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Highly-doped SiC resonator with ultra-large tuning frequency range by Joule heating effect

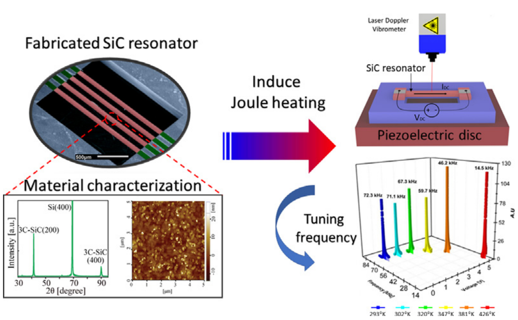
Pablo Guzman [a,*], Toan Dinh [a,b], Hoang-Phuong Phan [a], Abbin Perunnilathil Joy [d], Afzaal Qamar [e], Behraad Bahreyni [d], Yong Zhu [a], Mina Rais-Zadeh [e], Huaizhong Li [c], Nam-Trung Nguyen [a], Dzung Viet Dao [a]

[a] Queensland Micro and Nanotechnology Centre, Griffith University, West Creek Road, Nathan, QLD 4111, Australia
[b] University of Southern Queensland, 37 Sinnathamby Blvd, Springfield, Central QLD 4300, Australia
[c] School of Engineering and Built Environment, Griffith University, Parklands Drive, Southport, QLD 4222, Australia
[d] 4D LABS, Simon Fraser University, 8888 University Dr, Burnaby, BC V5A 1S6, Canada
[e] University of Michigan, 500 S State St, Ann Arbor, MI 48109, United States

HIGHLIGHTS

- Free-standing silicon carbide (SiC) bridges function as both resonant structure and heating element.
- Ultra-large frequency tuning capability of almost 80% was demonstrated for SiC resonators by thermal stress.
- Frequency tunability of SiC micromechanical resonators is dependent on stress induced by Joule heating.

GRAPHICAL ABSTRACT



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ABSTRACT

Tuning the natural frequency of a resonator is an innovative approach for the implementation of mechanical resonators in a broad range of fields such as timing applications, filters or sensors. The conventional electrothermal technique is not favorable towards large tuning range because of its reliance on metallic heating elements. The use of metallic heaters could limit the tuning capability due to the mismatch in thermal expansion coefficients of materials forming the resonator. To solve this drawback, herein, the design, fabrication, and testing of a highly-doped SiC bridge resonator that excludes the use of metallic material as a heating element has been proposed. Instead, free-standing SiC structure functions as the mechanical resonant component as well as the heating element. Through the use of the Joule heating effect, a frequency tuning capability of almost $\Delta f/f_o \approx 80\%$ has been demonstrated. The proposed device also exhibited a wide operating frequency range from 72.3 kHz to 14.5 kHz. Our SiC device enables the development of highly sensitive resonant-based sensors, especially in harsh environments.

1. Introduction

Microelectromechanical systems (MEMS) resonators have been employed in many important applications such as telecommunication, aerospace, biomedicine, and specialize measurement tools [1–4]. While silicon (Si) has been widely used for most commercial MEMS

* Corresponding author.
E-mail address: pablo.guzmanduran@griffithuni.edu.au (P. Guzman).

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resonators, silicon carbide (SiC) has also attracted considerable attention due to its superior properties as a high sublimation temperature, low mass density, and higher Young's modulus. The latter allows SiC devices to vibrate at higher frequencies compared to Si counterparts with the same dimensions [5,6].

A large number of applications have been developed based on MEMS/NEMS sensors such as temperature sensor [7,8], pressure sensors [9–11], mass sensors [12] and resonant accelerometer [13,14]. Due to its ubiquitous applications, the stability and reproducibility of resonators across many applications become increasingly important [15]. The ability to achieve a wide range of tuning frequency in resonators is imperative not only to overcome fabrication process uncertainties and changes in the environmental conditions but also to maintain the reliability and performance of the device [16,17]. Several tuning techniques have been developed to achieve this goal. Mechanical, electrostatic, and thermal tuning are some of the most common methods [18–21]. Among these methods, electro-thermal tuning stands out due to the simplicity of fabrication and application [22,23]. The use of a DC bias superimposed on an AC voltage allows to electrothermally actuate the resonator and also enable to tune the frequency of the devices [24–26].

Nevertheless, thermal tuning relies on two factors which are the heating element and the different thermal expansion coefficient (TEC) of the materials to induce mechanical strain and thus, frequency tuning. To the best of our knowledge, previous studies focused only on the use of metallic materials for heating element in MEMS resonators [20,25–27]. Although metallic materials are prevalently implemented as heaters for thermal tuning, the thermal fatigue damage generated by repetitive cycles is a risk that can degrade the electrodes and disable the resonator [28]. Additionally, most metals can not work reliably in harsh environments, including corrosion and high temperatures, limiting the applications of SiC resonators.

In this paper, for the first time an ultra-wide frequency tunable SiC resonator using SiC film concurrently as both heater and structural material is reported. The advances in the growth of single crystalline cubic silicon carbide on silicon at low cost and high quality for resonator applications was used. Deploying the advanced SiC materials and designs, SiC resonators with an ultra-high tuning range of 80% with respect to its original value were successfully demonstrated. The design, fabrication and characterization of a clamped-clamped bridge SiC resonator are presented. One of the main advantages of this work is the capability of growing high quality single crystalline SiC on Si with high electrical conductivity enabling the effective utilization of the Joule heating effect and hence the development of tunable electrothermal SiC resonator. Besides, from a device perspective, the use of SiC structure as a heating element itself allows for the operation at a higher temperature, opening the possibility of harsh environments applications, e.g. chemical corrosive condition. The frequency tunability of the resonator has been investigated experimentally and it is the highest compared with prior-art MEMS resonators found in the literature.

2. Growth of the SiC film and characterization

The p-type single crystalline 3C-SiC with a thickness of 220 nm was epitaxially grown on Si wafer using a low-pressure chemical vapor deposition process (LPCVD) at low temperature of 1000 °C [29]; As a precursor, propylene (C_3H_6) and silane (SiH_4) were used. The in-situ doping film was achieved by using Trimethyaluminium (TMAl) precursor as the source of Al p-type dopant. The process for obtaining a highly-doped 3C-SiC is described as follows. Si atoms were absorbed on the SiC surface and arranged in a self-assembled pattern when SiH_4 was applied. By adding TMAl, Al atoms were incorporated on the Si-terminated surface. In the end, the incorporated Al and Si atoms were transformed into a 3C-SiC layer by adding the C_3H_6. The aim of highly-doped SiC film with a carrier concentration of 5×10^{18} cm^{-3} is to achieve high conductivity and thus used SiC as a heater for Joule heating-electrothermal tuning. The resistivity of the p-3C-SiC film was measured to be approximately 0.14 Ωcm^{-1} by the hot probe technique and which is suitable for the utilization of the Joule heating effect in the structure.

The information on the crystal structure of 3C-SiC is revealed in the X-ray diffractogram (XRD) as shown in **Fig. 1**a. The pattern of the sample shows peaks with 2θ values of 41.4° and 90.1° corresponding to diffraction from the (200) and (400) crystalline planes of 3C-SiC and a peak in 69.2° of (400) crystalline plane for Si. These results confirm the epitaxial growth of SiC on Si. Furthermore, the 3C-SiC surface measured by an AFM (atomic force microscopy) in an area of 5 μm × 5 μm shows a roughness of approximately 20 nm (Fig. 1b).

3. Device fabrication and measurements

After SiC was deposited on the Si substrate, the device was fabricated using conventional photolithography and etching processes. **Fig. 2** describes the five main steps. Fig. 2a shows the deposition of 3C-SiC on Si. Next, the ICP (inductive coupled plasma) etching process was employed to open the backside of the sample (Fig. 2b). The etching rate was 100 nm min^{-1} under the reactive gas HCl at a flow rate of 50 sccm. Fig. 2c shows the wet etching of the Si to release the SiC structure

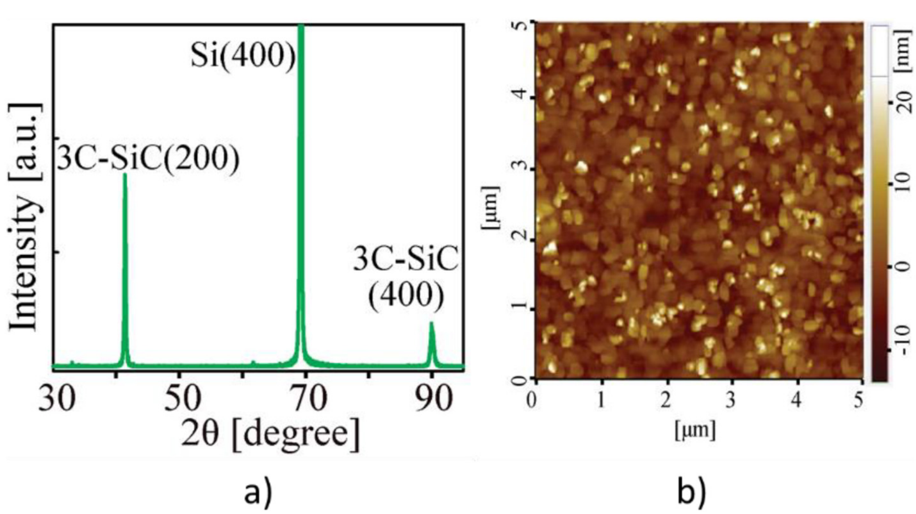

Fig. 1. a) XRD pattern and b) AFM surface characterization

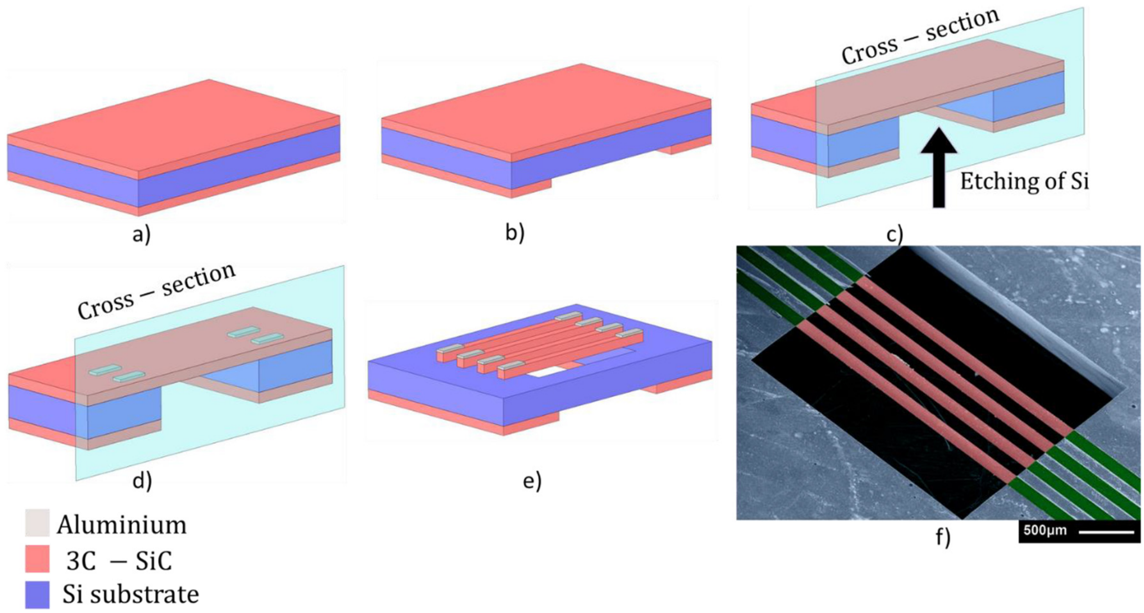

Fig. 2. Fabrication process of the resonator: a) Deposition of the 3C-SiC on Si substrate, b) Opening etching window by dry etching 3C-SiC on the backside, c) Wet etching of Si to release the 3C-SiC structure, d) Deposition and patterning of Al electrodes, e) Patterning and etching 3C-SiC to form 3C-SiC bridges, f) SEM image of the bridge resonators.

by using KOH at 80 °C with an etching rate of approximately 1.4 μm min^{-1}. Then, the sputtering deposition of a 300 nm-thick aluminium film and patterning of the electrodes (Fig. 2d). Finally, photoresist AZ1512 was used as etching mask during the dry etching process of SiC bridge. Cr mask was used in the photolithography process to pattern the SiC bridge which has a dimension of 1750 μm × 85 μm × 220 nm (length × width × thickness) and is shown in Fig. 2e. SEM micrograph of the resonator is shown in Fig. 2f.

To characterize the resonant frequency of SiC resonators, a laser Doppler vibrometer (LDV) to measure the out-of-plane flexural vibration was used (**Fig.** 3). The SiC structure was externally excited into vibration using a piezoelectric disc actuator. For frequency tunning, the suspended SiC bridge resonator is heated up by a DC bias current I_{DC} resulted from the DC bias voltage V_{DC} applied to the electrodes to tune the frequency by inducing stress as shown in Fig. 3. It is important to emphasize that the SiC structure itself is used as a heater while the function of the Al electrodes is only for interconnection. To comprehensively and systematically investigate the tuning efficiency in SiC resonators, the DC bias voltage V_{DC} was varied from 0 V to 5 V to further correlate the relationship between the increase in the device temperature and the resulting frequency modification. To calculate the device temperature, first, the temperature coefficient of resistance (TCR) was estimated. The TCR is described as, TCR = ΔR /ΔT where ΔR and ΔT are the resistance change and temperature change, respectively. By placing the device inside an oven with a controlled temperature chamber the change in the resistance related to change temperature was measured. Then TCR was correlated to the applied power to estimate the temperature of the SiC resonator.

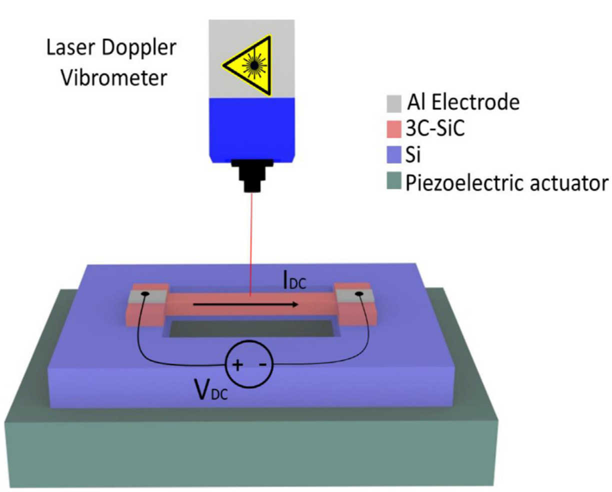

Fig. 3. Schematic illustration of SiC bridge resonator placed on the piezoelectric actuator, and the LDV measurement. The suspended SiC bridge is electrothermally heated up by a DC bias voltage.

4. Result and discussion

Fig. 4a shows the fundamental-mode resonant frequencies at different applied voltages V_{DC} from 0 V to 5 V. The measurements were carried out at room temperature of 20 °C. It can be seen that the resonant frequency can be tuned from 72.3 kHz to 14.5 kHz, or $\Delta f/f_o \approx 80\%$, when the voltage increases from 0 V to 5 V(Fig. 4b). This is the highest frequency tuning capability ever reported for 3C-SiC MEMS resonators. There are several effects contributed to the resonance characteristics of the SiC bridge as described as follows.

Firstly, the strong correlation between the applied voltage and temperature due to the Joule heating effect leading to thermal expansion of the structure. Since the bridge is clamped at both ends, the bridge can not expand and thermal expansion translates to compressive stress in the bridge [30].

Secondly, the frequency of the SiC bridge resonator is sensitive to stress induced by Joule heating; thus, the strain-dependent correction for the Euler-Bernoulli beam theory has to be made in the presence of residual stress within the resonator material and can be expressed by Eq. 1 [31].

$$f_n = \frac{k_n^2 t}{4\pi\sqrt{3}L^2}\sqrt{\frac{E}{\rho}}\sqrt{1+\gamma_n\frac{\sigma_T L^2}{Et^2}} \quad (1)$$

where k_n is the eigenvalue $k_n = (n + 1/2)\pi$ for bridges, $k_n^2/4\pi\sqrt{3}$ is the clamping coefficient. E, ρ, t and L are Young's modulus, mass density, thickness, and length of the beam, respectively. γ_n is a mode-

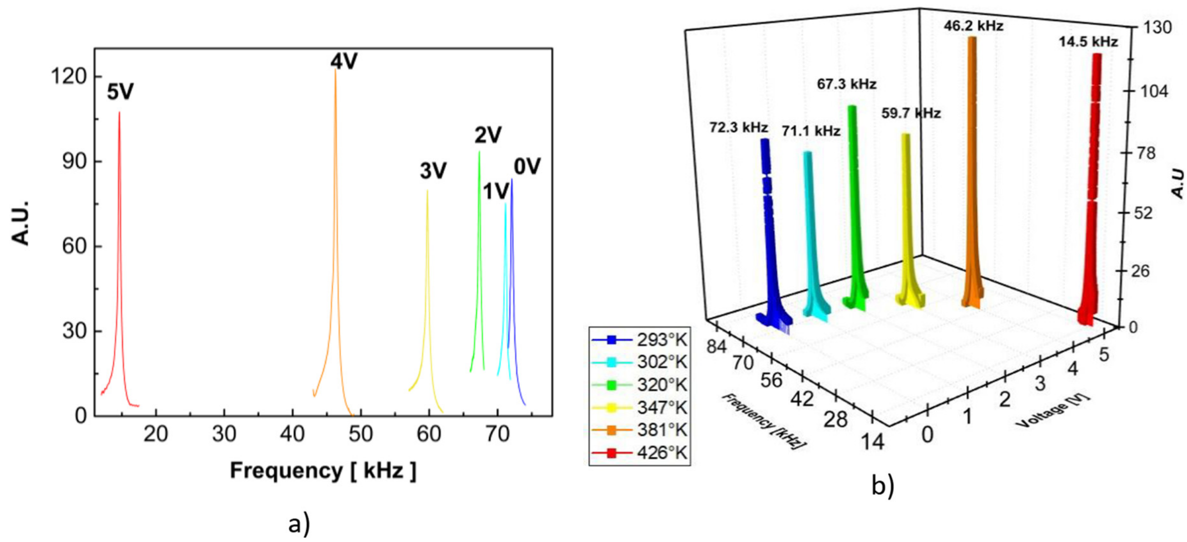

Fig. 4. a) Resonant frequency shift vs. applied voltage V_{DC}, b) Correlation between the applied voltage, frequency, and temperature in the bridge.

dependent coefficient ($\gamma_n = 12(k_n - 2)/k_n^3$) and σ_T is stress in the structure. In this study, σ_T is the total stress including the room-temperature residual stress σ_i generated in the SiC film during the growing process [32] and the thermal stress σ_{th} formed by the Joule heating effect. Hence, the total stress is given by Eq. 2 [33].

$$\sigma_T = \sigma_i + \sigma_{th} = \sigma_i - \alpha E \Delta T \quad (2)$$

where α is the thermal expansion coefficient and ΔT is the variation of temperature.

Finally, the dependence of Young's modulus on temperature can experience a reduction of less than 3% in the range from 300 K to 426 K for 3C-SiC film [34]. Thus, the temperature change affects the stiffness of the 3C-SiC structure and contributes to the higher tuning capability of the resonator [35–37]. Nonetheless, it is important to mention that the variable that dominates the tuning capability is the thermally-induced stress by Joule heating.

The resonant frequency of a bridge resonator without thermally-induced stress depends on both dimensions and mechanical properties of the material [37], the initial value of stress and Young's modulus for the structure was 150 MPa and 400 GPa, respectively [38]. At room temperature, the resonant frequency was calculated to be 70.4 kHz which is close to the measured value of 72.3 kHz. For a small power applied, σ_{th} is small and the resonant frequency is mainly determined by σ_i [32]. By gradually increasing the bias voltage and thus the power, an increment of temperature by Joule heating occurs. As a consequence of increasing the temperature, which is stable over time unless the power is varied, thermal stress σ_{th} increases, resulting in a reduction of the total stress σ_T (Eq. 2), causing the resonance migrating to a lower frequency. Under an applied voltage of 5 V, the resonant frequency decreased about 5 times compared to that of 0 V, as shown in **Fig.** 5. It can be seen that the frequency decreases in a nonlinear manner with the power or temperature increases. Also, it is worth noting that the use of the highly-doped SiC not only takes advantage of good electrical conductivity but also allows for a better thermal distribution owing to its large thermal conductivity. Accordingly, the induction of compressive stress by the thermal expansion in SiC leads to a significant change in the resonant frequency.

Table 1 shows the comparison of our 3C-SiC bridge resonator with existing devices that focused on tuning of frequency with electrothermal tuning. The principal parameters of the resonators such as material, type of structure, heater material, tuning method, size of resonator and maximum frequency shift are going to be evaluated.

In literature, the operating scheme of the resonators with electrothermal tuning is similar. A metallic material such as Al or Pt covers a part of the free-standing device and functions as the electrothermal electrode. The dissimilar thermal expansion coefficient between the elements induces stress and change the frequency. In more complex structures, Si crossbars can be used as a heating element. Nonetheless, the application of a dc voltage is widespread due to its simplicity. Besides, electrothermal tuning has the significant advantage of working with low DC bias voltages applied.

It is worth noting that the tuning range of our system is wider in comparison to those found in the literature. As an example, the tunning range of our SiC resonators is approximately 8 times higher than that reported in [29] which used aluminium as the heating element. This is reasonable since in the conventional structures, the ability to tune the frequency is limited by physical parameters such as position, size, and material of the heater [27]. In addition, the difference in the thermal expansion coefficient of the materials composing the electrode and the structure is another factor that hinders the tuning efficiency of the frequency [37]. In contrast to these resonators, our SiC device directly induces compressive stress through its own thermal expansion

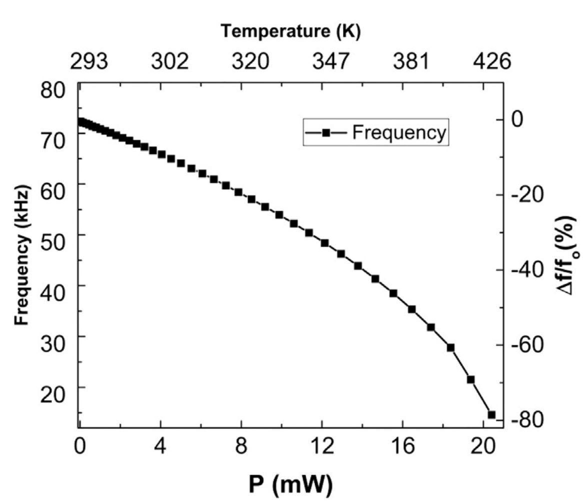

Fig. 5. Frequency change vs. applied power or temperature.

Table 1
Benchmarking of electrothermal tuning performance in SiC-MEMS resonators.

Material/Structure	Heater Material	Tuning method/ DC voltage	Size ($L \times w \times t^*$) [μm]	Tuning range/Maximum Shift [ppm]	Reference
3C-SiC/ Bridge	Al	Electrothermal/ 100 μV	12 × N.A × 0.03	12–10.8 MHz/100,000	[33]
3C-SiC/ Cantilever	Pt	Electrothermal/ 6 - 11V_{dc}	250 × 100 × 2	522.2–521.9 kHz 1300	[35]
3C-SiC/ Bridge	Pt	Electrothermal/ 1 - 4 V	300 × N.A × 2	N.A 35,000	[37]
3C-SiC/ Bridge	Pt	Electrothermal/ 2–6 V	200 × 50 × 2	0.85–1.05 MHz 300,000	[39]
3C-SiC/ Cantilever	Pt	Electrothermal/ 100 mV	200 × 15 × 3	889.812–889.80 kHz 12	[40]
3C-SiC/ Bridge	Pt	Electrothermal/ 1 - 7 V	100 × 50 × N.A	1.766–1.595 MHz 96,800	[41]
Comb-shape micro resonator	Si	Electrothermal/ N.A	N.A	31–28.985 kHz 65,000	[42]
Si/Resonator with a crossbar	Si	Electrothermal/ N.A	410 × 7 × 25	39.2–39.05 kHz 3800	[43]
VO_2 /Bridge	VO_2	Electrothermal/	300 × 40 × 2.2	247.6–227.6 kHz 80,700	[44]
3C-SiC/ Bridge	3C-SiC	Electrothermal/ 1 - 5 V	1750 × 85 × 0.2	72.3–14.5 kHz 798,800	This work

* L = Length, w = width, t = thickness.

(e.g similar to the bulking phenomenon) [26,44]. The deployment of SiC as both structural and heating element helps avoiding the dependence of the TEC between the materials. This property allows heating the entire structure and thus induces more compressive stress which enables the frequency tuning capability reaches almost 5 times with respect to the original value.

The low working frequency in the kHz order for our device is attributed to the greater length of the structure [45,46]. Our system has the widest range of tuning frequency compared to other resonators that operate based on electrothermal tuning technique. The tunable range of our method is almost 2.4 times higher than the highest value reported in the literature for 3C-SiC MEMS resonators. The use of highly doped SiC itself as a heating element opens the possibility for harsh environment applications due to the superior physical properties of SiC and the ability to work in high temperatures.

5. Conclusions

In summary, a SiC bridge resonator with an unprecedentedly large tunable frequency range has been developed. The significant frequency shift was realized by employing a highly-doped 3C-SiC as structural layer as well as heating element eliminating the requirement for the conventional metallic heaters. Experimental results showed a significant frequency tunability of 5 times under a small bias of 5 V. The tuning capability of our device is superior in comparison to those reported in the literature. Our innovative design and approach open a new pathway towards the development of low-power highly-tunable resonators for a broad range of MEMS applications.

Credit authorship contribution statement

Pablo Guzman: Conceptualization, Investigation, Writing - original draft, Writing - review & editing. **Toan Dinh:** Investigation, Resources, Writing - review & editing. **Hoang-Phuong Phan:** Investigation, Methodology, Writing - review & editing **Abbin Perunnilathil Joy:** Investigation, Visualization. **Afzaal Qamar:** Methodology, Writing - original draft, Formal analysis. **Behraad Bahreyni:** Investigation, Writing - review & editing. **Yong Zhu:** Resources, Writing - review & editing. **Mina Rais-Zadeh:** Resources, Writing - review & editing. **Huaizhong Li:** Validation, Supervision. **Nam-Trung Nguyen:** Supervision, Writing - review & editing. **Dzung Viet Dao:** Conceptualization, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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