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Kinetics of pulsed ultraviolet laser induced oxidation of *c*-Ge: The role of optical coupling and material losses

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Ultraviolet pulsed laser induced oxidation kinetics of crystalline germanium (*c*-Ge) is determined from real-time optical measurements in conjunction with absolute measurements of the oxygen incorporation performed by nuclear reaction analysis. Although the oxidation process can be triggered at laser fluences initially below the melting threshold of *c*-Ge, it is strongly activated when surface melting occurs and therefore the fast oxidation process observed is mainly a thermally activated process. Because an optical coupling between the oxide layer and the *c*-Ge underneath, the growth kinetics is complex and leads to nonconstant rates. The oxygen incorporation reaches a saturation value which depends both on the laser fluence and the oxygen pressure. The results show that the oxygen incorporation is limited by an overlapped laser-induced material removal process rather than by the diffusion length of oxygen species.

I. INTRODUCTION

There is an increasing interest in synthesize Si-based materials for photonic applications with new optical and electric properties. Because $\text{Si}/\text{Si}_{1-x}\text{Ge}_x$ and Si/Ge heterostructures have received much attention as potential candidates for the new generation of semiconductor devices, there is a renewed interest in Ge. Recent work involves the growth of epitaxial Ge films on Si^1 or laser-induced mixing in $\alpha\text{-Ge}/\text{Si}$ systems as a means to produce epitaxial SiGe films on Si^2 . In addition, semiconductor oxide films are needed as passivation layers and/or active insulating materials in metal-oxide-semiconductor structures. Consequently, there is also a revived interest in obtaining Ge oxide films on Ge and in characterizing the oxide properties.³ Low-temperature processing is desirable in order to prevent substrate degradation in the production of semiconductor-oxide overlayers. Many processes have been proposed for silicon which include chemical vapor deposition (CVD),⁴ laser-induced CVD,⁵ and ion⁶ and laser irradiation in an oxygen environment.

Among other advantages, laser processing offers the interesting possibility of local oxidation (to micron-sized areas). When pulsed laser irradiation is used, high temperatures are achieved at the near-surface region during short periods of time, which ensures rapid and localized processing. High growth rates have been obtained when irradiating near⁷ or above the melting threshold.⁸ It has been much discussion on the existence of a "photonic" or "non-thermal" component overlapped to the thermal process⁹⁻¹¹ during laser-induced oxidation. Several works have shown the existence of an enhancement of the oxidation process by irradiating with visible^{12,13} and ultraviolet (UV)^{14,15} light.

In an earlier work¹⁶ we studied the evolution of the optical properties of *c*-Ge upon multipulse UV laser irradiation in an oxygen atmosphere with an energy density above the melting threshold. The results were consistent

with the growth of an oxide layer at the surface. It was also evidenced that the optical coupling between the oxide layer and the bulk material underneath was responsible for the enhancement of a nonconstant growth rate. In this work, we will combine real-time optical measurements with nuclear reaction analysis (NRA) measurements. Whereas the former provides information on whether the process occurs in liquid or solid phase in addition to record the reflectivity changes as oxidation proceeds, the latter provides the absolute amount of oxygen incorporated to the surface. The aim of this work is to determine the oxidation kinetics of *c*-Ge under UV laser irradiation. In order to discuss the role of thermal and nonthermal contributions to the oxidation process, energy densities both below and above the melting threshold will be used.

II. EXPERIMENT

The samples used are $\langle 111 \rangle$ single-crystal Ge wafers (*c*-Ge) cleaned with organic solvents. No attempt to remove the native oxide is made in order to have a chemically inactive layer on the Ge surface. This guarantees the same initial conditions for all the irradiated areas.

An ArF excimer laser ($\lambda = 193$ nm, $\tau = 12$ ns) is used to perform the irradiations in multipulse regime at 0.1 Hz. Experiments are carried out both in vacuum (4×10^{-5} mbar) and in a controlled oxygen atmosphere (0.05–1.2 bar). The beam is focused by means of a 104-mm cylindrical lens and the sample is located after the focus position at normal incidence. The beam energy at the sample site is homogeneous within 5% and the energy densities used in the present work were in the 0.18–0.40 J/cm² range. Real-time reflectivity measurements are performed by means of a pulsed He-Ne probe laser beam ($\lambda = 633$ nm, $\tau = 1$ μ s) synchronized with the irradiation pulse. Further details of the experimental setup can be found elsewhere.¹⁶

The irradiated areas are analyzed by means of NRA using the $^{16}\text{O}(d,p)^{17}\text{O}^*$ reaction with a detection angle of

FIG. 1. Reflectivity transients recorded when irradiating *c*-Ge in vacuum (4×10^{-5} mbar) with a pulse energy density of 0.35 J/cm^2 after 1, 10, 30, and 100 laser pulses. The time profile of the laser pulse is also shown as a dashed line.

150° and a deuteron beam energy of 850 keV. The absolute number of oxygen atoms is obtained with an accuracy of 3% by using a standard oxygen reference target.¹⁷ A 3-nm GeO_2 native oxide thickness was measured in nontreated areas by assuming a density of 3.6 g/cm^3 .¹⁸

III. RESULTS

In order to understand the role of an oxygen environment in the evolution of the reflectivity as the sample is laser irradiated, it is convenient to characterize first its behavior when irradiating in vacuum and therefore reactive processes with the foreign atoms or molecules are minimized. The evolution of the transient reflectivity as the number of laser pulses is increased when irradiating *c*-Ge in vacuum (4×10^{-5} mbar) is shown in Fig. 1. An average laser pulse energy density of 0.35 J/cm^2 is used which is enough to induce surface melting under the first pulse as can be deduced from the first transient shown in Fig. 1. The shape of the first transients (1–10) corresponds to those typically observed under homogeneous melting^{19,20} with a sharp reflectivity increase (50% increase over the room temperature value²⁰) due to the metallic character of the melted material. The melt duration, as determined from the reflectivity transient width, decreases continuously as the number of pulses increases. After 30 laser pulses, the shape of the transients is consistent with an inhomogeneous melting process¹⁹ in which the solid and a small amount of liquid coexist at the sample surface. No significant changes are observed in the melt duration when the number of pulses is further increased. Figure 2 shows the evolution of the melt duration as a function of the number of pulses obtained at two different energy densities (0.35 and 0.40 J/cm^2). The highest energy density leads to the highest melt duration. It is worth noticing that the saturation melt duration values for a fixed pulse energy density, are in excellent agreement with those already reported for *c*-Ge when the native oxide layer was chemically removed prior to irradiation.²⁰ In addition, no measurable

FIG. 2. Melt duration as a function of the number of laser pulses when irradiating *c*-Ge in vacuum (4×10^{-5} mbar) with a pulse energy density of 0.35 J/cm^2 (O-O-O) and 0.40 J/cm^2 (Δ-Δ-Δ).

changes are observed in the reflectivity of the sample after solidification, which rules out the existence of any surface optical damage.

Two energy density regimes were studied when irradiating *c*-Ge in an oxygen atmosphere. The first one involved an energy density of 0.18 J/cm^2 which induced a first reflectivity transient typical of semiconductor surface heating since the maximum transient reflectivity value is lower than that of solid material at the melting temperature.²⁰ Figure 3 shows the evolution of reflectivity transients as the number of pulses increases when irradiating the *c*-Ge surface in an oxygen pressure of 1.2 bar and an average energy density of 0.18 J/cm^2 . The shape of the transients evolves from that expected in a semiconductor upon simple heating (pulse number 1) to that induced under inhomogeneous melting (pulse 15) and homogeneous melting (pulse 30).²⁰ The melt duration reaches its maximum value after 35 pulses. Further pulses lead to a change in the transient reflectivity shape together with a reduction in the melt duration.

FIG. 3. Reflectivity transients recorded when irradiating *c*-Ge in a 1.2-bar oxygen pressure with a pulse energy density of 0.18 J/cm^2 and after 1, 15, 35, 50, and 100 laser pulses. The time profile of the laser is also shown as a dashed line.

FIG. 4. Evolution of both, oxygen content (■-■-■) as measured by NRA and reflectivity change (□-□-□) normalized to the initial sample reflectivity (R_0) as a function of the number of laser pulses. Irradiations are performed in a 1.2-bar oxygen pressure with a pulse energy density of 0.18 J/cm^2 .

Another important feature which is not clearly seen in Fig. 3 refers to the sample reflectivity. It decreases after each pulse as can be deduced by comparing the initial sample reflectivity (R_0) and the final reflectivity value (R_f) in each transient in a longer time scale. The change of the sample reflectivity defined as $(R_0 - R_f)/R_0$ in order to make it positive is plotted in Fig. 4 as the number of pulses is increased. No significant changes are observed within the first 30 pulses. It changes instead very rapidly between 30 and 70 pulses and tends then slowly to reach a saturation level.

Since no measurable changes of the material reflectivity was observed when irradiating in vacuum, these results strongly suggest that the reflectivity evolution described above is most likely related to the incorporation of oxygen to the sample surface. The oxygen content is determined by means of NRA as a function of the number of pulses. The measurements were performed at different areas of the same *c*-Ge sample which are irradiated with different number of pulses in the same experimental conditions used in Fig. 3. The results are also plotted in Fig. 4 and it can be seen that only a slight increase of the number of oxygen atoms respect to the native oxide is obtained in the 1–30 interval. Then, the oxygen content increases rapidly as the number of pulses is increased up to 70 pulses. Next laser pulses induce a slower incorporation rate of oxygen. The oxygen content measured after 100 pulses is approximately 20 times higher than that corresponding to the native oxide. It is clearly seen that the reflectivity changes follow the oxygen incorporation being the inflexion points of both curves at the same number of pulses. A similar strong correlation between the oxygen incorporation and the reflectivity change is found when irradiations are performed as a function of the oxygen pressure. Figure 5 shows the results obtained from different areas of the same wafer irradiated with 100 pulses of 0.18 J/cm^2 at increasing oxygen pressures. The oxygen content becomes significant for

FIG. 5. Oxygen content (■-■-■) as measured by NRA and reflectivity change (□-□-□) normalized to the initial sample reflectivity (R_0) after 100 pulses of 0.18 J/cm^2 , as a function of the oxygen pressure applied during irradiation. The oxygen content corresponding to the native oxide is also indicated.

oxygen pressures above 0.1 bar and increases slightly as the pressure is increased to 1.2 bar.

The oxidation rate is not constant as evidenced in Fig. 4. A rough estimation of its average value can be obtained by assuming that the oxide layer is GeO_2 and using its density¹⁸ to convert the vertical axis in Fig. 4 in a thickness scale. The slope of the curve provides an average growth rate of 1 nm/pulse which is extremely high when compared with that obtained under conventional solid phase thermal processing. Furthermore, the optical transients show that the sharp increase of both the reflectivity change and the oxidation content in Fig. 4 is coincident with the occurrence of homogeneous melting. Therefore the oxidation process occurs mainly within the liquid phase which explains the high rates observed. They are slightly higher than those obtained on Si under similar experimental conditions.²¹

The second energy density regime studied (0.35 J/cm^2) was enough to induce surface melting under the first pulse. The evolution of the reflectivity transients as the number of pulses is increased has been already shown in a previous work.¹⁶ It is very similar to that shown in Fig. 3, except that the melt durations involved are higher and the first transient corresponds already to a melting process. The reflectivity decrease as the number of pulses is increased when irradiating at two different oxygen pressures (0.1 and 1 bar) are shown in Fig. 6. Results obtained at these oxygen pressures when irradiating below the melting threshold (0.18 J/cm^2) are also included for comparison. It is seen that the oxygen incorporation rate as deduced from the reflectivity change is faster when irradiations are performed with an energy density which is initially above the melting threshold (0.35 J/cm^2). However, the incorporation of oxygen seems to saturate after a short number of pulses. The oxygen incorporation is instead “active” in the whole range of pulses when irradiations are performed at an energy density which is initially below the melting threshold (0.18 J/cm^2) as it can be deduced by the non-

FIG. 6. Reflectivity decrease normalized to the initial sample reflectivity (R_0) as a function of the number of laser pulses when irradiating *c*-Ge in 0.1 (○,●) and 1.0 (□,■) bar of oxygen. Results are obtained with a pulse energy density of 0.18 (○,□) and 0.35 (●,■) J/cm².

zero slope of the corresponding curves after 100 pulses. Moreover, the results obtained after 100 pulses in an oxygen pressure of 0.1 bar are also consistent with a higher oxygen content when irradiating with the lowest energy density.

IV. DISCUSSION

The evolution of the reflectivity transients when irradiating in vacuum and in an oxygen atmosphere is different evidencing the growth of an oxide on the surface of *c*-Ge when irradiating in the latter conditions. As it was demonstrated in Figs. 4 and 5, a strong correlation exists between the oxygen incorporation and the reflectivity changes which proves that optical measurements are a powerful and simple means to analyze the oxidation process. This approach was previously used to analyze the laser-induced oxidation of both metals²² and semiconductors²³ although no absolute measurements of oxygen incorporation were reported. Another novel approach of this work is to perform the measurements in real time which allows us to obtain direct information about the kinetics of the process. Our results show clearly that the incorporation of significant amounts of oxygen occurs only upon surface melting.

The most stable Ge oxide is GeO₂ which has a dielectric nature and is transparent in the visible. The optical properties of a GeO₂/*c*-Ge system were simulated using the optical parameters reported elsewhere^{24–26} as the thickness of the oxide layer increases. As the oxide layer thickness increases, the reflectivity at the probe beam wavelength [Fig. 7(a)] decreases in agreement with our experimental observations when irradiating Ge in an oxygen environment (Fig. 6). The optical absorption at the irradiation wavelength [Fig. 7(b)] shows that the optical coupling between the oxide layer and the *c*-Ge underneath leads to a strong increase of the absorption of energy for small oxide layer thicknesses. Higher peak temperatures and melt durations are then expected for a certain oxide layer thickness. When irradiating at constant energy density in an oxygen environment, melt duration should increase ini-

FIG. 7. Simulation of the reflectivity at the probe beam wavelength ($\lambda = 633$ nm) (a) and absorption at the irradiation wavelength ($\lambda = 193$ nm) (b) of *c*-Ge covered by a GeO₂ layer as the thickness of the oxide layer is increased. The plotted values are normalized to the initial reflectivity and absorption values of *c*-Ge.

tially as the number of pulses is increased (see Fig. 3) accordingly with the increase of absorption [Fig. 7(b)] as an oxide layer grows at the surface.

The opposite behavior was observed when irradiating in vacuum (Figs. 1 and 2). Melt duration decreases to the values reported for clean Ge surfaces.²⁰ The shortening of the melt duration observed is consistent with a decrease of the laser energy absorbed and therefore a decrease of the native oxide layer thickness. The evolution of the melt duration with the energy density (Fig. 2) supports further this conclusion since the higher the energy density the shorter number of pulses needed to reach saturation. The existence of material removal during UV laser irradiation of metals and semiconductors covered by their native oxides^{27,28} has been reported as a useful technique to produce clean crystal surfaces. Compositional changes and material losses have been also observed when irradiating semiconductors and oxides^{28–30} even at temperatures well below the melting threshold.^{31,32} This removal process can be also active when irradiating both in vacuum^{27,29} and in an oxygen environment,³³ being the process significantly activated as the energy density increases above the melting threshold.

In our experimental conditions, the amount of oxygen which is incorporated to the material can be then the result of two competing processes: oxygen incorporation from the ambient (“indiffusion” process) and matrix atomic losses from the oxide layer or material evaporation (“removal” process). These mechanisms will control the oxidation kinetics and its relative importance is changing dynamically as the number of pulses increases since the experimental

scenario (oxide thickness, system absorption at the irradiation wavelength, surface peak temperature...) is also changing. As the oxide layer thickness increases a higher fraction of the laser energy is deposited on the oxide which will activate the oxide removal process. For a critical oxide thickness, this process may compensate the "indiffusion" process and the oxide layer thickness will tend to saturate. This interpretation is in agreement with the curves included in Fig. 6 where no further evolution of the sample reflectivity is measured after 20–30 laser pulses when irradiating at 0.35 J/cm². Nevertheless, the "indiffusion" process is still active after 100 pulses when irradiating at a lower-energy density (0.18 J/cm²) as evidenced by the nonzero slope of the reflectivity change curve. This result is explained because at this energy density, most of the process takes place at energy densities close to the melting threshold and therefore the contribution of the removal process (if it exists) is small. It can be then concluded that the process which limits the oxide growth is mainly the laser-induced removal process.

It is not straightforward to determine which are the microscopic mechanisms responsible of the oxygen "indiffusion" and oxide removal processes. Difficulties arise from the fact that the absorption of the laser energy is dynamically changing because of the optical coupling between the growing oxide layer and the bulk material. Thermal explanations based on the high diffusion coefficient of oxygen species in liquid semiconductors were given to explain the fast oxide growth rate achieved when the sample surface is melted.^{34,35} Within this model the material losses should be then related to thermal evaporation.³⁶ This explanation can account for the sharp increase of oxygen incorporation which we observe upon melting [Figs. 4 and 6], but does not explain the processes produced during the first laser pulses when irradiating at energy densities below the melting threshold. They are nevertheless responsible for the triggering of the oxidation process later on.

It has been reported that SiO₂ undergoes some microscopic changes prior to macroscopic changes even when irradiating at low energy density or under low-temperature annealing.^{29,37,38} These changes involve oxygen desorption and the formation of oxygen-vacancy related defects and voids. The reflectivity transients are consistent with solid-phase surface heating (first ten pulses, Fig. 3) when irradiating at energy densities below the melting threshold (0.18 J/cm²). Heating at temperatures below melting threshold will most likely involve the production of defects like those mentioned above. These defects will enhance the absorption of next laser pulses in addition to provide preferential sites for diffusion. High density of defects have been reported to give rise to diffusivities which may exceed those in the bulk by several orders of magnitude³⁹ in addition to decrease the melting temperature. Under these conditions, the incorporation of small amounts of oxygen together with the defect formation will contribute to an enhancement of the laser energy absorption after a few number of pulses and therefore, to laser melting.

Several authors^{10,11} have proposed the existence of a "photonic" or "nonthermal" activation of the oxidation

process. Oxidation occurs by diffusion of foreign oxygen atoms through vacancies to the oxide layer/bulk material interface where reaction takes place. Our results cannot rule out a possible contribution of such "nonthermal" mechanisms to the material transformations occurring during the first pulses when irradiating at low-energy density. Nevertheless, the comparative analysis of optical transients and oxygen incorporation amounts shows clearly that the oxygen content increases significantly only upon surface melting. It allows us to conclude that the oxidation of Ge observed under our experimental conditions occurs primarily via a thermal process.

V. CONCLUSIONS

Oxidation of Ge has been induced by multipulse UV laser irradiation of *c*-Ge in an oxygen environment with laser fluences both below and above the melting threshold. It is demonstrated that transient reflectivity features are directly related to the oxygen incorporation and therefore real-time optical measurements are a suitable means to follow dynamically and control the oxidation process. Since significant oxygen incorporation is only observed upon surface melting, we conclude that the oxidation process occurs primarily via a thermal process. The results also show that the amount of oxygen tends to saturate depending on the laser fluence and the oxygen pressure being the limiting mechanism for further oxygen content increase, the existence of an overlapped laser-induced removal process. The lower the energy density, the lower the contribution of the removal process and therefore thicker oxide layers can be obtained at lower-energy densities.

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