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РЕСПУБЛИКИ КАЗАХСТАН

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X-RAY ANALYSIS OF SIC EPITAXIAL FILMS GROWN BY METHOD OF ATOM REPLACEMENT ON LOW DISLOCATION SILICON SUBSTRATE

Abstract. In this work, SiC films were synthesized by method of atoms replacement in the silicon lattice of on the surface of low dislocation silicon substrates M-5168 brand. By methods of X-ray diffraction, ellipsometry and profilometry, the surface roughness, phase composition, thickness and quality of SiC films synthesized through the substitution of atoms in high-resistance monocrystalline (111) oriented n-type silicon wafers in a mixture of gases CO and SiH_4, were studied. It is shown that the films contain the both nanocrystalline and single crystalline 3C-SiC layers with β-SiC crystallites of high degree of perfection. Dimensions of silicon carbide nanocrystals in the transition region "film-substrate" constitute values of 3 – 5 nm. Dimensions of large crystals of silicon carbide or monolayers reached values within 35 – 365 microns with a thickness of SiC films ~ 95 – 110 nm and the quantity of Si vacancies about 5 – 6.5 %. The results can be used in nano- and microelectronics and in the production of solar cells.

Keywords: thin films, silicon carbide, structure, crystallization, X-ray diffraction.

Introduction

Silicon carbide is a wide gap semiconductor which has a high thermal conductivity, hardness and high values of intensity breakdown of electric field. It is one of the most promising materials for use in the electronics industry. The physical and electrical properties of SiC led to great interest in electronic devices and sensors on the basis of silicon carbide for use in high temperature and radiation [1-4]. Amorphous and crystalline SiC films are also used in photovoltaic [5,6].

In recent years, it was theoretically developed and experimentally implemented a new method of growing thin low defective SiC films on Si [7-9]. The method is based on the replacement of matrix part of silicon atoms to carbon atoms with formation of silicon carbide molecules: 2Si + CO = SiC + SiO. SiC films were synthesized in special equipment described in [9]. SiC films investigated in [8,9] were grown on standard silicon substrates p- and n-type conductivity. In [8] it was shown that the higher quality of the original substrate Si, the higher the quality of the grown SiC layer structure. In this regard, in this study there was investigated the formation of SiC films on the surface of low defective M-5168 grade silicon substrates.

Experiment

For this purpose, a series of films was prepared (# I), grown at 1250 ° C and at pressure of CO gas at 264 Pa low dislocation silicon surface. The growth time of these films was 15 min. Another series of II films was synthesized for 7 minutes at a temperature of 1330 ° C and at gas pressure of 395 Pa CO [10].

The substrates were silicon wafers of high quality n-type (111) with a resistivity of 1987 - 3165 ohm·cm, 1300 microns in thickness and 20 mm in diameter. By bilateral grinding and polishing there been removed 100 mm on each side of the silicon wafer. Further II Series samples were subjected to chemical

etching in acid mixture in a ratio of HF:HNO3 = 1:10 to 870 micron, and then in an alkaline KOH solution. Samples of I series were subjected only in the alkaline etching solution KOH.

The roughness of the films was investigated with the help of the profilometer NewView 6000 (company Zygo). Phase composition and structure of the films were studied by a highly sensitive photographic X-ray diffraction using narrow collimated (0,05×1.5 mm^2) monochromatic (CuKα) X-rays directed at an angle 5° to the sample surface [11, 12]. The intensity of X-ray reflections was measured every 0,1° on MD-100 microdensitometer. In order to determine the physical parameters of the films we used ellipsometer M-2000D J.A. Woollam, which allows reading the ellipsometric spectra in the range of 0,7 - 6,5 eV.

Results

As research shows, the roughness on the profilometer NewView 6000 (company Zygo), at considerable area of silicon 701 × 526 mm (Fig. 1 a), in the treatment of the mixture of I series sample acids leads to an increase in the average surface roughness R_a of the silicon surface in the 88.5 / 3 7 = 24 times and an average surface roughness of synthesized silicon carbide films on I series sample estimated to 21.168 / 5.684 = 3.72 times. Thus, chemical treatment in a mixture of HF acid: HNO_3 removes the deep scratches (Figure 1 b), but leads to increase the average surface roughness R_a (figure 1).

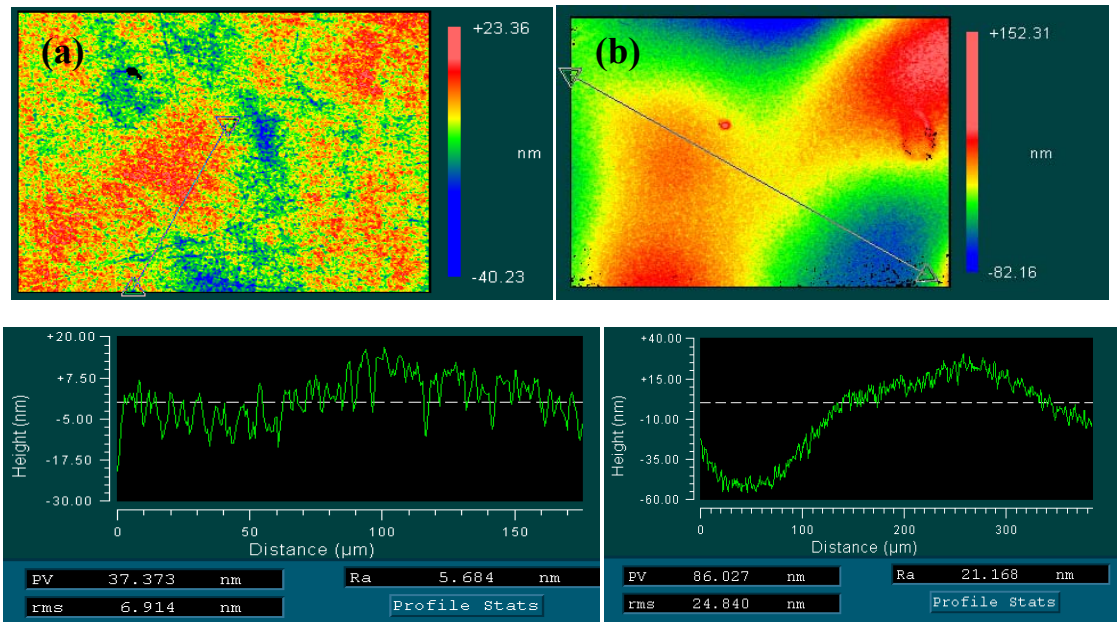

Figure 1 – The surface profile and roughness of the SiC films of II series sample (a) and II series sample (b) at areas 350×263 microns

Figure 2 a, b clearly shows the X-ray debayegrams for the samples of SiC series I and II contain almost all of the known β-modification of silicon carbide (3C-SiC).

With Jones method [13] of the x-ray line broadening (Fig.2 c) by the Scherrer formula (1) [14] we determined average size of β-SiC nanocrystals in different planes:

$$\varepsilon = \frac{R\lambda}{\beta \cdot \cos\theta}, \qquad (1)$$

where ε – average crystallite size (nm); R = 2,86 cm – camera radius (cm); λ = 0,1540 nm – wavelength of CuK_α-ray (nm); θ – Bragg angle; β – line broadening. The value of the x-ray line broadening β is determined from meaning $\beta = \sqrt{\beta_S \beta_W}$ [15], $\beta_S = B - b$ [14], $\beta_W = \sqrt{B^2 - b^2}$ [16], where B – half-width of the X-ray line adjusted for doublet line CuK_α, b – instrumental half-width component of line.

Nano crystallite size SiC for sample I comprised values of 4,5 nm in planes (111), 3,1 nm in planes (220) and 3,0 nm in planes (311).

As it was shown in [9], SiC layer consists of SiC film layer covering triangular and shrink pores. At larger value of super saturation, the critical radius of nucleus pore has a size of a few nanometers, the critical pore radius shrinkage is of the order of atomic dimensions. This means that SiC embryo is surrounded by vacancy clusters that can merge into thin cracks surrounding the seed embryo. [9] Thus, measurement data on the nanocrystals of silicon carbide indicate that SiC nucleation sizes in the transition region "film-substrate" constitute value of 3-5 nm.

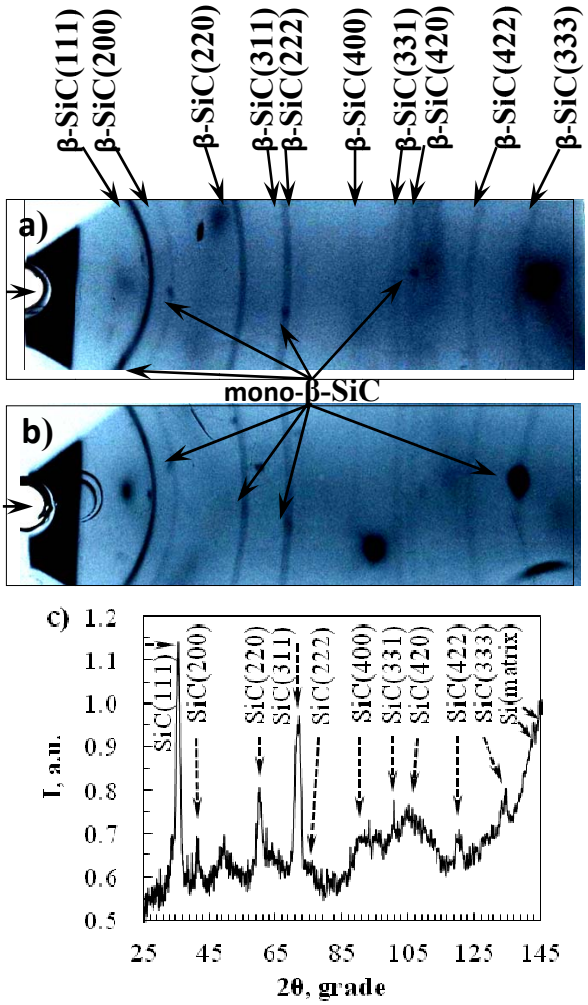

Figure 2 – X-ray powder patterns of thin film of silicon carbide I (a) and II (b)
and the intensity of the X-ray reflections *I*(2θ) (c), synthesized by substitution of atoms

Some lines of nanocrystalline phase β-SiC show single reflex overlay resulting from a large crystal reflection of silicon carbide formed in the area of X-ray beam insertion into narrow collimated surface. The method of Clark and Zimmer, based on the measurement of the size of spots and described in [15] is used, according to this method, changes in reflex sizes from 0.20 to 1.20 mm corresponds to a linear change from 0.010 to 0.085 mm grain size. With this method we determined size of β-SiC large crystals. For a sample of I series, sizes of large crystals or monolayers of silicon carbide totaled value of 130×35 mm in the plane (111), 70×60 mm in the plane (200), 85×70 mm in the plane (311) and 60×85 mm in the plane (420). In contrast, for the sample of II series, we observed reflex, comparable in size to the reflex of the Si substrate and the beam size. This reflex, which lies on a line β-SiC (333) corresponds to a crystal 365×220 mm and indicates the presence of the layer of β-SiC monocrystalline. Thickness of SiC films synthesized by substitution of atoms not normally exceed 100-150 nm [9].

Fig. 3 a, b shows elliptical plots, in other words dependence of ε_1 real and imaginary ε_2 parts of samples I and II series of the SiC film. Elliptical plots show that there are differences in structure of the SiC films of samples I and II series. According ellipsometric spectra of SiC layer thickness is approximately on a sample of series I - 95 nm, on a sample of series II - 110 nm. Calculations using ellipsometric model [17] have shown that series of samples I contain only 5% Si vacancies, in the samples of II series - about 6.5%.

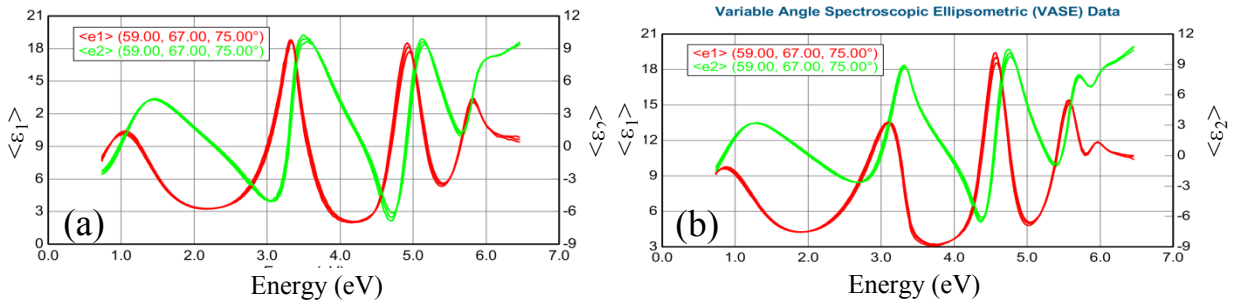

Figure 3 – Dependence of ε_1 real and $\varepsilon_2$imaginary parts of dielectric permeability of films SiC / Si (111) from the photon energy for the samples of Series I (a) and II (b)

Conclusion

With the help of the method of substitution of atoms in the silicon lattice we synthesized two series of SiC samples on the surface of the low defective silicon substrate of n-type (111) orientation (mark M-5168) grown in a mixture of gases CO and SiH4. A series of samples (№ I), was synthesized at a temperature of 1250°C and at gas pressure of CO 264 Pa on the narrow dislocation silicon surface. The growth time of these samples was 15 min. Another series of samples (№ II) was synthesized for 7 minutes at a temperature of 1330°C and at gas pressure of CO 395 Pa.

With the help of the method of profilometry it was found that treatment of HF acid in a mixture: HNO3 = 1: 10 silicon substrate results in the removal of deep scratches, polishing their surfaces. On the other hand, the chemical treatment causes etch pits and the increase in roughness in the whole surface.

With the help of the method photographically XRD it was showed that the synthesized film comprises a single crystal, nanocrystalline layers and β-modification of silicon carbide (3C-SiC). Dimensions of nanocrystals of silicon carbide in the transition region "film-substrate" constitute values of 3-5 nm. The dimensions of large crystals of silicon carbide or monolayers reached values within 35-130 microns up to 365 microns.

Calculations performed using ellipsometric model [17] showed that samples of I series only contain 5% of Si vacancies and in series of II samples - about 6.5%. According to ellipsometric spectra, the thickness of SiC layer on the sample of I series is 95 nm, on a sample of series II is 110 nm.

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АҚАУЫ АЗ КРЕМНИЙ МАТРИЦАЛАРЫНДАҒЫ АТОМДАРДЫҢ ОРНЫН БАСУ ӘДІСІМЕН АЛЫНҒАН ЭПИТАКСИАЛДЫ SiC ҚАБЫРШАҚТАРЫН РЕНТГЕНДІК ТАЛДАУ

Аннотация. Ақауы аз М-5168 маркалы кремний матрицаларының бетіне, кремнийдің кристалдық торындағы атомдардың орнын басу әдісі арқылы SiC қабыршақтары синтезделді. Рентгендік дифракция, профилометрия және эллипсометрия әдістері арқылы, CO және SiH4 газ қоспаларының атмосферасында жоғарыомды монокристалды n-типті Si(111) матрицада синтезделген SiC қабыршақтарының фазалық құрамы, қалыңдығы және сапасы зерттелінді. Синтезделген қабыршақтар құрамында, жетілу дәрежесі жоғары β-SiC кристаллиттеріне ие монокристалды және нанокристалды 3C-SiC қабаттар бар екендігі көрсетілді. «Қабыршақ-матрица» өтпелі аймақтағы кремний карбиді нанокристалдарының өлшемдері 3 – 5 нм құрайды. SiC қабыршақ қалыңдығы ~ 95 – 110 нм және Si вакансия мөлшері ~ 5 – 6,5 % болған жағдайда, кремний карбидінің ірі кристалдарының немесе моноқабаттарының өлшемдері 35 – 365 мкм құрайды. Жұмыстың нәтижелері нано- және микроэлектроникада, күн элементтерін өндіруде пайдаланылуы мүмкін.

Түйін сөздер: жұқа қабыршақтар, кремний карбиді, құрылым, кристалдану, рентгендік дифракция.

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РЕНТГЕНОВСКИЙ АНАЛИЗ ЭПИТАКСИАЛЬНЫХ ПЛЕНОК SiC, ВЫРАЩЕННЫХ МЕТОДОМ ЗАМЕЩЕНИЯ АТОМОВ НА ПОДЛОЖКАХ НИЗКОДЕФЕКТНОГО КРЕМНИЯ

Аннотация. В работе на поверхности низкодефектных подложек кремния марки М-5168 синтезированы пленки SiC методом замещения атомов в решетке кремния. Методами рентгеновской дифракции, эллипсометрии и профилометрии исследованы шероховатость поверхности, фазовый состав, толщина и качество пленок SiC, синтезированных в высокоомном монокристаллическом кремнии n-типа ориентации (111) в смеси газов CO и SiH4. Показано, что синтезированные пленки содержат в себе монокристаллический и нанокристаллические слои 3C-SiC с кристаллитами β-SiC высокой степени совершенства. Размеры нанокристаллов карбида кремния в переходной области «пленка-подложка» составляют величины 3 – 5 нм. Размеры крупных кристаллов или монослоев карбида кремния составили величины в пределах 35-365 мкм при толщине пленок SiC ~ 95 – 110 нм и количестве вакансий Si ~ 5 – 6,5 %. Результаты могут быть использованы в нано- и микроэлектронике, в производстве солнечных элементов.

Ключевые слова: тонкие пленки, карбид кремния, структура, кристаллизация, рентгеновская дифракция.

CONTENTS

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Social sciences

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