

# High-order resonance of single-crystal diamond MEMS with high-quality factor at high temperatures

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**Abstract**—MEMS resonators are typically engineered to function at their first vibration mode, and extensive research has been dedicated to understanding and optimizing this fundamental mode. However, despite the demonstrated effectiveness of operating at high modes for achieving high sensitivity in MEMS sensing applications, there is currently no reported research on higher-order modes at high temperatures. This paper reports an investigation on the resonance properties of higher-order vibration modes of single-crystal diamond (SCD) MEMS, specifically focusing on their performance at elevated temperatures up to 500°C. It is shown that the second mode of SCD cantilever exhibits a high-quality factor of 9,541 and high thermal stability of frequency less than 10 ppm/°C at 500°C.

**Keywords**—diamond MEMS, high-order mode, high temperature, high quality factor

## I. INTRODUCTION

Nano-electromechanical systems (NEMS) and micro-electromechanical systems (MEMS) have attracted considerable attention as emerging technologies and are becoming increasingly important in both research and engineering applications [1]. For MEMS/NEMS devices, high frequency and low dissipation or high quality ( $Q$ ) factor are typically desirable, resulting in high sensitivity, high speed and increased resilience to mechanical noise sources [2]. Single-crystal diamond (SCD) is emerging as an ideal semiconductor material for MEMS resonant sensors in challenging environments contributed to its exceptional properties, i.e., the highest mechanical properties, such as the highest Young's modulus, extreme hardness, and thermal stability of resonance frequency [3-5].

In our previous work, we successfully accomplished the mass production of SCD MEMS resonators by a smart-cut method [6]. And we investigated the higher-order resonance of SCD cantilever sensors toward high  $fQ$  products [7]. Additionally, we achieved diamond MEMS resonators magnetic sensing up to 500°C by operating the first mode [8]. Furthermore, higher-order resonance modes have been demonstrated to achieve high performance in MEMS resonators [9]. Nevertheless, the challenge of efficiently exciting these higher order modes, mainly due to their small amplitudes, has significantly limited their systematic investigation as a method to enhance the performance of MEMS resonators. Therefore, it is of great importance to examine the higher-order modes of MEMS resonators at high temperatures. In this work, we aim to investigate the performance of higher-order modes of SCD MEMS

cantilevers in high temperature environments. We measured the resonance frequency and  $Q$  factors of diamond cantilevers in a higher-order vibration mode at high temperatures up to 500°C.

## II. DESCRIPTION OF METHODS

In this study, SCD MEMS cantilevers with high  $Q$  factors were fabricated by a smart-cut method using ion implantation assisted lift-off (IAL) technique [10]. Prior to homoepitaxial growth of the SCD, a graphite-like sacrificial layer was embedded in the SCD substrate using high-energy ion implantation. The SCD cantilevers were then released by a combination of photolithography, reactive ion etching and wet etching to remove the sacrificial layer.

We report resonance properties of the high-order vibration mode in SCD MEMS cantilevers at high temperatures up to 500°C, using an in-situ measurement equipment. We investigated the resonance performance and thermal stability of the diamond cantilevers of the second mode in high-temperature environment. The resonance frequency of cantilevers is characterized using the Euler-Bernoulli beam theory,

$$f_n = \frac{\omega_n}{2\pi} = \frac{\lambda_n^2 t}{2\pi L^2} \sqrt{\frac{E}{12\rho}} = k_n \frac{t}{L^2} \sqrt{\frac{E}{\rho}} \quad (1)$$

wherein  $n$  denotes the resonance mode number,  $\lambda_1 = 1.875$ ,  $\lambda_2 = 4.694$ ,  $k_1 = 0.162$ ,  $k_2 = 1.012$ ,  $E$  and  $\rho$  represent the Young's modulus and mass density of diamond,  $E = 1,100$  GPa,  $\rho = 3.5$  g/cm<sup>3</sup>,  $\omega$ ,  $L$ , and  $t$  represent the angular resonance frequency, length, and thickness of diamond cantilever, respectively [11]. All resonance spectra were recorded after several cycles until stability was achieved and frequencies were determined by fitting the resonance spectra to a Lorentzian function. Energy dissipation, quantified as the inverse of  $Q$  factor, denoted as  $Q^{-1}$ , is a key parameter for MEMS cantilevers, which determines the properties of MEMS devices, particularly their sensitivity. Dissipation represents the proportion of mechanical energy lost per oscillation cycle ( $\Delta W$ ) relative to the overall mechanical energy ( $W_0$ ) possessed by the resonator, defined as

$$Q^{-1} = \frac{\Delta W}{2\pi W_0} \approx \frac{\Delta f_r}{f_r} \quad (2)$$

Here,  $Q$  factors are estimated by analyzing from the full-width at half maximum (FWHM) through Lorentzian fitting,  $f_r$  represents the resonance frequency, while  $\Delta f_r$  corresponds to the FWHM [12]. The temperature dependence of the

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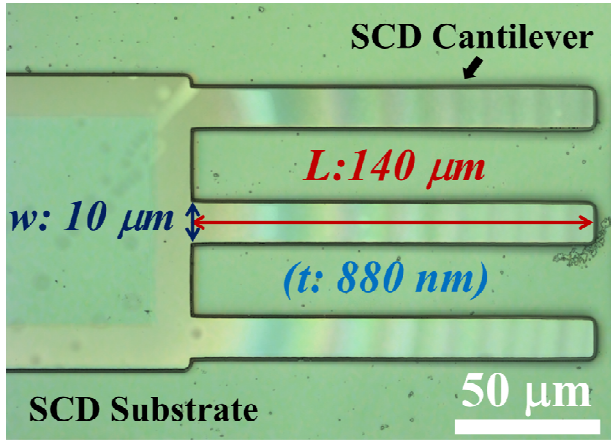


Fig.1. Optical image of typical diamond MEMS cantilevers with length of 140  $\mu\text{m}$ , fabricated by a smart-cut method.

resonance frequency and  $Q$  factor variations for the SCD cantilevers in high-order mode were analyzed.

### III. RESULTS AND DISCUSSIONS

The optical image of the SCD cantilevers is depicted in Fig. 1, which was on a SCD substrate with an epitaxial diamond layer. These MEMS cantilevers were fabricated by the smart-cut method, possessing dimensions of 140  $\mu\text{m}$  in length ( $L$ ), 880 nm in thickness ( $t$ ), and 10  $\mu\text{m}$  in width ( $w$ ). In Fig. 2, the resonance frequency spectra of the diamond cantilever operating in the first two vibration modes at 500°C is presented. The resonance frequency of the high-order is measured at 2.073 MHz, with a high  $Q$  factor of 9,541 calculated by Lorentz fitting.

Fig. 3 shows the resonant frequency shift of the cantilever in the second order vibration across the temperature range from 25 to 500°C. It can be seen that the resonance frequency decreases with increasing temperature because of the decrease in Young's modulus, as described in (1). Due to the ultra-low thermal expansion coefficient of SCD, the temperature effect on the cantilever dimensions and the mass density can be neglected [13]. The resonant

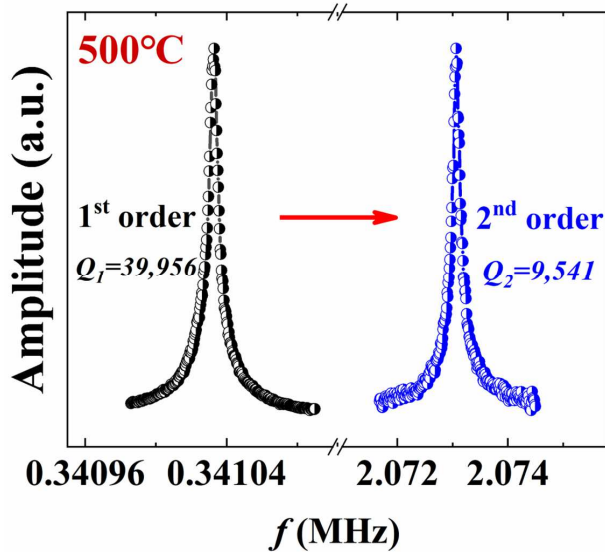


Fig.2. The resonance frequency spectra of a 140  $\mu\text{m}$ -length SCD cantilever in the first two vibration modes at 500°C.

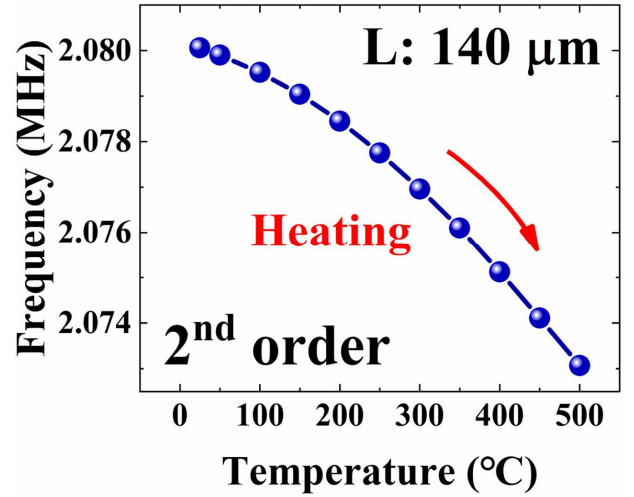


Fig.3. Dependence of resonance frequency at high temperatures in the 2nd order vibration mode of a 140  $\mu\text{m}$ -length diamond MEMS cantilever.

frequency is therefore primarily determined by the modulus of the Young's modulus under different temperatures. A model has been proposed to explain the effect of temperature on Young's modulus through the equation

$$E = E_{T0} - A \exp(-T_0 / T) \quad (3)$$

where  $E_{T0}$  represents the Young's modulus of the material at temperature  $T_0$ , and  $A$  is a constant [14].

The temperature coefficient of the resonant frequency (TCF) of the SCD cantilever is expressed as

$$TCF = (\Delta f / f_0) / \Delta T \quad (4)$$

where  $\Delta f = f - f_0$ ,  $f_0$  represents the resonant frequency with the temperature of 300 K. Despite the resonance frequency shifting towards lower frequencies with increasing temperature, the TCF for the diamond MEMS cantilever remains below 10 ppm/°C up to 500°C, as depicted in Fig. 4.

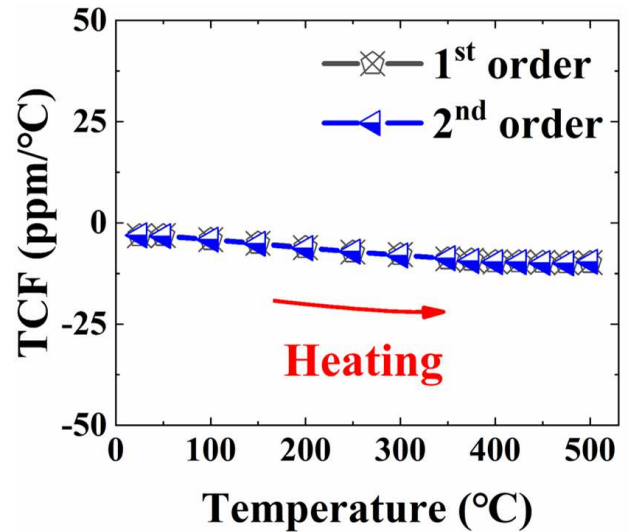


Fig.4. Temperature coefficient of resonance frequency (TCF) of the diamond MEMS cantilever in the first two orders.

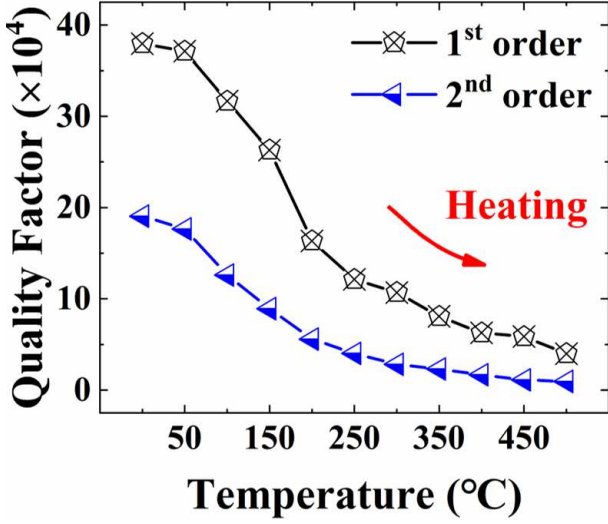


Fig.5. Dependence of  $Q$  factors at high temperatures in the first two vibration modes of the diamond MEMS cantilever.

This performance surpasses that of silicon, which has a  $TCF$  value of 35 ppm/°C [15]. The lower  $TCF$  value in diamond indicates superior thermal stability of the second-order mode, similar as that of the fundamental mode. The resonance frequencies of these cantilevers in both the first and second modes exhibit a slightly temperature-dependent behavior.

Fig. 5 exhibits the temperature dependence of the  $Q$  factors in the first two orders. It is evident that the  $Q$  factors of the diamond cantilever in both modes decreased at high temperatures, attributed to the energy dissipation of thermoelastic damping ( $Q_{TED}$ ). The dissipation mechanism for mechanical resonators working in vacuum is described as below

$$Q^{-1} = Q_{clamp}^{-1} + Q_{TED}^{-1} + Q_{MD}^{-1} + Q_{surface}^{-1} \quad (5)$$

Here,  $Q_{clamp}$  means clamping losses,  $Q_{MD}$  means mechanical defects, and  $Q_{surface}$  means surface loss. The clamping loss is defined as the energy transferred at the nodes into the support. Additionally, mechanical defects, including substitutional impurities, interstitial motion, grain-boundary sliding, and point defects, play a role in dissipation. This occurs because these defects reconfigure between equilibrium and metastable states within the dynamic strain field [16]. Energy losses due to air damping are negligible due to measurement in high vacuum ( $\sim 10^{-4}$  Torr). Thermoelastic losses are not dominant at room temperature as the  $Q_{TED}$  exceeds  $10^7$  [17]. However, at high temperatures, thermoelastic losses become dominant. The higher temperature, the less difference of  $Q$  factors between the first order and the second order, as shown in Fig. 5.

The product of resonance frequency and  $Q$  factor, denoted as  $f \cdot Q$ , stands as another key figure employed for evaluating the response speed and sensitivity of MEMS devices. Its enhancement can be facilitated by elevating the frequency within higher orders of vibration modes. The sensitivity of MEMS cantilevers in measuring small forces is additionally influenced by thermomechanical noise, defining  $F_{min}$ , expressed as

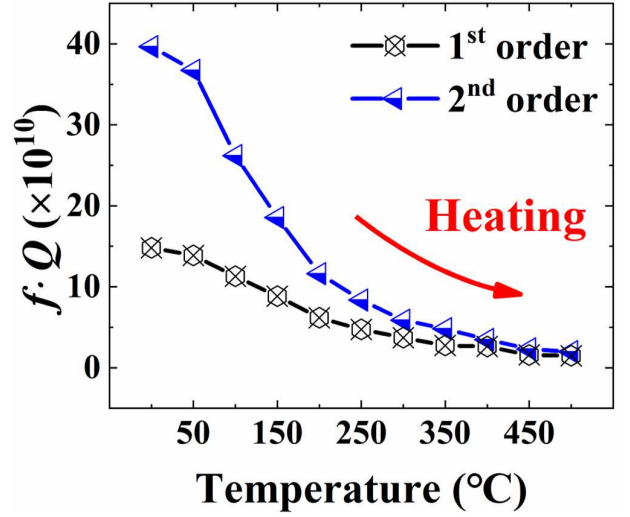


Fig.6. Dependence of  $f \cdot Q$  at high temperatures under the second order vibration mode of diamond MEMS cantilevers.

$$F_{min} = \sqrt{\frac{4k_B T k_C}{2\pi f Q}} \quad (6)$$

assuming a bandwidth ( $B$ ) of 1, where  $k_B T$  represents thermal energy and  $k_C$  stands for the spring constant. This equation demonstrates that  $F_{min}$  decreases as the  $f \cdot Q$  product increases. Consequently, in the design of mechanical resonators, the goal is to maximize the  $f \cdot Q$  product to achieve optimal outcomes.

We illustrate the relationship between working temperature and the  $f \cdot Q$  product in Fig. 6. It is noteworthy that the  $f \cdot Q$  product for the second order mode significantly exceeds that of the first order mode. It is evident that the  $f \cdot Q$  for the SCD cantilevers undergoes a significant increase with higher order resonances. Indeed, the second order mode exhibits an  $f \cdot Q$  value of approximately  $4 \times 10^{11}$  Hz at room temperature, indicating an almost doubling of the  $f \cdot Q$  compared to the first order mode. It is clear that the  $f \cdot Q$  product demonstrates a decrease with rising temperature in both the first and the second order. Nevertheless, even at the high temperature of 500°C, the  $f \cdot Q$  product persists at approximately  $10^{10}$  Hz. This emphasizes the potential for enhancing the  $f \cdot Q$  value by employing higher-order resonance mode.

#### IV. CONCLUSION

In conclusion, we investigated the higher-order resonances in SCD cantilevers at high temperatures up to 500°C. We observed that for both the first and second order modes, the resonance frequency decreases with increasing temperature. The high-order mode of the diamond cantilever exhibits low  $TCF$  of 10 ppm/°C and high  $Q$  factor of 9,541 at high temperature of 500°C. The high thermal stability of the resonance frequency and the high  $Q$  factors in the higher-order mode of diamond cantilevers ensure high performance, including reliability and sensitivity for MEMS resonant sensors, particularly in demanding environments.

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