Process for Growth of Group-IV Alloys Containing Tin by Remote Plasma Enhanced Chemical Vapor Deposition

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A remote plasma enhanced chemical vapor deposition (CVD) process using GeH₄, SiH₄, and SnCl₄ precursors has been developed for epitaxial growth of group-IV alloys directly on Si (100) substrates, without the need for buffer layers. X-ray diffraction measurements of a representative Ge₁₋ₓSnₓ sample which is 233 nm thick, with x = 9.6% show it to be highly oriented along the [001] direction and nearly relaxed, with 0.37% compressive strain. Ellipsometry measurements provide a pseudo-dielectric function which is well fitted by a 3-layer (substrate/alloy/surface oxide) model. Cross-sectional transmission-electron-microscope images show a highly defective interface layer, ~ 60 nm thick, containing edge dislocations and stacking faults; above this layer, the lattice is well-ordered, with a much lower density of defects. Atomic force microscopy measurements show an RMS roughness of 1.2 nm for this film.

Keywords: RPECVD, characterization, growth process, GeSn and GeSiSn alloys, crystalline

INTRODUCTION

Semiconductor devices based on the group-IV elements Si and Ge, as well as their alloys, have dominated many applications in the electronics industry, such as microprocessors for logic circuits, cell phones, global positioning systems (GPS), and wireless communications (WiFi). However, similar progress in optical and electro-optical devices has been limited by the indirect nature of the fundamental energy gap in these materials. In contrast, cubic α-Sn is a zero bandgap, group-IV semiconductor which, when combined in sufficient concentration with Ge, results in a Ge₁₋ₓSnₓ alloy with a direct bandgap (Mathews et al., 2010; Moontragoon et al., 2012; Gupta et al., 2013; Tonkikh et al., 2013; Zaima et al., 2015; Fernando et al., 2018). These Ge₁₋ₓSnₓ alloys have attracted attention as potential low-cost detectors (Chang et al., 2016; Maczko et al., 2016; Tsai and Chang, 2017) and emitters (Wirths et al., 2015; Zhou et al., 2016; Margetis et al., 2018) in the short- and mid-wave-infrared (SWIR and MWIR) spectral regions and can be incorporated into a variety of components (Sun et al., 2007; Soref, 2014; Wirths et al., 2016) for Si-based integrated photonics. Further alloying Ge₁₋ₓSnₓ with Si produces a ternary alloy, Ge₁₋ₓ−ySiₚSnₓ, which allows independent control of lattice constant and bandgap energy (Kouvetakis et al., 2006, 2008).
One significant challenge in the synthesis of Sn-containing group-IV alloys is that the solubility of Sn is \( \sim 1\% \) in Ge (Olesinski and Abbaschian, 1984a) and \( \sim 0.1\% \) in Si (Olesinski and Abbaschian, 1984b). As a result of these limitations, low temperature, non-equilibrium growth methods are required to deposit Ge\(_{1-x}\)Sn\(_{x}\) and Ge\(_{1-x}\)Si\(_{x}\)Sn\(_{y}\) alloys. Early attempts to produce Ge\(_{1-x}\)Sn\(_{x}\) films by sputtering (Shah et al., 1987; Ishida et al., 1989; Maruyama and Akagi, 1998) or molecular beam epitaxy (Asom et al., 1989; Höchst et al., 1989; Pukite et al., 1989; Piao, 1990; Fitzgerald et al., 1991; Rojas-López et al., 1998; Bratland et al., 2005; Kasper et al., 2012; Oehme et al., 2013) showed limited success, with growth on different substrates (Ge, GaAs) to minimize the lattice mismatch. Many of these samples were amorphous or the crystalline quality was not very high, and film growth was susceptible to Sn segregation.

A major advance in Sn-containing group-IV alloy synthesis was the development of a chemical vapor deposition (CVD) process (Taraci et al., 2001; Bauer et al., 2002, 2003) using SnD\(_4\) as the Sn precursor. High quality samples were prepared and characterized, and photoluminescence (PL) was demonstrated for the first time (Soref et al., 2007; Mathews et al., 2010). Higher order germanes and silanes have subsequently been used to increase the reactivity of the precursors, allowing growth at lower temperatures than those used in conventional CVD processes. This alternative process for precursor activation allows for thin-film deposition of group-IV alloys at reduced substrate temperatures.

This work describes a process for synthesizing Sn-containing group-IV alloys, directly on Si substrates, using RPECVD to activate GeH\(_4\), SiH\(_4\) and SnCI\(_4\) precursors. The physical, structural, and optical characteristics of a representative Ge\(_{1-x}\)Sn\(_x\) sample grown using this RPECVD process are also presented. A more detailed study of the properties of Sn-containing group-IV alloys grown by RPECVD as a function of composition will be reported separately (Grzybowski et al., unpublished).

**EXPERIMENT**

Epitaxy of group-IV alloys containing Sn, grown directly on 2-inch Si substrates, without buffer layers, has been performed via an RPECVD process in a custom designed, ultra-high-vacuum (UHV) chamber using a top-mounted Advanced Energy Litmas RPS 3001 inductively coupled remote plasma tube, as shown schematically in Figure 1. Equipment details for this RPECVD reactor will be published separately (Grzybowski et al., unpublished). Growth of Sn-containing group-IV alloy films have been demonstrated over a range of substrate temperatures, from 295 to 340°C as measured by a thermocouple, and chamber pressures, from 200 to 800 mTorr. X-ray photoelectron spectroscopy (XPS) measurements using a PHI 5500 ESCA system were used to confirm the growth of different Sn-containing alloys. The Si substrates receive a dilute (2.5%) HF:H\(_2\)O dip to remove their native surface oxide prior to loading into the growth.
chamber and are heated to the deposition temperature in flowing He prior to flowing precursor gases and initiating the remote plasma.

For a representative Ge$_{1-x}$Sn$_x$ film growth on a 2-inch, p-type, Si (100) substrate, a flow of He gas at 50 SCCM was injected upstream of the RF coil and excited with 1 kW of plasma power. A 50 SCCM flow of 2% GeH$_4$ diluted in He was introduced through a gas dispersal ring, downstream from the plasma as shown in Figure 1. Similarly, a 12.5 SCCM flow of He carrier gas was used to entrain liquid SnCl$_4$ vapor through a bubbler, resulting in a ∼ 2% concentration which was injected through a separate gas injector ring, downstream of the He plasma. A substrate temperature of 325°C and a chamber pressure of 535 mTorr were maintained throughout this film growth.

Film thickness and uniformity of this Ge$_{1-x}$Sn$_x$ sample were measured by optical interference with a Filmetrics F50 Thin Film Mapper tool. The pseudo-dielectric function of the film was determined at a 70° angle of incidence from 0.6 to 4.7 eV in 0.01 eV steps using a Jobin Yvon Horiba UVISEL phase modulated spectroscopic ellipsometer. The data were fitted in a three-layer (substrate/alloy/surface oxide) model using the WVASE32 software of the J. A. Woollam Company. The optical constants of the alloy were described using a parametric semiconductor model (Fernando et al., 2018). Reciprocal space maps (RSM) of the (224) reflection and 2θ–ω scans of the (004) reflection were performed using a PANalytical Empyrean X-ray diffractometer. Film composition and strain were determined (McSkimin, 1953; Xu et al., 2017) using these measured values of lattice parameters and the elastic constants for Ge and Sn. Cross-sectional, bright field, transmission-electron-microscopy (TEM) along the <110> direction was used to evaluate the crystalline structure of the layer as well as to investigate the substrate/film interface. Finally, surface roughness of the sample was measured using a Bruker Dimension atomic force microscope (AFM) in tapping-mode.

RESULTS AND DISCUSSION

The RPECVD process provides a robust and reproducible method to synthesize group-IV alloys containing Sn over a range of compositions: Ge$_{1-x}$Sn$_x$ films have been produced with Sn concentrations as high as 10%, without surface Sn segregation, as well as Ge$_{1-x-y}$Si$_y$Sn$_x$ and Si$_{1-x}$Sn$_x$ layers. Examples of each of these alloys films as measured by XPS are shown in Figure 2. The relative incorporation of each constituent generally reflects the gas flow ratios of the corresponding precursors. Furthermore, RPECVD provides considerable flexibility in process development to optimize specific material properties of interest.

A representative example using this RPECVD process is a Ge$_{1-x}$Sn$_x$ sample with a thickness of 233 nm, grown at a rate of 2.2 nm/min. Thickness uniformity is typically better than 10% across a 2 inch wafer as a result of the flow dynamics of the reactor, even without the use of sample rotation. It should be noted that without plasma excitation, there is no film deposition under the growth conditions used. Ellipsometry data were well fitted with a 3-layer model (substrate/alloy/surface oxide), as shown in Figure 3, giving an alloy thickness of 217 nm and a surface oxide thickness of 3.9 nm. The oscillations below 2 eV in the pseudo-dielectric function are the result of interference fringes from the film.

An X-ray 2θ–ω scan along [001] superimposed on a corresponding (224) RSM for the representative Ge$_{1-x}$Sn$_x$ sample are shown in Figure 4. A (004) Ge$_{1-x}$Sn$_x$ peak at 2θ = 64.85° is observed and the RSM shows an asymmetrical (224) reflection that lies close to the relaxation line, indicating that the film is nearly strain relaxed. From the measured lattice constant values, a 9.6% Sn content and 0.37% compressive strain are inferred for this sample. Quantitative values of Sn content derived from X-ray measurements for Ge$_{1-x}$Sn$_x$ films grown by RPECVD have been confirmed by Rutherford backscattering (RBS) measurements (not shown) on a number of separate samples.
A cross-sectional TEM image of the representative Ge$_{1-x}$Sn$_x$ alloy is shown in Figure 5. An ~60 nm thick interface layer contains a high density of stacking faults as well as edge dislocations at the substrate interface. Above this interface layer, the crystalline quality of the film improves dramatically and the defect density decreases so that individual threading dislocations can be imaged. The defective interface layer accommodates much of the strain due to the lattice mismatch of Ge$_{1-x}$Sn$_x$ and Si, allowing the bulk of the film to relax, consistent with the X-ray results. Most of the stacking faults within this ~60 nm layer appear to annihilate, resulting in the improved crystallinity and lower defect density observed above it. The electrical and optical properties of this interface layer require further investigation since the defects may serve as non-radiative recombination centers or as electrical traps which could impact device applications.

Figure 6 shows a 10 μm × 10 μm AFM image of the sample surface. The film is quite smooth, with an RMS roughness of 1.2 nm. There is no indication of the crosshatching which is typically seen in the growth of thick Ge films on Si as a result of strain relaxation from misfit dislocation formation and motion along the Ge/Si interface. This lack of structure on the surface indicates that a different strain relief mechanism dominates in RPECVD alloy films, consistent with the TEM results.

**CONCLUSION**

An RPECVD process using GeH$_4$, SiH$_4$, and SnCl$_4$ precursors has been developed for growth of nearly strain relaxed, group-IV alloys containing Sn, directly on Si substrates, without the need for buffer layers. Single crystal alloy films with up to 10% Sn incorporation have been demonstrated without surface segregation of Sn. This remote plasma process uses energy transfer from plasma-excited He to activate the chemical precursors, thereby removing the need for their thermal activation on the substrate surface. This decoupling of the substrate temperature from precursor decomposition allows for independent control of the growth temperature to optimize desired film characteristics. In addition, RPECVD provides a simple process for group-IV alloy growth on Si since buffer layers are not required to produce good structural quality. Finally, the RPECVD process uses a precursor partial pressure ratio that is commensurate with the target film composition, resulting in a much more efficient use of these precursors.

A representative Ge$_{1-x}$Sn$_x$ sample synthesized using this RPECVD process has $x = 9.6\%$ Sn incorporation and is nearly strain relaxed. Cross-sectional TEM shows a highly defective interface layer ~60 nm thick resulting...
in a nearly strain relaxed film. The sample surface is very smooth, with an RMS roughness of 1.2 nm as measured by AFM.

**DATA AVAILABILITY STATEMENT**

The datasets generated for this study are available on request to the corresponding author.

**AUTHOR CONTRIBUTIONS**

BC, GG, and AK contributed to conception and design of the study. BC, GG, MW, and SZ provided the analysis and interpretation of data for the work. BC wrote the first draft of manuscript. GG, SZ, and BC made substantial contributions to acquisition, analysis, and interpretation of data for the work. All authors contributed to manuscript revision, read and approved the submitted version.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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