

# Enhanced nano-mechanical and wear properties of polycarbosilane derived SiC coating on silicon



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## ABSTRACT

Liquid Polycarbosilane derived  $\beta$ -SiC and  $\alpha$ -SiC coating was deposited at three differently moderate high temperatures by CVD method on Silicon wafers. The nano-mechanical properties such as hardness, reduced Young's modulus, elastic recovery and plasticity index of the coatings were evaluated and compared at different deposition temperature. It was observed that with the increase in deposition temperature the nano-mechanical property of the coating enhances, the reason being the increase in crystallinity along with good adherence at higher temperature. Moreover the tribological properties of the coated samples show similar trends with increase in deposition temperature although it were found to be much better for the  $\beta$ -SiC than that of the  $\alpha$ -SiC coated samples. All the coated samples shows brittle mode of failure during tribological testing. It was found that the 900 °C deposited film has better nano-mechanical as well as good tribological properties compared to other temperature of deposition due to better crystallinity and adherence. Thus the enhanced mechanical and tribological properties of this coating can be used as protective films on silicon for application in micrometers and other micro devices.

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## 1. Introduction

Hardness, toughness, adhesion and wear are some of the reliability issues imposed on Microelectromechanical systems (MEMS). Silicon is an attractive structural material for the microscale devices, owing to its high elastic modulus and low density. But due to its brittleness and substantially low wear resistance parameter a small crack can result in catastrophic failure [1,2]. Hence to increase the life of these parts it is necessary to have hard coatings on silicon for better wear resistance parameters.

Recently hard coatings on silicon also attract considerable attention for thermo-mechanical applications where the material is subjected to wear under high temperatures [3]. As silicon is an important material for MEMS application hence hard coating on silicon is essential to strengthen the Si substrate and protect it from damage caused due to environmental influence such as particle

impact or moisture. DLC films are commonly used in aeronautics space and high precision machining because of their high hardness and low wear [4,5]. However, the use of a DLC Film has some limitations, (a) above 500 °C, its properties rapidly degrade due to loss of hydrogen from the carbon matrix [6] and (b) DLC films has a high residual compressive stress of 10 GPa (maximum) which causes weak bonding between the film and the substrate thereby making the film delaminated [7]. Hence one needs a better hard coating in place of DLC coating. SiC is an attractive material which is used in mechanical and high temperature applications [8]. Thin SiC coating shares many of the desirable properties of bulk SiC, such as exceptional tribological properties and corrosion resistance. Thus a thin hard coating of SiC on Si is an alternating but attractive mechanism to protect Si from harsh environment at high temperatures [9].

There is a strong view that the conventional method for heteroepitaxial growth of 3C-SiC on Si based on a dual precursor chemical vapor deposition (CVD) requires high growth temperatures (typically » 1000 °C) [10]. Since polycrystalline silicon (polysilicon) microstructures is unable to withstand such high temperatures without deformation so the conventional CVD method cannot be used to coat existing polysilicon devices [1]. It has also

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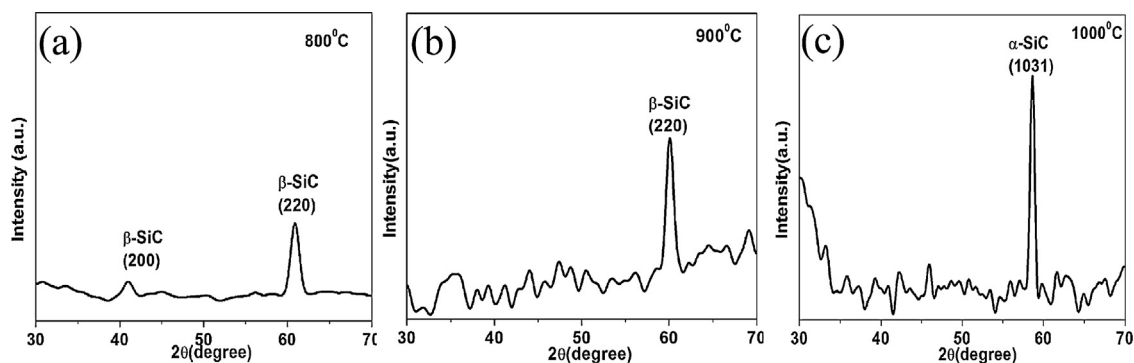


Fig. 1. GA-XRD plot of SiC coating at (a) 800 °C, (b) 900 °C and (c) 1000 °C [13].

been reported that the prime advantage of single source CVD method being its deposition in much lower temperature compared to conventional CVD method [11,12].

Keeping this in mind we reported in our previous work deposition of  $\beta$ -SiC and  $\alpha$ -SiC coating on Si using a single precursor (liquid polycarbosilane) at three different moderately high temperatures [13,14]. It is well known that determination of mechanical properties of thin coating on substrates by indentation is a complex function of the elastic and plastic properties of both film and substrate [15,16]. We therefore focused our present work on the mechanical properties of different phases of SiC thin film deposited using nanoindentation technique and to correlate and compare the tribological properties of the composite at different deposition temperatures. The main aim of this paper is to provide a hard wear resistance coating on Si which can protect it from harsh environment and high temperature.

## 2. Materials and methods

Liquid polycarbosilane (LPCS) used as the precursor of SiC with highly branched structure was synthesized and supplied by Defence Materials and Stores Research and Development Establishment (DMSRDE), Kanpur, India. The polymer precursor (LPCS) have the structure as  $[\text{Si}(\text{CH}_3)_2\text{CH}_2\text{SiH}(\text{CH}_3)\text{CH}_2]_n$  and its molecular weight as measured by GPC was found to be ( $M_n$ ) 500. The experiment was carried out with silicon (111) wafers procured from Montco silicon Technologies, Inc. having thickness of (975–1025)  $\mu\text{m}$ . The wafers were sliced into pieces of 1.5 cm  $\times$  1.5 cm and cleaned ultrasonically. The samples were then placed inside the inert atmosphere furnace, where continuous flow of Argon gas at a flow rate of 12 ml min<sup>-1</sup> was maintained. The experiment was carried out at a pressure of 76 cm of Hg. The rate of heating was maintained at 4 °C min<sup>-1</sup> under flowing Argon. At different temperature the Argon gas was channelized through LPCS (at 12 ml min<sup>-1</sup>) so as to carry the PCS vapors. The PCS vapor was channelized inside the furnace for 1 h which decomposed to form SiC coating on silicon (111) substrate. Finally the furnace was slowly cooled down at a rate of 4 °C min<sup>-1</sup> to room temperature, keeping all the parameters same. The coating was deposited at three different substrate temperatures of 800 °C, 900 °C and 1000 °C. Glancing Angle X-ray diffraction (GA-XRD) (PANalytical, UK) at a  $\Omega$  of 1.5° and using CuK $\alpha$  radiation at 40 kV/30 mA was used to observe phases of the coated samples at three different temperatures. The nano-mechanical properties of the coating were measured using depth sensing nanoindentation with berkovich indenter (Hysitron TI 950 Triboindenter, USA). Finally, tribological properties of the coated samples were estimated using Ducom Scratch tester (TR-101, India) having Vickers indenter.

## 3. Results and discussions

### 3.1. GA-XRD

The GA-XRD (Fig. 1) of the coating at different deposition temperature shows the increase in crystallinity along with phase transition from  $\beta$ -SiC to  $\alpha$ -SiC with the increase in deposition temperature. The percentage of crystallinity was calculated to be 37.1577% for the 800 °C deposited film and subsequently increases to 44.54% and 57.859% for the film deposited at 900 °C and 1000 °C, respectively [13]. The surface roughness ( $R_a$ ) was measured with optical profilometer for Si wafer (0.476231 nm) and SiC film deposited at 800 °C (2.5415 nm), 900 °C (4.51143 nm) and 1000 °C (8.83873 nm). One can see the increase in crystallinity as the surface roughness increases which is in consonance with Cho et al. [17].

### 3.2. Nanoindentation

Fig. 2 shows the load–displacement curve for the SiC films deposited at three different temperatures at a maximum load of 1 mN and a holding time of 10 s. The maximum indentation depth is maintained within 1/10th of the film thickness to avoid the influence of the Si substrate. There was no pop-ins observed in any of the coated samples even at a maximum load of 8 mN. It was observed that the indentation depth is least for the coating deposited at 1000 °C and maximum at 800 °C deposition. The results clearly corroborates increased in crystallinity of the films with temperature is the cause for increase in hardness. The percentage of elastic

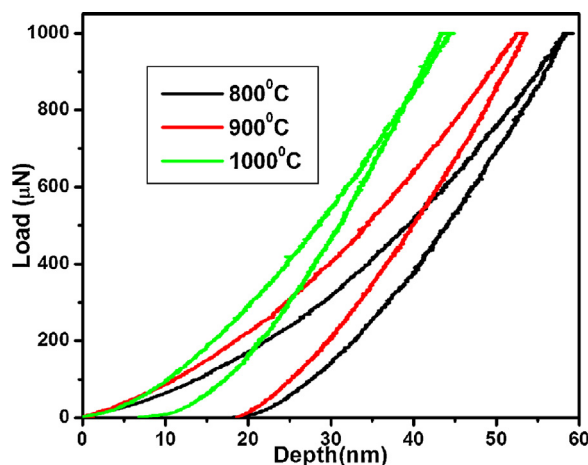


Fig. 2. Load–displacement plot for SiC coating deposited at three different temperatures at 1 mN load.

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