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Optical and electrical characterizations of 4H-SiC–oxide interfaces by spectroscopic ellipsometry and capacitance–voltage measurements

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ABSTRACT

4H-SiC–oxide interfaces formed by various oxidation methods on SiC (0 0 0 1) Si- and (0 0 0 $\bar{1}$) C-face substrates have been characterized by performing spectroscopic ellipsometry in wide spectral region including deep UV spectral range and capacitance–voltage measurements. The results exhibit that the refractive indices of the interface layers well correlate with interface state density in all the cases of oxidation processes. To investigate the difference in interface characteristics between wet and dry oxidation, we compared to the sample fabricated by wet oxidation followed by heating in Ar or O_2 atmosphere, aiming to remove hydrogen related species at the interface. We also tried to make clear the difference in the interface characteristics between Si- and C-faces by lowering the oxidation rate of C-face down to those for Si-face. Putting together with all of the results obtained, we discuss the origins that determine the interface characteristics in terms of both the optical and electrical characterizations.
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1. Introduction

Among wide band-gap semiconducting materials, only silicon carbide (SiC) can be thermally oxidized, and insulating SiO_2 layers, known as superior dielectric material for metal-oxide-semiconductor (MOS) applications, can be grown on it, similar to Si. In addition, its physical properties, such as high-breakdown electric field and high thermal conductivity, compared with Si, are good for high-speed switching and low-power-loss electronic devices [1]. For these reasons, SiC MOS field-effect transistors (MOSFETs) are expected to have superior specifications that cannot be obtained using Si. However, the electrical characteristics of SiC MOSFETs, such as on-resistance, are seriously poorer than those predicted from SiC bulk properties [2]. It has been considered that these poor device characteristics result from a high interface state density [3]. While, it has been well known that the characteristics of SiC–oxide interfaces such as interface state density strongly depend on the oxidation method and the surface orientation of the substrates used [4–6]. The electrical properties, such as interface state density and/or oxide fixed charges should relate strongly to the interface structures. However, the structures of the interfaces have not be completely clarified yet, resulting in insufficient understanding of the origin of interface states which brings about the poor electrical properties of SiC-MOSFETs.

Many studies have been carried out to investigate the SiC–oxide interfaces electrically, such as capacitance–voltage (*C–V*) measurement [3–6], and structurally, such as X-ray photoelectron spectroscopy (XPS) [7–9] and secondary ion mass spectroscopy (SIMS) [10]. However, since only a few studies that combine the structural study with the electrical characteristics have been carried out [9,11,12], their relation has not been sufficiently clarified yet. Moreover, since, in XPS and SIMS measurements, the samples are etched down to the interfaces in order to get the information from the interfaces, we fear that the interface structures are changed by exposing to vacuum and/or by etching with, for example, ion bombardment during the measurements. In contrast, optical methods, like spectroscopic ellipsometry, can evaluate the

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interfaces through transparent oxide layers without any change in the interface structures.

In the previous works [13,14], we have evaluated the oxide–SiC interfaces by spectroscopic ellipsometry measurements in the spectral range between 1.5 and 4.2 eV using slope-shaped SiC oxide films. We have found that an interface layer with high refractive index, larger than those for SiC and SiO_2, around 1nm in thickness, exists between the oxide and SiC [13], and the values of refractive indices depend on the oxidation method and oxidation temperature [14]. In the case of Si oxidation, oxide–Si interfaces have been studied by using spectroscopic ellipsometry in the absorption region, i.e., the visible and ultraviolet spectral region, where Si shows optical properties reflecting the direct optical transitions. In the case of SiC, the bandgap energy of 4H-SiC, for example, is 3.2 eV which is covered by our measurements reported. However, as in the case of Si, SiC is an indirect bandgap semiconductor, and its absorption is very small up to around 4 eV. Therefore, it is expected that the extension of the photon energy range of the measurements to that corresponding to the direct transition, i.e., to the deep UV spectral range, may bring about more information on the structures of the interfaces, especially the information from the part closed to SiC substrates, as well as those from the part near the oxide layers.

In this report, we have characterized the SiC–oxide interfaces fabricated by various oxidation methods grown on SiC (0 0 0 1) Si- and $(0\,0\,0\,\bar{1})$ C-face substrates. We have performed spectroscopic ellipsometry measurements in the wide spectral range from the visible to deep ultraviolet (DUV) spectral range, i.e., between 1.5 and 6.4 eV. *C–V* measurements have been also carried out for the same samples used in the optical measurements in order to the direct comparison between optical and electrical characteristics. Based on the relation between optical and electrical characteristics of the interfaces and their differences in the dependence on the oxidation method and surface orientation, we have discussed the interface structures related to the interface states.

2. Experiments

4H-SiC (0 0 0 1) Si-face and $(0\,0\,0\,\bar{1})$ C-face epitaxial layers with 8° off-angles were used in this study. After standard RCA cleaning was executed, SiC epilayers of Si-face were oxidized in dry and wet oxygen flow at 1000 and 1100 °C, and those of C-face were oxidized in dry oxygen flow at 900, 1000, and 1100 °C, and in wet oxygen flow (O_2: H_2O = 2:1) at 900, 950, and 1000 °C. We have also performed the oxidation of SiC under the reduced oxygen pressure, where the Ar and O_2 mixture gas of 1 atm was introduced into the furnace. The thicknesses of the oxide layers used in this study were around 20 nm.

The photon energy dependences of ellipsometric parameters (Ψ, Δ) were measured by use of a spectroscopic ellipsometer (GESP5; *Sopra*) in the photon energy range between 1.5 and 2.0 eV at an angle of incidence of 70° and between 2.0 and 6.4 eV at an angle of incidence of 75°. We have changed the angle of incidence in the two energy regions, because the angle of incidence leading to high sensitivity changes by the photon energy range measured. In the optical analysis of oxide layers on SiC, we have assumed that the oxides are composed of 2 layers, i.e., a silicon oxide layer having stoichiometric composition and a interface layer, and derived the thickness of the oxide layer and the optical constants of the interface layer. As it is hard to determine the thicknesses of the interface precisely, we have assumed the layer to be 1 nm, followed by the results from the XPS studies [15].

As the interface layers are considered to be composed of Si, O, and C, any combination of these atoms gives the materials having no or very small absorption in the photon energy rage from 1.5 and 2 eV, which suggests that the extinction coefficient can be assumed to be zero in his energy range. Therefore, in this range, we can determine the two parameters, thickness of oxide layer and refractive index of the interface layer from the ellipsometric parameters (Ψ, Δ) measured. We have used the Sellmeier's dispersion relation to the wavelength dependence of the refractive indices of the interfaces. Using the thickness of the oxide layer determined from the data in 1.5–2.0 eV, we have derived the optical constants (n, k) of the interface layers at each photon energy in the range between 2.0 and 6.4 eV from the ellipsometric parameters (Ψ, Δ) measured.

High-frequency *C–V* measurements were carried out for the same samples used for the optical measurements to evaluate interface state density D_{it}. Al electrodes, 0.5 mm in diameter, were formed on the oxide surfaces by vacuum evaporation to fabricate MOS capacitors. *C–V* measurements were performed using an *LCR* meter (Agilent Corp. 4284A) with a 1 MHz driving frequency and a time constant of 10 s. The measurements were done at room temperature. The values of D_{it} were estimated by the Terman method [16].

3. Results and discussion

3.1. Dependences on oxidation method, surface polarity and oxidation temperature

Fig. 1(a–c) shows the optical constants of the interface layer, *C–V* characteristic and interface state density (D_{it}), respectively, for Si-face oxidized by wet and dry oxidation at the oxidation temperatures of 1000 and 1100 °C. The optical constants of SiO_2 and SiC are also shown in Fig. 1(a). It is found from Fig. 1(a) that the values of n are much larger than those for SiC and SiO_2 at all the photon energies measured, and increase with photon energy. The values of k are almost zero below about 5 eV and increase with photon energy steeply above 5 eV, which is quite similar to those of SiC. The figure also shows that the values of n do not change little with oxidation temperature, while change remarkably with oxidation method, i.e., those for wet oxidation are larger than those for dry oxidation, while energy dispersions of k show no or little change with oxidation method and temperature. Looking more precisely, however, we can see the changes of k with the oxidation method in the DUV spectral region, i.e., those for wet oxidation are a little larger than those for dry oxidation at around 6 eV, though little dependence in the temperature. Fig. 1(b) shows the capacitance normalized by the capacitance corresponding to the oxide (C_{ox}) as a function of gate voltage. The figure reveals that only the dry oxidized 1100 °C sample has a large flat-band voltage shift. In our previous work, we found that the flat-band voltage for dry oxidized Si-face sample is intrinsically large but abruptly decreases with concentration of vapor in the oxygen ambient used in the oxidation [17]. Therefore, we believe that the small flat-band voltage shift for 1000 °C is due to the slight concentration of vapor in the oxygen ambient. Fig. 1(c) shows the values of D_{it} as a function of energy from the conduction band edge. In comparison between wet and dry oxidation, both of n and D_{it} for wet oxidation are larger than those for dry oxidation. The values of D_{it} for wet and dry oxidation are scarcely dependent on temperature just as in the case of n, which is different from the tendency in flat-band voltage. Taking together with these results into account, we can say that there exists a good correspondence between (n, k) and D_{it} in the dependencies of oxidation method and temperature. To our knowledge, in the case of Si, there is no article that reports the correlation between optical constant and D_{it}. There are few articles those report the oxidation temperature dependence of D_{it} [18,19]. According to these articles, in general, the lower oxidation temperature brings about the lower D_{it}, like our results. In contrast, there have been many articles those compare wet

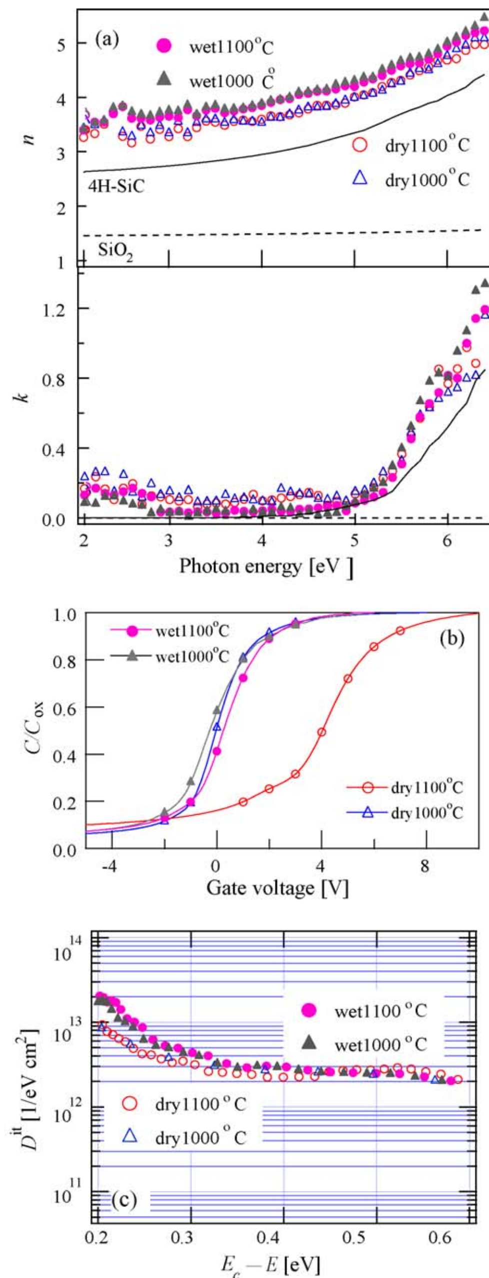

Fig. 1. Refractive index, n, and extinction coefficient, k, of the interface layer (a), C–V characteristic (b), and interface state density, D_{it} (c) on Si-face. The solid and broken lines in (a) show the optical constants of SiC and SiO_2, respectively.

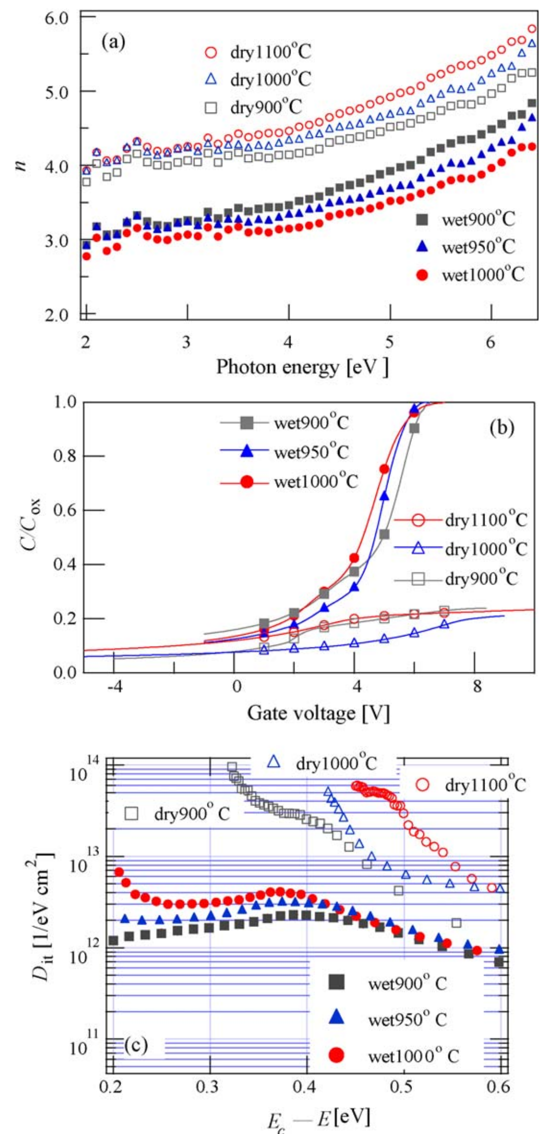

Fig. 2. Refractive index of interface layer, n (a), C–V characteristic (b), and interface state density D_{it} (c) on C-face.

oxidation with dry oxidation, and the results therein agree with our results [20,21].

Fig. 2 shows the values of optical constants n and k, C–V characteristic and interface state density D_{it} for C-face oxidized by wet oxidation between 900 and 1000 °C and those by dry oxidation between 900 and 1100 °C. In the case of dry oxidation, as shown in Fig. 2(b), the capacitance does not reach the accumulation region, which is probably due to the Fermi level pinning caused by extremely high D_{it}. In this case, since we did not obtain the oxide capacitance from C–V curve, we used the oxide capacitance derived from the oxide thickness, measured by spectroscopic ellipsometry measurements. In the case of C-face, wet oxidation, n and k values cannot be obtained unless we assume the thickness of the interface to be not 1 nm but around 1.5 nm, which suggests that the thickness of the interface for C-face, wet oxidation is larger than the other samples. Thus we assumed the thickness of the interface to be 1.5 nm only for C-face, wet oxidation. Since the C–V data in the plateau region cannot exactly estimate the D_{it} values, we used the C–V data except for this region for the D_{it} analysis. The value of D_{it} for wet oxidation is smaller than that for dry oxidation, which is opposite relation to that for Si-face. It is very remarkable that the values of D_{it} for dry oxidation are very high compared not only with those for C-face, wet oxidation but with those for Si-face. The figures also show that both of n and D_{it} for wet and dry oxidations depend on oxidation temperature, which is different from the case of Si-face. In addition, the values of D_{it} both for dry and wet oxidation and those of n for dry oxidation increases with temperature, and, in contrast, those of n for wet oxidation decreases with temperature.

From the results for the oxidation method-, oxidation temperature- and polar face-dependences of optical constants of interface layers and interface state density, it is obvious that the refractive indices of the interface layers have some information on the interface states. The fact that the dispersion relation of n is similar to that of SiC but its absolute value is higher than that of SiC and the value of k is almost equal to that of SiC may mean the presence of materials having the similar band structure as that of

SiC at the interface. Namely, the interface layer is considered to be not the transition layer between SiC and SiO_2, like SiO_x or SiC_xO_y, but a layer having the modified structure and/or composition from SiC, locating at the SiC-side of SiC–oxide interface. Nguyen et al. have derived the dielectric functions of the interface between Si and oxides using spectroscopic ellipsometry in the direct transition energy (E_0) region for Si, i.e., from 1.5 to 6 eV, and found the presence of the interface layers having the dielectric constants just like Si, but the E_0 energy shifts in the order of 0.04 eV, to smaller photon energy. They have attributed this shift to the strained Si layers, around 1 nm in thickness, formed due to the expansion of the Si–Si atom distance by oxidation. In the case of SiC oxidation, Si–Si atom distances are also expanded by oxidation, and thus there is a possibility of existing strained SiC layers at the interface. If so, as the E_0 energy for 4H-SiC is reported to be around 5.6 eV [22], the larger values of k in the DUV spectral region may mean the presence of largely stained SiC layers at the interface, which may bring about the red shift of E_0 energy, resulting in the large n and k at the visible and near UV region. Another candidate as the interface structure is SiC layer with interstitials taking into account the dispersion relations of n and k [23]. It is plausible that the existence of such a stained layer or a SiC layer with interstitials relates to the electrical properties of interface, like interface state density. In order to discuss about the existence of stained layers and their influence on the interface properties, more precise measurements of the optical constants of interfaces in the DUV spectral region are required.

In the case of C-face, wet oxidation, the correlation between refractive indices and the interface state density is contradictory against the other cases. We have no explanation for it, so far. However, taking into account the fact that it is necessary to change the interface layer thickness from 1 nm to around 1.5 nm for adjusting the calculated values to measured ones, we may have to discuss not only from the values of refractive indices, but also from the change of interface layers thickness, which will be carried out in future.

We will discuss the dependence of oxidation method and surface polarity in detail in the successive sections.

3.2. Dry oxidation vs. wet oxidation

As shown in Figs. 1 and 2, both n and D_{it} for wet oxidation on Si-face are larger than those for dry oxidation, while on C-face, both of them for wet oxidation are much smaller. In the case of wet oxidation, species related to hydrogen, like -H and/or -OH radicals may play an important role for the properties of MOS structures. We examined the effect of hydrogen related species for the optical and electrical properties of SiC–oxide interfaces by removing the hydrogen related species from the interfaces intentionally. For this purpose, the samples fabricated by wet oxidation were heated in Ar and O_2 atmosphere at 600 °C for 3 h, which is known to be sufficiently effective to change the interface characteristics [24]. Figs. 3 and 4 show n and D_{it} values for Si- and C-face, respectively after heating as well as those before heating. From the figures, both for Si- and C-faces, the interface characteristics close to those for dry oxidation by heat treatment, hence it is considered that before heating, hydrogen related species terminate the dangling bonds of Si or C at the interface, and then they are removed by heating, resulting in the interface characteristic closed to those for dry oxidation. In addition, the reduction in D_{it} for C-face by heating are

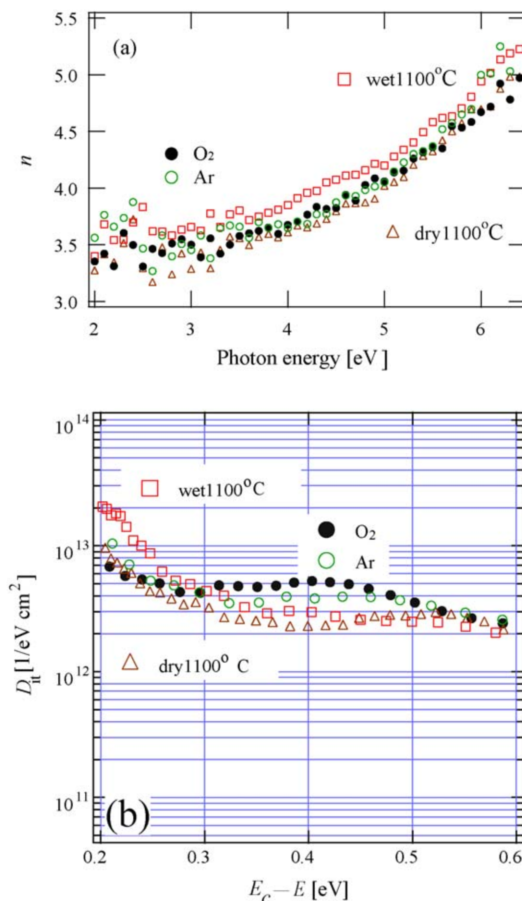

Fig. 3. Change in n (a) and D_{it} (b) on Si-face due to heating after wet oxidation.

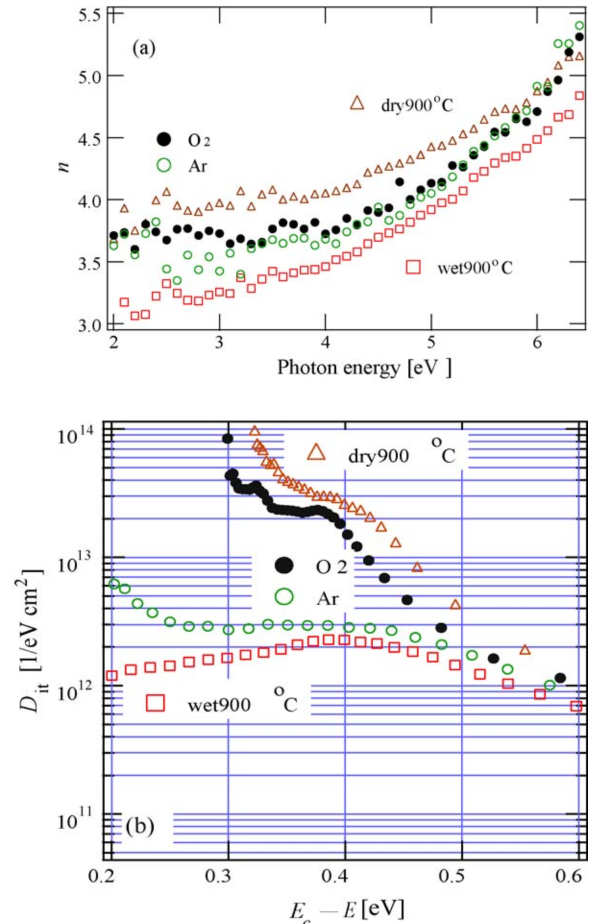

Fig. 4. Change in n (a) and D_{it} (b) on C-face due to heating after wet oxidation.

quite large compared with those for Si-face, as shown in Fig. 2(b), which suggests that there are much more dangling bonds in the interface layer on C-face compared with that on Si-face. It is also considered that the lower D_{it} at lower temperature oxidation in the case of wet oxidation of C-face means that the hydrogen termination are enhanced by lowering the oxidation temperature. As mentioned in Section 3.1, the interface state density for Si-face is higher for wet oxidation than for dry oxidation, which is contrary to C-face. We speculate that the interface layer for Si face contains somewhat defects that cannot be terminated by hydrogen, such as interstitial. We are now investigating the structural difference of interface layer between Si- and C-face in the light of SiC oxidation mechanism [25,26]. The electron paramagnetic resonance (EPR) studies suggested that the dangling bond originates from not Si but C [27]. Putting together with the EPR studies, our results suggest that C hydride is resolved by heating at 600 °C, which leads to the interface characteristics closed to those for dry oxidation.

By comparing between the heating in Ar and O_2, for Si-face, the changes by heating in O_2 are almost the same with each other. While in the case for C-face, the changes by heating are much larger than that in Ar atmosphere. This can be attributed to the occurrence of oxidation even at very low temperatures around 600 °C for SiC C-face.

3.3. Si-face vs. C-face

In the case of dry oxidation, both n and D_{it} for C-face are larger than those for Si-face. However, there is a possibility that these differences between for Si- and C-faces are due to the difference in the oxidation rate, because the oxidation rate for C-face is around 10 times larger than that for Si-face. Thus, to make clear the origin of the differences, we have compared the Si- and C-face samples oxidized with same oxidation rate by lowering the oxidation temperature for C-face. According to the previous work [28,29], the oxidation rate for Si-face at 1100 °C is almost same as that for C-face at 850 °C. Fig. 5 shows n and D_{it} values for dry oxidation of C-face between 800 and 1100 °C as well as those of Si-face at 1100 °C. As show in the figure, n and D_{it} for C-face considerably reduce with lowering oxidation temperature and approach towards those for Si-face. These results reveal that the lowering in the oxidation rate is quite effective for reducing the interface states. However, the interface characteristics for C-face, oxidized at 850 °C does not agree with that for Si-face at 1100 °C. In addition, the values for C-face oxidized even at 800 °C, whose oxidation rate is lower than that for Si-face at 1100 °C, are still larger than those for Si-face. These results suggest that only the difference in the oxidation rate cannot account for the difference in the interface characteristics between Si- and C-face.

We fear that the change of oxidation temperature affects not only on the oxidation rate but also on the oxidation-reaction-process. Therefore, we have prepared the samples for C-face oxidized at same temperature but with low oxygen partial pressure, in order to avoid the influence from the change of oxidation temperature. C-faces of SiC were oxidized at 900 °C under the oxygen partial pressures between 0.4 and 0.6 atm. Fig. 6 shows n and D_{it} values for dry oxidation for C-face at reduced oxygen partial pressures as well as those for Si-face at 1100 °C and 1 atm. Similar to the tendency in the case of lowing oxidation temperature, the values of n and D_{it} reduce with decreasing pressure and approach towards those for Si-face, but do not reach those for Si-face. These results also suggest that the influence of the growth rate to the interface characteristics is very large, but only the difference in oxidation rate cannot account for the difference in the interface characteristics between Si- and C-face. In conclusion, the oxidation rate is most crucial factor for C-face that determines the interface characteristics, however, not only the oxidation rate determines the interface characteristics, but also the difference in the interface structures existing intrinsically between

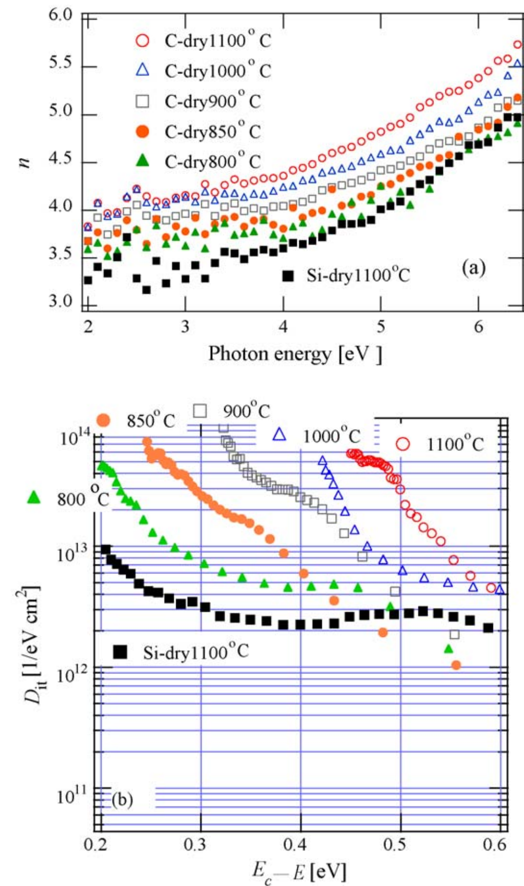

Fig. 5. n (a) and D_{it} (b) on C-face at oxidation temperatures between 800 and 1100 °C.

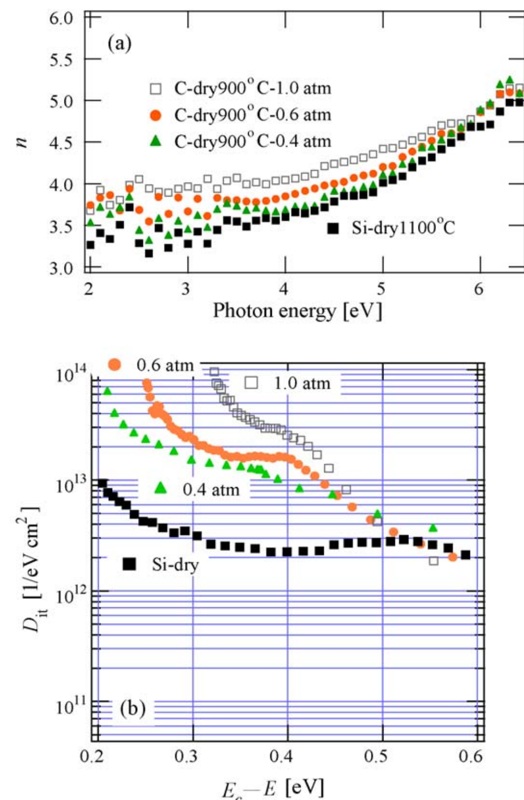

Fig. 6. n (a) and D_{it} (b) on C-face at oxygen-partial-pressures between 0.4 and 1.0 atm.

Si- and C-face may affect the difference in the interface properties. According to the recent work performed by Ray et al. [30], MOS characteristic is independent of oxidation pressures in the range of equal or lower than 1 atm, which is discrepant to our results. This discrepancy is considered to be due to, for example, the difference in oxide thickness of the samples measured.

4. Summary

Optical and electrical evaluation based on the wide-spectral-range spectroscopic ellipsometry and C–V measurements have been carried out for SiC–oxide interfaces formed on Si- and C-faces of 4H-SiC by dry and wet oxidation at various oxidation temperatures. It is found that refractive indices of the interface layers correlate well with the interface state density all in the case of oxidation processes, suggesting that the presence of the interfaces having large refractive indices is related to the generation of interface states. It is also found that the dispersion relation of extinction coefficient of the interface layers in the entire energy region is similar to that of SiC, suggesting the presence of materials having a little different band structures from that of SiC, like strained SiC layers. From the studies in the effect of post-annealing in Ar atmosphere on the refractive indices of interface and D_{it} for the samples fabricated by wet oxidation, it is concluded that hydrogen related species terminate the dangling bonds of Si or C at the interfaces for wet oxidation, which may bring about the low D_{it} for wet oxidation, comparing with that for dry oxidation. The studies of lowering of the oxidation rate of C-face oxidation down to those for Si-faces suggest that the low oxidation rate brings about the low refractive index and interface state density, though only the difference in the oxidation rate cannot account for the difference in the interface characteristics fully between Si- and C-faces.

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