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Defects in Czochralski-grown Si–Ge annealed under high hydrostatic pressure

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ABSTRACT

Defect structure of Czochralski-grown oxygen-containing silicon–germanium (Si–Ge) samples with Ge content $1.5\times10^{18}\,cm^{-3}$, subjected to processing for 5 h at up to 1400 K under hydrostatic Ar pressure up to 1.1 GPa, has been investigated by synchrotron topography (at HASYLAB), high-resolution X-ray diffraction, photoluminescence and related methods. Ge admixture prevents formation of extended defects at processing done under 10^5 Pa. Such defects are created, however, in Si–Ge processed at 1400 K under high pressure (1.1 GPa).

1. Introduction

Silicon–germanium (Si–Ge) single crystals with germanium content $c_{Ge}\leq10^{19}\,cm^{-3}$ are of interest for microelectronics, because the presence of Ge in a low concentration has been reported to affect favourably the creation of extended defects (Yang, 2005).

Annealing of Czochralski-grown Si–Ge (Cz–Si–Ge) at $\geq$1000 K (*HT*), especially under enhanced pressure (*HP*) of inert ambient gas (up to 1.2 GPa), results first of all in stimulated precipitation of oxygen interstitials (O_i's) always present in Cz–Si–Ge and so in the creation of oxygen-related defects (Misiuk et al., 2008). Almost no extended defects are, however, formed in Cz–Si–Ge with low Ge content if processed at *HT–HP*; contrary to the case of Ge-lean Cz–Si are processed at the same conditions (Misiuk, Yang, et al., 2006; Misiuk, Londos, et al., 2006). This unusual effect is now investigated in more detail.

2. Experimental

The (0 0 1)-oriented, p-type, 2-mm-thick single crystalline Cz–Si–Ge samples with Ge and O_i contents at 1.5×10^{18} and $1\times10^{18}\,cm^{-3}$ levels, respectively (Fig. 1), were chemically etched to a depth of about 120 μm and subjected to processing for up to $t=10$ h at 1000–1400 K under $HP\leq1.1$ GPa in Ar atmosphere.

The samples were investigated by the synchrotron diffraction topography (at HASYLAB), high-resolution X-ray diffraction (HRXRD), secondary ion mass spectrometry (SIMS), photoluminescence (PL) and infrared (IR) methods.

3. Results and discussion

HP-promoted precipitation of oxygen interstitials (O_i's) occurs in Si–Ge at $HT\geq1000$ K (Fig. 1). An amount of precipitated interstitial oxygen (Δc_{oi}) after processing of Si–Ge at particular *HT* for time *t* increases with *HP*. For example, processing for 5 h at 1070 K under 10^5 Pa resulted in precipitation of about 10% of initially present O_i's, while the same processing under 1.1 GPa - in precipitation of about 15% of oxygen interstitials. After processing at 1273 K, the respective Δc_{oi} values corresponded to about 5% and 50% (Fig. 1).

Enhanced concentration of oxygen in the near-surface region of the as-grown and processed Si–Ge samples (Fig. 1) is related to surface oxidation by air as well as to gettering of O_i's diffusing from the sample bulk.

The *HT–HP* processed Si–Ge samples absorb IR light at $1107\,cm^{-1}$ (this absorption evidences the presence of O_i's) and at

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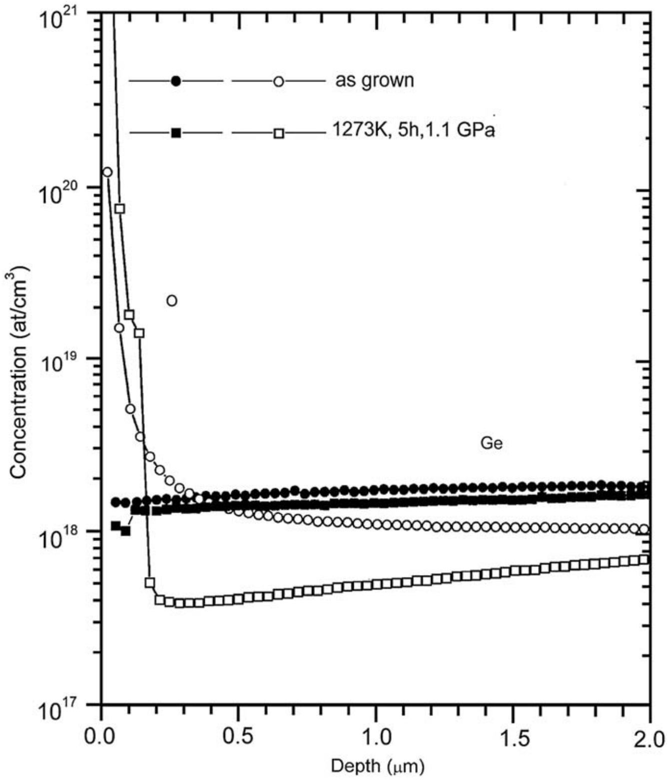

Fig. 1. SIMS depth profiles of germanium (black dots) and of oxygen (open marks) in Si-Ge samples, as-grown (top curves) and processed for 5 h at 1273 K under 1.1 GPa (bottom curves).

about $1060\,cm^{-1}$ (as it has been indicated by Borghesi et al. (1995), such absorption is related to a creation of SiO_x precipitates). The relative intensity of IR at $1060\,cm^{-1}$, $I_{1060\,cm^{-1}}/I_{1107\,cm^{-1}}$, depends on the processing conditions and increases with *HP* confirming the *HP*-stimulated creation of SiO_x. For example, the value of $I_{1060\,cm^{-1}}/I_{1107\,cm^{-1}}$ was equal to 0.13 in the case of processing of Si–Ge at 1073 K for 5 h under 10^5 Pa, while to 0.16 if processed at same conditions but under 1.1 GPa.

As seen in the X-ray reciprocal space maps (XRRSM) of the Si–Ge samples processed at 1073 K (Fig. 2), as well as of these ones processed at up to 1400 K (compare Misiuk, Londos, et al., 2006), the samples annealed under *HP* indicate the high intensity of diffusively scattered X-rays. This corresponds to considerable disturbance of their structure.

The PL spectra of Si–Ge processed at 1073 K do not evidence the presence of the strongest dislocation—related lines, at about 0.81 and/or 0.87 eV, observed for the case of dislocated single crystalline silicon. The same concerns the Si–Ge samples processed for 5 h at up to 1400 K (compare Misiuk, Yang, et al., 2006). Intensity of the FE(TO) and BE(TO) PL lines decreases after the treatment under *HP*. These as well as the EHD lines (Fig. 3 and its caption) are related to the presence of specific point-like (not extended) defects in a high concentration.

No dislocations or other extended defects, distinctly related to intrinsic precipitation of interstitial oxygen, have been revealed by X-ray topography in the case of Si–Ge processed at ≤ 1273 K both under 10^5 Pa and *HP* as well as at 1400 K under 10^5 Pa (Fig. 4A–C). Dark spots in these topographs visualize scratches and similar mechanically introduced irregularities. Only the sample processed at 1400 K under *HP* indicates the presence of strain fields related to oxygen precipitates (Fig. 4D).

As follows from now presented and earlier reported results (Misiuk, Yang, et al., 2006; Misiuk, Londos, et al., 2006), the

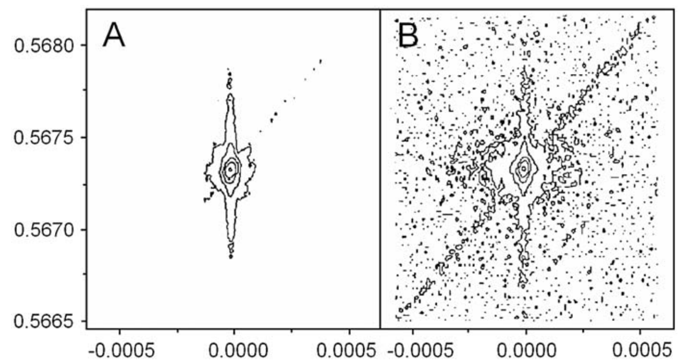

Fig. 2. XRRSM's of Si–Ge samples processed for 5 h at 1073 K under 10^5 Pa (A) and 1.1 GPa (B).

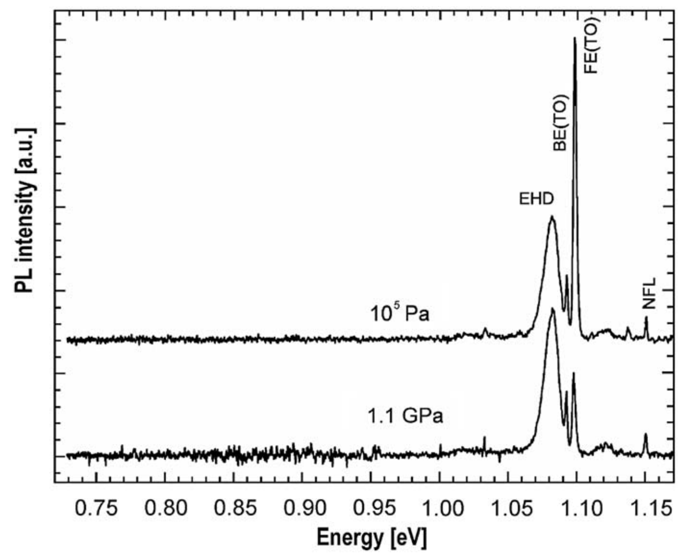

Fig. 3. PL spectra of Si–Ge samples processed for 5 h at 1073 K under 10^5 Pa and 1.1 GPa. EHD lines correspond to recombination of so-called electron–hole droplets, probably associated with nano-dimensional defects; BE(TO) and FE(TO) lines are related to emission involving transverse optical phonon replicas of boron-bound exciton and of free exciton; NFL—emission related to non-phonon recombination of excitons.

presence of Ge admixture in Czochralski-grown silicon a concentration of about $1\times10^{18}\,cm^{-3}$ prevents the formation of extended oxygen-related defects at annealing, also under *HP*. Extended defects were created only in Si–Ge processed at the highest temperature applied, 1400 K, and only in the case of enhanced *HP*. One can suppose that the Ge admixture forms very numerous small nucleation centres for oxygen precipitation. In effect, mostly nano-dimensional oxygen clusters are created during annealing, as confirmed by XRRSM and PL measurements. Their dimensions are so low so they do not reach the critical conditions for the creation of dislocations (Ashby and Johnson, 1969). Such oxygen clusters, created during processing, are also too small to be detected by X-ray topography.

4. Conclusions

Processing of Si–Ge samples at enhanced temperatures/pressures results in massive precipitation of interstitial oxygen creating small SiO_x clusters; this effect is more pronounced under *HP*. Contrary to the case of processing under atmospheric

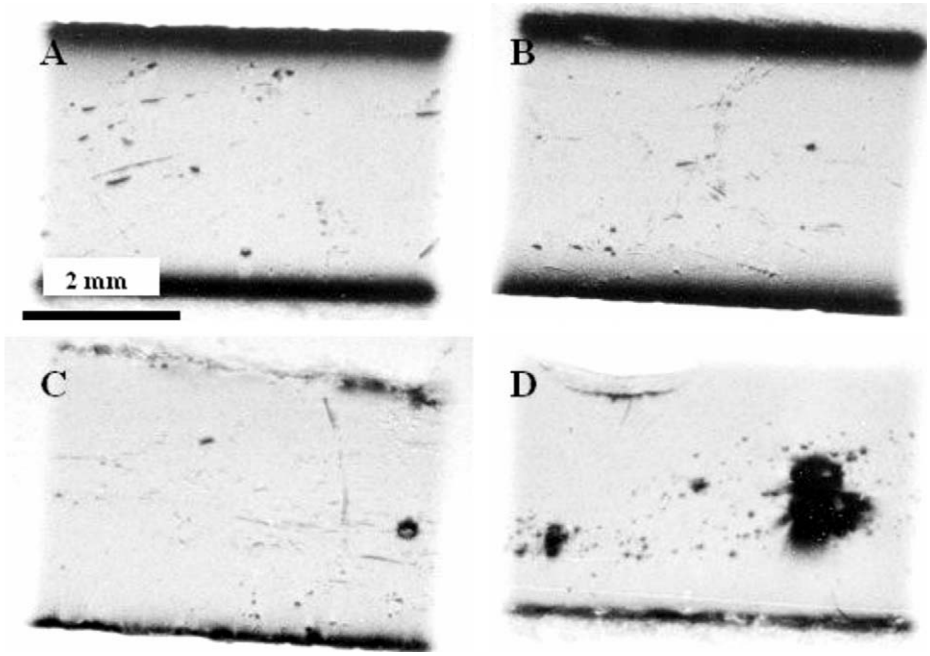

Fig. 4. White beam projection topographs of Si–Ge samples processed for 5 h at 1273 K (A and B) or at 1400 K (C and D) under 10^5 Pa (A, $c_o = 6.4\times10^{17}$ cm^{-3}; C, $c_o = 5.9\times10^{17}$ cm^{-3}) or under 1.1 GPa (B, $c_o = 6.1\times10^{17}$ cm^{-3}; D, $c_o = 5.7\times10^{17}$ cm^{-3}).

pressure, the treatment of Si–Ge at 1400 K under 1.1 GPa produces extended defects (dislocations).

The effect of *HT–HP* processing on Si–Ge may be related in part to stress-induced activation of some primary existing defects acting under *HP* as nucleation centres for precipitation of O_i's; Ge atoms may be the components of such centres.

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