

# Mechanical Properties of Polycrystalline 3C-SiC Heteroepitaxial Layers

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## ABSTRACT

Silicon carbide (SiC) is widely recognised as the leading candidate to replace silicon in Micro Electro-Mechanical Systems (MEMS) devices operating in harsh environments. In this work, cantilevers and bridges in SiC are designed, fabricated and evaluated between room temperature (RT) and 600 °C. The active material is a cubic poly SiC layer deposited on a poly-Si layer which is separated from the Si substrate by a thermal oxide. The structures are excited either mechanically or electrostatically. Their resonance frequency is measured by Laser Doppler Velocimetry and used to derive the Young's modulus and residual stress in the heteroepitaxial layer (330±45 GPa and 200±20MPa, respectively). The temperature coefficient of Young's modulus is found to be -53±2 ppm/K in the range RT to ~300 °C, whilst an analytical expression is given for the temperature dependency of the Young's modulus between RT and 500 °C.

**Keywords:** silicon-carbide, MEMS, temperature coefficient of Young's modulus, harsh environment.

## 1 INTRODUCTION

In recent years, silicon carbide (SiC) has emerged as a valid alternative to silicon (Si) for Micro Electro-Mechanical Systems (MEMS) operating in harsh environments due to its superior mechanical, physical and chemical properties [1, 2]. The cubic polytype (3C-SiC) is superior to the other polytypes in some respects (*e.g.* higher electron mobility), but it is proving more difficult to grow in high quality single crystals [3]. A two-step technique, carburization followed by chemical vapour deposition, has been refined in the past decade to deposit 3C-SiC films on Si. Although a considerable mismatch between the lattice parameters of Si and SiC renders the interface highly defective, the properties of the deposited film are still superior to those of silicon. Furthermore, the deposition technique permits the build-up of layered materials which can prove more flexible for the fabrication of MEMS.

Although SiC is advantageously more stable at extreme temperatures than Si, mechanical, electronic and

dimensional changes can be important when the fabricated devices are required to operate over a wide temperature range. It is therefore necessary to characterize the material not only at a fixed temperature, but also as a function of temperature.

From the mechanical point of view, the dependence of the Young's modulus with temperature should be a primary concern. In a simple 'static' device, like an accelerometer with proof mass, a decrease in the Young's modulus with temperature means that a systematic overestimation of the acceleration will take place at high temperature. A similar effect would be seen with a membrane pressure sensor. Dynamic sensors and resonators are also significantly affected. One of the first determinations of the temperature coefficient of Young's modulus (TCYM) of 3C-SiC films is due to Su et al. [4]. Their work focused on single crystal 3C-SiC epitaxially grown on Si, for which they found a value of -46 ppm/K for the undoped material in the range between room temperature and ~450 °C. However, polycrystalline 3C-SiC is more commonly found in material structures suitable for surface micromachining. The focus of this paper, therefore, is a layered structure where the 3C-SiC is deposited epitaxially on poly-Si, which functions as the sacrificial layer in the MEMS fabrication process.

Because of the widespread acceptance of Si and the relative novelty of SiC, the difference in quantity and quality of available data about the two materials is very significant. The aim of this paper is to contribute to bridging this gap, by reporting on the characterization of a multilayered poly-3C-SiC structure suitable for surface micromachining. In particular, we have determined the residual stress, stress gradient, the value of Young's modulus and its dependence on temperature, of a polycrystalline 3C-SiC film grown according to the two-step technique mentioned above.

## 2 EXPERIMENTAL DETAILS

The material is a 2.7- $\mu\text{m}$  thick SiC undoped layer heteroepitaxially deposited on a structure composed of a single crystal Si substrate (~500  $\mu\text{m}$ ), a SiO<sub>2</sub> insulating layer (~1.5  $\mu\text{m}$ ) and a polysilicon sacrificial layer (~3.7  $\mu\text{m}$ ), as shown schematically in Fig. 1.

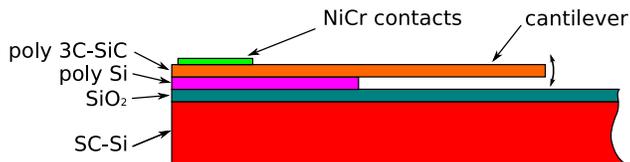


Fig. 1. Schematic cross-section view of the material used to fabricate the devices.

Cantilever structures were used to determine the Young's modulus and its dependence upon temperature (TCYM). Bridges, together with a strain measuring device, were used to measure the residual stress and strain, and this provided an alternative method to derive the value of Young's modulus.

Fabrication of the devices was performed by etching the SiC layer using a one-step inductively coupled plasma (ICP) etching technique that also removes the underlying poly-Si, releasing the devices [5]. Silicon dioxide was used as the etching mask. NiCr contacts were deposited near the edges of the die to permit the electrostatic actuation of the SiC devices by virtue of the electric field between the SiC beams and the Si substrate, see Fig. 1.

The experimental apparatus used to conduct dynamic tests on the SiC beam structures is shown in Fig. 2. An evacuated chamber contains a heating element on which the sample is placed. The walls of the small cavity housing the sample are covered in silver foil to reflect thermal radiation. Virtually all the remaining volume of the chamber is filled with thermally insulating ceramic (duratec®). The structures can be viewed from above through a 'hot mirror', whose special coating reflects most of the infra-red radiation but is transparent to the visible light. The collimated beam of a laser Doppler vibrometer is focused onto the devices through a modified microscopic objective that enables simultaneous optical observation. The LDV output was digitized with an oscilloscope PCI card. Specially developed software performed a Fast Fourier Transform (FFT) of the temporal data and enabled automatic detection of the peaks in the computed spectrum. The peaks position was plotted in real-time to enable the observation of their evolution with time during the heating steps. The beams were forced into off-plane flexural vibration using either electrostatic excitation or by means of the piezoelectric ring actuator placed at the bottom of the apparatus, as in Fig. 2.

Precise temperature control of the die was achieved by placing the die on a specially designed resistive heater. The heater was constructed from two 1-mm thick plates of macor® ceramic and a serpentine of NiCr wire, bonded together as a sandwich by a filling of suitable high temperature binder. The die was placed directly onto the top ceramic plate and was clamped firmly down by two small steel clips. During electrostatic excitation a thin copper sheet was laid between sample and heater to enable electrical contact to the substrate.

To monitor the surface temperature of the die, a mineral insulated type K thermocouple with external diameter

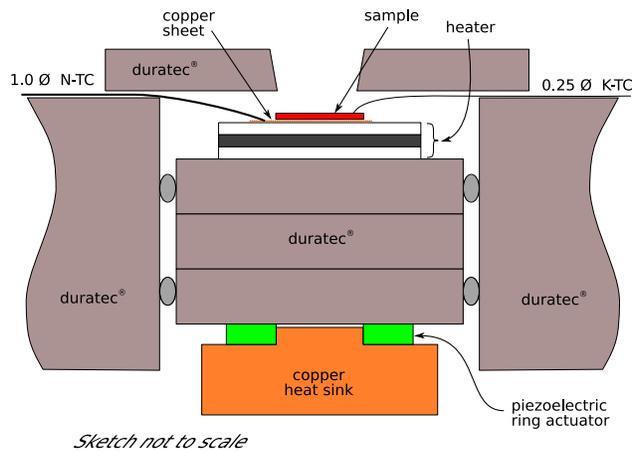


Figure 2. Sketch of the cross section of the experimental setup used for the characterization of the devices at different temperatures.

250  $\mu\text{m}$  was placed in contact with the top surface of the sample and was held in position by elastically preloading it against the surface. A second mineral insulated thermocouple (type N, diameter 1 mm) was placed a couple of millimetres away from the die, in close contact with either the ceramic plate or copper sheet. The type K thermocouple was used to provide feedback to a PID-based temperature controller whilst the type N thermocouple was independently monitored. It is reasonable to assume that the real temperature of the devices ( $T_R$ ) lies near the two values read ( $T_N$  and  $T_K$ ), with  $T_K$  providing a more accurate reading.

Following these considerations, it was decided to assume the temperature of the devices to be equal to  $T_K$ , with an estimated uncertainty ranging from  $\pm 5$  K at low temperature to  $\pm 25$  K at the highest temperature – these also include the intrinsic uncertainties of the thermocouple wire and of the measuring equipment.

### 3 RESULTS AND DISCUSSION

Initial observation with a surface profilometer showed that the cantilevers are bent upwards, with a radius of curvature of  $r = 4.0 \pm 0.1$  mm. This indicates the existence of a strain gradient through the thickness. A simple calculation, based upon elementary bending theory, reveals that the bending can be explained by the relaxation of an average stress gradient of  $\Delta\sigma/\Delta t = E/r = 92 \pm 10$  MPa/ $\mu\text{m}$  (assuming  $E = 370 \pm 40$  GPa).

One device was removed from the chip and placed flat on a reference surface; by profiling the area of contact it was possible to measure the thickness, which was found in excellent agreement with the SEM measurements. A value of thickness equal to  $2.7 \pm 0.1$   $\mu\text{m}$  was calculated by combining these two values.

A total of 47 bridges of varying lengths between 400  $\mu\text{m}$  to 4 mm were manufactured on the same chip to

measure the residual tensile stress. This stress is caused by the lattice mismatch between Si and SiC and the thermal strain due the fact that the SiC layer was deposited on Si at high temperature – their coefficients of thermal expansion are significantly different [6, 7]. A very good linear correlation was observed between the period of vibration and the length when the bridges were mechanically excited in their first mode. The equation fitting the data ( $l$  in  $\mu\text{m}$ ):

$$T_r = a \cdot (l + l_0) = (7.95 \pm 0.02) \cdot 10^{-9} \cdot [l - (59 \pm 4)]$$

$$R^2 = 0.9997$$

The linear dependence, evidence of string-like behaviour, is expected as the bridges are very long compared to their thickness and are also under considerable tension. From simple vibration theory, the fundamental resonance frequency of a taut string is:

$$f_n = \frac{1}{2l} \sqrt{\frac{\sigma}{\rho}}$$

So that the slope of the linear fit is given by:

$$a = 2 \sqrt{\frac{\rho}{\sigma}}$$

Assuming a tabulated value of  $\rho = (3150 \pm 10\%) \text{ kg/m}^3$  one can calculate that the bridges are under an average tensile stress  $\sigma = 200 \pm 20 \text{ MPa}$ .

The strain-measuring device fabricated on the same chip showed that the thermal strain relaxing upon etch-release is approximately equal to  $5 \cdot 10^{-4}$ . Using this strain value with the tensile stress measured from the bridges, a value of Young's modulus equal to  $E = \sigma/\varepsilon = 400 \pm 50 \text{ GPa}$  is found.

A total of 35 cantilevers of varying lengths between  $50 \mu\text{m}$  and  $500 \mu\text{m}$  were fabricated on the die. The period of their fundamental mode is plotted against length in Fig. 3 and follows the quadratic behaviour expected from simple vibration theory. The equation used for the fit is:

$$T_r = a(l + l_0)^2 = (2.195 \pm 0.01) \cdot 10^{-10} \cdot [l + (10.5 \pm 0.8)]^2$$

$$R^2 = 0.99986$$

where a parameter  $l_0$  is introduced inside the squared term to signify that the length of the cantilevers may be viewed

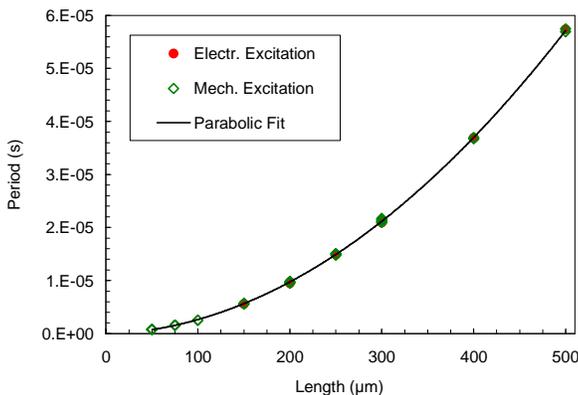


Figure 3. Period of first normal mode of vibration of 35 cantilevers as a function of their length.

as the total of the design length and a fixed length which is related to the undercut. The parameter  $a$  found by the least-squares-fit is related to the thickness  $h$ , the Young's modulus  $E$  and the density  $\rho$  by:

$$a = \frac{4\pi}{(1.875)^2 h} \sqrt{\frac{3\rho}{E_e}}$$

where  $E_e = (1-\nu^2) E$  and  $\nu = 0.16$  is the Poisson's ratio. This equation can be used to deduce that  $E = 330 \pm 45 \text{ GPa}$ . This value of Young's modulus is in reasonable agreement with the value of  $E = 400 \pm 50 \text{ GPa}$ , obtained from the data provided by the bridges and the strain measuring device.

For the determination of the TCYM, the resonance frequency of a subset of cantilevers was measured at different steady temperatures. Fig. 4 reproduces the averages of the normalized resonance frequencies of eight cantilevers for one particular run, where the temperature was ranged between  $22$  and  $600 \text{ }^\circ\text{C}$ . Only data up to  $500 \text{ }^\circ\text{C}$  are used in the analytical fit, as an anomaly clearly appears beyond that value. The fit is almost linear, but the data are accurate enough to clearly bring out a second order dependence. It is therefore worthwhile making a nonlinear analysis, also because the coefficient of thermal expansion is known to be non-linear [7].

The fundamental resonance frequency of a cantilever of thickness  $h$ , length  $l$  and density  $\rho$  can be written as:

$$f \approx \frac{(1.875)^2}{4\pi} \frac{h}{l^2} \sqrt{\frac{E}{3\rho}} \quad (1)$$

If the temperature dependence of a generic linear dimension is written as  $l(T) = l_0 \gamma(T)$  and we also write  $E = E(T)$ , so that  $\gamma(T_0) = 1$  and  $E(T_0) = E_0$  for an arbitrary reference temperature  $T_0$ , equation (1) yields:

$$E(T) = E_0 \frac{(f/f_0)^2}{\gamma(T)} \quad (2)$$

The scaling factor  $\gamma(T)$  is a function of the instantaneous coefficient of thermal expansion (CTE),  $\alpha(T)$ :

$$\gamma(T) = \exp \left[ \int_{T_0}^T \alpha(T) dT \right] \quad (3)$$

Equation (2) completely describes the dependence of the Young's modulus with temperature in the range from RT to  $\sim 800 \text{ K}$ , as  $f/f_0$  is the experimental curve plotted in Fig. 4. A least-square fit of these data with a 2<sup>nd</sup>-order polynomial gives (with  $T$  in kelvin):

$$f/f_0 = (1.0063 \pm 0.0002) + (-1.95 \pm 0.06) \cdot 10^{-5} T + (-5.7 \pm 0.5) \cdot 10^{-9} T^2$$

$$R^2 = 0.9999 \quad (4)$$

As for  $\alpha(T)$ , the semi-empirical formula from [7] can be used. Experimental errors in the CTE data are not so important for the present derivation because the total effect of the CTE on the calculated TCYM is less than 10%.

In some situations it may be more convenient to have an expression giving the instantaneous TCYM. One way to derive it is by differentiating the logarithm of equation (2):

$$TCYM(T) \equiv \frac{dE(T)}{E(T)} = 2 \frac{df(T)}{f(T)} - \alpha(T)dT \quad (5)$$

A simple 2<sup>nd</sup> order approximation to equation (5) can be written as (with T in kelvin):

$$TCYM(T) = 7.6 \cdot 10^{-12} T^2 - 3.8 \cdot 10^{-8} T - 3.7 \cdot 10^{-5}$$

This is found by fitting a polynomial to the analytical curve and yields excellent results between room temperature and approximately 800 K.

The slopes of  $f/f_0$  vs.  $T$  for data collected in several runs for temperatures between RT and 300 °C fall in the range from -24.3 ppm/K to -26.6 ppm/K, with different degrees of uncertainty. Thus the best estimate for the  $df/f_0$  for this temperature range is (-25±1) ppm/K. As the temperature range now considered is relatively narrow, a linear approximation is advisable and therefore Eq. 3 can be written without including the temperature dependence. By taking an average value of 3±1 ppm/K for the CTE, the temperature coefficient of Young's modulus in the range RT to 300 °C can be written as:

$$TCYM = 2 \frac{df_n}{f_n} - \alpha = -(53 \pm 2) \text{ ppm/K}$$

This value compares well with the value of -46 ppm/K found by Su et al. [4] on single crystal material. It is also interesting to compare it with the values for poly-silicon, which range between -32 ppm/K [8] and -75 ppm/K [9].

The uncertainties given above were determined for a 67% confidence level.

## 4 CONCLUSIONS

Conscious design of SiC MEMS devices operating at high temperatures requires knowledge of the mechanical

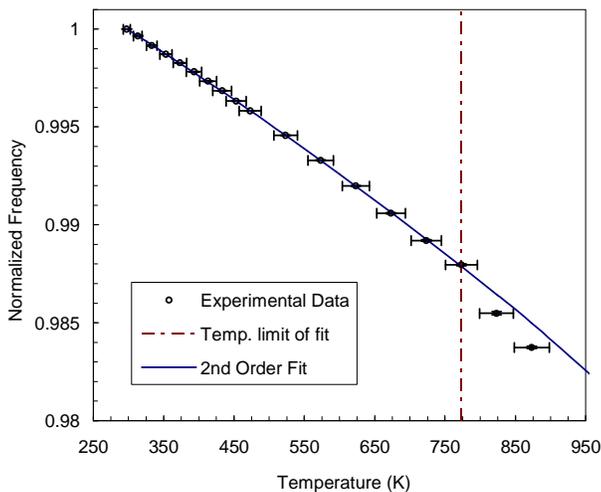


Figure 4. Effect of temperature on cantilevers. The y-value of each point is the average  $f/f_0$  of eight cantilevers. The vertical error bars (barely noticeable) are equal to the corresponding standard deviations. The horizontal error bars reflect the uncertainties in the temperature measurement.

properties of the material as a function of temperature. In this work, test devices in SiC were designed, fabricated and evaluated over a wide temperature range.

The Young's modulus of SiC was seen to decrease with increasing temperature at a rate of  $-53 \pm 2$  ppm/K between RT and  $\sim 300$  °C, in good agreement with results previously reported by other authors. For the first time a more accurate non-linear expression is given to describe the temperature dependence of Young's modulus for the range between RT and  $\sim 500$  °C. This information is deemed to be valuable when designing devices capable of predictable operation over a wide range of temperatures.

The results reinforce the view that although SiC is a mechanically superior material to Si for application at high temperatures, the designer must be aware of the change of its mechanical properties with temperature, as this will cause variations in performance at different temperatures.

## 5 ACKNOWLEDGMENTS

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