Vacuum-Ultra-Violet and Ozone Induced Oxidation of Silicon and Silicon-Germanium

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UV radiation has been used to induce low temperature oxidation of crystalline silicon (c-Si) surfaces and silicon-germanium (Si–Ge) strained layers on Si. For c-Si at 550°C, growth rate enhancements of up to 50 times the dry thermal oxidation rate are achieved, equivalent to what is normally only obtainable thermally at 900°C. When irradiated under identical conditions by the UV light, SiGe layers were found to oxidise two times faster than c-Si. Prolonged oxidation of SiGe produced a dielectric structure consisting of a pure SiO_2 film on top of a SiO_2 layer containing several percent of trapped Ge, with the remaining SiGe layer snow-ploughed at the Si interface but retaining its epitaxial structure. Simple modelling qualitatively explains the observed features of the silicon oxidation and has led to further studies of ozone oxidation of c-Si.

1. Introduction

The relentless progress towards ultra large scale integrated (ULSI) technology has produced immense interest in developing low temperature processing techniques that reduce the overall thermal budgets associated with silicon microfabrication. Where oxidation is concerned, the use of UV radiation has been explored as a means of providing a more reactive environment thereby enabling lower temperatures to be used.[1-3] In this respect, light which can induce a large electron-hole density within the crystalline Si (c-Si), or can directly dissociate the O_2 bond (5.1 eV) and liberate atomic species and lead to ozone (O_3) formation, is of particular interest. We have previously shown that radiation from a low pressure mercury (Hg) lamp can significantly enhance the low temperature oxidation of Si.[4,5] This work has been extended to the growth of dielectrics on silicon-germanium (SiGe) strained layers[6] which are of particular interest because of their potential optoelectronic applications,[7,8] where there is a stringent need to avoid thermal relaxation of the strained layers during conventional thermal oxidation. Moreover, because the presence of Ge atoms at the Si–SiO_2 interface is known to modify the kinetics of Si oxidation,[9,10] there is also a fundamental interest associated with such studies.

In this paper, we present data concerning the UV oxidation of single crystal Si (c-Si). Films up to 10.8 nm in thickness are produced at 550°C in 6 hours. A simple model which empirically can explain the observed behaviour, is also developed. The oxidation characteristics of $Si_{0.8}Ge_{0.2}$ strained layers during UV irradiation times of up to 9 hours are also presented. In particular, the composition and microstructure of the layers formed are described. Initial results of rapid ozone (O_3) oxidation of silicon, without UV radiation, are also shown.

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2. Experimental Details

The UV oxidation system used consisted of a UV lamp contained within a stainless steel chamber through which high purity VLSI grade oxygen (O_2) was pumped. The photon source was a 20 × 20 cm water-cooled low pressure Hg lamp which emitted strongly in the UV at 254 nm ($h\nu = 4.98$ eV), with a weaker output in the vacuum-ultra-violet at 185 nm ($h\nu = 6.7$ eV). In this paper, for simplicity, we use the term UV to describe the collective output from the lamp. The substrates used were either p-type (100) oriented c-Si with a resistivity of 2–10 Ω cm, or MBE grown $Si_{0.8}Ge_{0.2}$ strained layers, measured by Rutherford backscattering (RBS) to be 15–16 nm thick, on 1000 Ω cm p-type (100) c-Si substrates. These were placed on a heater at a distance of some 4.5 cm below the lamp and could be resistively heated to 550°C. Prior to UV exposure in 1 atmosphere of O_2, the samples were cleaned in HF (48%) and rinsed in deionised water.

After UV exposure for various times, the thickness of each oxide grown was measured by optical ellipsometry, while RBS and Auger electron spectroscopy (AES) were employed to determine the distribution of Si, Ge and O species in the $Si_{0.8}Ge_{0.2}$ strained layers as a function of depth from the surface. RBS (random and channelling) measurements were performed using 2 MeV He⁺ ions and a scattering angle of 170° and the spectra were interpreted using the RUMP simulation program.[11] Cross-sectional transmission electron microscopy (X-TEM) was carried out using a Philips CM30 microscope at an accelerating voltage of 250 kV.

3. Results

3.1 UV oxidation of c-Si

Figure 1 shows the oxide thicknesses grown on c-Si as a function of exposure time for substrate temperatures from 400°C to 550°C. In the present conditions, oxidation below 400°C was particularly slow and therefore not studied. The data points include the estimated ± 0.5 nm error associated with measurement and ex-
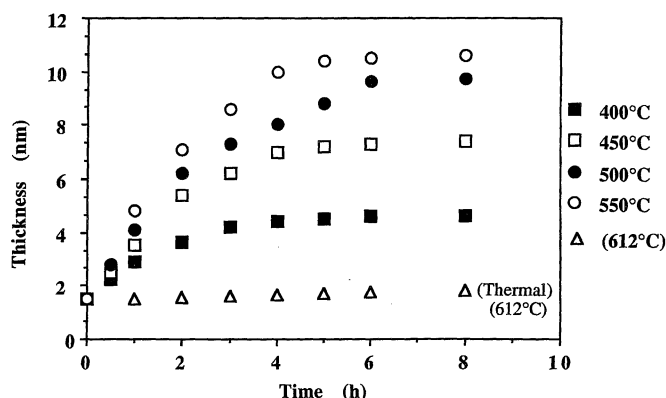
Fig. 1.   Oxide thickness grown on c-Si as a function of UV exposure time at different temperatures.

Fig. 2.   High resolution X-TEM of the SiO_2/Si interface of a sample oxidised for 4 hours at 500°C. Poly-Si was deposited on top of the grown SiO_2 in order to permit an accurate measurement of layer thickness.

perimental reproducibility. As can be seen from the figure, an initially rapid linear growth regime is followed by a transition region and finally saturation at each temperature used. (We shall refer hereafter to the characteristic time after which oxidation becomes negligible as the "saturation time"). It is already well established that conventional thermal oxidation of Si at a temperature of 612°C produces less than 0.5 nm of growth within the timescale used here. The growth rates induced by the UV over the first hour at 550°C (≈ 0.05 nm/min) are more than 50 times faster than those obtained thermally at 612°C.

Several factors may contribute to the observed reaction enhancement. Some of the UV light (particularly that whose photon energy is greater than 5.1 eV) will be absorbed by the O_2 in the gas phase, leading to photodissociation, the release of oxygen atoms, and ultimately the formation of ozone, which is known to be a stronger oxidising agent than its allotropic cousin, molecular oxygen. Any radiation reaching the Si surface will also induce photoelectron ejection from the Si into the SiO_2. The relative importance of these mechanisms is difficult to assess because of the lack of knowledge concerning even the conventional thermal reaction of O_2 with Si. However, several data were recorded for Si oxidation under conditions where the substrate was not *directly* illuminated by the UV lamp yet still in the presence of the photo-generated ozone. Whilst geometric factors discourage quantitative analysis, it was found that in all cases the growth rate in the shadow area decreased yet remained well above the thermal growth rate. A tentative model to explain these enhancement and saturation features is developed in the Theory section of this paper.

Fourier transform infrared spectroscopy (FTIR) measurements of the oxides grown indicated, from the position of the Si–O stretching vibration at 1075 cm^{-1} and the full width at half maximum) (FWHM) value of 74 cm^{-1}, that the layers were essentially identical to those conventionally prepared by thermal methods. Interface roughness, a parameter which can affect both the stoichiometry of the first oxide layers and thickness measurements, was studied using cross-sectional high resolution TEM. An X-TEM image of the interface
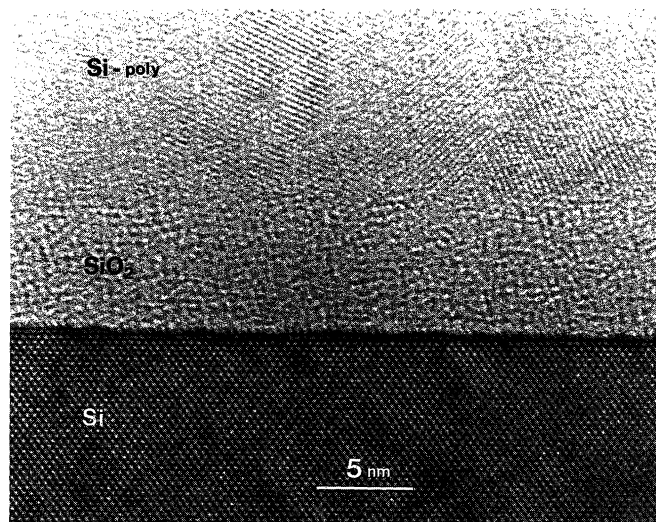
region for an oxide grown at 500°C for 4 hours is presented in Fig. 2. The average roughness was found to be of the order of 1 nm, a value corresponding to approximately 3 layers of SiO_2. Such roughness is not unusual for Si–SiO_2 interfaces on oxidised samples prepared using even the classical RCA cleaning method[12,13] and can in fact limit the accuracy of any thickness measurements performed on oxidised Si samples.

3.2   SiGe oxidation

The thickness of the oxide layers grown on $Si_{0.8}Ge_{0.2}$ as a function of the UV exposure time is presented in Fig. 3 together with the data for c-Si at 550°C. The most immediately striking feature of the results is the further enhanced oxidation rate of SiGe over that of c-Si (roughly a factor of two) under identical irradiation conditions. Although such an enhancement of the Si oxidation rate in the presence of Ge atoms has been observed for wet oxidation conditions,[14,15] these present
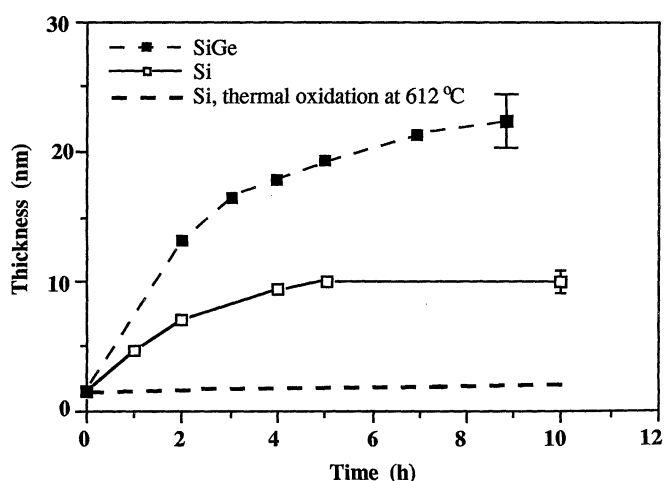

Fig. 3.   The thickness of SiO_2 films on Si and SiGe strained layers as a function of irradiation time at 550°C.

observations are the first yet reported that show such an effect during *dry* conditions. Furthermore, it can also be seen that while the oxidation of c-Si appears to be a self-limited process, for $Si_{0.8}Ge_{0.2}$ oxidation there is no such reaction limitation, although a slight decrease in the reaction rate for longer oxidation times is apparent.

RBS measurements confirmed the continuous growth of the oxide in the $Si_{0.8}Ge_{0.2}$ for longer irradiation times. In Fig. 4, where the RBS spectra of the SiGe layers irradiated for 6 and 9 hours are superimposed, an increase in the size of the integrated oxygen peak, which is proportional to the thickness of the oxide layer, can be seen. It is also worth noting from these spectra, that the leading edge of the Ge profiles does not change after the extra 3 hour oxidation. This strongly implies that the Ge atoms in the SiO_2 layer are completely immobile.

The structure of the oxidised samples, as revealed by simulations of the RBS spectra and confirmed by the AES measurements, consists of three discrete regions. The outermost layer of 4–5 nm thickness is always pure SiO_2. This covers a region of SiO_2 containing between 5% and 7% of trapped Ge. The remaining unoxidised SiGe lies beneath these SiO_2 layers. Channelled RBS spectra of the irradiated samples revealed that the remaining SiGe layer always retained epitaxial order.

Several general conclusions can be drawn from these observations. Firstly, we have found the first instance where SiGe oxidation proceeds at a higher rate than that of c-Si in identical dry conditions. Secondly, the UV induced SiGe oxidation is not a self-limited process, at least up to oxidation times two times longer than the saturation time found in the case of UV oxidation of pure Si. Thirdly, the structure of the grown layers is quite different to that found for thermal wet or dry oxidised SiGe strained layers.[8,14-16]
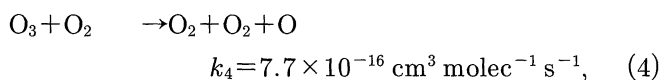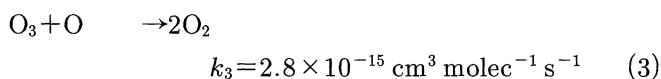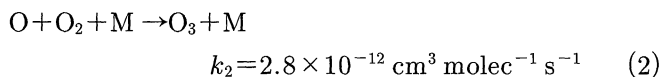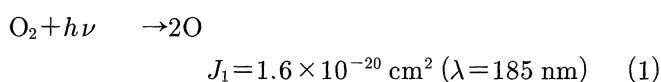
Several questions are also raised by these results. It remains intriguing as to why so few Ge atoms are trapped inside the growing SiO_2 layer when thermodynamic considerations and other experimental data[16-18] suggest that at the relatively low temperature

used, almost all the Ge should have been incorporated into the growing oxide as GeO_2. From the RBS simulation of our oxidised samples an interdiffusion coefficient of about 10^{-18} cm²/s is estimated. When extrapolating the diffusion coefficients measured in the $850°–1100°C$ temperature range[18-20] to our oxidation temperature of $550°C$, however, one obtains a maximum possible diffusion coefficient of the order of $10^{-20}–10^{-21}$ cm²/s. It is thus very clear that not only during the usual thermal oxidation[21] but also now during UV assisted oxidation, Ge and Si atoms attain much larger than expected diffusion coefficients, even taking into consideration the dependence of the coefficients on Ge concentration and strain.

4. Theory

The mechanisms operating during SiGe oxidation are evidently very complicated. It is also widely recognised that dry thermal oxidation of c-Si is not yet fully understood for layers thinner than 20 nm. With this in mind, we tentatively propose a simple empirical model that is capable of explaining some of the salient features associated with UV oxidation of c-Si only. Of course, it is recognised that agreement between the model and the experimental results does not prove *per se* the dominant mechanism controlling the reaction. Rather, like the case for pure thermal oxidation of c-Si, it could easily be one of many that can provide qualitative agreement with the data. More experimental evidence remains to be accumulated before a reliable picture can be formulated for the UV oxidation process. The requirement is even more so for the case of SiGe oxidation.

In framing a simple initial picture of the process, we shall assume that oxidation proceeds when the relevant oxidant reaches the Si–SiO_2 interface and reacts with any available Si atoms. For UV radiation (where $\lambda < 240$ nm) passing through O_2 it is well established that the following reactions will take place in the gas phase.[22,23]

$$O_2 + h\nu \quad \rightarrow 2O$$
$$J_1 = 1.6 \times 10^{-20} \text{ cm}^2 \ (\lambda = 185 \text{ nm}) \quad (1)$$

$$O + O_2 + M \rightarrow O_3 + M$$
$$k_2 = 2.8 \times 10^{-12} \text{ cm}^3 \text{ molec}^{-1} \text{ s}^{-1} \quad (2)$$

$$O_3 + O \quad \rightarrow 2O_2$$
$$k_3 = 2.8 \times 10^{-15} \text{ cm}^3 \text{ molec}^{-1} \text{ s}^{-1} \quad (3)$$

$$O_3 + O_2 \quad \rightarrow O_2 + O_2 + O$$
$$k_4 = 7.7 \times 10^{-16} \text{ cm}^3 \text{ molec}^{-1} \text{ s}^{-1}, \quad (4)$$

where J_x and k_x are reaction rate constants and M is a third body. Thus, not only O_2, but also O and O_3 impinge upon the SiO_2 surface. It must be emphasized that the numbers given for the reaction rates k_2, k_3 and k_4 are only applicable at room temperature and whilst values exist for temperatures up to 100°C there is disagreement over values for higher temperatures.[23] Since radiation at $\lambda = 254$ nm is also emitted by the lamp, the following reaction also occurs:
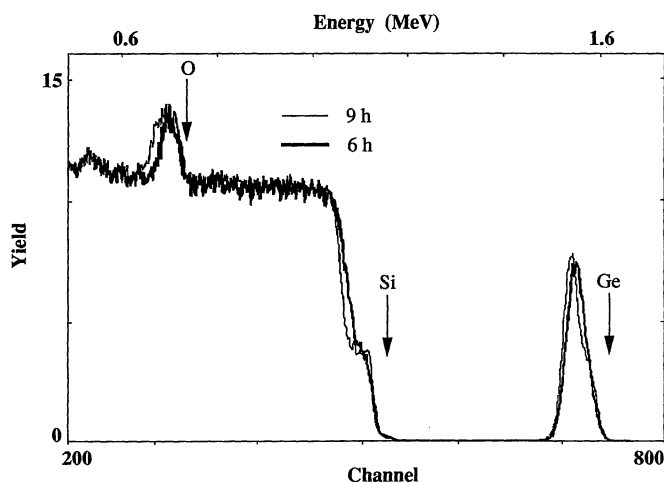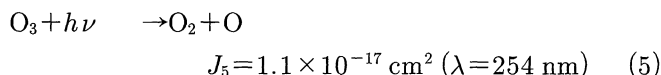

Fig. 4. RBS spectra of the $Si_{0.8}Ge_{0.2}$ samples oxidised for 6 and 9 hours.

$$O_3 + h\nu \rightarrow O_2 + O$$

$$J_5 = 1.1 \times 10^{-17} \text{ cm}^2 \ (\lambda = 254 \text{ nm}) \quad (5)$$

Therefore during UV exposure, the SiO_2 layer is supplied with molecular and atomic oxygen as well as ozone from the surrounding gas. As it is well known that O_3 decomposes extremely rapidly at temperatures above 100°C we proceed by assuming that the role of ozone is predominantly to act as a supplier of O and O_2 species to the oxide.

Clearly molecular oxygen *per se* cannot induce the rapid reaction rates obtained in our experiments. One must therefore consider which other species might be formed that would be capable of driving such an oxidation reaction. Cabrera and Mott have previously discussed the influence of ionic species during oxidation of many metals[24] as have Deal and Grove to explain the initial rapid thermally growth of SiO_2 at high temperatures in dry oxygen.[25] The latter suggestion has by no means been universally accepted. However, the possibility exists for both O^- and O_2^- to form by interacting with any electrons thermally or photonically ejected from the Si into the oxide. It is reasonable to expect that during UV irradiation the electron population in the oxide will be higher than the usual thermal equilibrium concentration. For example, it has been shown by Goodman[26] and Williams[27] that electrons can be photo-injected into the SiO_2 conduction band from the Si valence band provided $E_{\text{photon}} > 3.15$ eV. This process has a quantum efficiency of $(5\pm 3).10^{-5}$ electrons/photon which is comparable to that measured for vacuum photo-emission at the same photon energy.[27] Therefore, as a direct consequence of the UV radiation, O atoms and O^- and O_2^- ions can be formed. These species will exist in addition to the usual species present during thermal oxidation.

Since one would expect their diffusion to be much more rapid than the larger molecular species, enhanced oxidation could conceivably occur simply because of the presence of the O atoms. So long as the UV radiation was present at and above the sample surface, then these species would be continually supplied for the reaction, and one would expect a linear-parabolic type characteristic for the reaction kinetics, with no obvious "saturation" effect.

If ions played a significant role, then their mechanism of formation, i.e. the interaction of the injected electrons with the neutrals, both O and O_2, would be important, as would be the influence of the well known space-charge region. Such species would be created most easily during the initial stages of oxide growth when the electrons ejected from the Si could readily interact with the neutral oxygen. However, as oxidation proceeded, this would become progressively more difficult as the build-up of space-charge restricted charge movement. In the initial stages of the reaction, therefore, the drift of the ionic species towards the Si–SiO_2 interface will be much more rapid than the thermal diffusion of their neutral counterparts. Progressively, however, the ionic contribution to the reaction will

slow down and ultimately stop when the field totally restricts the photo-generated current and ceases to ionise sufficient species. For this reason, in the model formulated here we invoke the domination of ionic species as the main oxidants in the UV oxidation enhancement process.

We shall now consider the growth rate within the linear regime of the reaction. The form of the charge distribution and associated field in the oxide is known to be exceedingly complicated and subject to much debate. As a first step in our analysis, therefore, we shall assume a simple form of electric field, E (*e.g.* approximately constant) across the oxide layer of thickness, x. More detailed form are currently being explored. The linear growth rate of the oxide, dx/dt, may be therefore written as:

$$(dx/dt) = C.E = A \quad (6)$$

where A and C are constants. The solution gives growth behaviour of the form:

$$x = x_0 + At \quad (7)$$

where x_0 is the native oxide thickness. Such a linear behaviour usually suggests that the growth of SiO_2 is interface reaction limited rather than diffusion limited. If one assumes that A exhibits Arrhenius behaviour, a plot of $\ln (A)$ against $1/T$ for our data gives an activation energy, E_a, of 0.27 eV.

During the growth of the oxide within the linear region, the build up of space-charge in the oxide with time will gradually reduce the photo-injected electron density reaching the SiO_2/gas interface and decrease the rate of formation of ionic oxidant. This will cause the linearity of the reaction rate to be lost, and eventually with time, will hinder any further oxidation from taking place by this mechanism. The form of this blocking effect cannot be readily formulated owing to a lack of knowledge concerning the precise charge distribution and subsequent field profile within the dielectric and is currently subject to further research. Nevertheless, with this approach, the basis for broad agreement with the data would seem to be a realistic possibility.

5. Discussion

UV induced oxidation produces some interesting and potentially useful characteristics. Not only are the reactions significantly faster than the thermal equivalents at the same temperature, but for Si it is self-limiting to a predetermined thickness governed primarily by the substrate temperature. Our simple model proposes the dominating effect of ionic species formed in the oxide by the electrons ejected from the Si. The effective activation energy of 0.27 eV is quite different from the E_a value of >1.5 eV for thermal oxidation of Si using oniy O_2.[25] In fact it is reminiscent of that usually found for plasma oxidation of Si, where the reported values for O^- field enhanced diffusion through SiO_2 are $0.14 < E_a < 0.7$ eV.[28-30] The variation in these values is due to the precise nature of the space charge in each experimental set-up. Joseph *et al.*[31] have recently used
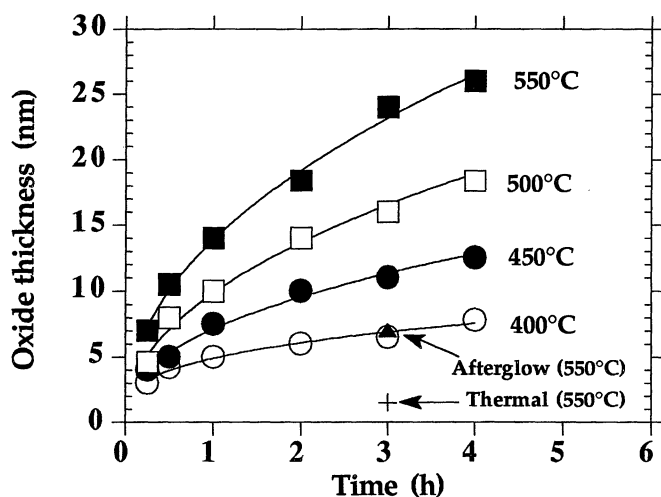
Fig. 5.   Thickness of oxides grown using remotely produced ozone, as a function of exposure time, for a range of processing temperatures.

oxygen radicals from a plasma source to oxidise Si at 450°C and found an activation energy of 0.18 eV. Their suggestion that O⁻ is the *only* oxidant in the process makes it tempting to propose a similar identity here.

In order to isolate the influence of the photo-injected current on the reaction more clearly than the earlier shadow experiments, an alternative approach was devised whereby *only* ozone (i.e. no UV) was present during the reaction. In this way, the specific role of ozone could be more readily isolated. These studies were performed using O_3 produced remotely by the barrier discharge method.[32] The ozone concentration generated by this technique was not only some 6–8 times higher (determined by actinometric analyses) than that produced by the Hg lamp, but also was delivered much closer to the c-Si surface. Preliminary results have already indicated a strong enhancement over thermal oxidation where O_3 enriched oxygen was used for a fixed time at several temperatures.[33] Figure 5 shows the oxide thicknesses produced for a range of temperatures under these conditions. Not only is the growth rate significantly faster (some 0.35 nm/min) than previously achieved, but no saturation is apparent on the timescale used. It is important to emphasise again here that no UV radiation is present in the oxidation chamber. Hence, we can conclusively state that O_3 plays an extremely crucial role in the reaction enhancement, as a supplier of O atoms to the oxide surface. This also suggests that the UV radiation is not only influential in initiating a rapid oxidation reaction by producing ozone, but also in limiting the process. Further work is in progress to explore the $Si + O_3$ reaction more fully.

6.   Conclusion

We have shown, for the case of c-Si, that UV radiation is capable of dramatically enhancing the oxidation reaction, by up to a factor of 50 at low temperatures.

Both photo-generation of ozone in the gas phase and photo-injection of carriers into the oxide are central to the enhancement process. The role of the ozone is to supply oxygen atoms to the oxide surface. The UV radiation provides a further mechanism for enhancement, but eventually causes self-limitation of the reaction. An effective activation energy of 0.27 eV is indicative of an O⁻-controlled reaction as found in several plasma-induced processes. Further experimentation has shown that much larger ozone concentrations (without UV light) can also induce rapid oxidation of Si, where contributions due to ionic species are effectively eliminated.

The microstructure of dielectric layers formed by low temperature UV assisted dry oxidation of SiGe strained layers has been studied. The initial oxidation rate is higher than that of pure Si under identical conditions and does not self-limit up to twice the saturation time found for c-Si. A 3-layered structure is found after prolonged oxidation of SiGe consisting of pure SiO_2 on top of Ge-rich SiO_2 under which lies the unoxidised SiGe. The diffusion coefficients of Si and Ge extrapolated to the low temperatures used in our investigations cannot account for this Ge enrichment behaviour.

These experiments provide exciting evidence for the possibility of realistic application of UV radiation and/or ozone for producing rapid low temperature oxidation of silicon and Si alloys. Clearly, the techniques can be more widely applied to other materials.

Acknowledgements

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