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Preparation and study of tin and gallium thin films on silicon substrates

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ARTICLE

Preparation and Study of Tin and Gallium Thin Films on Silicon Substrates

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Abstract

In this research, the ideal procedures were obtained for growth epitaxial layers of tin and gallium solutions which are solid solutions, and the two solutions of the molten material were dissolved on a silicon substrate. The results were to obtain the lowest dislocation density of thin films. The relationship between the dislocation densities of thin films and the thickness of the thin film was an exponential relationship between its variable values. Thin films with a compound containing a smooth structure varied in order were obtained by smoothly changing the lattice parameters of the gradient gap solid solution, and an ideal structure of epitaxial layers of gallium on silicon substrate was also obtained.

Keywords: Thin films, Substrate, Lattice, Melt, Solution, Solid solution, Dislocation

1. Introduction

O ne of the ways to obtain epitaxial layers in semiconductor instrumentation is their crystallization from melt solutions, called liquid-phase epitaxy (LPE), which makes it possible to obtain them with different contents of composition components.

The solvent can be a melt of one of the main components of the crystallizing compound and a solid solution, for example, a melt of gallium in the preparation of Si_1-xGe_x epitaxial layers. They can also serve as a melt of a low-melting alloying element, for example, a tin melt in the preparation of tin-doped Si_1-xGe_x epitaxial layers.

At present, to create semiconductor devices, heterostructures based on solid solution of compounds of the $A^{IV}-B^{IV}$ class are used. Twocomponent solid solutions have the feature that it is possible to preserve a crystalline structure while varying Eg and crystal lattice period (a) of the films in a gradual and negligible manner along the direction of growth $[1-4]$ $[1-4]$ $[1-4]$ $[1-4]$ $[1-4]$. However, usually semiconductors and heterostructures of this class obtained on their basis have a significant drawback, which consists in a mismatch in the value of the parameter lattice (a) and the coefficient of thermal expansion (α) (CTE) of the substrate and

crystallized layer $(a_{5i} = 5.4307 \times 10^{-10} M_{\odot}$ $a_{\text{Ge}} = 5.66 \times 10^{-10}_{\text{M}}$, $a_{\text{Si}} = 5.1 \times 10^{-6} \text{ K}^{-1}$, $a_\mathrm{Ge} = 6.1 \times 10^{-6} \mathrm{~K}^{-1}$). Because of this, many defects are formed in the film.

When two- and multicomponent solid solutions are grown from the liquid phase, the following factors play an important role in formation of defects: composition inhomogeneity along the thickness of the epitaxial layers dC/dx; and the difference between the lattices period Δ a and CTE Δ a, conjugated at the material interface. In two component hetero-structures, defects are also heavily influenced by the inheritance of substrate defects. Charge carriers dissipate on defects, drastically worsening the properties of semiconductor devices built on their foundation.

To obtain a hetero-structure matched in terms of the crystal lattice period and CTE, it is necessary to switch to two-component smoothly varying gradedgap compositions of solid solutions.

The use of $Si_{1-x}Ge_x$ semiconductor solid solutions grown on silicon substrates makes it possible to control the band gap Eg , lattice period a, and CTE α , which is especially important for devices based on radiative recombination.

In this regard, the study of $Si_{1-x}Ge_x$ solid solutions grown on silicon substrates (which is a cheap

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semiconductor material compared to others) is very important.

The $Si_{1-x}Ge_x$ solid solution's characteristics, including the band gap and photo-sensitive spectral region, can be controlled by gradually modifying the composition. For instance, if solid solutions made of Si and Ge components are created, it will have a band gap of its own and will therefore have wider spectral photo-sensitivity ranges than Si and Ge separately. This is of great importance for the manufacture of photovoltaic devices, and also serves as a buffer layer for obtaining structures $Si-Si_{1-x}$ Ge_x $-(Ge_2)_{1-x}$ $(GaAs)_x Si-(Si₂)_{1-x-y} (Ge₂)_x (GeAs)_w$ and others [[5,](#page-5-0)[6](#page-5-1)].

In addition, for the intensive development of microelectronics, semiconductors are needed with much higher breakdown electric fields than those of silicon Si, band gap, higher electron mobility and high resistance to $T^{O}C$.

2. Materials and methods

The experiment was conducted utilizing chemically pure materials of Si, Ge, and Zn by ATS Optical Material Co., Ltd. in Taizhou. The substrate was formed of a silicon plate in the direction of <100>. A kind of K thermometer with four channels was used to determine T 0C during the experiment, performed in EPOS-style equipment (Pd-15Tpurifier) with hydrogen gas at $T^{O}C$ ranging from 1023 K to 1323 K. The weight of the substances was determined using an analytical balance manufactured by AE ADAM NBL 214e (Germany). The equipment was utilized to measure the component composition on the thickness of the crystals by Jeol-JSM-LV1059 (Japan).

3. Research method

On single-crystal Si substrate with a diameter of 40 mm and a thickness of about 400 m, we have grown epitaxial films of the Si1-xGex solid solution via liquid-phase epitaxy in a wide concentration range from a restricted Sn and Ga solution-melt $(1 > x > 0)$.

To grow the $Si_{1-x}Ge_x$ solid solutions, we used a quartz reactor of the vertical kind and prepared on horizontal substrates in an EPOS-type setup. The epitaxial layer was grown from a tiny volume of Solutions of Sn and Ga melt, in a hydrogen atmosphere purified by Pd, restricted by two substrates, allowing for a reduction in the amount of solutionsmelt consumed. First, pure hydrogen was circulating through the reactor for 20 min before the heating procedure started, after which a vacuum was first established inside the reactor to a remaining pressure of 10^{-2} Pa. The equipment

switched to automated mode as the T ⁰C touched the ideal level. For $40-60$ min, the melt solutions were homogenized. As soon as the graphite holder's substrates come into link with the melted solutions, they raised 1 cm a level over the solution by filling in the gaps between them with the melt solution. The correct moment was found to terminate the formation of $Si_{1-x}Ge_x$ epitaxial layers by centrifugally draining the melted solutions since the substrate. The state diagram of alloys that are binary $Ga-Si$, $Ga-Ge$ and $Sn-Si$, $Sn-Ge$ allowed for the determination of the composite of the melted solutions made up of Sn, Si and Ge, as well as Ga, Si and Ge,. The solubility of Ge in Sn and Si were investigated in T ⁰C variety of 450–1100 °C in order to prepare a liquid solution-melt [[7,](#page-5-2)[8](#page-5-3)]. The $Si_{1-x}Ge_x$ epitaxial films were produced at T^0C between 1100 °C and 500 \degree C, which corresponded to the beginning and finish of crystallization, respectively.

Experiments were conducted with a modification in the necessary parameters to determine how the composition, crystallization onset T^0C , and forced cooling rate affect the films' quality. According to studies, the best epitaxial films of the $Si_{1-x}Ge_x$ solid solution can be made by cooling the melted solutions at a rate of $1.5-2.5^{\circ}/$ minute through the thickness of melted solutions being $0.75 \div 1.5$ mm, or the width between two horizontally positioned substrates. As substrates, single-crystal Si (111) with n-type conductivity variation of $-(0°15' \div 0°30')$ was utilized.

A MIM-8M metallographic microscope was used to examine surfaces of the produced film's morphology. We have selected the composition of the etchant (we used mixtures concentrated hydrofluoric (HF), nitric (HNO₃), and (CH₃COOH) acetic, the ratios of acids are 1 h HF $+$ 3 h HNO3 $+$ 4 h CH3COOH). The sample was solution-etched, it diluted by deionized water, and then washed to expose dislocation etch pits on the (111) plane. $6-7$ counts were made on each layered sample to evaluate the dislocation density (N_D) .

4. Measurement results

The results of the experiment demonstrated that all technological factors, including growth T^oC , enforced cooling ratio, substrate orientations and purities, and solvent form, affect the dislocation density (N_D) of formed $Si_{1-x}Ge_x$ semiconductor solid solutions. We grow an epitaxial layer with grad gaps where the band gaps (ΔE) varies smoothly from silicon $(\Delta E_{Si} = 1.13 \text{ eV})$ to germanium $(\Delta E_{Ge} = 0.66 \text{ eV})$, starting from the Si-Si1-xGex substrate-film boundary, where silicon content

declines along the growth direction and the germanium content increases.

The outcomes of a morphological analysis investigation of the surface of four different film samples produced from Sn and Ga melted solution at $T^{O}C$ of the onset (T_{OC}) and termination (T_{TC}) of crystallization are shown in [Figs. 1 and 2.](#page-3-0)

[Figs. 1 and 2](#page-3-0) show that the relationship between the Ni concentration of the films and its thickness, and consequently, its composition content, is exponential and is given by $Ni = ae^{bd}$ (section 1 in the figures). Coefficient a and power coefficient b is two mathematical factors that depend on the chemical structure of the film because they depend on the amount of Si or Ge components in the solid solution:

 $a(x,y) = (\Psi_{i1}x + \Psi_{i2}y) \cdot 10^{\gamma},$ b $(x,y) = (\Psi_{i3}x + \Psi_{i4}y)$, $N_{i0}\left(x,y\right) = \left(\Psi_{i5}x + \Psi_{i6}y\right) \cdot 10^5$, and $\delta > 0 < \gamma$.

The coefficients ψ_{ik} and the degree γ , δ can be (-) or $(+)$ and are determined by experiment (i -sample (S) number, x, y - a solid solution's concentration of Si and Ge, respectively).

Utilizing both experimental results and mathematical calculations, it has been demonstrated that the N_D concentration in the films exponential reduces in the path of the solid solutions thickness, rises to a confirmed values (section 1 in [Figs. 1 and](#page-3-0) [2\)](#page-3-0), and then either stays nearly unchanged (section 2 and [Figs. 1 and 2\)](#page-3-0) or has an unnoticeable linear character to the surface [[9\]](#page-5-4).

The functional dependency in this situation is N_i $=N_i$ (d) could be expressed through dual analytical formulas on the interval (0; d_k] $N_i = ae^{bd}$, and on the interval $d_k n b s p$; d or $(d_k; d] - N_i = N_{i0}$, where N_{i0} is calculated from the plot using experimental results (see [Figs. 1 and 2\)](#page-3-0):

Fig. 1. The dislocation density (Ni) of solid solution (Sn-solvent) of the film as a function of the thickness (d).

Fig. 2. The dislocation density (Ni) of solid solution (Ga-solvent) of the film as a function of the film thickness (d).

 S_1 : $N_i = 114213e^{-0.092d}$ at $T_{OC} = 950^{\circ}$ C of Snsolvent;

 S_2 : $N_i = 979283e^{-0.092d}$ at $T_{OC} = 1100^{\circ}$ C of Snsolvent;

 S_3 : $N_i = 114235e^{-0.043d}$ at $T_{OC} = 850^{\circ}$ C of Gasolvent;

 S_4 : $N_i = 136578e^{-0.032d}$ at $T_{OC} = 1100^{\circ}$ C of Gasolvent.

The distribution pattern of the constituents over the depth of the epitaxial layer was established using the results of the X-ray microprobe analysis. [Figs. 3 and 4](#page-4-1) show how epitaxial Si layers are first grown on the Si substrate and that their content gradually decreases along the direction of growth. In response, the Ge content rises up to the film surface (this is made possible by the small volume of melt solution), forming a $Si_{1-x}Ge_x$ solid solution based on the growth mode. By varying $T^{O}C$ of the onset (T_{OC}) and termination (T_{TC}) of crystallization of the grown solid solution, we obtain Si_1-xGe_x films of different thicknesses, which have different degrees of vari-gap ([Figs. 3 and 4](#page-4-1)).

In our graded-gap $Si_{1-x}Ge_x$ solid solution, the composition of the films changed in thickness, so we can consider $d_i = f(x)$ (see [Figs. 3 and 4](#page-4-1)). Then, as a complex function, the dependency of dislocations Ni on composition x takes the following form: $N_i = ae^{bf(x)}$.

For a specific $Si_{1-x}Ge_x$ sample, in this case, the semi-empirical formula is expressed as how:

 $N_i = 114{,}213\ e^{-0.092(-0.0721\ x^2-0.0875\ x+101.09)}.$

With an accuracy of 9.8%, this calculation can be used to determine how much the film dislocation density is affected by the composition.

Following these tests, it was determined that the solid solution component content depends on the film thickness and this was known as the function

Fig. 3. The composition of solid solution (Sn-solvent) as a function of the thickness (d) of films.

 $di = f(x)$. The experiment demonstrated that while the formula is almost linear for film thicknesses (d) less than $20 \mu m$, it demonstrates a quadratic dependence as d increases. This shows that with an increase in $T^{O}C$ onset of crystallization $(T_{O}C)$ of the solid solution, due to silicon and germanium being highly soluble in the solvent at the beginning of development, the silicon content moves slowly in the direction of growth while the germanium content moves gradually up until the lowest T_{OC} .

During growth, single-crystal Si(111) were used as substrates. Growth is carried out from a partial melted solutions (here, the gap between the substrates, in which the melted solutions was located, was left unchanged $\delta = 1$ mm), so the composite of the $Si_{1-x}Ge_x$ stable solid solution modifies the growth direction and is graded-gap.

Si(111) single crystal substrates were used during the growing process. In this case, since there is only

Fig. 4. The composition of solid solution (Ga-solvent) as a function of the thickness (d) of films.

a small amount of solution-melt available for growth (in this case, the gap in the substrate where the melted solutions were located was left unchanged $\delta = 1$ mm), the composite of the $Si_{1-x}Ge_x$ solid solution is graded-gap.

The law of Vegard, which could be represented as a first approximation as a linear relation, states that the value of the crystal lattice constant of the semiconductor solid solution $Si_{1-x}Ge_x$ (where a_{Si1-x} , a_{Si} , and a_{Ge} were the solid solution's lattice parameters, Si and Ge) can vary smoothly into a particular variety of values according to the composite:

$$
a_{\mathrm{Si}_{1-x}\mathrm{Ge}_{x}}=a_{\mathrm{Ge}}-xa_{\mathrm{Ge}}+xa_{\mathrm{si}}.
$$

Due to this, we are able to avoid a sharp discrepancy between the lattice parameter on the Due to this, we are able to avoid a sharp
discrepancy between the lattice parameter on the
"substrate-film" structure as the substrate's epitaxial layer grows.

On a silicon substrate, observations and semiempirical mathematical computations have demonstrated that the dominant factor is T^O C onset of crystallization (T_{OC}) .

With an increase in $T^{O}C$ onset of crystallization (T_{OC}) , the solid solution's epitaxial layer contains a rise in dislocation density. It can be seen from the graph that the slope at different T_{OC} is almost the same, i.e. in the regular dependence of N_d on T_{OC} .

5. Conclusions

The experiments were carried out in a variety of solvents to decrease T_{OC} . The best regime by lesser dislocation (9 \times 10⁴–10⁵ cm⁻²), which satisfies the requirements of instrumentation, was obtained at disfocation $(9 \times 10 - 10 \text{ cm}^2)$, which satisfies the
requirements of instrumentation, was obtained at
the "substrate-lenka" interface in the T_{OC} mode = 900 °C from tin, in the T_{OC} mode = 800 °C from gallium melt solutions, on silicon substrate by < 111> orientation, forced cooling was applied in the 0.5 mm -1.5 mm gap between the substrates..

These experimental data can be used to obtain $Si_{1-x}Ge_x$ solid solutions on a silicon substrate with the lowest densities and dislocations with given electro-physical parameters in the manufacture of devices based on them.

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