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Structural Property Study for GeSn Thin Films

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Abstract: The structural properties of GeSn thin films with di erent Sn concentrations and thicknesses grown on Ge (001) by molecular beam epitaxy (MBE) and on Ge-bu ered Si (001) wafers by chemical vapor deposition (CVD) were analyzed through high resolution X-ray di raction and cross-sectional transmission electron microscopy. Two-dimensional reciprocal space maps around the asymmetric

(224) reflection were collected by X-ray di raction for both the whole structures and the GeSn epilayers. The broadenings of the features of the GeSn epilayers with di erent relaxations in the ! direction, along the !-2 direction and parallel to the surface were investigated. The dislocations were identified by transmission electron microscopy. hreading dislocations were found in MBE grown GeSn layers, but not in the CVD grown ones. The point defects and dislocations were two possible reasons for the poor optical properties in the GeSn alloys grown by MBE.

Keywords: GeSn; structural property; XRD

1. Introduction

Since the first planar silicon (Si) transistor was invented in 1959 [1], the Si electronic industry has developed prosperously. According to the Moore's law the number of components on integrated circuits (ICs) has doubled every 18 to 24 months for the last 50 years [1]. However, the continuous scaling of the transistors has led to several problems, such as signal delays, higher power consumption, quantum limitations, etc. Moreover, without new technologies, the shrinking of the transistors will stop around 2021 [2]. Si photonics, which utilizes photons instead of electrons for information transmission and processing, is one of the promising solutions for these problems and shows a bright future [3,4].

It is di cult to fabricate light sources using Si due to its indirect bandgap nature. The light sources used in the commercial Si based circuits are made of group III-V materials, and then bonded to the circuits at present [5]. However, the bonding technology hinders the future large-scale integration [3]. Monolithic light sources compatible with the complementary metal-oxide-semiconductor (CMOS) processing platform are in urgent need. Ge is also an indirect bandgap material, but the energy di erence between its Γ and L valley is only 140 meV [6], much smaller than that of Si. It is found that incorporation of Sn in Ge can reduce the energy of both the Γ and the L valleys in the conduction band, while the Γ valley goes downward faster. Eventually, the Ge 1-xSnx alloy becomes a direct bandgap semiconductor with the Γ valley lower than the L valley, when the Sn concentration is



above about 8% [7]. When tensile strain is applied on Ge, a similar transformation of the band structure in the conduction band can happen [7]. However, devices based on tensile-strained Ge require complex processes, such as integration of Si₃N₄ stressor layers [8], or selective wet underetching [9]. Moreover, GeSn is predicted to own high carrier mobility [10], making it a potential candidate for fabricating both electronic and optoelectronic devices integrated on the Si platform. GeSn p-MOSFETs on Si [11–13] and GeSn lasers [7,14–17] are already successfully realized. Especially, the successful demonstration of the GeSn lasers has drawn significant attention and confidence on the realization of CMOS technology compatible light sources monolithically integrated on Si platform.

The electrical property and (or) optical property of the as grown materials are critical for the performance of devices, both of which are highly related to the structural properties. For GeSn alloys, Sn incorporation is the largest growth challenge, due to the large miscibility gap of the Ge-Sn binary system [18] and the large lattice mismatch between Ge and -Sn [7]. Molecular beam epitaxy (MBE) and chemical vapor deposition (CVD) are the main methods for the growth of GeSn alloys [19,20]. Generally speaking, the MBE-grown GeSn thin films hold advantages on high Sn concentration [21] while the CVD grown ones show high optical property [7,14–17,22–24]. So far, to the best of our knowledge, all GeSn lasers were grown by CVD [7,14–17,25,26]. The causes for the di erence in the optical properties of GeSn alloys grown by MBE and CVD are not fully clear. Structural property is usually a fatal factor on optical property. Crystalline fluctuations on a microscopic scale, line defects like dislocations and point defects such as vacancies can all result in degradation of optical property.

In this work, we investigate the structural property di erences of GeSn alloys with di erent Sn concentrations and thicknesses. The structural properties of two groups of GeSn samples grown by MBE and CVD, respectively, are investigated through high resolution X-ray di raction (XRD) and cross-sectional transmission electron microscopy (XTEM). We further study the di erences of optical properties of GeSn samples grown by MBE and CVD and their connections with the structural properties.

2. Materials and Methods

The first group of six GeSn samples (M1-M6) were grown on Ge (001) substrate by MBE with Sn concentrations varying from 3.4% to 7.6%. The Ge (001) substrate was firstly deoxidized at 550 C for 30 min. Secondly, a Ge bu er layer of about 100 nm was grown on the Ge (001) substrate at 500 C. Thirdly, the temperature was lowered to 200 C and the GeSn layer was grown. The detailed growth procedure was described elsewhere [27]. Another group of three GeSn samples (C1-C3) were grown by CVD with the Sn concentrations of 6.8%, 7.6% and 10.4%, respectively. A relaxed Ge virtual substrate (VS) of about 2.5 m was firstly grown on the Si (001) substrate at 750 C and 20 Torr. Secondly, to reduce the dislocation densities in the Ge-VS, the Ge-VS was annealed at 890 C for 5 min and then the annealing was repeated for three times. Thirdly, the GeSn layer was deposited under a fixed precursor partial pressure ratio for the digermane (Ge₂H₆): tintetrachloride (SnCl₄) = 220. The Sn content was controlled by adjusting the growth temperature. The detailed growth methods for Ge-VS and the GeSn layer can be found in [28] and [29], respectively.

Figure 1 shows the sample structures grown by MBE (a) and CVD (b), respectively. The six GeSn samples grown by MBE are of di erent thicknesses and di erent Sn concentrations. For the CVD grown GeSn thin films, the three samples are grown on fully relaxed Ge VS. Threading dislocations were found in the annealed Ge VS, with the density of about 10⁷ cm ² [28]. If the dislocation densities in the Ge VS is significantly reduced or the GeSn thin films are directly grown on Ge substrate by CVD, the structural and optical properties of the GeSn thin films might be further improved. The thicknesses and Sn contents of all the samples can be found in Table 1.

Crystallographic properties were measured by high resolution XRD with an X'Pert PRO di ractometer (Philips/Panalytical, Almelo, The Netherlands). An X-ray source with Cu K 1 (= 1.54)



Å) was used and traveled to the GeSn samples and then di racted. There were two di erent optical paths (Optics1 and Optics2) for the detections. In Optics1, the di racted beam directly traveled to the detector. In Optics2, the di racted beam traveled to a three-crystal Ge (002) analyzer and then reached

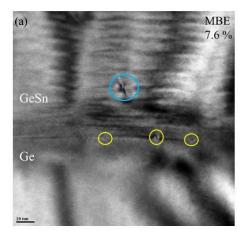
the detector. The thicknesses of GeSn layers were inferred from the calibrated growth rate and the XTEM data. The Sn concentrations were extracted from the Pendellösung interference fringes from symmetric (004) rocking curve (!-2) scans. When getting the (004) !-2 rocking curves, Optics1 was used. The applied voltage and current were 30 KV and 25 mA, respectively. The angle step in the !-2 direction were both 0.0005 while the time for each step was 0.5 s. Then the broadenings of the GeSn epilayer features were investigated by two-dimensional reciprocal space mapping (2DRSM) around the asymmetric (224) reflections. When collecting the 2DRSM, Optics2 was used. The applied voltage and current were 40 KV and 40 mA, respectively. The angle steps along the ! direction and the !-2 direction were both 0.002 while the times for each step were 1.2 s. Furthermore, the structural properties of the GeSn layer were analyzed through XTEM using a Tecnai G2 F20 system (FEI, Hillsbro,



Figure 1. The sample structures of GeSn thin films grown by MBE (a) and CVD (b), respectively.

the mismatch dislocations significantly increases indicating that the critical thickness of strain relaxation is reached. The ω -broadenings of C1–C3 are larger than that of M1–M6, due to larger relaxations of C1–C3. However, the strain relaxation through dislocation process is complicatedly associated to lattice mismatch, film thickness, type of dislocations, etc. A three-dimensional plot of the variations of the broadenings with different Sn contents and thicknesses can be found in Figure Materials **2020**, 13, 3645

A1 in the 'Appendix A' section. A more intensive analysis is as follows.



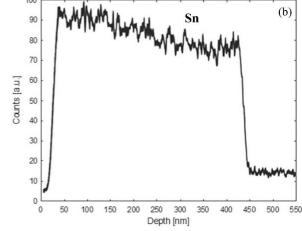




Figure 4. (a) XTEM and (b) the energy dispersivespectroscopy (E(EDX) of the GeSn sample with the Sn

4. (a) XTEM and (b) the energy of the GeSn with the Sr concentration of 7.6% grown by MBE. The EDX is collected from the surface of GeSn layer down to the concentration of 7.6% grown by MBE. The EDX is collected from the surface of GeSn layer down to the Ge layer. Gelayer.

The broadening in the ! direction shows a similar trend to the broadening parallel to the surface. Figure 4a shows the XTEM results of the M6. The blue circle is the threading part of 60° Since the thickness of the GeSn epilayers is very small compared with that of the Ge substrates or dislocations. Yellow circles are the edge dislocations or the misfit part of 60° dislocations. For the Ge-VS, the wafer curvature caused by strain in the GeSn layer is negligible. Therefore, dislocation is the CVD grown GeSn samples, mostly pure edge dislocations together with small amount of 60° most probable reason for the ! broadening. When the Sn concentration is above 7%, the density of the dislocations bending into the Ge layer were observed in TEM measurements, as shown in reference mismatch dislocations significantly increases indicating that the critical thickness of strain relaxation [7]. Figure 4b shows the EDX line- scan result of Sn content in M6. Sn content increases from the

is reached. The !-broadenings of C1–C3 are larger than that of M1–M6, due to larger relaxations of bottom of the GeSn layer to the surface, indicating a segregation of Sn atoms. Due to the low thermal

C1–C3. However, the strain relaxation through dislocation process is complicatedly associated to lattice equilibrium solid solubility of Sn in Ge, Sn segregation is a big challenge for the GeSn growth [27]. mismatch, film thickness, type of dislocations, etc. A three-dimensional plot of the variations of the The nonuniform distribution of Sn atoms in M6 explains the large broadenings along the ω -2 θ broadenings with di erent Sn contents and thicknesses can be found in Figure A1 in the 'Appendix A' direction measured by the 2DRSM.

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2thedirectionconentsmeasuredinthebracketbyhein 2DRSMexpression. (2) couldThbe mismatchequivalentdislocationtothedegrensityeofstrainiscalculatedrelaxationas

(follows, *R*), so the according mismatch to Hu's dislocation expression ensity [33]: is estimated as follows:

$$\begin{array}{ccc}
& = b \sin \sin ? & & _{h}c & ! \\
& \underline{f} & \underline{h}
\end{array} \tag{2}$$

In the above expression, is the angle between the dislocation line and the Burgers vector and is the angle between the slip plane and the interface. In GeSn systems, there are basically two types of dislocations, 60 and pure edge dislocations [34]. For 60 dislocations, is 60 and the slip plane is the (111)plane, so is 54.7 . For edge dislocations, is 90 and the slip plane is the (010) plane, so is 90 . Considering the relaxation rate measured by XRD, the contents in the bracket in expression (2) could be equivalent to the degree of strain relaxation (R), so the mismatch dislocation density is estimated

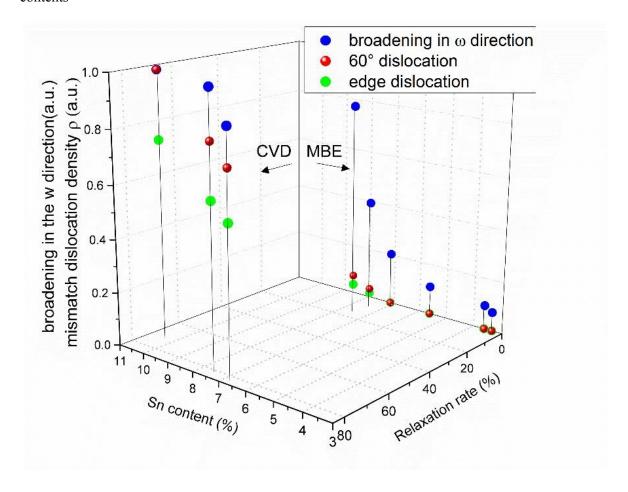
as follows:



 $= b\sin \sin ? \tag{3}$

The broadening in the ! direction (blue) and the dislocation density based on all 60 (red) and all edge dislocation (green) assumptions of the samples are shown in Figure 5. A three-dimensional plot of

Figure 5. Normalized broadening in the ! direction (blue), and the dislocation density calculated based on all 60 dislocation (red) and all edge dislocation (green) assumption with di erent Sn contents





was obtained [35]. For other lattice properties, such as lateral incoherence, the CVD grown samples show no advantages. Although MBE-grown GeSn shows a higher compositional nonuniformity, it is commonly believed not an important factor for optical property. Local states may even enhance photoluminescence. Another important factor, which can't be directly measured by XRD, is the point defect density. The point defects, such as vacancies, can act as nonradiative recombination centers. The CVD process to grow GeSn uses a much higher growth temperature than that by MBE. It is generally true that the higher growth temperature the lower point defect density. The "hidden" factor who causes the broadening in! direction for the MBE samples could be an important factor. To sum up, the high density of threading dislocation and point defects could be the most probable reasons for the worse optical property of MBE-grown GeSn than the CVD grown ones.

The major limitation of this experiment is that the substrates and the sample structure of the MBE and the CVD grown samples are di erent, leading to di culty in direct comparison.

4. Conclusions

In this paper, structural properties of GeSn alloys with di erent Sn contents and thicknesses grown by MBE (low relaxation) and CVD (high relaxation) are analyzed through XRD and XTEM. The GeSn layers fully strained to Ge substrate shows the best structural properties. The structural properties, such as lateral incoherence and the density of mismatch dislocations of GeSn layers partially relaxed to Ge substrate are found better than that of GeSn layers almost fully relaxed to GeVS. However, the GeSn alloys grown by MBE has higher threading dislocation density than that grown by CVD. Meanwhile, GeSn layers partially relaxed to Ge substrate grown by MBE show larger compositional nonuniformity than that of GeSn layers almost fully relaxed to Ge-VS grown by CVD. Point defects and the threading dislocations are two possible reasons for the poor optical property of the GeSn grown by MBE.

Author Contributions: L.Z. was responsible for the XRD mapping, analysis of data and preparation of the manuscript. Y.S. supervised the entire study and revised the manuscript. Z.Z. grew the GeSn samples by MBE. N.v.d.D., D.B. and D.G. grew the GeSn samples by CVD. S.W. participated in the manuscript revision. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflict of interest.

Appendix A

Figure A1 shows the variations of the broadenings of the GeSn epilayer features with di erent Sn contents and thicknesses. The tendency for the variations of the broadenings parallel to the surface, along the !-2 direction and in the ! direction for M1–M6 are similar. The thicknesses for M1–M5 are 200 nm, the broadenings increases with the Sn contents. For M5 and M6, the Sn contents are 7.5% and 7.6%, almost the same, the thicknesses are 200 nm and 400 nm, respectively. The larger broadenings of M6 than that of M5 is mainly due to the increases of the thickness. In Figure A1a,c, C1–C3 show larger broadenings parallel to the surface and in the ! direction than M1–M6, respectively. It indicates a worse lateral incoherence and larger amount of mismatch dislocations in C1–C3.

In Figure A1b, C1–C3 show smaller broadenings along the !-2 direction than M5,M6, indicating a nonuniform distribution of Sn atoms in the MBE samples with high Sn content, which is in consistent with the EDX line-scan result shown in Figure 4b.



Figure A2 shows the variations of the broadening in the ! direction and the dislocation density based on all 60 degree and all edge dislocation assumptions of the samples with di erent thicknesses and Sn contents. The dislocation density and the broadening in the ! direction are both normalized and share the z axis.

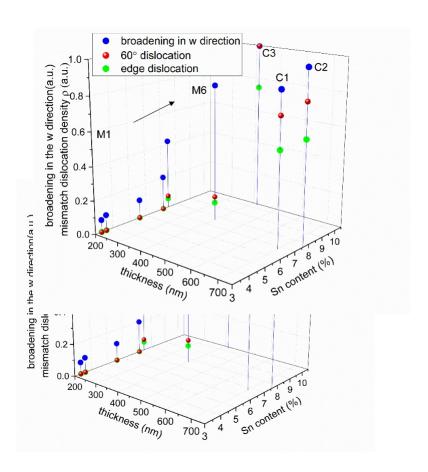




Figure A1. 3D plot of the broadenings of the GeSn epilayer features parallel to the surface (a), along the ω -2 θ direction (b) and in the ω direction (c). The blue and red balls represent the samples grown by MBE and CVD, respectively.

In Figure A1b, C1–C3 show smaller broadenings along the ω -2 θ direction than M5,M6, indicating a nonuniform distribution of Sn atoms in the MBE samples with high Sn content, which is in consistent with the EDX line-scan result shown in Figure 4b.

Figure A2 shows the variations of the broadening in the ω direction and the dislocation density based on all 60 degree and all edge dislocation assumptions of the samples with different thicknesses

Figure A1. 3D plot of the broadenings of the GeSn epilayer features parallel to the surface (a), along

and Sn contents. The dislocation density and the broadening in the ω direction are both normalized **Figure A1.** 3D plot of the broadenings of the GeSn epilayer features parallel to the surface (a), along

the !-2 direction (**b**) and in the ! direction (**c**). The blue and red balls represent the samples grown by and the samples grown by and the samples grown and in the ω direction (**c**). The blue and red balls represent the samples grown MBE and CVD, respectively. by MBE and CVD, respectively.

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Figure A2 shows the variations of the broadening in the ω direction and the dislocation density based on all 60 degree and all edge dislocation assumptions of the samples with different thicknesses and Sn contents. The dislocation density and the broadening in the ω direction are both normalized and share the z axis.

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