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Electrochemical deposition of PbSe and CdTe nanoparticles onto p-Si(100) wafers and into nanopores in SiO_2/Si(100) structure

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Abstract

Electrochemical deposition of PbSe and CdTe nanoparticles onto p-Si(100) wafers and into nanopores in SiO_2 layer grown thermally on p-Si(100) substrates (SiO_2/Si(100) structure) under illumination was studied. To produce nanopores we used SiO_2 layer with tracks developed by irradiation of 350 MeV Au ions. Pores structure was formed by chemical etching of the irradiated SiO_2 layer in dilute HF. The pores were shaped like truncated cones with a base diameter of 200 and 250 nm and height of 200 nm. Photoelectrochemical deposition of PbSe and CdTe was carried out with constant cathodic potentials from the water solutions containing Pb^{2+}, Cd^{2+} cations and H_2SeO_3, H_2TeO_3 acids. The potentials applied were more positive than the equilibrium E_{Me^{n+}/Me^0} redox potential. The underpotential deposition of Pb (or Cd) occurs only onto the co-deposited Se (or Te) atoms due to electrons photogenerated in Si substrate. The average sizes of PbSe and CdTe particles electrodeposited onto Si(100) wafers were dependent on the duration of electrodeposition, changing from 50 to 200 nm for PbSe and 30–80 nm for CdTe. According to XRD data, PbSe particles formed at ambient conditions had the crystalline structure. To deposit CdTe nanoparticles we used an electrolyte heated up to 80 °C. Variations in the electrodeposition time enabled one to form either separate chalcogenide particles or a polycrystalline film-like layer in case of electrodeposition onto p-Si(100) wafers and to control the degree of pore filling in case of electrodeposition into the nanopores of SiO_2/Si(100) structure.
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1. Introduction

PbSe is a narrow bandgap semiconductor (E_g=0.27 V at 300 K [1]). It is widely used for manufacturing of photodetectors, photoresistors and IR photoemitters (3–5.5 μm) [2–5]. PbSe can be formed as epitaxial or polycrystalline films, quantum dots, superlattices on various substrates: Si [1,4,6–9], PbTe [10], BaF_2 and CaF_2 [5,11–14], GaSb [15], etc. PbSe layers were epitaxially grown on PbTe, BaF_2 and CaF_2 single crystals.

PbSe deposition on the surface of Si and the formation of PbSe/Si heterostructures is of great interest from the practical point of view because silicon is the basic material for modern electronics. PbSe/Si heterostructures are formed by different methods including MBE [1,6–8,11,13,14,16–18], thermal evaporation in vacuum [3], liquid-phase epitaxy [19], pulsed laser deposition [9], etc. However, direct epitaxy of PbSe on Si substrates is rarely used due to a large lattice mismatch (11.9%) and also thermal expansion mismatch causing strains in PbSe layer when Si substrate is cooled from the growth temperature to the room temperature [4]. To make perfect epitaxial layer CaF_2 and BaF_2 are commonly used as intermediate layers to mitigate the lattice mismatch between PbSe and Si substrate [2,4,6,7,14,16]. A rare exception has been recently reported concerning high-quality p-type PbSe thin films grown directly on Si(111) and Si(100) substrates without any buffer layers using the pulsed laser deposition technique [9].

The electrochemical deposition technique takes a specific place among other growth methods. While requiring relatively simple and inexpensive equipment, it is energy-efficient, provides convenient process control by the

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electrode potential or current and is characterized by high ecological safety. Moreover, the majority of physical deposition methods fail to cover the inner surface of porous substrates, whereas electroplating has been successfully used for this purpose.

The works devoted to PbSe electrodeposition are few in number [20–27]. PbSe electrodeposition was realized onto polycrystalline Ti [20], SnO_2 [21], Au, Pt [22–24,27], single crystals of InP(111) and InP(100) [25,26]. Polycrystalline PbSe films with 20–40 nm particles were formed in the case of Au and Pt substrates [22–24]. And epitaxial PbSe film was grown on InP(111) surface [25,26].

Unlike PbSe electrodeposition, the deposition of CdTe has been studied extensively. The research activities carried out in the 1970s and in the 1980s are presented in the review papers [28,29] and in some later works [30–37] (see also references therein). Metallic electrodes (Au, Pt, Ti, Ni, stainless steel) are commonly used as substrates for CdTe electrodeposition, whereas semiconductor substrates are much less common [37,38]. Since the works devoted to electrodeposition of CdTe on Si are few [39–46], a study of CdTe electrodeposition on Si electrodes is of considerable interest.

In the present work, electrochemical deposition of PbSe and CdTe onto p-Si(100) wafers and into nanopores in SiO_2 layer formed on p-Si(100) under illumination has been studied. The use of p-type Si as an electrode enables electrodeposition by photoelectrochemical processes involving electrons photogenerated in the substrate.

2. Experimental procedure

PbSe and CdTe particles were electrochemically deposited onto the surface of single crystalline p-Si(100) wafers (B-doped with resistivity of 40 Ω·cm) and into porous SiO_2 layer thermally grown on p-Si(100) substrate. Before electrodeposition onto Si wafers (with linear sizes of $5\times5\times1$ mm^3) the latter were subjected to a preliminary etching in 6% HF solution to remove the native oxide and a subsequent 3-min etching in 40% NH_4F solution, resulting in the atomically flat, H-terminated Si surface.

To produce nanopores in SiO_2 layer we used tracks manufactured by irradiation of SiO_2/Si structures by 350 MeV Au ions. SiO_2 layer was thermally grown on B-doped p-type silicon (40 Ω·cm) with (100) crystallographic orientation. Pores were formed after chemical etching of the irradiated SiO_2 layer in dilute HF. Etching with 2.7% concentration of HF for 20 min at 20 °C caused the formation of pores randomly distributed over the surface and shaped like a truncated cones with the base diameters of 200 and 250 nm and height of 200 nm (Fig. 1).

Thin aluminum films ($d\sim0.1$ μm) as ohmic contacts were deposited by vacuum evaporation onto the back surface of Si electrodes. During electrodeposition procedure the aluminum contacts were protected from the electrolyte by a chemically resistant lacquer.

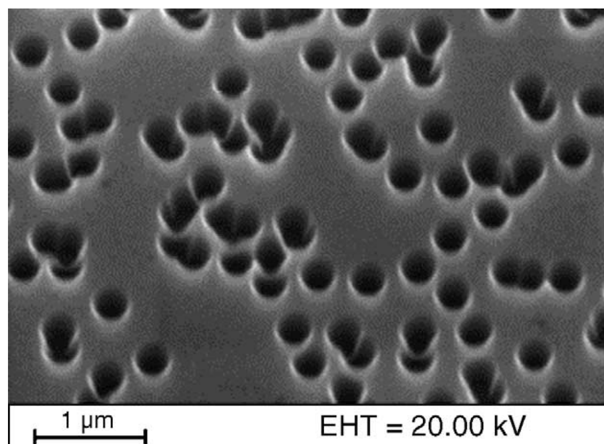

Fig. 1. SiO_2/Si(100) structures with nanopores produced by HF etching of the tracks created in the thermally oxidized silicon wafers by 350 MeV Au ions irradiation.

Electrochemical and photoelectrochemical measurements were carried out in a three-electrode two-compartment cell (20 cm^3) with an optical quality window, platinum counter-electrode and Ag|AgCl|KCl$_{(sat.)}$ reference electrode (+0.220 V vs. SHE). All potentials in this work are given with respect to this reference electrode. The potentials were controlled by a conventional programmable potentiostat. The potential scan rate was 0.02 V·s^{-1} in all potentiodynamic experiments. Before electrochemical measurements the dissolved oxygen was removed from the electrolyte using Ar bubble flow through the electrolyte during 10 min. The halogen lamp (50 W) with IR filter was used as a light source. The light flux power was $J=7$ mW·cm^{-2}.

PbSe deposition was performed from a water solution containing 50 mM $Pb(NO_3)_2$+2 mM H_2SeO_3+0.1 M HNO_3 at the constant potential ($E=-0.3$ V) and temperature of 20±2 °C. CdTe was electrochemically deposited from 1 M $CdSO_4$+1 mM H_2TeO_3+50 mM H_2SO_4 solution at $E=-0.55$ V and at temperature 80±2 °C. The solutions were prepared employing high purity reagents and double distilled water.

SEM and EDX investigations were carried out using LEO 1455 microscope (ROENTEC GmbH). X-ray diffraction analysis of the films was performed with HZG-4A diffractometer (Co–Kα radiation, Ni filter).

3. Results

3.1. Photoelectrochemical deposition of PbSe onto p-Si(100) wafers and into nanopores in SiO_2/Si(100)

Our investigations have demonstrated that PbSe electrodeposition from acid water solutions containing $Pb(NO_3)_2$ and H_2SeO_3 is possible at the applied potentials more positive than E_{Pb^{2+}/Pb^0} (so-called underpotential deposition—UPD) [22–24]. It was proposed to combine lead UPD and chalcogen deposition in overvoltage conditions (so-

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