EFFECT OF SILICON CARBIDE PASSIVATION FILM ON THE RESONANCE CHARACTERISTICS OF CANTILEVER

KI YONG CHOI a, c, DAE SUNG YOON a, TAE SONG KIM a & DUCK KYUN CHOI b

a Microsystem Research Center, Korea Institute of Science & Technology, Seoul, Korea
b Division of Advanced Materials Science, Hanyang University Seoul 133-791, Korea
c Also at Division of Advanced Materials Science, Hanyang University Seoul, 133-791, Korea

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Effect of Silicon Carbide Passivation Film on the Resonance Characteristics of Cantilever

Ki Yong Choi,1,*,** Dae Sung Yoon,1 Tae Song Kim,1 and Duck Kyun Choi2

1Microsystem Research Center, Korea Institute of Science & Technology, Seoul, Korea
2Division of Advanced Materials Science, Hanyang University Seoul 133-791, Korea

ABSTRACT

We have used silicon carbide (SiC) thin films as an insulating material of the PZT microcantilevers for electrical and biological passivation. The use of SiC thin films as a passivation layer of the PZT microcantilevers is also seemingly viable to insure the high mass sensitivity as well as the stable passivation. In this study, we report the effect of SiC passivation layer on the performance of the PZT microcantilevers. The micromachined PZT microcantilevers having a structure of SiNx/Ta/Pt/PZT/Pt were fabricated through MEMS processes. In order to improve the mass sensitivity and the passivation, SiC thin films of the high elasticity material were deposited on the cantilever using plasma-enhanced chemical vapor deposition (PECVD) at the temperature of 400°C. Plane-strain modulus of SiC thin film was measured by nanoindentation. We observed that SiC thin films showed higher Young’s modulus than Si and SiO2. Before and after the deposition of SiC thin films, the end-tip deflection and the resonant frequency change of microcantilevers were measured by a confocal microscope and an impedance analyzer. It was confirmed that end-tip deflection of microcantilever was reduced by 13~18% through the deposition of SiC thin films, indicating the stress relaxation of the microcantilevers.

Keywords: Microcantilever; biosensor; PZT; silicon carbide thin film

INTRODUCTION

In recent years, silicon carbide (SiC) has emerged as an important material for MEMS application [1–2]. SiC is a material with very attractive properties for
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microsystems applications. Many advantages of high mechanical strength, high thermal conductivity, high elasticity, high temperature stability and extreme chemical inertness in several liquid electrolytes make SiC an attractive candidate for MEMS applications, both as a structural material and as a passivation layer. The thermal and mechanical properties or the chemical inertness of SiC can significantly improve device performance. In particular, the high elasticity and the chemical inertness are important requirements of the passivation layer of the microcantilever biosensor. However, high temperature deposition of the SiC is known to be a drawback inducing the failure of device fabrication and the performance problem. Recently, it has been demonstrated that SiC can also be deposited as an amorphous phase using low temperature deposition techniques [3–4]. Despite of the lower deposition temperature, many of the attractive properties of this material are still preserved. The applicability of conventional micromachining processes and the possibility of using it as coating in relation to the type of SiC used (single-crystal, polycrystalline or amorphous) are indicated in Table 1 [2].

In the case of piezoelectric microcantilever sensor, we have used parylene-C or SiO$_x$ film as an insulating material of the PZT microcantilever for electrical and biological passivation [5–6]. However, in order to improve sensitivity in sensor, it is necessary to use high elastic modulus material. Because SiC thin films have higher elastic modulus than Si or SiO$_x$, use of SiC as a passivation layer is seemingly viable to improve the sensitivity. There is a broad range of application of SiC thin films as a passivation layer due to good step coverage and its high chemical resistance against wet etchants like KOH or HF.

In this work, the mechanical properties of deposited amorphous SiC thin films have been studied using nanoindentation. On behalf of the cantilever performance, we used amorphous SiC thin films as a passivation layer of the cantilever to improve the sensitivity and relieve the residual stress. The micro-machined PZT microcantilevers having a structure of SiNx/Ta/Pt/PZT/Pt were fabricated through MEMS processes. The SiC thin films with the high elasticity were deposited on the microcantilevers using plasma enhanced chemical vapor

<table>
<thead>
<tr>
<th>SiC</th>
<th>Bulk micromachining</th>
<th>Surface micromachining</th>
<th>Coating</th>
</tr>
</thead>
<tbody>
<tr>
<td>Monocrystalline</td>
<td>✓ ✓</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>Polycrystalline</td>
<td>✓ ✓</td>
<td>✓</td>
<td>✓$^b$</td>
</tr>
<tr>
<td>Amorphous</td>
<td>✓ ✓</td>
<td>✓</td>
<td>✓</td>
</tr>
</tbody>
</table>

✓ = Possible.
X = Not possible or very difficult.
$^a$Epitaxial layer of crystalline SiC on Si.
$^b$Depending on substrate and thermal budget allowed.
deposition (PECVD). In order to evaluate how the SiC coating influences property enhancement, the young’s modulus and the hardness were measured and discussed in terms of the cantilever performance.

**EXPERIMENTAL PROCEDURES**

500 nm thick SiC films were deposited on the Si substrate by PECVD using hexamethydisilane (HMDS) and C$_2$H$_2$ as a main precursor. The SiC thin films were deposited under plasma power of 300 W, and at the temperature of 400°C. In order to obtain high elastic modulus material, SiC thin films were deposited under various C$_2$H$_2$ gas flow rates between 3 and 10 sccm. The mechanical properties of SiC thin films were measured using a Nano-indentation (Triboscope, Hysitron, USA) equipment with a berkovich indenter. Hardness ($H$) and Plane-strain modulus ($E/(1-v^2)$) were obtained from unloading curves based on the Oliver and Pharr method [7]. $E/(1-v^2)$ represents the plane-strain modulus where $E$ is elastic modulus and $v$ Poisson’s ratio of the SiC thin films.

In order to observe the passivation effect of SiC, we fabricated the monolithic PZT thin film microcantilever as shown in Fig. 1. As the substrates for the PZT capacitors, we used 100 mm diameter p-doped Silicon (100) wafers covered with 1.2 μm thick low-stress silicon nitride (SiNx), which was deposited by low-pressure chemical vapor deposition (LPCVD). Then the bottom electrode having a Pt(150 nm)/Ta(30 nm) were deposited using an rf magnetron sputter. The PZT films with a thickness of 0.5 μm were deposited on the Pt/Ta/SiNx/Si substrates by a diol-based sol–gel route shown elsewhere [8]. For the metal–ferroelectric–metal capacitor structure, a Pt layer for the top electrode was formed using the rf magnetron sputter.

In order to construct the cantilever geometry, subsequent etching processes of Pt top electrode, PZT thin film and Pt bottom electrode were performed.

![Figure 1](See Color Plate V)

*Figure 1.* The fabrication flow chart for self-sensing PZT nanomechanical cantilever. (See Color Plate V)
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Figure 2. A SEM photograph of the micromachined PZT cantilever arrays and (b) close-up of the cantilever designed for simultaneous self-actuating and sensing. (See Color Plate VI)

using an inductively coupled plasma etcher (Multiplex AOE, STS). Then, SiO₂ thin film was deposited for electrical and biological passivation and photolithographically patterned for via-hole. Cr/Au with a 30/100 nm for electrical pads were formed by evaporation and lift-off process. In order to pattern the rear Si₃Nₓ window, we used reactive ion etching (RIE). The bulk silicon was then wet etched with a KOH silicon etchant. Finally, Si₃Nₓ was etched with RIE to form the microcantilever. Figure 2 shows a scanning electron microscopy (SEM) photograph of the micromachined PZT cantilever arrays designed for simultaneous self-actuating and sensing. After fabricating the nanomechanical PZT microcantilever, we deposited 100 nm SiC thin films for the electrical and chemical passivation. The sensing signal of a monolithic PZT microcantilever can be measured by changing the sweeping frequency. Before and after the deposition of SiC thin films, the end-tip deflection change and the resonant frequency changes of microcantilevers were measured using a confocal microscope (LSM 5 Pascal, Carl Zeiss) and an impedance analyzer (4294A, Agilent).

RESULTS AND DISCUSSION

In order to analyze the mechanical properties of the deposited SiC thin films on Si wafer, nanoindentation analysis was conducted. Figure 3 shows plots of (a) the hardness and (b) the plane-strain modulus of the SiC thin films as a function of C₂H₂ flow rate. It was observed that the hardness and the plane-strain modulus have maximum values (approximately 12.8 and 215 GPa, respectively) at 5 sccm of C₂H₂ flow rate. Plane-strain modulus of Si₃N₄, SiO₂ thin film and Si wafer was estimated to be 233.3, 75, and 182 GPa, respectively. From the result, it was found that the plane-strain modulus of SiC thin films was higher value than those of Si and SiO₂.

Scanning probe microscope (SPM) images of the deformation area under nanoindentation are shown in Fig. 4. There is a triangular dent at the center of
surface. The conservation of volume in the thin film during such microplastic deformation results in significant pile-up of material around the indent which is seen in the present work on SiC thin films, as well as atomic force microscopy (AFM) of nanoindentation response in Al thin films on Si substrates [9]. Figure 4(a), (c) and (d) show that contact morphologies of the sample were excessively deformed in surface area. It indicated that surface of SiC thin films were deformed by behavior of plastic deformation during loading. On the contrary, the SiC thin films deposited at 5 sccm C2H2 showed a characteristic behavior of elastic deformation in Fig. 4(b).

In general, the as-fabricated cantilever beams are deflected either upward or downward because of residual stress. A positive stress gradient bends the released beam upward, whereas a negative gradient bends the beam downward. If we assume that the stress in the beam varies linearly across the film thickness, then the stress gradient can be determined from [10]

$$\sigma' = \frac{d\sigma}{dh} = \frac{2E_e\delta}{L^2}$$  \hspace{1cm} (1)

where $\sigma'$ is the stress gradient in the beam, and $\delta$ is the end-tip deflection of the released cantilever. From Eq. (1), the stress gradient in the beam can be evaluated if we measure the end-tip deflection of the microcantilevers with a confocal microscope. In order to evaluate the effects of SiC thin films deposited as passivation layers, the end-tip deflections of cantilever before and after SiC deposition were measured using a confocal microscope. Figure 5 shows the end-tip deflection change of microcantilever with the dimension of $L = 150 \, \mu m$ and $W = 50 \, \mu m$ before and after the deposition of 100 nm SiC. From the result, the as-fabricated cantilever beams were bent upwards from the substrate about 18.44 $\mu m$, indicating that there is a positive stress gradient field across the cantilever thickness. On the other hand, the end-tip deflection of SiC-coated microcantilever was reduced to about 15.32 $\mu m$. 

![Figure 3](image-url)  
Figure 3. Nonindentation data of hardness and plane-strain modulus value as a function of C2H2 of flow rate. (See Color Plate VII)
Figure 4. SPM image of the deformation area under nano-indentation as a function of $C_2H_2$ of flow rate (a) 3 sccm, (b) 5 sccm, (c) 7 sccm and (d) 10 sccm.

Figure 6 shows the effect of SiC coating on the end-tip deflections of microcantilevers with respect to the cantilever length. Measured initial deflection of the cantilever beam increases with the increase of the beam length. The maximum deflection reaches about 20% of the beam length for a 200 $\mu$m long beam. The end-tip deflection of microcantilevers was reduced by about 13\textasciitilde18\% by

Figure 5. Confocal microscope image of change in end-tip deflections of microcantilever as deposited with SiC thin films; (a) Before deposited with SiC (18.44 $\mu$m) (b) After deposited with SiC (15.32 $\mu$m). (See Color Plate VIII)
the deposition of SiC thin film. From the result, it was shown that the SiC passivation induced the decrease of $\delta$, indicating that the stress of the micro-cantilever was relaxed.

The dynamic characteristics of a beam structure are well understood. The mechanical resonance frequency of a cantilever or bridge structure is related to the dimension of the beam and the material properties, such as Young’s modulus, residual stress, and material density. The fundamental resonance frequency of a cantilever is given by [11]

$$f_c = 0.16154 \frac{h}{L^2} \sqrt{\frac{E_e}{\rho}}$$ (2)

where $L$ and $h$ are the length and the thickness of the cantilever beam, respectively, $E_e$ the effective modulus, and $\rho$ is the density of the material. The effective modulus, $E_e$, is replaced by $E/(1 - \nu^2)$, where $\nu$ is Poisson’s ratio, if the beam width, $d$, is relatively large compared to its thickness, $h(d \geq 5h)$, to account for plane-strain conditions [12]. Figure 7 shows the resonant frequency shifts plot of the SiC-coated microcantilever as function of cantilever length. It was observed that the resonant frequency of microcantilever increases with the deposition of SiC thin film. Because the plane-strain modulus of SiC is higher than SiO$_2$ or Si, The effective modulus of cantilever with SiC thin film was increased. From Eq. (2), the increase of $E_e$ was followed by increase of resonance frequency. From this result, by enhance the value of resonant frequency, we have shown possibility for increase of mass sensitivity. From the above results, it was revealed that the SiC thin film is applicable to the insulation of the microcantilever for electrical and biological passivation.
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Figure 7. Resonant frequency shift plot as function of length of cantilever with SiC thin films.

CONCLUSIONS

In this paper, we studied the effect of SiC thin film as a passivation layer to improve mass sensitivity and to relieve surface residual stress. SiC thin films have been deposited on Si wafer using the PECVD technique, and mechanical properties of SiC thin films were measured by the nanoindentation method. We obtained the SiC film with higher Young’s modulus at 5 sccm C2H2 than those of Si and SiO2. The hardness and the plane-strain modulus of the SiC thin film were approximately 12.8 and 215 GPa, respectively. The micromachined PZT microcantilevers having a structure of SiNx/Ta/Pt/PZT/Pt were fabricated through MEMS processes. We observed that the as-fabricated cantilever beams were bent upwards from the substrate, indicating that there is a positive stress gradient field across the cantilever thickness. In order to relieve the surface stress, the SiC thin films with high elasticity were deposited on the microcantilevers. The end-tip deflection of microcantilevers was reduced by about 13~18% by the deposition of 100 nm SiC thin film. From the result, it is revealed that the stress of the microcantilevers was relieved through the SiC deposition. We observed that the resonant frequency of microcantilever increased with the deposition of SiC thin film. From these results, we obtained that possibility for increase of mass sensitivity and release of surface stress. From the above results, it was revealed that the SiC thin film is applicable to the insulation of the microcantilever for electrical and biological passivation.

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