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# Growth and characteristics of polycrystalline 3C–SiC films for extreme environment micro/nano-electromechanical systems

Kang-San Kim, Gwiy-Sang Chung\*

School of Electrical Engineering, University of Ulsan, San 29, Mugeodong, Namgu, Ulsan 680-749, South Korea

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#### ABSTRACT

This paper presents the growth conditions, physical, chemical, mechanical and optical characteristics of polycrystalline 3C–SiC films for extreme environment micro/nano-electromechanical systems (M/NEMS). The growth of the polycrystalline 3C–SiC thin film on oxidized Si wafers was carried out by using atmospheric pressure chemical vapor deposition (APCVD) with a single-precursor of hexamethyldisilane (HMDS:  $Si_2(CH_3)_6$ ). The growth temperature and the HMDS flow rate were adjusted from  $1000 \text{ to } 1200\,^{\circ}\text{C}$  and from 6 to 8 sccm, respectively. The effect of  $H_2$  carrier gas addition was also evaluated to reduce surface roughness and improve mechanical properties. The grown polycrystalline 3C–SiC films grown in this work had very good crystal quality without twins, defects, or dislocations. Therefore, it is expected to have applications in harsh environment, RF and bio M/NEMS devices.

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#### 1. Introduction

In recent years, many electronic and mechanical micro-devices that can be used at high temperature and in corrosive environments have been in demand, particularly in automotive and aerospace engineering, ships, nuclear power instrumentation, satellites, space exploration, and geothermal wells. Silicon-carbide (SiC), a wide band gap semiconductor, is suitable for these applications because of its merits including high thermal conductivity and high saturation velocity properties [1,2]. Moreover, SiC has an electrical breakdown field strength over eight times greater than Si or GaAs, making it suitable for high-voltage and high-power devices [3]. SiC is an excellent candidate for micro/nano-electromechanical system (M/NEMS) operating in harsh-environments because of its superior chemical, thermal, and mechanical properties at high temperatures [4]. Additionally. SiC is inert to most chemicals at room temperature and has been found to be a biocompatible material [5]. Furthermore, owing to its large ratio of its Young's modulus to its density, SiC has attracted interest in ultra-high frequency M/NEMS for wireless signal-processing systems [6].

To date, over 200 SiC types have been identified, and the technique of producing large-area 3C–SiC films on Si substrates by using heteroepitaxial growth has been developed, making high-volume batch processing possible and allowing for the use of highly advanced Si bulk or surface micro-machining for M/NEMS fabrica-

tions [7,8]. The application of heteroepitaxial 3C–SiC films grown on Si wafers in M/NEMS fields can be of great interest in fabricating new micro-sensors and micro-actuators for harsh environments, RF and bio fields, and Si micro-machining technology.

3C–SiC is classified into single-crystalline or polycrystalline types by its crystal structure. The growth processes and the properties of single-crystalline have been extensively studied since 1983 [12]. Although single-crystalline 3C–SiC films have high crystallinity and excellent electrical properties, they can only be epitaxially grown on restricted crystalline substrates at high temperatures. The growth temperature of single-crystalline 3C–SiC films are higher than that of polycrystalline 3C–SiC. Thus, single-crystalline 3C–SiC films have large residual stress, cracks, lattice mismatching (20%) due to the mismatch in thermal expansion coefficients (8%) at the Si/SiC interface at the high growth temperature. These defects seriously affect their applications in M/NEMS and other technologies.

In contrast, polycrystalline 3C–SiC thin films can be grown on insulating materials at a temperature lower than that of single-crystalline 3C–SiC films so that the problems mentioned above can be reduced [9].

Up to now, significant progress has been made in the growth of single-crystalline SiC bulk films, with special emphasis on the 6H-and 4H-hexagonal polytypes and the 3C-cubic polytype. The most common precursors for 3C–SiC films are double-source precursors, such as SiH<sub>4</sub> (or SiHCl<sub>3</sub>) and C<sub>3</sub>H<sub>8</sub> (or CH<sub>4</sub>) with a carrier gas (H<sub>2</sub>), usually at temperatures higher than 1200 °C [10]. However, strict safety control is required for the use of silane gas source owing to its pyrophoric nature, flammability, and toxicity. Thus, recent efforts have focused on the growth of 3C–SiC thin films utilizing

<sup>\*</sup> Corresponding author. Tel.: +82 52 259 1248; fax: +82 52 259 1686. E-mail address: gschung@ulsan.ac.kr (G.-S. Chung). URL: http://nfs.ulsan.ac.kr (G.-S. Chung).

single-precursors that contain both Si and C atoms with a reduced activation barrier for SiC formation [11].

Single organosilane precursors, such as 1,3-disilabutane (1,3-DSB, SiH $_3$ -CH $_2$ -SiH $_2$ -CH $_3$ ), tetraethylsilane (TMS, Si(CH $_3$ ) $_4$ ) [12,13], and hexamethyldisilane (HMDS, Si $_2$ (CH $_3$ ) $_6$ ), have been used recently in growing 3C–SiC thin films due to their safety, ease of handling, low growth temperature, and accurate stoichiometry. The typical deposition temperature ranges of 1,3-DSB, TMS, HMDS was 800–1350 °C.

Therefore, this work utilized a HMDS precursor to deposit polycrystalline 3C–SiC thin films at lower deposition temperatures for M/NEMS applications. Moreover, low-pressure chemical vapor deposition (LPCVD) at pressure below 10 Torr is superior to atmospheric pressure CVD (APCVD) because it is possible to grow thin films with uniform and smooth morphology. However, in spite of these advantages, LPCVD has disadvantages of low growth rate and additional cost for establishing and maintaining low-pressure [14,15]. In this paper, we report the deposition of polycrystalline 3C–SiC thin films on the oxidized Si wafers by using a commercial APCVD reactor with a single-precursor. The physical, chemical, mechanical and optical characteristics of 3C–SiC thin films were also investigated in this paper.

#### 2. Experimental

APCVD was used for the growth of 3C–SiC films on oxidised Si substrates. The oxidized Si substrate at center of the reactor tube was horizontally parallel to gas flow. After 10 slm (standard litters per minute) of Ar as carrier gas had been injected into the reactor tube, an Ar purging cycle for removing residual air was performed 3 times. A graphite susceptor was heated by using an RF coil, and the ramping up time for reaching the growth temperature was less than 2 min. In order to obtain an optimal growth temperature, we varied the growth temperature from 1000 to 1200 °C while maintaining the HMDS flow rate at 8 sccm. The deposition time and growth rate were 30 min and 0.2  $\mu m/min$ , respectively.

Additionally, variations in the film composition, the bonding structure, and the crystallinity of 3C–SiC thin films with growth temperature were investigated using several analyses: X-ray diffraction (XRD), Fourier transform-infrared spectroscopy (FT-IR), reflection high energy electron diffraction (RHEED) and Raman scattering.

XRD patterns obtained by operation in  $\theta$ -2 $\theta$  geometry were employed to determine the crystal structure of the 3C–SiC film deposited on the oxidized Si substrate. The FT-IR wave number was in the range from 400 to  $1000\,\mathrm{cm}^{-1}$ . The crystallinity of 3C–SiC was characterized by using RHEED measurements. We used X-ray photoelectron spectroscopy (XPS) to determine the chemical nature and elemental compositions of the deposited film, and glow discharge spectrometry (GDS) to investigate the depth profiling for component analysis with changing thickness. The surface roughness of the 3C–SiC film and the quality of the SiC/SiO $_2$  interface were investigated by using atomic force microscopy (AFM) and tunneling electron microscopy (TEM), respectively.

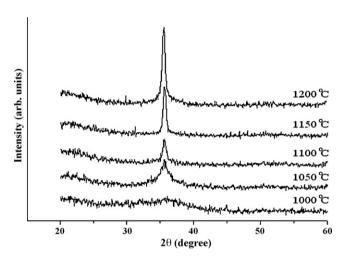
The root mean square (RMS) of as deposited polycrystalline 3C-SiC thin films was too high for M/NEMS applications. Thus, we used high purity  $H_2$  gas (0–100 sccm) to improve the surface roughness.

The mechanical properties, elastic modulus and hardness, of the polycrystalline 3C–SiC thin film were measured using the CSM (continuous stiffness measurement) model of Nano-Indentor. The indentor tip used for this experiment was a Diamond Berkovich tip. The interval of position of Nano-Indentor is 80 µm in order to minimize influence of near indent depths. For the mechanical data of 3C–SiC, it was assumed that Poisson's ratio of the poly-

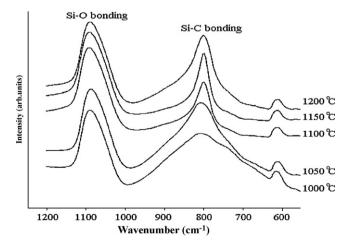
crystalline 3C–SiC is 0.16. In order to measure Raman spectrum at room temperature, Renishaw Ramanscope 1000 with resolution of 1 cm<sup>-1</sup> was employed. The system was equipped with an Ar<sup>+</sup> ion laser ( $\lambda$ =514.5 nm). The spot size of incident laser light was 1.5 mm or larger and microscope (20× objective) was used to focus on reflected laser light. Here the Raman spectrum value was the average value of three scan measurements.

#### 3. Results and discussion

Fig. 1 shows the XRD spectra for polycrystalline 3C–SiC deposited on oxidized Si substrates. The Si peak was removed to reduce the errors caused by the differences in the intensities of the peaks. The 3C–SiC and the Si peaks corresponded to  $2\theta$  =  $36^{\circ}$  and  $69^{\circ}$ , respectively. The Miller index of polycrystalline 3C–SiC is (111) due to  $2\theta$  =  $36^{\circ}$ . The shape of the 3C–SiC peak changed as a function of the growth temperature, and its full width at half maximum (FWHM) became narrower with increasing growth temperature. At  $1000^{\circ}$ C, the 3C–SiC thin film was amorphous due to a lack of thermal energy. However, with increasing growth temperature, the crystal phase of 3C–SiC thin film became polycrystalline because of the increased thermal energy. This means that polycrystalline 3C–SiC (111) was improved by decreasing the lattice disorder and that high-quality 3C–SiC was grown on a SiO<sub>2</sub> buffer layer.



**Fig. 1.** The XRD spectra of the polycrystalline 3C–SiC thin films grown on the oxidized Si substrates according to temperature.



**Fig. 2.** FT-IR spectra of polycrystalline 3C–SiC thin films grown on the oxidized Si substrate.

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