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Photoemission studies of initial oxidation for ultra-thin zinc film on 6H-SiC(0 0 0 1) surface with synchrotron radiation

C.W. Zou [a,b], Y.Y. Wu [a], B. Sun [a], P.S. Xu [a,*], H.B. Pan [a], F.Q. Xu [a]

[a] National Synchrotron Radiation Lab, University of Science and Technology of China, Hefei 230029, People's Republic of China
[b] Wuhan Institute of Physics and Mathematics, Chinese Academy of Sciences, Wuhan 430071, People's Republic of China

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Abstract

The thermal oxidation process of metallic zinc on 6H-SiC(0 0 0 1) surface has been investigated by using atomic force microscopy (AFM), synchrotron radiation photoelectron spectroscopy (SRPES) and XPS methods. The AFM images characterize the surface morphology of ZnO film formed during the thermal oxidation and SRPES record the valence band, Si 2p and Zn 3d spectra at different stages. The O 1s peak is recorded by XPS because of the energy limit of the synchrotron radiation. Our results reveal that the silicon oxides layer of SiC substrate can be reduce by hot metallic zinc atom deposition. The oxygen atoms in the silicon oxides are captured by the zinc atoms to form ZnO_x at the initial stage and as a result, the oxidized SiC surface are deoxidized. After the zinc deposition with the final thickness of 2.5 nm, the sample is exposed in oxygen atmosphere and annealed at different temperatures. According to the evolution of peaks integrated intensities, it is considered that the Zn/SiC system will lose zinc atoms during the annealing in oxygen flux at high temperature due to the low evaporation temperature of pure zinc. After further annealing in oxygen flux at higher temperature, the substrate is also oxidized and finally the interface becomes a stable SiC–SiO_x–ZnO sandwich structure.
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1. Introduction

In recent years, ZnO has attracted considerable attention all over the world for its many potential applications due to its special characteristics [1–5]. For example, ZnO is a pressure-sensing ceramic and can be used as nonlinear varistor. ZnO with high resistivity and perfect *c*-axis orientation texture shows a piezoelectric effect and thereby can be applied on surface and bulk acoustic wave devices. Due to its high thermal and chemical stability, direct wide band gap (~3.3 eV at RT) and large exciton binding energy (~60 meV), this material is also suitable for UV laser-emitting devices operational at high temperatures and extreme radiation conditions.

Many traditional techniques such as pulsed laser deposition (PLD), metal organic chemical vapor deposition (MOCVD) and molecular beam epitaxy (MBE) have been employed in the preparation of ZnO thin films [6–8]. Recently, an easy and useful method for ZnO preparation by thermal oxidation of metallic Zn has been applied and obtained great success [9–14]. This method can prepare high-quality ZnO thin film with excellent crystallinity and strong UV emission even at low temperature on SiC substrate for their close lattice parameters and expansion coefficient. However, the initial oxidation process of this method on the SiC substrate has not been investigated.

In this paper, we study the oxidation process of thin metallic zinc film on the 6H-SiC substrate with different temperatures by using atomic force microscopy (AFM), synchrotron radiation photoelectron spectroscopy (SRPES) and XPS. The AFM images characterize surface morphologies, while SRPES and XPS results reveal the evolution of valence bands and core levels during the oxidation process.

2. Experimental

The experiment is performed at the surface physics station of National Synchrotron Radiation Lab (NSRL) in Hefei, China.

* Corresponding author. Tel.: +86 551 3602037; fax: +86 551 5141078.
E-mail address: psxu@ustc.edu.cn (P.S. Xu).

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This experiment station is mainly composed of a three-chamber VG multi-technique UHV system. It consists of a deposition chamber, a pre-treatment chamber with a fast entry lock and an analysis chamber equipped with ARUPS10 hemispherical analyzer. The base vacuum of the whole system is better than 2×10^{-10} mbar. Sample can be cleaned by cycles of Ar^+ ions etching and e-beam annealing in the pre-treatment chamber and transferred to the analysis chamber for SRPES. The beamline covers the energy range from 10 to 300 eV and the energy resolution ($E/\Delta E$) is better than 1000. More details of the experiment station and the related beamline are described elsewhere [15].

The substrate in this experiment is 6H-SiC single crystal from Cree. Research. To obtain clean surface, the sample is treated chemically using the conventional organic solvents and then dipped into the HF solutions particularly to remove the silicon oxides. Then the sample is blown dry by pure N_2 and immediately introduced into the UHV chamber followed by degassing at 550 °C for about 5 h with e-beam heater. After above treatment clear spots are observed in low energy electron diffraction (LEED) pattern. High purity zinc threads (better than 99.99%) are evaporated in the K-cell at 200 °C in the deposition chamber and the evaporation rate is about 1 Å per 16 s measured by quartz crystal thickness monitor. The total film thickness is measured to be around 2.5 nm. Then the zinc films are exposed in oxygen flux with the pressure of 2.0×10^{-6} mbar and annealed at 180 and 600 °C for 10 min, respectively. At each stage the sample is transferred to the analysis chamber for SPRES and XPS measurement to evaluate the chemical reaction process. In the specific experiment, SRPES record the valence band (including Zn 3d peaks) with photon energy of 32 eV and Si 2p spectra with photon energy of 150 eV, respectively. The O 1s peak is recorded by XPS with Mg Kα line because of the energy limit of our synchrotron radiation source. *Ex situ* AFM investigations are also performed to characterize the final surface morphology of the sample.

3. Results and discussion

Chemically cleaned 6H-SiC substrate surfaces are investigated by contact-mode AFM just as shown in Fig. 1(a). The AFM image is really dim in looking with minimal nano-scale scratches and quite consistent with the report of Yao and his co-workers [16]. Actually the chemically cleaned SiC surface are not atomic-ordering and oxygen-free surface though it can express quite clear LEED pattern. The extreme clean and ordered surface for 6H-SiC(0 0 0 1) plane should be obtained by annealing the sample in Si flux under UHV conditions [17–19]. However, in the method of thermal oxidation of metallic Zn for ZnO preparation, only chemically cleaned 6H-SiC substrate is used. So here we just investigate the SiC treated by HF solutions. Fig. 1(b) shows the final surface morphology of ZnO film grown on the SiC substrate prepared by thermal oxidation of metallic zinc at 600 °C. Few irregular islands remain while large blurry areas almost cover the whole substrate region. These blurry areas are regarded as the formation of ZnO micro-crystal. Our XRD measurement also proves the formation of polycrystalline ZnO film though the intensities of related diffraction peaks are quite small. It is perhaps due to the thickness of ZnO films.

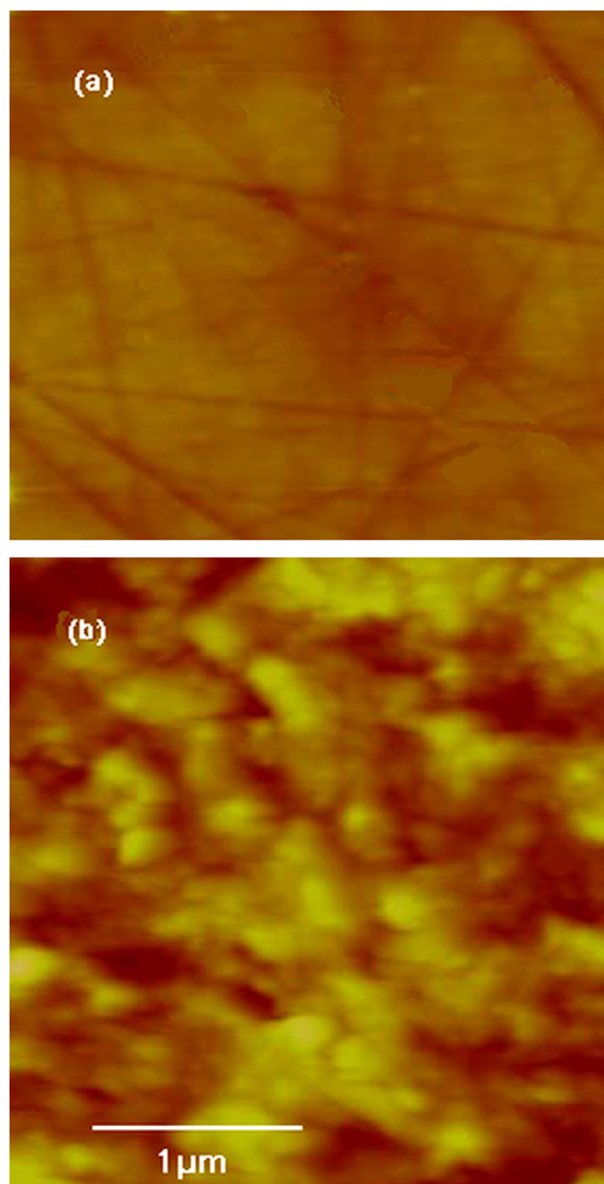

Fig. 1. The AFM images for 6H-SiC substrate treated by chemical method (a) and the ZnO thin film prepared by thermal oxidation of metallic Zn at 600 °C (b).

Fig. 2 shows the valence band spectra at different stages with photon energy of 32 eV. This picture clearly reveals the evolution process during the formation of ZnO film. When metallic zinc of 0.5 nm is deposited on 6H-SiC surface, a broad peak located at −10.0 eV appears and it is regarded as Zn 3d level. When further metal deposition is up to 2.5 nm, the valence band of substrate is almost depressed by the Zn 3d spectra. After the deposited Zn film are annealed at 180 °C in oxygen flux with the pressure of 2×10^{-6} mbar, the Zn 3d peak becomes broader and another peak at −6.6 eV appears, which is originated from the contribution of O 2p in the ZnO valence band [20]. When the annealing temperature is increased up to 600 °C, these two peaks become much sharper and indicate the

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