of the area dominated by silicon line (‘520 cm⁻¹’), the detailed analysis of the line shape is not possible. In the broad part of the main band two maxima can be found: at ∼333 and ∼438 cm⁻¹. In the range of Raman shift corresponding to the broad part of the main band, strong lines of expected pseudo-crystalline species are placed. There are strong lines of tridymite at 320, 355, 403, 422, 449 and 457 cm⁻¹ [24], as well as those of α-cristobalite and α-quartz at 421 and 465 cm⁻¹ [24], respectively. Strong lines of coesite (522 cm⁻¹) and moganite (501 cm⁻¹) [24] are placed in the range of Raman shift dominated by silicon line so they can be masked by signal coming from silicon substrate. In the next band three main components can be recognized. The maxima of components are placed at the following values of Raman shift: 600, 624 and 664 cm⁻¹. The main maximum 624 cm⁻¹ corresponds to the position of maximum of D₁ line observed for densified structure of bulk material [10]. The maximum placed at 600 cm⁻¹ corresponds to the position of D₂ line observed for non-densified structure in bulk silica. The maximum placed at 664 cm⁻¹ has the best correlation with the weak line of coesite 661 cm⁻¹ [24]. Generally, the multicomponent band with maximum at 624 cm⁻¹ can be treated as shifted towards larger values of Raman shift and broadened D₂ line of non-densified structure in bulk material. This kind of behavior was observed for densified bulk material [23]. The next band with maximum placed at 815 cm⁻¹ is similar to the band ‘800 cm⁻¹’ observed for densified structures [24]. This similarity involves two aspects: the shift of the maximum towards larger values of Raman shift and the change of the shape of the band (increase of the contribution of higher frequencies in the total intensity). The band with maximum at 1084 cm⁻¹ can be correlated with weak lines of α-quartz ~1080 cm⁻¹ or α-cristobalite ~1070 cm⁻¹ [24]. The band centered around 1201 cm⁻¹ shows the best correlation with weak lines of α-tridymite ~1190 cm⁻¹ or α-cristobalite ~1180 cm⁻¹ [24]. Correlation of bands 1084 and 1201 cm⁻¹ with bands of bulk material 1065 and 1200 cm⁻¹ should probably be excluded. Two bands mentioned above observed in bulk silica are shifted downwards for densified structures [23].

5. Conclusion

To sum up: the structure of silicon oxide film is similar to that of densified silicon oxide bulk material. D₂ line and the band ‘800 cm⁻¹’ show features typical for densified SiO₂. There are also bands that can be correlated with pseudo-crystalline structures like cristobalite, coesite, tridymite or quartz. Detailed study of Raman spectra of silicon oxide layer, including mathematical analysis with Gaussian or Lorentzian profiles as well as measurements with application of controlled stress, should deliver detailed knowledge about the structure of the oxide layer. This knowledge should be helpful in the understanding of non-uniform distribution of electric parameters in MOS-type circuits [7]. Investigation of the
silicon oxide layer can be also treated as an introduction to the study of dielectric layers made from materials like gadolinium oxide, lanthanum-lutetium oxide or hafnium oxide.

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