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D.J. Olego

H. Baumgart
Old Dominion University, hbaumgar@odu.edu

G.K. Celler

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D. J. Olego, H. Baumgart, and G. K. Celler

Strains in Si-on-SiO_2 structures formed by oxygen implantation: Raman scattering characterization

D. J. Olego and H. Baumgart[a)]
Philips Laboratories, North American Philips Corporation, Briarcliff Manor, New York 10510

G. K. Celler
AT&T Bell Laboratories, Murray Hill, New Jersey 07974

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Low-temperature Raman scattering measurements were carried out to characterize Si-on-SiO_2 structures formed by oxygen implantation and subsequent furnace or lamp annealing. The experiments were conducted with 413.1 nm laser light to probe only the thin Si layers at the top of the structures. The Raman spectra of the furnace-annealed samples are red shifted and broadened when compared with a virgin Si surface. The shifts and broadenings decrease with increasing annealing temperatures but they are still present in samples annealed above 1250 °C for 3 h. No shifts or broadenings affect the Raman peaks of the layers, which were lamp annealed at 1405 °C for half an hour. The red shifts indicate that the recrystallized Si layers are under tensile strains, whose origin is attributed to oxide precipitates. Quantitative estimates of the strains and associated stresses are obtained from the measured Raman shifts.

Great efforts are being devoted to the development of silicon-on-insulator (SOI) technologies for high-speed and radiation-hardened devices. Among the potential technologies, the process of creating a buried insulator by ion implantation into the Si wafer is attracting considerable attention because of the promising device results obtained so far. Typical structures consist of a Si substrate, a 200–400-nm-thick buried insulator under a 100–300-nm-thick Si layer at the top. The direct synthesis of a buried SiO_2 layer by the implantation process is known by the acronym SIMOX (for separation by implanted oxygen).[1] Implantation of N to form Si_3N_4 has also been studied.[2] In these methods a dose of ions, usually in excess of 1×10^{18} cm^{-2} and with energies in the range of 100–200 keV, is implanted deep into the Si wafer, which is kept during the implantation at a temperature above 450 °C to minimize damage in the bombarded Si layer. After implantation a thermal treatment above 1100 °C is required to improve the stoichiometry of the buried insulator, to sharpen the interfaces between the insulator and the Si below and above it, and to fully restore the crystallinity of the top Si layer. Conventional furnace annealing and a novel approach utilizing high-intensity lamps for radiant heating have been investigated.[3]

Characterization of the properties and quality of the top Si layer above the buried insulator becomes important, because the active devices are fabricated in it. Many diagnostic techniques, such as transmission electron microscopy (TEM), Rutherford backscattering (RBS), secondary ion mass spectrometry (SIMS), and x-ray photoelectron spectroscopy (XPS), have yielded important information about structural and chemical characteristics of the top Si films, and elucidated the relationship between the film properties and the thermal history of the sample.[3,4] Optical characterization techniques have been applied only recently to the study of SIMOX,[5,6] in spite of the fact that they are nondestructive and provide fast analysis. In this letter we present a Raman scattering characterization of the top Si layers of SIMOX samples. By a careful selection of the incoming laser wavelength, one can achieve the spatial resolution required to probe only the top layers of the structures. Under these conditions the Raman data can be related to the quality and intrinsic properties of the recrystallized layers. We have detected the presence of strains in the top Si films of SIMOX structures and followed them as a function of the annealing procedures. We present results for samples that have been annealed by the conventional furnace method and with the novel lamp furnace system.

The SIMOX samples were obtained by implanting 160 keV O^+ ions at a dose of 1.8×10^{18} cm^{-2} into (100) surfaces of Si wafers, which were maintained at 560 °C during the implantation. Before annealing, the implanted surfaces were coated for protection purposes with a 550-nm-thick cap of SiO_2 deposited by low-pressure chemical vapor deposition. Conventional furnace heat treatments were then performed in a N_2 atmosphere for 3 h at temperatures covering the span between 1100 and 1300 °C in increments of 50 °C. Structures were also annealed at 1405 °C for 30 min by using the special lamp arrangement described in Ref. 3. In this case the wafers were suspended between a bank of high-intensity lamps and a water cooled base with the back side of each sample held at the melting temperature of Si (1412 °C) by means of a natural feedback based on the optical properties of Si. The implanted side of the wafers was then about 7 °C cooler because of the temperature gradient imposed by one-sided radiant heating. After the annealing procedures, selected samples were checked by TEM in order to gain insight into the evolution of the thickness d_{Si} of the top Si layer and d_{SiO_2} of the buried oxide. It was found that d_{Si} is 184 nm for the as-implanted sample, 170 nm for the samples annealed at 1150 and 1200 °C, and 142.5 nm for the lamp-annealed surfaces. Conversely, d_{SiO_2} increases from 248 nm for the as-implanted sample to 300 nm for the sample annealed at 1150 °C, to

[a)] Present address: Philips Research Laboratories, P.O. Box 80000, 5600 JA, Eindhoven, The Netherlands.

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330 nm for the 1200 °C case, and finally to 365 nm for the lamp-annealed sample. The trends in thickness variations with annealing temperatures are in agreement with similar observations reported by others.[3,4] The structural properties of the Si layers also follow patterns established previously. The as-implanted Si surface is highly defective with a considerable amount of disorder, particularly in regions close to the interface with the buried oxide. The annealed layers are crystalline, and the dominant defects are oxide precipitates and threading dislocations. The number of precipitates decreases with increasing annealing temperature, and they are almost nonexistent in the lamp-annealed sample. The dislocation density is on the order of 3×10^{9} cm^{-2} for the 1150 °C annealed sample. This value decreases with annealing temperature, but reaches a saturation value of about 3×10^{8} cm^{-2} for samples annealed at or above 1250 °C.[3]

The Raman scattering measurements were performed in backscattering configuration with near ultraviolet incoming laser photons generated by a Kr^{+} -ion laser operating at 413.1 nm. These photons are absorbed within a skin depth of $\approx$150 nm,[7] which is comparable to d_{Si}. The Raman light originates only in the top Si layer, and therefore one can probe this part of the sample without interference from the underlying Si substrate or the buried SiO_2 layer. The Raman scattered light was analyzed in frequency with a double-monochromator and detected with photon-counting electronics. The position of the Raman peaks was measured with an accuracy of $\pm$0.1 cm^{-1} and the linewidths with a spectral resolution better than 0.6 cm^{-1}. To improve on the ability to establish frequency shifts and line broadenings, we performed the Raman experiments at a low temperature with the samples mounted in a cold finger of a closed cycle cryostat and kept at $\approx$12 K.

Figure 1 shows typical first-order Stokes–Raman spectra for several of the investigated SIMOX samples, including also for comparison data taken from a virgin (not implanted, not annealed) Si (100) surface. The spectra arise from scattering by long-wavelength transverse optical phonons.[8] For clarity, the Raman peaks have been displaced vertically with the horizontal solid lines representing the background levels in each case. The following experimental conclusions are apparent from the result in Fig. 1: The position and linewidth of the Raman peak corresponding to the lamp-annealed sample are within the experimental uncertainties the same as those of the virgin Si surface. On the other extreme, the implanted and not-annealed sample shows a Raman band with a pronounced shift to lower frequencies (red shift) and a clear asymmetrical broadening compared to the reference Si surface. The low-frequency wing rises above the background level faster than the high-frequency one. The spectra of the furnace-annealed samples are also red shifted and broadened with respect to the virgin case. However, the red shifts and broadenings decrease with increasing annealing temperature T_A. Important to point out is the fact that these broadenings are symmetrical, as opposed to the as-implanted case. The measured peak positions ω and linewidths Γ (corrected for instrumental widths), as well as the shifts $\Delta\omega$ and broadenings $\Delta\Gamma$ with respect to the control Si sample, are summarized in Table I for all the investigated samples. The values of ω and Γ for the virgin Si surface are in excellent agreement with those reported in the literature.[9] We also scanned the spectral region at around 480 cm^{-1} to look for the typical Raman signal of amorphous Si. No scattering intensity readily identifiable as due to amorphous Si was observed at these frequencies in the Raman spectra of the SIMOX samples.

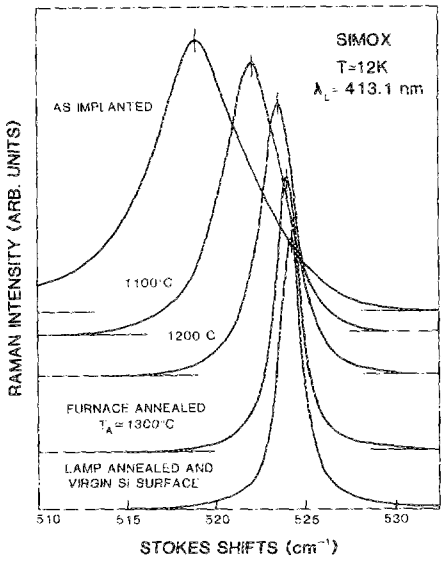

FIG. 1. First-order Raman spectra of the top Si layers of SIMOX structures for different annealing treatments. The temperature in the lamp-annealing case was 1045 °C. For comparison, data are included for an as-implanted (not annealed) and virgin Si surface (not implanted, not annealed).

In what follows we discuss the results of Fig. 1, particularly the fact that the Raman lines of the annealed samples are symmetrical, but nevertheless red shifted. The symmetrical Raman line shapes confirm the crystalline nature of the top Si layer. If a high degree of disorder were present in the network because of microcrystalline grains, then wave vector nonconservation will dominate the scattering process.[8] The Raman spectra would then be asymmetrically broadened, which is indeed the case for the as-implanted layer. Ruling out the effects of structural disorder in the Raman data of the annealed layers is important, because it excludes phonon confinement as the origin of the phonon shifts.[8,10] We attribute $\Delta\omega$ to the presence of strain in the crystalline layers, and we will show that the strains are of tensile nature.

TABLE I. Measured parameters of the Raman lines at 12 K of SIMOX samples: peak positions ω, linewidths Γ, and shifts $\Delta\omega$ and broadenings $\Delta\Gamma$ with respect to the virgin sample. All these parameters are in units of cm^{-1}. Also included are the determined values of the strain ϵ and stress X. The latter are in kbars.

Sample	ω	$\Delta\omega$	Γ	$\Delta\Gamma$	ϵ	X
Virgin	524.2	0	1.3	0	0	0
As-implanted	518.9	−5.3	6.2	4.9		
1100 °C furn. ann.	521.9	−2.3	4.1	2.8	0.007	11.5
1150 °C furn. ann.	522.3	−1.9	3.7	2.4	0.005	8.3
1200 °C furn. ann.	523.5	−0.7	2.4	1.1	0.002	3.3
1250 °C furn. ann.	523.8	−0.4	1.4	0.1	0.001	1.7
1300 °C furn. ann.	523.9	−0.3	1.3	0	0.0009	1.5
1405 °C lamp ann.	524.2	0	1.3	0	0	0

Softening of the lattice modes has been observed in other semiconductor systems implanted at very high doses and attributed to large volume expansions in the bombarded regions along directions perpendicular to the surfaces of the layers.[10,11]

We can obtain quantitative estimates of the maximum strains expected in the top Si layers from the measured $\Delta\omega$, and theoretical expressions and parameters of Ref. 12. If we assume that the layers are allowed to relax only in the z direction,[10,11] then the relationships $\Delta\omega = -323\epsilon = -0.195X$ hold between $\Delta\omega$ (in cm^{-1}), the strain $\epsilon_{zz} = \epsilon$, and the associated stress $X = \sigma_{zz} = C_{11}\epsilon$ (in kbar). C_{11} is the corresponding elastic stiffness constant of Si. The convention adopted is that ϵ is positive for tension, z is perpendicular to the implanted surface, and x and y lie in the plane of the layer. Replacing $\Delta\omega$ by the measured quantities in the expressions above yield the values of ϵ and X given in Table I. The strains are of tensile nature, and their magnitude decreases with increasing annealing temperature T_A. However, even the highest furnace-annealing temperatures are not effective in removing all traces of strain. Within the accuracy of the Raman determination, strain-free material is obtained only in the lamp-annealed specimens. We have not attempted to estimate the strain in the as-implanted layer, because the contribution of phonon confinement has to be elucidated first.[10] The task implies a detailed fitting of the line shape which is beyond the scope of the present work. However, we point out that $\Delta\omega$ for this sample is larger than the shifts measured in polycrystalline Si with comparable $\Delta\Gamma$, and therefore some strain is also built in the as-implanted layer.[13]

We ascribe the origin of the strain in the annealed layers to the presence of the oxide precipitates. Their inclusion in the Si matrix occurs at the expense of creating a volume expansion of the layers along the z direction. The decrease of ϵ with T_A certainly tracks the reduction of the density of precipitates until they are almost eliminated in the lamp-annealed sample for which $\epsilon = 0$. The magnitude and sign of the strains in Table I are comparable to those reported in other SOI structures, such as recrystallized Si directly on quartz (SiO_2).[14] In these systems the strains are attributed to the mismatch in thermal expansion between Si and SiO_2. In the SIMOX case this effect cannot be invoked, because d_{SiO_2} and the thermal mismatch increase, and d_{Si} decreases with increasing T_A, and therefore larger values of ϵ should be expected for the samples annealed at the highest temperatures. This is in contradiction with the experimental findings. The broadening $\Delta\Gamma$ of the Raman peaks can be understood in terms of inhomogeneities in ϵ. In the interpretation of the results, we have not involved the threading dislocations. We estimate that they do not affect the positions or widths of the Raman peaks, because their separation is much larger than any expected correlation lengths for the phonons.[8]

In summary, we have shown how Raman spectroscopy can be applied to the characterization of SIMOX structures. We established that the recrystallized Si layers are under tensile strains, which are attributed to the oxide precipitates in the Si matrix. Strain measurements by Raman scattering can be used to gauge rapidly and nondestructively the quality and uniformity of SIMOX wafers. Such determinations by means of the conventionally used structural characterization techniques are cumbersome and destructive. The realization that strains can be present is relevant for the analysis of the transport properties of devices built in the recrystallized layers or for the subsequent growth of epitaxial Si on Si. Since the implantation parameters and annealing temperature and time settings selected in this study are quite standard, we expect that our findings can be extended to SIMOX structures fabricated in other laboratories.

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[1] K. Izumi, M. Doken, and H. Ariyoshi, Electron. Lett. **14**, 593 (1978).
[2] K. J. Reeson, P. L. F. Hemment, C. D. Meekison, G. R. Booker, J. A. Kilner, R. J. Chater, R. J. Davis, and G. K. Celler, Appl. Phys. Lett. **50**, 1882 (1987).
[3] G. K. Celler, Solid State Technol. **30**, 93, (1987).
[4] B. Y. Mao, P. H. Chang, H. W. Lam, B. W. Shen, and J. A. Keenan, in *Mater. Res. Soc. Symp.* **53**: *Semiconductor-on-Insulator and Thin Film Transistor Technology*, edited by A. Chiang, M. Geis, and L. Pfeiffer (Materials Research Society, Pittsburgh, 1986), p. 251; P. H. Chang and B. Y. Mao, Appl. Phys. Lett. **50**, 152 (1987).
[5] S. N. Bunker, P. Sioshansi, M. M. Sanfacon, and S. P. Tobin, Appl. Phys. Lett. **50**, 1900 (1987).
[6] J. Narayan, S. Y. Kim, K. Vedam, and R. Manukonda, Appl. Phys. Lett. **51**, 343 (1987).
[7] D. Aspnes and A. A. Studna, Phys. Rev. B **27**, 985 (1983).
[8] M. Cardona, in *Light Scattering in Solids II*, edited by M. Cardona and G. Güntherodt (Springer, Heidelberg, 1982), p. 19.
[9] M. Balkanski, R. F. Wallis, and E. Haro, Phys. Rev. B **28**, 1928 (1983).
[10] G. Burns, F. H. Dacol, C. R. Wie, E. Burstein, and M. Cardona, Solid State Commun. **62**, 449 (1987).
[11] D. R. Myers, P. L. Gourley, and P. S. Peercy, J. Appl. Phys. **54**, 5032 (1983).
[12] E. Annastassakis, A. Pinczuk, E. Burstein, F. H. Pollak, and M. Cardona, Solid State Commun. **8**, 133 (1970).
[13] I. H. Campbell and P. M. Fauchet, Solid State Commun. **58**, 739 (1986).
[14] S. A. Lyon, R. J. Nemanich, N. M. Johnson, and D. K. Biegelsen, Appl. Phys. Lett. **40**, 316 (1983).