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EFFECT OF ARGON ION BOMBARDMENT ON THE COMPOSITION, ELECTRONIC STRUCTURE AND PHYSICAL PROPERTIES OF CADMIUM FLUORIDE

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Abstract. In the paper, the effect of bombardment with Ar^+ ions on the composition, electronic and crystal structure of the surface layers of bulk single crystal samples and CdF_2(111) films has been studied using the methods of Auger electron and ultraviolet photoelectron spectroscopy, high-energy electron diffraction and recording the angular dependences of the reflectance factor of inelastically reflected electrons. The effect of this bombardment on the density of states of valence electrons and energy band parameters of CdF_2 was investigated for the first time. The degree of disorder of CdF_2 into components and evaporation of fluorine from the surface layers was established to depend on the energy and dose of Ar^+ ions. The complete evaporation of F in the form of a diatomic gas was shown for the first time to be observed in the energy range of 1 – 2 keV at a saturation dose.

Keywords: epitaxial layer, heterostructures, ion bombardment, Auger spectrum, photoelectron spectrum, disordered layer, electron density of state

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ВЛИЯНИЕ БОМБАРДИРОВКИ ИОНАМИ АРГОНА НА СОСТАВ, ЭЛЕКТРОННУЮ СТРУКТУРУ И ФИЗИЧЕСКИЕ СВОЙСТВА ФТОРИДА КАДМИЯ

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Аннотация. В работе изучено влияние бомбардировки ионами аргона Ar^+ на состав, электронную и кристаллическую структуру поверхностных слоев объемных монокристаллических образцов и пленок фторида кадмия CdF_2(111). Для этого использованы методы оже-электронной и ультрафиолетовой фотоэлектронной спектроскопии, дифракции быстрых электронов и регистрация угловой зависимости коэффициента отражения неупругоотраженных электронов. Впервые изучено влияние указанной бомбардировки на плотность состояния валентных электронов и энергетические зонные параметры CdF_2(111). Установлено, что степень разупорядочения CdF_2 на составляющие и испарение фтора с поверхностных слоев зависит от энергии и дозы ионов Ar^+. Впервые показано, что полное испарение фтора в виде двухатомного газа наблюдается в области энергий 1 – 2 кэВ при дозе насыщения.

Ключевые слова: эпитаксиальный слой, гетероструктуры, ионная бомбардировка, оже-спектр, фотоэлектронный спектр, неупорядоченный слой, плотность электронных состояний

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Introduction

The great interest in epitaxial fluoride layers is associated both with the unique properties of the latter and with the wide potential possibilities of their application in opto- and microelectronics [1 – 13]. In particular, metal fluorides are widely used in the creation of special semiconductor – dielectric – semiconductor (SDS) structures in the three-dimensional integrated circuits. Of particular interest are CdF_2/Si(111) heterostructures with a CaF_2 buffer layer [9, 14, 15]. The minimum thickness of the CaF_2 buffer layer was 0.9 nm [5]. In this case, CaF_2 plays the role of a barrier layer for the chemical reaction between CdF_2 and Si substrates [9]. At the same time, trivalent germanium turned out to be the most promising for doping CdF_2 [16].

Single-crystalline cadmium fluoride is a solid dielectric that can be converted into a semiconductor by doping with donor impurities and subsequent heating in a reducing atmosphere [16 – 18].

In Refs. [19, 20], the energy position of the levels of rare earth (RE) elements in the band diagram of BaF_2 and CdF_2 crystals was determined. The role of RE^{3+} and RE^{2+} ions in the carrier capture, luminescence, and the formation of radiation defects was assessed. It was shown that the significant difference in the luminescent properties of BaF_2:RE and CdF_2:RE was due to the position of excited energy levels in the band diagram of the crystals. In Ref. [21], Shubnikov – de Haas oscillations and a quantum staircase of the Hall resistance were discovered in a p-CdF_2 quantum well limited by $CdB_xF_{2-x}\delta$ barriers on the n-CdF_2 surface. Thanks to the low effective mass of two-dimensional holes, the observation of the quantum Hall effect became possible at room temperature.

Studying the influence of various external influences, especially ion bombardment, on the composition, structure and physical properties of fluorides is of both fundamental and applied interest. In recent years, we have thoroughly studied the effect of ion bombardment on the composition, electronic and crystal structure, emission and optical properties of dielectric films and samples [12 − 27]. However, to date, the effect of low-energy ion bombardment on the composition and properties of CdF_2 films has been practically unstudied.

In this work, changes in the composition, electronic and crystal structure of CdF_2 (111) upon bombardment with Ar^+ ions were studied for the first time.

Experimental methods

The subject of research was a single-crystal sample of CdF_2 (111) with a thickness of about 0.5 mm and molecular beam epitaxial (MBE) films of CdF_2/Si(111) with a thickness of about 500 Å. Before ion bombardment, the samples under study were degassed at $T \approx 1000$ K for 3 hours in a vacuum (pressure $P \approx 10^{-7}$ Pa). The elemental and chemical compositions of the samples were determined by Auger electron spectroscopy (AES). The degree of amorphization of the CdF_2 film upon bombardment with Ar^+ ions and its crystallization during annealing, the type and parameters of the lattice were studied by high-energy electron diffraction (HEED) method and by measuring the angular dependences of the reflectance factor η of inelastically reflected electrons. To study the density of state of valence electrons and determine the parameters of energy bands, the method of ultraviolet photoelectron spectroscopy (UPS) was used. All measurements were carried out after the target was cooled to room temperature, in a vacuum with a pressure of at least 10^{-7} Pa. The choice of the (111) plane has been due to the fact that the CdF_2(111) surface has the lowest free energy ($E_{CdF2} \approx 5 \cdot 10^{-7}$ J·cm^{-2}, $E_{Si} \approx 1.35 \cdot 10^{-4}$ J·cm^{-2} and is atomically smooth.

Experimental results and their discussion

The Auger spectrum of a well-cleaned CdF_2(111) surface is shown in Fig. 1. It can be seen that the CdF_2 surface contains mainly an impurity of oxygen atoms with a concentration of no more than 1 at.%. The CdF_2 film surface has high crystalline perfection and an atomically smooth surface with a (1 × 1) structure reflection high-energy electron diffraction (RHEED) image (see inset in Fig. 1).

An analysis of the dependence of the intensity IF of the Auger peak from fluorine at an energy of 646 eV on the irradiation dose to the CdF_2(111) surface bombarded by Ar^+ ions with different energies E_0 (see Fig. 2) allows us to conclude the following. The intensive desorption of fluorine from the surface of CdF_2 occurs, starting from the irradiation dose $D = (1 - 5) \cdot 10^{13}$ cm^{-2} and up to $D = 10^{16}$ cm^{-2}; the rate of decrease in the intensity I_F depending on the energy E_0. In particular, for $E_0 = 0.5$ keV at $D = 5 \cdot 10^{16}$ cm^{-2}, the intensity of the Auger peak corresponds to a minimum, but I_F is not equal to zero even at $D = 10^{17}$ cm^{-2}, and thus $D = (4 - 5) \cdot 10^{16}$ cm^{-2} is the saturation dose D_s for $E_0 = 0.5$ keV. For $E_0 = 1.0$ keV, a decrease in I_F to zero is observed at $D = (6 - 8) \cdot 10^{16}$ cm^{-2}. A decrease in I_F to zero occurred up to $E_0 = 2$ keV. At E_0 more than 2 − 3 keV, the I_F value, even at dose D more than 10^{17} cm^{-2}, was above zero. Apparently, at high energies of Ar^+ ions, the decomposition of CdF_2 predominantly occurs in the surface layer and complete evaporation of fluorine atoms from these layers does not occur. Or, the evaporation of Cd and CdF_2 as a whole may begin simultaneously with the evaporation of fluorine atoms.

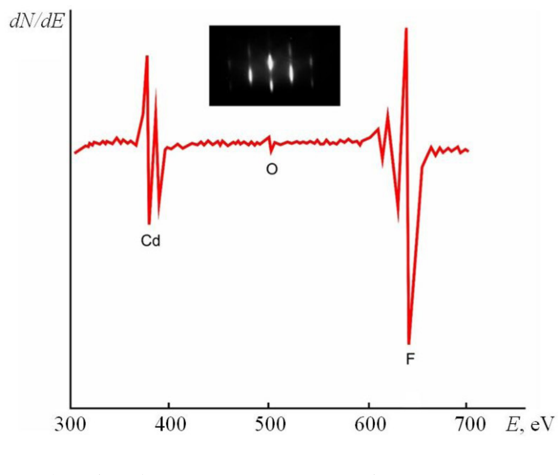

Fig. 1. The Auger spectrum and RHEED images (inset) of the pure CdF_2(111) surface

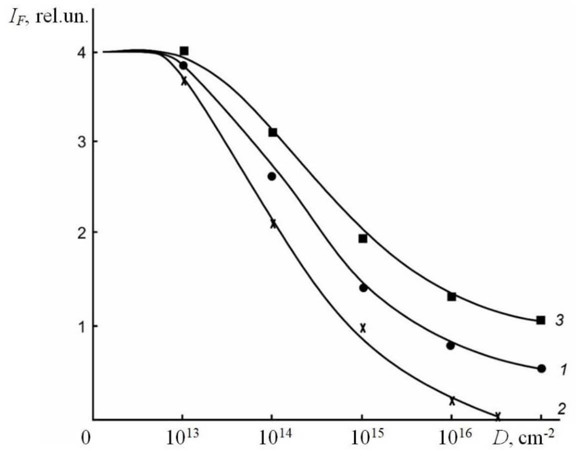

Fig. 2. Plots of the Auger F peak intensity (at E = 646 eV) versus the Ar^+ irradiation dose D for CdF_2 bombarded by Ar^+ ions with different energy values; E_0, keV: 0.5 (*1*), 1.0 (*2*), 2.5 (*3*)

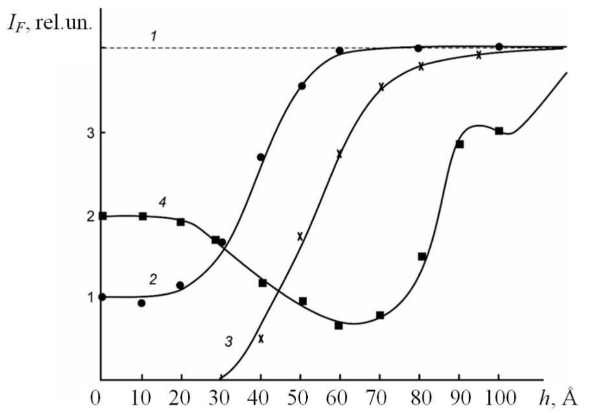

Fig. 3. The depth distribution profiles of F atoms from CdF_2 bombarded by Ar^+ ions at $D = D_s$ with different energy values; E_0, keV: 0.0 (*1*) 0.5 (*2*), 1.0 (*3*), 5.0 (*4*)

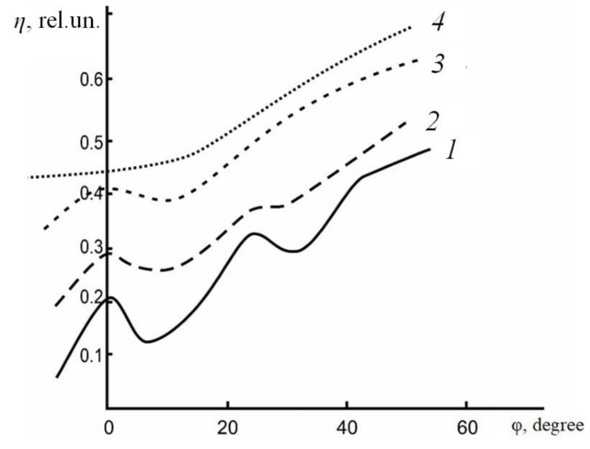

Fig. 4. The dependence of η on the angle φ (the angle of incidence of the primary beam) for amorphous CdF_2 films on Si(111) substrates; the film thickness values, Å: 10 (*1*), 20 (*2*), 40 (*3*), 50 (*4*). E_p = 0.8 keV

To answer this question, we studied the dependence of I_F on the depth h of a CdF_2 layer bombarded with Ar^+ ions with different energies at $D = D_s$ (Fig. 3). The plots in Fig.3 show that the intensity of the I_F peak decreases sharply (by ~ 4 times) at E_0 = 0.5 keV, and it practically does not change until the layer depth h = 20 – 80 Å, that corresponds to the projected range of Ar^+ ions.

Apparently, all F atoms in the form of diatomic gas F_2 evaporate from these layers. In the region $h \approx 25 - 50$ Å, the I_F concentration increases and, starting from $h \approx 50$ Å, the stoichiometric composition of CdF_2 is completely established. However, further studies showed that the CdF_2 layers were highly disordered to a depth of $h \approx 130 - 150$ Å. In the case of E_0 = 1.0 keV, the surface layers of the cadmium fluoride are completely decomposed into components to a depth of h = 30 – 40 Å, and almost all F atoms evaporate from these layers, and hence an amorphous cadmium film with a thickness of d = 30 – 40 Å is formed on the surface (see curve *3* in Fig. 3). When CdF_2 is bombarded by Ar^+ ions with E_0 = 5 keV, the greatest decomposition occurs at the depth of the projected range of Ar^+ ions ($h \approx 60 - 70$ Å). Apparently, most of the fluorine atoms go into vacuum, and the other part diffuses deep into the target. Therefore, the concentration of F increases significantly at a depth of h = 80 – 100 Å.

It is known that the thickness of disordered layers of a single crystal under ion bombardment is very difficult to determine experimentally. In this work, the depth of such layers was assessed by investigation of the angular dependences of the reflectance factor η of inelastically scattered electrons at the different primary electron energies E_p.

The dependences of η on the angle of incidence of the primary beam on the surface, for the Si(111) system with amorphous CdF_2 films of various thickness values are presented in Fig. 4. They were recorded at E_p = 0.8 keV. It can be seen that the main Si(111) peak is completely smoothed out at a film thickness of about 50 Å. A similar method was used to determine the thicknesses of the CdF_2 films at which the main Si peak was smoothed out in the range E_p = 1 – 10 keV. The results are given in Table.

Using the data from Table, the thickness of disordered layers d_p in the CdF_2(111) was estimated using the condition when bombarded with Ar^+ ions with different energies E_0 at doses $D = D_s$ (Fig. 5). This plot shows that

Table

The dependence of the thickness of the amorphous CdF_2/Si(111) films on the primary electron energies E_p

E_p, keV	0.8	1.0	3.0	5.0	10
d, Å	50	80	200	350	500

Footnote: d values were found under the condition when the main peak on the curve η(φ) was smoothed out at a given value of energy E_p.

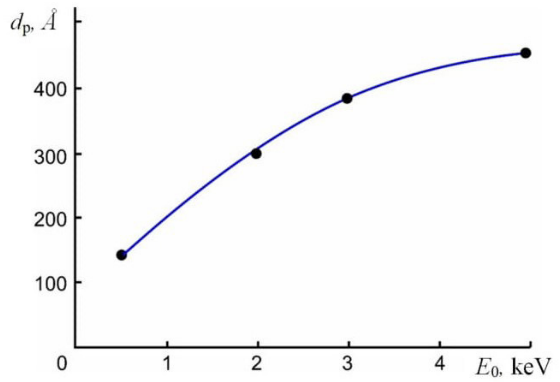

Fig. 5. A plot of the thickness of disordered layers versus the energy of Ar^+ ions

d_p increases exponentially from about 130 to 450 Å as E_0 increases from 0.5 to 5 keV.

The photoelectron spectra of a CdF_2/Si(111) epitaxial film before and after bombardment by Ar^+ ions at E_0 = 1 keV with different doses, taken at $h\nu$ = 21.2 eV (Fig. 6). These spectra provide information about the density of state of the valence band electrons, and the area under the energy distribution curve is proportional to the quantum yield Y of photoelectrons. The top of the valence band E_v of CdF_2 is taken as the reference point. In the spectrum of pure CdF_2, there are three clearly defined maxima (peaks) at energies E_{cv} = −1.6 eV, −3.8 eV, and −8.1 eV. It can be assumed that peak E_1 appears due to hybridization of the 5s level of Cd with the 2p level of F; the main contribution to the appearance of peak E_2 is made by 5s levels of Cd, and that to E_3 is made by 2p levels of F.

When CdF_2 film is bombarded by Ar^+ ions, a slight broadening of the curvilinear energy distribution of photoelectrons is observed at the dose D = 10^{14} cm^{-2}, and a decrease in the intensity is observed at the peak of E_{cv} = −1.8 and 8.1 eV. Also, the shift of the peak in the initial state of the spectrum (E_{cv} = 0.6 − 1.0 eV) to the right leads to

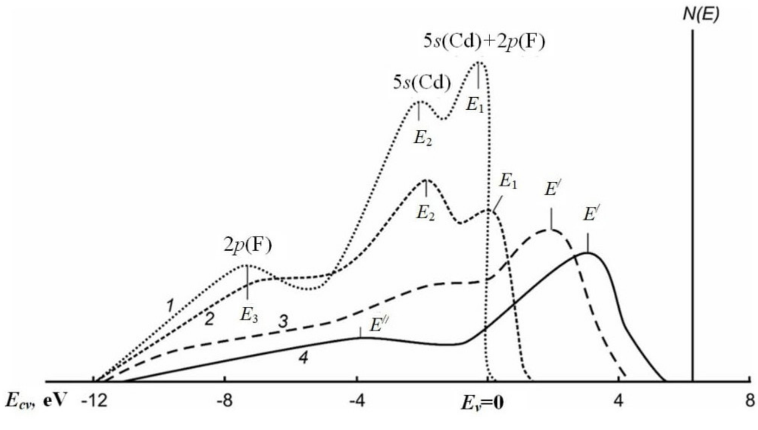

Fig. 6. The photoelectron spectra of the CdF_2 bombarded by Ar^+ ions with energy E_0 = 1 keV. Doses D, cm^{-2}: 0 (*1*), $1\cdot10^{14}$ (*2*); $5\cdot10^{15}$(*3*), $5\cdot10^{16}$(*4*)

a decrease in the gap width E_g of the CdF_2 film. At a dose of $5\cdot10^{15}$ cm^{-2}, a new peak E' characteristic of cadmium appears instead of E_1 and E_2 peaks, and the value of Y decreases by about 2 times in this case [19, 23].

Conclusion

In this work, the effect of bombardment with argon ions on the composition, electronic and crystal structure of the surface layers of single-crystal samples and CdF_2(111) films has been studied. The molecular beam epitaxial film of the CdF_2/Si(111) with a thickness of 500 Å was shown to have high stoichiometric and crystalline perfection with a surface structure of 1 × 1.

It was established that the intense desorption of fluorine atoms occurred, which continued up to dose $D = (5 - 10)\cdot 10^{16}$ cm^{-2}. The concentration of fluorine in the surface layers were found to go down to zero in the region $E_0 = 1 - 3$ keV, and not decrease to zero when E_0 being greater than 4 keV. The main mechanisms of these changes were clarified.

For the first time, the thickness of layers enriched with cadmium atoms and the thickness of highly disordered CdF_2/Si(111) layers were estimated. The change in the density of state of CdF_2 valence electrons upon bombardment by Ar^+ ions with $E_0 = 1$ keV was also studied for the first time using ultraviolet photoelectron spectroscopy (UPS) at different doses.

The results obtained in this work can undoubtedly be useful in the development of modern generation communications-electronics equipment.

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