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Optical Materials 27 (2005) 935–941
OPTICAL Materials
www.elsevier.com/locate/optmat

Ga-doping for β-$FeSi_2$ films prepared by molecular beam epitaxy

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Available online 12 October 2004

Abstract

β-$FeSi_2$ is expected as a new material for light emitting diode and optical sensor operating at 1.5 μm. However, doping techniques suited for various fabrication methods of β-$FeSi_2$ films have not been established. In this report, we present the results of Ga doping as a p-type impurity into β-$FeSi_2$ films using three molecular beam epitaxy (MBE)-associated growth methods. They are, (i) standard MBE method by depositing simultaneously Fe, Si and Ga molecular beams, (ii) alternating Fe/Si multilayers deposition method with an interval time between the Fe and Si depositions (migration enhanced epitaxy, MEE) in which Ga was deposited only in Si layer, (iii) standard alternating Fe/Si multilayers deposition (superlattice, SL) method in which Ga was deposited only in Si layer. All β-$FeSi_2$ films were prepared on Si substrates at room and elevated temperatures in MBE chamber and were subjected to post-annealing. Ga concentration was adjusted by changing its Knudsen effusion cell temperature or opening time of shutter for K-cell. Unintentionally doped β-$FeSi_2$ films prepared by SL method were n-type, having residual net electron concentration, $|N_D–N_A|$ of $\sim 3\times 10^{16}\,cm^{-3}$ and mobility of $\sim 400\,cm^2/V\,s$. When these β-$FeSi_2$ films were doped with Ga above a critical concentration, they exhibited p-type conductivity. By varying Ga concentration, net hole concentration, $|N_A–N_D|$ ranging from 7×10^{16} to $2\times 10^{18}\,cm^{-3}$ with hole Hall mobility, μ_h from 200 to $10\,cm^2/V\,s$ were obtained. These results demonstrate that Ga is an effective p-type dopant for β-$FeSi_2$ films to fabricate various electronic and optoelectronic devices.
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PACS: 78.66.Li; 68.55.Ln
Keywords: Ga-doped β-$FeSi_2$; MBE growth; MEE growth; SL structure; p-type conductivity; Hall effect measurements; Net hole concentration; Hall mobility

1. Introduction

β-$FeSi_2$ has a band gap energy of 0.83–0.87 eV (λ = 1. 3–1.5 μm) and a large optical absorption coefficient of $\sim 10^5\,cm^{-1}$. Using these features extensive studies have been started to fabricate light emitting diode (LED) and photosensors operating at 1.33–1.55 μm that are suited for optical fiber communication systems. Efforts have been launched to make extremely thin solar cells to cover rather longer wavelength [1,2]. It is a very attractive feature as a semiconductor that β-$FeSi_2$ can be epitaxially grown on Si substrate which leads to the integration of LEDs and photosensors on Si-LSI circuits, operating as optical interconnects [3–5]. For the growth of β-$FeSi_2$ thin films on Si substrate, various methods have been proposed and described in the last decade.

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doi:10.1016/j.optmat.2004.08.039

They are reactive deposition epitaxy (RDE) using the chemical reaction of thin Fe layer deposited on Si substrate to form eventually β-$FeSi_2$ layer [6–8], ion beam synthesis (IBS) using high energy ion-implantation with high temperature post-heat treatment [9], molecular beam epitaxy (MBE) using effused Fe and Si molecular beams [6,10], pulsed laser deposition (PLD) using ablated elemental Fe and Si, and compound $FeSi_x$ beam fluxes from appropriate alternative Fe/Si/β-$FeSi_2$ targets [11,12], metal-organic chemical vapor deposition (MOCVD) [13] and several sputtering methods including RF-sputtering [14] and face-target sputtering (FTS) methods [7,15]. These research have been mostly dedicated for the elucidation of crystallographical features β-$FeSi_2$ films [6–15]. To extract detailed optical and electrical features from β-$FeSi_2$, and further to make the aforementioned devices, one should make high quality β-$FeSi_2$ films. At present, there are however a number of technological difficulties to be overcome in terms of precise control of stoichiometry ratio between Fe and Si (1:2), surface and interface smoothness to make well-defined p–n junction, reduction of residual carrier concentration to a reasonably acceptable level ($\lesssim 1 \times 10^{16}\,\mathrm{cm}^{-3}$) and so forth. Among them, the most important research target crossing these multiple difficulties is to establish an impurity doping technology to fabricate tailored n-type and/or p-type thin layers with desired carrier concentration and maximum mobilities [16]. This technology should be also suited for the individually selected thin film fabrication method. Further, we began recently to notice peculiar difficulties associated with significant impurity diffusion of n- and p-type dopant atoms in β-$FeSi_2$ films. In this situation, we have also to remind that Fe atoms exhibit anomalous diffusion into Si [6]. It is interesting to note that the control of stoichiometry adjustment and the removal of dopant atoms together with achievement of epitaxial growth can be carried out by the formation of an appropriate template layer between depositing β-$FeSi_2$ layers and Si substrate [6–8]. Systematic studies on the impurity doping along the above purposes, particularly by developing customized and optimized doping technologies for a specific film growth method have not been presented. There are still numerous arguments dealing with the most appropriate methods to control conductivity type in β-$FeSi_2$. The fabrication of slightly Fe-rich and Si-rich films for both p-type and n-type conductivity is unrealistic to make devices due to its extremely uncontrolled stoichiometry nature [17].

Controversial discussions are on-going as for the sites of introduced doping atoms in β-$FeSi_2$, both experimentally and theoretically [18,19]. There are a few reports on the control of the conductivity type of β-$FeSi_2$ films by substituting Si by impurities, and particularly gallium (Ga) in β-$FeSi_2$, acting as acceptor. Practically bulk β-$FeSi_2$ crystal made by temperature gradient solution growth using Ga solvent and $FeSi_2$ solute at 900 °C, presented p-type conductivity with $|N_A–N_D| = 0.9–2.0 \times 10^{19}\,\mathrm{cm}^{-3}$, suggesting that some amount of residual Ga atoms in the bulk crystal act as acceptor impurities. Some works have been made for Mn, and Ni and Co atoms to be substituted at Fe-sites as p-type and n-type impurities, respectively [20,21].

In this report, we indicate the results of Ga doping as a p-type impurity that were prepared principally by using MBE technologies. The advantages of using MBE technologies are: (i) a high background vacuum pressure ($\gtrsim 10^{-8}$ Pa) that is ideal to reduce residual carrier concentrations, (ii) the Fe to Si stoichiometric ratio that can be tuned precisely in a wide range, (iii) doping time schedule and accordingly depth profile of dopant concentration that can be freely designed, (iv) in situ real-time monitoring of surface crystallographical structures (homo- and hetero-substances, β- and α-phases, orientations and so forth) through the observation of reflection of high energy electron diffraction (RHEED) is that can be accomplished, and (v) if necessary, in-site high temperature annealing that can be carried out without exposing the as-grown samples to the atmospheric ambient. Since there are many informative research reports as for the epitaxial growth mechanisms of GaAs by MBE, it is of great help to use them to obtain high quality β-$FeSi_2$ layers.

As techniques for impurity introduction, we developed three methods by varying doping time schedule to incorporate Ga atoms at desired depth position in β-$FeSi_2$ layers. Owing to our experimental results these methods allow the formation of alternating Fe/Si multiple layers and decide a powerful solution to adjust precisely Fe/Si stoichiometry. We varied widely the temperature of Knudsen cell for Ga effusion. We revealed that although Ga can be introduced effectively as p-type dopant with satisfying tunable $|N_A–N_D|$ and μ_h, the activation efficiency of the dopant atoms is very much dependent upon the doping method. We also discuss the surface morphology related to heavy doping of Ga atoms [10,22].

2. Experimental

In this research, n-Si(1 1 1) substrates with $\rho = 3681–6829\,\Omega\mathrm{cm}$ were used for the growth of β-$FeSi_2$ layers. Prior to the insertion of the substrates into the MBE deposition chamber, to form hydrogen-terminated layer, the cleaning of the surface was made by RCA washing and HF etching. Substrates were then introduced into the MBE chamber with background vacuum of $\sim 10^{-6}$ Pa. As Knudsen effusion cells, Al_2O_3 and p-BN crucibles were used for the evaporation of Fe and Ga atoms, respectively. For the evaporation of Si, e-gun was used but to reach a highly stabilized Si molecular

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