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RF sputtering of epitaxial lead chalcogenide films in argon and krypton plasma

S P Zimin¹, I I Amirov² and E S Gorlachev^{1,2}

¹ Microelectronics Department, Yaroslavl State University, Sovetskaya Street 14, Yaroslavl 150000, Russia

² Yaroslavl Branch of the Institute of Physics and Technology, Russian Academy of Sciences, Universitetskaya Street 21, Yaroslavl 150007, Russia

E-mail: zimin@uniyar.ac.ru

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Abstract

The measurements of sputtering etch rates for monocrystalline (1 1 1)-oriented epitaxial films of semiconductor binary compounds PbTe, PbSe, PbS in RF high-density low-pressure inductively coupled argon and krypton plasma were performed. Films with 1–5 μ m thickness were grown on Si(1 1 1) and BaF₂(1 1 1) substrates using molecular beam epitaxy. Sputtering was carried out with the energy of Ar⁺ and Kr⁺ ions of 20–400 eV. The sputtering etch rates of the binary lead chalcogenides are demonstrated to have abnormally high values in comparison with the basic semiconductor materials of microelectronics. The sputtering yield values for PbTe, PbSe, PbS for the average energy of the argon ions of 200 eV are practically equal (0.46 ± 0.05 molecule/ion) and vary linearly with the variation of the ion energy. Substitution of the plasma discharge gas from the argon to krypton does not result in a significant change in the sputtering yield of lead chalcogenides. The physical principles of the observed phenomena are discussed.

1. Introduction

In recent years, the processes of etching and sputtering in plasma have been effectively applied in the technology of IV-VI semiconductors for formation of mesastructures, laser structures, thermoelectric elements, photonic crystals and other devices [1, 2]. Additionally, plasma techniques have proven successful in the analysis of a complex structure of superlattices [3] and the fabrication of micro- and nanostructure arrays [4, 5]. The application of plasma etching processes in distinction to commonly used wet etch processes allows one to fabricate anisotropic microstructures with low defect density. This is especially important during the fabrication of infrared (IR) emitters and photodetecting devices on the basis of IV-VI materials, which have a low threshold of radiation damage. Among the various optical materials of IR-optoelectronics, the etching processes of $Hg_{1-x}Cd_x$ Te materials are most studied (e.g. see [6]), while the number of works dedicated to plasma-assisted nanoand microstructurization of the surface of lead chalcogenides

is relatively small [1, 5]. Investigations of $Hg_{1-x}Cd_xTe$ material etching have shown that for the H₂/CH₄ reactive ion etching, their sputtering rate achieved 0.4 μ m min⁻¹ [7]. The close values of the etching rate (0.6–0.7 μ m min⁻¹) were obtained [2] during the etching of PbSnSeTe and PbTe films in $CH_4/H_2/Ar_2$ plasma. However, such high etching rates turned out to be typical not only for the plasma-chemical treatment, but also during the sputtering of these materials in argon plasma, when the material removal takes place due to the physical ion sputtering. The etching rates for $Hg_{1-x}Cd_xTe$ with an ion energy of 300 eV reached 1.0 μ m min ⁻¹ [7] and for lead telluride with an ion energy of 500 eV at 0.38 μ m min⁻¹ [8]. Obviously, the high etching rates of these materials are determined by the high sputtering yields of the components of these compounds, which are among the highest of all the elements [9]. These results show that in the fabrication of nano-, microstructures on the surface of lead chalcogenide materials, it is possible to apply the plasma treatment with an inert gas discharge. In this case the influence of the chemically active radicals of the plasma (CH, H, etc) on the fabricated structures can be avoided. From that standpoint, for the etching of mesa- and microstructures, the most attractive is the use of the high-density low-pressure radio-frequency inductively coupled plasma (RF ICP), which allows one to achieve higher etching rates even with the low ion energy. Recently, a sputtering rate reaching 0.9 μ m min⁻¹ in high-density Ar plasma with the average ion energy of 400 eV was demonstrated [4]. The aim of the present work is to investigate the RF ICP sputtering of PbSe, PbTe, PbS in Ar and Kr plasma and to determine the sputtering yields for the lead chalcogenide binary compounds depending on their composition and the energy of the bombarding ions.

2. Experimental details

Epitaxial films of binary compounds PbTe, PbSe, PbS grown by molecular-beam epitaxy on Si(111) or BaF₂(111) were investigated. In the former case for the lattice parameter mismatch compensation between the film and the Si substrate, the CaF₂ buffer layer with the thickness of 2–4 nm was used. The substrate temperature during the growth of the films was 350–400 °C. The thickness of the films of lead chalcogenides was 1–5 μ m. The layers had the orientation [111] along the growth axis and were characterized by a monocrystalline structure as confirmed by x-ray diffraction studies. Detailed information for the structural properties of the studied thin films is given in [4, 10, 11].

The experiments on the film sputtering were carried out in the RF ICP reactor as described in detail in [10]. The plasma reactor consisted of discharge and reaction chambers. Air evacuation of the chamber was performed using a turbomolecular pump. The maximum residual vacuum was 2×10^{-6} Torr. The pressure p of the inert gas (argon or krypton) was in the range of 0.07–0.98 Pa, and the gas flow Qwas varied in the range of 5-50 sccm. RF ICP was generated in the discharge chamber (f = 13.56 MHz, with inductive RF power P = 800 W) and in the magnetic field of two coils was transported to the reaction chamber, where the lower aluminum RF electrode with the surface area $S = 201 \text{ cm}^2$ was positioned and served as a sample holder for the studied samples, which were placed on it via the gateway device. The RF bias power applied on the electrode/substrate was $P_{\rm sb} = 0-400$ W (f = 13.56 MHz).

It is well known that in ICP discharges, it is possible to independently control the energy of the ions and the ion flux [12, 13]. The ion flux is determined by the RF power *P* applied on the inductor and the ion energy is controlled by the RF bias power P_{sb} applied on the substrate. The application of the RF bias power on the electrode results in the emergence of the negative self-bias potential U_{sb} , which controls the energy of the bombarding ions. The function of the ion energy distribution (IED) on the substrate surface for RF ICP has a double energy maxima and not a monoenergetic single-peak shape, as in the case of the constant negative potential on the electrode [14–16]. In general, the IED shape depends on the ion mass and the frequency of the applied RF field [14]. The experimental measurements of the IED in the RF ICP reactor showed that the self-bias potential is positioned



Figure 1. The negative self-bias potential on the substrate as a function of the applied RF bias power in Ar (open circles) and Kr (solid squares) plasma. P = 800 W, $Q_{Ar,Kr} = 10$ sccm, p = 0.08 Pa, $J(Ar^+) = 7.3$ mA cm⁻², $J(Kr^+) = 7.5$ mA cm⁻².

symmetrically between the two maxima of the IED curve [15, 16]. The average energy of the bombarding ions E_i in this case is determined as [13]

$$E_{\rm i} = e(U_{\rm p} - U_{\rm sb}),\tag{1}$$

where e is the elementary charge, and U_p is the plasma potential, which in current experiments was 25 V.

Another main characteristic of the plasma sputtering—the ion current density *J*—was determined from the dependence of the self-bias potential on the applied RF power. In the case of the RF ICP, when the entire RF bias power applied to the discharge is expended on the acceleration of the ions in the sheath, it can be assumed that $P_{\rm sb} \sim J U_{\rm sb}$ for the electrode with area *S* [13]. The linear dependence of the self-bias potential $U_{\rm sb}$ on the RF bias power $P_{\rm sb}$ in Ar and Kr plasma (figure 1) confirms this clause. Hence

$$J = \frac{\Delta P_{\rm sb}}{\Delta \left| U_{\rm sb} \right| S}.$$
 (2)

It should be noted that, of course, not the entire RF power is spent for the acceleration of the ions, since a certain percentage of it can be lost in a coupling device. It is believed that in the case of a full coupling, up to 95% of applied RF power can be transmitted to plasma [17]. A typical value of the ion current density *J* calculated using (2) during our experiments was in the range of 5.5–7.5 mA cm⁻², which corresponded to the value of the ion flux F_i of (3.4–4.7) × 10¹⁶ ion cm⁻² s⁻¹.

The sputtering etch rate of the films was determined by the etch step measurement using a Talystep profilometer. The etch step was obtained using a silicon wafer stencil mask. The etch step height depended linearly from the treatment duration in the time range of 30-120 s, indicating that the sputtering rate of PbSe, PbTe, PbS films was constant in time. Nonuniformity of the sputtering rate of the material of the samples along the radius of the sample holder did not exceed 5%. This is indicative of the uniformity of the ion flux incident on the electrode.

It is known that the treatment of the free-laying samples in plasma is accompanied with their heating to a high temperature. Reported measurements of the temperature of a Si wafer using a laser interferometric method showed that it is



Figure 2. Scanning electron microscopy image of the circular air holes fabricated using the RF ICP etching technique in the lead chalcogenide films (stage is at 70°).

heated to 500 K after 30 s treatment with the $P_{sb} = 200$ W [18]. Therefore, it was essential to experimentally test the influence of the sample temperature on the etching rate. We have found out that the etching step heights for the thermostated lead chalcogenide samples placed on a massive sample holder using vacuum glue and for the free-laying samples were identical, which indicates the independence of the sputtering rate from the temperature of the sample.

3. Results and discussion

It was experimentally confirmed that in the case of the initial thickness of the PbTe, PbSe, PbS films of $1-5 \mu m$, the substrate cannot influence the sputtering process. This did not allow us to differentiate the substrate type and the film thickness for the studied samples during further experiments.

Based on the numerous comparative measurements, it was established that the quantitative parameters of the obtained etching steps are adequately reproduced during treatment under the same conditions, indicating good stability of the applied etching technique. Figure 2 demonstrates an example of a PbSe film test structure on Si with an array of circular air holes with a diameter of 75 μ m and a depth of 1.7 μ m fabricated using RF ICP treatment.

The results of the experimental measurements of the sputtering etch rate V for the lead chalcogenide binary compounds during sputtering in Ar RF plasma with average Ar⁺ ion energy $E_i = 200 \text{ eV}$ (Q = 10 sccm, p = 0.08 Pa, $J = 5.5 \text{ mA cm}^{-2}$) are given in table 1, from which it follows that the sputtering rates of the studied materials are in the range of 8.8–10.4 nm s⁻¹ and are distinctly surpassing the sputtering rates for other well-known binary semiconductors. Thus, the value of the sputtering rate for PbTe, PbSe, PbS, reduced to the ion current density of 1 mA cm⁻², is in the interval of $1.6-1.9 \text{ nm s}^{-1}$. The reduced sputtering rates for Si [19], InN [20], GaN [21], InSb [22], and GaAs [23] for the Ar⁺ ion sputtering under similar conditions are noticeably smaller and are 0.02, 0.2, 0.4, 0.6, and 0.7 nm s^{-1} , respectively. High sputtering rates of lead chalcogenides and $Hg_{1-x}Cd_xTe$ (see the introduction) may be related to the fact that these narrowgap materials have lower damage threshold and greater defect density in comparison to wide gap semiconductors [24], as

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Table 1. Sputtering rates and sputtering yields for PbTe, PbSe, PbS in argon RF plasma^a.

Material	<i>V</i> (nm s ⁻¹)	$ ho (g cm^{-3})$	$M(g mol^{-1})$	Y (molecule/ ion)	E_{subl} (kcal mol ⁻¹) [30]
PbTe PbSe PbS	$\begin{array}{c} 10.4 \pm 0.3 \\ 8.9 \pm 0.3 \\ 8.8 \pm 0.3 \end{array}$	8.2 8.1 7.6	334.79 286.15 239.26	$\begin{array}{c} 0.45 \pm 0.02 \\ 0.44 \pm 0.02 \\ 0.49 \pm 0.02 \end{array}$	53.5 54.0 56.7

^a E = 200 eV, Q = 10 sccm, p = 0.08 Pa, J = 5.5 mA cm⁻², $F_i = 3.4 \times 10^{16}$ ion cm⁻² s⁻¹.

well as a relatively small sublimation energy in the case of lead chalcogenides, as will be discussed in detail below.

The main parameter describing the effectivity of the process of the interaction of ions with a solid substrate is the sputtering yield Y, which shows how many atoms are leaving the surface due to the impact of a single bombarding ion. For the experimental determination, the sputtering yield Y of a material consisting of one kind of atom is calculated using [25]

$$Y(\text{atom/ion}) = \frac{V\rho e N_{\text{A}}}{JM},$$
(3)

where ρ is the density of the studied target, N_A is the Avogadro constant and M is the molecular mass of the target. It is known that in the case of polyatomic targets, the overall sputtering yield Y in general will be determined by the sputtering yields and the concentrations of the atoms of each kind. There are many models to determine the sputtering yield of two or more component compounds as described in the most recent thorough review [26] and in [27–29].

It is important to mention that for the studied lead chalcogenide materials, a unique situation takes place, which noticeably simplifies the applicable physical models. In contrast to other compound semiconductors, for lead chalcogenides the values of the molecule dissociation energy in the crystalline and gaseous states exceed the values of the sublimation energy E_{subl} at 298 K [30]. As a result, during the sputtering of lead chalcogenides not individual atoms, but the entire molecules and molecular complexes are leaving the surface [31]. Additionally, it is well known that the evaporation of the lead salt compounds takes place predominantly in the form of a binary molecule and the degree of the dissociation is only a few percent [32].

In the case of the sputtering of the entire molecules for the determination of the sputtering yield *Y*, it is possible to modify equation (3) used for a single component substrate by inserting the values of the density and the molecular mass for PbX (X = Te, Se, S) compounds. The dimension of the estimated value under these conditions will be molecule/ion:

$$Y(\text{molecule/ion}) = \frac{V\rho_{\text{PbX}}eN_{\text{A}}}{JM_{\text{PbX}}}.$$
(4)

The values of density, molecular mass and sputtering yield for the energy of the primary Ar^+ ions of 200 eV for the studied lead chalcogenides are given in table 1. In the traditional sequence PbTe–PbSe–PbS with decreasing size and mass of the molecule, no noticeable difference in the sputtering yield takes place, and for all these three compounds, *Y* is in the narrow range of 0.46 ± 0.05 molecule/ion. This value is significantly smaller than the sputtering yields of Pb, Te, Se, S with argon ions, which for the ion energy of 200 eV equal approximately 1.4, 2.5, 2.8, 1.8, respectively [9]. The lower value of the sputtering yield of lead chalcogenides can be explained by the influence of the binding energy of the molecule on their sputtering yield [26]. Additionally, since the sputtering yields of the elements of the compound are higher than the sputtering yield of the compound itself, any segregation and accumulation of lead or chalcogen on the surface of the studied films should not take place during the sputtering process. This is confirmed by the results of the local chemical microanalysis of the surface performed using SUPRA 40 Carl Zeiss with the energy dispersive xray spectrometer Oxford Instruments INCAx-act, when the concentrations of lead and chalcogen on the surface of the initial and the plasma treated films remained approximately constant.

The obtained results of the close values of the sputtering yields for the binary lead chalcogenide compounds qualitatively can be explained in the framework of Sigmund's model [33] of the sputtering of a random monoatomic target in the linear collision cascade regime, based on transport theory. The applicability of this model in the case of the lead chalcogenide binary compounds is determined by the possibility of a simple substitution of the sputtered atoms of one kind by the molecules of one kind. For the sputtering yield with an ion energy of \sim 1 keV, Sigmund has obtained a general equation [33]

$$Y = \frac{0.042\alpha S_{\rm n} (E_{\rm i})}{U_0},\tag{5}$$

where α is the factor depending on the ratio of the masses of the target atoms and of the primary ion, $S_n(E_i)$ is the nuclear stopping cross section, U_0 is the surface binding energy, which is often substituted by the sublimation or cohesion energy. The replacement of the chalcogen X in PbX (due to a lower mass with respect to lead) does not significantly change the value of αS_n , and the values of U_0 of the lead chalcogenide binary compounds are close and differentiate at most by 6% (the values of E_{subl} at 298 K are given in table 1).

During the increase of the ion energy in the energy range of 200–400 eV the dependence $Y = f(E_i)$ had a near linear shape, which is typical for an interval of small energy primary ions in Sigmund's theory [33]. For $E_i = 400 \text{ eV}$ the sputtering yields of the PbTe, PbSe, PbS were still close to each other and laid in the range of 0.91 ± 0.10 . Close Y values for the Ar plasma sputtering for the studied compounds are explained by the similarity of the physicochemical and thermodynamic properties of lead chalcogenides and the small ratio of the mass of the primary ion to the mass of the sputtered molecule. That is why the sputtering rates of PbTe in Ar and Kr plasma are similar (figure 3), although the masses of Kr^+ and Ar^+ ions differ almost two-fold. In (5), the parameter α when replacing argon with krypton remains practically unchanged [33], since the ratio of the mass of the bombarding ion to the mass of the PbX molecule remains much smaller than 1. The parameter $S_n(E_i)$ in the framework of different known models [33] should also not change significantly, as a result



Figure 3. The sputtering rate of the PbTe film in Ar (open circles) and Kr (solid squares) plasma as a function of the applied RF bias power. P = 800 W, $Q_{Ar,Kr} = 20$ sccm, p = 0.15 Pa.



Figure 4. The sputtering rate of the PbTe (open circles) and the PbSe (solid squares) films in Kr plasma as a function of the applied RF bias power. P = 800 W, $Q_{\rm Kr} = 10$ sccm, p = 0.08 Pa, $J({\rm Kr}^+) = 7.5$ mA cm⁻².

of which the possible variation of the sputtering rate does not exceed the experimental error. Similar experimental facts of the invariability of the sputtering yield for Cu depending on the composition of the inert gas at low energies (<1 keV) are analyzed in detail by Sigmund in [33].

Further investigations of the processes of the sputtering in Kr plasma were carried out for the lead telluride and lead selenide films only. The dependence of the sputtering etching rate on the RF bias power for lead telluride and lead selenide binary compounds in krypton plasma had a linear shape, as shown in figure 4. The decrease of the sputtering rate for PbSe in comparison with PbTe at higher energies of Kr⁺ ions was observed in Ar plasma as well (see table 1). This is explained by the close sputtering yields of the materials and by minor differences in M_{PbX} in (4). It should be noted that without the RF bias power application, when the energy of the ions is less than 20 eV, the sputtering of PbTe does not take place. In contrast, a minor effect of the film surface swelling (by 40-60 nm) after 120 s treatment was observed. From these data we can conclude that the ion energy threshold for the sputtering of lead chalcogenides is 25-30 eV.

It is interesting to compare the obtained values of the sputtering rates in argon RF ICP with the rates of the physical Ar^+ ion sputtering according to the published data for (111)-oriented PbTe crystals [8, 34, 35], which were measured with the standard ion energy of 500 eV and the ion



Figure 5. The sputtering rate of the PbTe film (open circles) and the negative self-bias potential (solid squares) in Ar plasma versus the pressure with the constant power density of the ion flux of 0.5 W cm^{-2} .

current density of 1 mA cm⁻². In order to obtain V for RF ICP etching for such parameters, the value of $P_{\rm sb}$ was extrapolated from figure 1 for $-U_{sb} = 475$ V (self-bias potential corresponding to 500 eV ion energy) for the case of argon plasma, then it was normalized for the ion current density of 1 mA cm⁻² and finally used to determine the value of sputtering rate V in Ar from the approximation of data in figure 3. Thus, it was determined that for the ion energy of 500 eV and the constant ion density current of 1 mA cm^{-2} the sputtering rate for PbTe in RF plasma would amount to 5.4 nm s⁻¹, which is in the range 5.0–6.3 nm s⁻¹ of experimental results of PbTe(111) etch rates for Ar⁺ ion beam sputtering [8, 34, 35]. A comparison of the normalized sputtering rate of PbTe in the RF plasma with the ion energy of 200 eV and the ion current density of 1 mA cm^{-2} (as determined using table 1) with the data [34] for the ion sputtering showed that the rates of ion sputtering and plasma sputtering are almost identical and are 2.8 and 2.7 nm s^{-1} , correspondingly. It should be noted that during plasma sputtering with a low vacuum along with the inevitable desorption of the adsorbed water molecules, oxygen, carbohydrates from the reactor walls, their influence on the nature of the sputtering can be quite strong [28]. However, the present experimental results show that the presence of any trace atmospheric compounds in the plasma does not affect the sputtering rate of the material. In addition, the same sputtering rates for PbTe for the Ar⁺ ion beam and in the Ar RF plasma with the same ion current density as determined by (2) indicate the fairness to use (1) and (2) to estimate the parameters of the ion current density in RF ICP plasma discharge.

Figure 5 demonstrates the experimental dependence of the sputtering rate of the PbTe film and of the negative selfbias potential from the Ar pressure p. With the increase of the inert gas pressure from 0.07 to 1.03 Pa at a constant power density of the ion flux equal to 0.5 W cm⁻² ($P_{sb} =$ 100 W), the sputtering rate of the PbTe film remained almost constant. At the same time, the negative self-bias potential increased more than five times (from 60 up to 320 V), and the ion flux decreased 5.3 times. This is explained by the fact that with increasing pressure, the density of the ion flux to the electrode was decreasing. Therefore, at constant RF bias power, an increase in negative self-bias potential was observed. It should be noted that the RF power applied on the inductor was constant (800 W) with the increase of the discharge gas pressure, but the RF power applied on plasma from the inductor actually can be decreasing. The behavior of the sputtering rate suggests that the yield of the PbTe molecules depends linearly on the ion energy. This is confirmed by the linear dependence of the etching rate of the PbX materials from the energy of ions, which can be obtained from the data presented in figures 1 and 4. However, the simultaneous increase in ion energy and decrease in ion flux density still are not completely understandable, since for the low energies of the ions (20-200 eV) the sputtering yields of Pb and Te increase by more than ten times with an increasing ion energy [9]. Quite possibly, the constant sputtering rate of the PbX films takes place due to a synergetic effect of the influence of the energy and of the ion flux, i.e. etching of the film is determined by the power density of the ion flux.

4. Conclusions

The processes of the sputtering of the polyatomic targets in general have certain specific features associated with the different sputtering ability of the elements, which can result in a change in the chemical composition of the surface and in the processes of segregation. At the present time, due to the diversity of the occurring processes, there is no universal theory describing the sputtering of binary compounds and ternary solid solutions. At this stage the process of accumulation of experimental data is extremely important and will help to solve the problem of creating a universal theory of the sputtering of the polyatomic targets, including ternary and quaternary semiconductor solid solutions. A special feature of the etching of lead chalcogenide binary compounds in Ar and Kr plasma is the sputtering of the entire molecules, which nullifies the segregation processes in the surface layer and allows one to apply the classical models and formulae as used for the single-element targets for a qualitative analysis of the sputtering process of the PbX compound substrates. It is established that the sputtering yields for the lead chalcogenide binary semiconductor compounds PbTe, PbSe, PbS during the RF sputtering in Ar and Kr inductively coupled plasma are practically the same and do not depend on the plasma discharge gas. The sputtering rates of lead chalcogenides are depending linearly on the RF bias power. The same sputtering rate for PbTe in RF high-density low-pressure inductively coupled Ar plasma and with the Ar⁺ ion beam with the same ion current density, as estimated according to (2), indicates the adequate use of (1) and (2) for the estimation of the ion parameters of the RF plasma. High sputtering rates of PbTe, PbSe, PbS make the application of the RF ICP etching technique very promising for the formation of mesastructures in the thermoelectric and optoelectronic semiconductor device fabrication technology.

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