1. Introduction

Atomically thin two-dimensional (2D) materials such as graphene and transition metal dichalcogenides (TMDC) are attractive for nano-electro-mechanical systems (NEMS) due to their excellent electrical and mechanical properties [1–5]. In particular, mechanical resonators (MRs) composed of atomically thin 2D materials have widely been investigated [5–20]. In addition, stacked 2D materials of graphene and MoS2 have also applied to MRs, which is expected for exploiting mechanical effects of heterojunction and interlayer interactions [21]. Owing to their extremely light weight, they show a very high resonance frequency, which is beneficial for force sensor applications. Broad dynamic range of the linear oscillation regime on the electrically driven MoS2-MRs, which is also important not only for sensing application but also other novel applications such as ultralow power information processing, has been reported [22]. In addition, oscillators with a tunable resonance frequency [22, 23] are candidates for the base-clock element in digital circuits. Toward computing applications, atomically thin 2D MRs exhibit energy transfer across the oscillation modes [24, 25], but depending on the temperature, the thermal stress on atomically thin 2D membranes of MR modulates the resonance frequency [7, 18, 19].

This modulation results in unexpected fluctuations in the resonance frequency. These temperature induced fluctuations prevent stable operations of MR devices. For instance, strong perturbations make accurately measuring the force using atomically thin 2D MR difficult. Although suppression of the temperature dependence of the resonance frequency at a specific gate bias condition has been reported [7], the tunability of the resonance frequency is limited in this case. Thus, the tuning of the resonance frequency should be independent of the bias condition.

Thermal stress is a general problem not only in atomically thin 2D MR but also in conventional bulk NEMS. Temperature fluctuations easily affect the stability of NEMS-MR. To suppress the thermal stress, the thermal expansion coefficient (TEC) of MRs, which consist of bulk materials, have been tuned by chemical doping or introducing defects in the materials [26–28]. However, both approaches may degrade the superb electrical and mechanical properties of atomically thin 2D materials. Especially, the introduction of defects degrades the quality factor of the resonance [29], which is the most important figure of merit for MR, even in nanoscale resonators. Hence, establishing simple techniques to tune TEC for atomically thin materials is desirable.

Tuning of the electrical properties in atomically thin 2D materials is widely investigated by stacking...
different kinds of atomically thin 2D materials [30–34] to form a van der Waals (vdW) heterojunction, which results in the promising applications such as ultra-high-gain photodetectors [35]. In a vdW, the stacked 2D materials have different electrical properties such as electronic band structures. In this regard, we expect that MRs with a vdW heterojunction [21] can tune the mechanical properties of atomically thin 2D materials. Herein we demonstrate that stacking graphene and MoS2, which have different signs of TECs, suppresses the temperature dependence of the frequency shift in atomically thin 2D MR.

2. Experimental section

Figure 1(a) schematically illustrates the MoS2/graphene stacked MR, which is drum type resonator with a typical diameter of 6 μm. The stacked MoS2/graphene is put on a pair of electrodes, which act as source and drain electrodes. The heavily doped Si (n−-Si) substrate acts as a back gate. Trenches at both sides of the drum were served as an evacuation channel for air between the graphene drum and the substrate during measurement of resonance characteristics in vacuum. The fabrication process as follows. First, metal electrodes consisting of Cr/Au (5 nm/30 nm) as a support of the graphene drum were fabricated on a heavily doped n-type Si substrate (<0.02 Ωcm) with a 300 nm-thick SiO2 layer using conventional photolithography. After the monolayer graphene was transferred onto the substrate using polymethyl methacrylate, the sample was trimmed using oxygen plasma etching to form the drum, where the graphene was synthesized using low-pressure chemical vapor deposition at 1050 °C and Cu foil as a catalyst [36, 37]. MoS2 flakes were transferred on graphene by the conventional polydimethylpolysiloxane (PDMS) gel stamp method [34], where bulk MoS2 was mechanically exfoliated with a thermal release tape and subsequently transferred onto the PDMS gel stamp.

To form the drum-type graphene/MoS2 (G/MoS2)-MR suspended by metal electrodes, the SiO2 layer underneath the graphene drum was etched using buffered HF, where the metal electrodes were used as the metal mask for etching [17]. Trenches on both sides of the drum are necessary for uniform etching of the SiO2 layer. The samples were finally dried using supercritical drying to prevent sticking of the suspended structure induced by the surface tension of water.

Figure 1(b) shows a scanning electron microscope (SEM) image of the prepared sample. The gap between the G/MoS2 membrane and the substrate is 300 nm, which corresponds to the SiO2 layer thickness. The diameter of the drum part of the G/MoS2-MR is 6 μm. As for control, we prepared a graphene-only MR (G-MR) as well as a G/MoS2-MR. Figures 1(c) and (d) show the Raman spectra for MoS2 and graphene after the fabrication process, respectively. For MoS2, first-order Raman active modes at 383.8 cm⁻¹ (E₂g) and 406.9 cm⁻¹ (A₁g) are clearly observed. The difference between the peak positions depends on the number of layers [38]. From the peak position difference of 23.1 cm⁻¹, we determined that there are three MoS2 layers. The intensity ratio of 2D/G bands is larger than 2, where G and 2D bands are peaks observed at 1590 and 2690 cm⁻¹, respectively [39]. This indicates that the graphene is monolayer. It should be noted that the D band peak for graphene observed at 1350 cm⁻¹ originating from defects is hardly observed, indicating that the fabrication process induces few defects.

3. Results and discussion

For the resonance property measurements, the AM modulation down mixing method [40] was used. As shown in figure 2(a), the graphene mainly acts as the FET channel. All measurements were performed in a vacuum (less than 10⁻³ Pa) after annealing at 150°C for 2 h. Figure 2(b) shows the DC transfer characteristic of the G/MoS2-MR without AC modulation at a source-drain voltage, V_{DS}^DC = 5 mV. The gate voltage, V_{gs}, successfully controls the drain current, I_{DS}^{DC}, even for the suspended graphene/MoS2 stacking channel after the sample preparation process. It should be noted that the drain currents for MoS2 FETs with a similar structure (less than 100 nA) show much smaller channels than that for graphene under V_{DS}^{DC} = 5 mV. Thus, the current passing through the graphene layer mainly contributes to the measured I_{DS}^{DC}. To prevent the G/MoS2 membrane from sticking onto the substrate, the gate bias is limited to less than 5 V.

Figure 2(c) shows the frequency response curves of the G-MR under various V_{gs} when V_{DS}^{AC} = 7 mV_{rms} without a DC component is applied at a modulation frequency of 1 kHz and a modulation depth of 99%. The small arrows denote the resonance frequency, f_r. As V_{gs} increases, f_r shifts toward a higher frequency due to the increased internal strain of the graphene membrane.

Figure 2(d) shows the frequency response curves of G/MoS2-MR. Similar to G-MR in figure 2(c), the resonance frequency shifts toward a higher frequency with increasing V_{gs}. The mixed down current (I_{MOD,AM}) measured for G/MoS2-MR is attributed to the current passing through the graphene layer due to the higher conductivity of graphene than that of MoS2 as mentioned above.

These results demonstrate that an electrical method can measure the resonance curves of G/MoS2-MR. The frequency responses for both samples are well fitted to the linear response curve calculated with a parameter for the phase differences between V_{DS}^{AC} and I_{AM,MOD} for all V_{gs}. Thus, we conclude that the frequency responses observed in these experiments are in the linear response regime. It should be noted that the low cutoff frequency (less than 1 MHz) of the MoS2...
Figure 1. Drum-type graphene/MoS$_2$ MR. (a) Schematic of G/MoS$_2$-MR. (b) SEM image of G/MoS$_2$-MR. Raman spectrum for the drum area of G/MoS$_2$-MR for (c) MoS$_2$ and (d) graphene related peaks.

Figure 2. Electrical measurement of the atomically thin 2D MR. (a) Schematic of the measurement setup for the mechanical resonance properties. (b) DC transfer characteristic of the suspended G/MoS$_2$ FET. Frequency response curves measured under various $V_{gs}$’s for (c) G-MR and (d) G/MoS$_2$-MR. Baselines of $I_{MOD\_AM}$ for respective $V_{gs}$ are shifted artificially in the horizontal axis to clarify the resonance frequency shift. Small arrows in the respective curves are the resonance frequencies under each $V_{gs}$. 
FET in the experimental conditions prevents measurements of the frequency response of MoS_2-only MR.

Figures 3(a) and (b) show the V gs dependences of f_0 for the G-MR and G/MoS_2-MR measured under various temperatures, ΔT, above room temperature (296 K) in a vacuum, respectively. For both samples, the resonance frequencies increase with increasing V gs at all temperatures. At V gs < 4 V for G-MR, f_0 depends on ΔT, and increases with increasing ΔT. This is most likely due to the negative TEC of graphene [7]. At V gs = 4.5 V, the ΔT dependence of f_0 almost vanishes. Further increasing V gs corresponds to the higher strain and results in the opposite temperature dependence of f_0. A similar behavior [7] has been already reported in the low temperature regime as discussed later. In contrast to G-MR, the temperature dependence of f_0 for G/MoS_2-MR is greatly suppressed, even under a lower strain (V gs < 4 V). However, like G-MR, the temperature dependence of G/MoS_2-MR seems to decrease at higher V gs. Note that the graphene and MoS_2 show the different temperature dependences of the electrical resistance. Although this might affect the experimental results obtained here, the current passing through the MoS_2 layer is negligible small as mentioned before. As a result, the temperature dependence of the electrical resistance is ignorable in the case of the evaluation of the resonance frequency. To discuss the temperature dependence of the oscillation amplitude, we would consider the temperature dependence of the electrical properties. This is a subject for further study.

To clarify the temperature dependence, figures 4(a) and (b) plot Δf_0/f_0 against ΔT for G-MR and G/MoS_2-MR, respectively, where Δf_0 is the resonance frequency shift measured at room temperature for each V gs. The temperature dependence of Δf_0/f_0 for G-MR strongly depends on V gs at V gs < 4 V, as mentioned in figure 3(a). At V gs = 4.5 V, the temperature dependence almost disappears and becomes negative at higher V gs. Thus, the temperature dependence Δf_0/f_0 can be tuned by V gs. However, it is hard to tune the resonance frequency by V gs with remaining the minimum temperature dependence, limiting the practical applications for a tunable oscillator. In the case of G/MoS_2-MR shown in figure 4(b), the temperature dependence is greatly suppressed to less than 2% for ΔT = 10 K, even
at low $V_{go}$. This is one order of magnitude smaller than that for G-MR. Hence, the resonance frequency can be tuned by $V_{gs}$ with small fluctuations in the resonance frequency against the temperature.

Figure 4(c) summarizes the temperature coefficient of $\Delta f_0/f_0$ for the respective $V_{gs}$ estimated from figures 4(a) and (b). As mentioned before, the temperature coefficient of $\Delta f_0/f_0$ decreases with increasing $V_{gs}$, which corresponds to the increase in the internal strain. The reported values of TEC for graphene $\alpha_{gra}$ and MoS$_2$ $\alpha_{MoS_2}$ at room temperature are in the range of $-3 \sim -8 \times 10^{-6}$ K$^{-1}$ [7, 41] and $-5 \times 10^{-6}$ K$^{-1}$ [4], respectively. Although the opposite signs of TECs cause the membrane to bend, this thermal stress is relaxed due to the presence of many wrinkles, as observed in figure 1(b). In addition, the MoS$_2$(3L)/graphene(1L)-MR with a 6 $\mu$m diameter examined here can be treated as a membrane [2, 13], where the membrane tension is the dominant parameter in the resonance frequency. If this model is valid, membrane bending has a limited impact on the temperature dependence of the resonance frequency.

The fundamental resonance frequency is given as

$$f_0 \approx \frac{E_{gra}^{\infty} \alpha_{gra} + E_{MoS_2} \alpha_{MoS_2} n_{MoS_2}}{E_{gra}^{\infty} + E_{MoS_2} n_{MoS_2}}$$

(1)

where $E_{gra}$ and $E_{MoS_2}$ are the monolayer Young’s moduli, and $\alpha_{gra}$ and $\alpha_{MoS_2}$ are the number of layers of graphene and MoS$_2$, respectively. In our experiments, the apparent TECs are in the range of $-1.8 \sim 1.3 \times 10^{-6}$ K$^{-1}$ owing to the compensation of $\alpha_{gra}$ and $\alpha_{MoS_2}$ upon stacking, which results in a smaller temperature dependence of the resonance frequency of G/MoS$_2$-MR at a wider range of $V_{gs}$.

Table 1. Parameters used for the numerical calculations.

| $d_0$ (µm) | $d_1$ (µm) | $E_{gra}$ (TPa) | $E_{MoS_2}$ (TPa) | $n_{gra}$ | $n_{MoS_2}$ | $\rho_{gra}$ (kg m$^{-3}$) | $\rho_{MoS_2}$ (kg m$^{-3}$) | $\epsilon_{gra}$ (nm) | $\epsilon_{MoS_2}$ (nm) | $T_{gra}$ (nm) | $T_{MoS_2}$ (nm) |
|------------|------------|-----------------|-------------------|-----------|-----------|----------------|----------------|----------------|----------------|--------------|--------------|
| 6          | 0.3        | 1.0             | 0.3               | 1         | 3         | 2250           | 5060            | 0.34           | 0.67           |              |              |

where $\Gamma$ is the tension of the membrane in plane, $\rho$ is the apparent mass density of the resonator, and $t$ is the thickness. $\epsilon_0$ is the electric constant and $d_t$ is the gap between the membrane and the substrate for the electrostatic attraction. The apparent mass density is given by $\rho = \frac{\rho_{gra} n_{gra} + \rho_{MoS_2} n_{MoS_2}}{t_{gra} + t_{MoS_2}}$, where $\rho_{gra}$ and $\rho_{MoS_2}$ are the mass densities, and $t_{gra}$ and $t_{MoS_2}$ thicknesses of graphene and MoS$_2$, respectively. The tension, $\Gamma \left( V_{go}, \Delta T \right)$, is expressed as

$$\Gamma \left( V_{go}, \Delta T \right) \approx \Gamma_0 + \frac{Et d_0^2 \epsilon_0}{\Gamma_0} V_{go}^4$$

(3)

where

$$\Gamma_0 \left( \Delta T \right) \approx \Gamma_0 (1 - \frac{Ed_t}{\Gamma_0} \alpha_{eff} \Delta T)$$

(4)

$\Gamma_0$ is the initial tension at $\Delta T = 0$ and $V_{gs} = 0$. $E$ is the apparent Young’s modulus of the membrane, and $\alpha_{eff}$ is the effective TEC of entire device, including the membrane, substrate, and electrodes. We further assumed that graphene and MoS$_2$ have the same Poisson’s ratio. Thus, the apparent Young’s modulus of G/MoS$_2$-MR is $E \approx \frac{E_{gra} n_{gra} + E_{MoS_2} n_{MoS_2}}{n_{gra} + n_{MoS_2}}$. Finally, inserting equations (3) and (4) into (2) gives

$$f_0 \left( V_{go}, \Delta T \right) = f_0^0 \left( 1 - \frac{b \Delta T}{\Gamma_0} \right) + \frac{a}{\Gamma_0^2 (1 - \frac{b \Delta T}{\Gamma_0})}$$

$$\times V_{go}^{4} - \frac{d_0^2 \epsilon_0}{\pi T_{00}} V_{go}^{4 \frac{1}{2}}$$

(5)

where $f_0^0 = \frac{1}{2} \left( \frac{\Gamma_0}{d_0^2 \epsilon_0} \right)^{1/2}$, $a = \frac{E \epsilon_0 d_t^2}{96 d_t}$, and $b = Ed_t$. We fitted the experimental results to equation (5) (figures 3 and 4, solid lines). Note that because we assumed the drum is rectangular, the geometrical factors considered in equation (5) are invalid.

Even under this rough assumption, both the $V_{gs}$ and $\Delta T$ dependences qualitatively agree well with equation (5), except for G/MoS$_2$-MR at $\Delta T = 0$ (figures 3 and 4). From the fitting parameters, we roughly evaluate the apparent TECs of G-MR and G/MoS$_2$-MR to be $-1.6 \times 10^{-6}$ K$^{-1}$ and $-5.9 \times 10^{-7}$ K$^{-1}$, respectively, Table 1 summarizes the parameters used in this fitting. As expected, the apparent TEC of G/MoS$_2$-MR is greatly suppressed to 1/3 of G-MR by creating a vdW heterojunction (figure 4). In this way, the temperature dependence of the resonance frequency of the atomically thin MR is successfully manipulated without tuning the resonance frequency by the gate bias.
4. Conclusions

This study investigated the apparent TEC of atomically thin membranes using an electrostatically actuated MR from their mechanical resonance frequencies. Stacking two different atomic layers, graphene and MoS$_2$, which have the opposite signs of TECs, suppresses the apparent TEC of the atomically thin drum-type MR. Due to the reduction in the apparent TEC, the temperature dependence of the resonance frequency shift decreases without the detraction of the tunability of the resonance frequency by V$_{gs}$. Consequently, the resonance frequency shift shrinks from 0.25 to 0.15% K$^{-1}$ as the electrostatic attraction increases. We believe that this strategy, which manipulates TEC, will realize additional applications of atomically thin MR and eventually achieve highly accurate NEMS sensors with an improved temperature stability.

Acknowledgments

This work was partially supported by KAKENHI Grant Numbers 16H00920, 16K14259, 16H06504, and 17H01040.

Author contributions

TI and SA conceived and designed the experiments, led the research, and wrote the paper. YM contributed to data analysis. All authors discussed the results and assisted in manuscript preparation. KT and TA contributed to sample preparation. KT and TA contributed to data analysis. All authors discussed the results and assisted in manuscript preparation.

Competing interests

The authors declare that they have no competing interests.

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