BIAXIAL STRAIN IN SUSPENDED GRAPHENE MEMBRANES FOR PIEZORESISTIVE SENSING
A.D. Smith¹, F. Niklaus¹, S. Vaziri¹, A.C. Fischer³, M. Sterner¹, F. Forsberg¹, S. Schröder¹, M. Östling¹, and M.C. Lemme²
¹KTH – Royal Institute of Technology, Stockholm, Sweden, ²University of Siegen, Germany

ABSTRACT
Pressure sensors based on suspended graphene membranes have shown extraordinary sensitivity for uniaxial strains, which originates from graphene’s unique electrical and mechanical properties and thinness [1]. This work compares through both theory and experiment the effect of cavity shape and size on the sensitivity of piezoresistive pressure sensors based on suspended graphene membranes. Further, the paper analyzes the effect of both biaxial and uniaxial strain on the membranes. Previous studies examined uniaxial strain through the fabrication of long, rectangular cavities. The present work uses circular cavities of varying sizes in order to obtain data from biaxially strained graphene membranes.

INTRODUCTION
Graphene has a number of interesting properties, which make it suitable for pressure sensor applications. It consists of a monolayer of carbon atoms and its extraordinary thinness gives graphene membranes a high sensitivity to forces applied to it. In addition, it has a high carrier mobility [2, 3]. Further, it is mechanically stable with a Young’s modulus of 1 TPa [4, 5] and can be elastically strained by up to 20% [6]. Since graphene is one atom thick, graphene membranes will be deflected by small changes in pressure – resulting in changes to its electronic properties. A further advantage of graphene is its impermeability to gasses: not even helium can permeate a graphene membrane [7, 8].

The piezoresistive effect in graphene has been demonstrated by uniaxially straining monolayer graphene membranes, which are sealing air filled cavities [1]. The uniaxial strain was induced by geometry, i.e. with thin rectangular cavities and suspended graphene membranes sealing the cavities. These devices were then placed into a pressure chamber. As air is pumped out of the chamber, the pressure difference between the air in the chamber and the air trapped underneath the graphene membrane changes. This creates a strain on the membrane as the trapped air presses against it. Typical values of the extracted gauge factors for graphene range from 1 to 4 [1, 9, 10]. Very high sensitivity has been achieved for these devices with values that are tens to hundreds of times more sensitive per membrane area than the sensitivity of conventional silicon pressure sensors. This allows for aggressive scaling of graphene-based sensors [1]. Simulations further corroborate measurement data for both uniaxially and biaxially strained graphene [1]. In uniaxially strained graphene, the graphene is strained in only one direction whereas in biaxial strained graphene, the graphene is strained in two directions (dimensions) simultaneously. This work for the first time provides measurements on the piezoresistive effect for biaxially strained graphene. The data is compared to previous simulations as well as uniaxial strain data. The data yields an average measured gauge factor of 6.1. Devices with circular membranes of 18, and 24 µm diameter were investigated in order to assess how the size of the cavity affects sensitivity.

FABRICATION
Devices were fabricated beginning with a p-type doped silicon substrate with a 1.5 µm layer of thermally grown SiO₂. Cavities of various sizes were etched 1.5 µm into the oxide to the substrate (Figure 1) using reactive ion etching (RIE). A second set of 603 nm deep cavities were then etched into the oxide layer for the contact metallization. These cavities were filled with Ti/Au (Figure 2) using metal evaporation and lift off: This creates electrical contacts, which are embedded in the SiO₂ layer. The reason for embedding contacts is to eliminate additional process steps after the graphene is transferred. The contacts are made of 150 nm of Ti to act as an adhesion layer to the oxide and 500 nm of gold. Chemically vapor deposited (CVD) graphene was then transferred from copper foil onto the top of the contacts (Figure 1) using a conventional poly(bisphenol A) carbonate (PC) transfer process and FeCl₃ to etch the copper foil. The graphene was then patterned with a photore sist mask using O₂ plasma and placed in a chip package (Figure 1). SEM images of wire bonded devices are shown in Figure 3. The main image shows a 24 µm circular cavity as used for measurements with biaxially strained graphene. The inset shows a rectangular cavity from previous experiments used for measuring uniaxial strain.

EXPERIMENTAL SETUP
In order to measure the devices, a pressure chamber equipped with a reference commercial pressure sensor was used. The graphene devices were connected to a Wheatstone bridge and current was pulsed in order to eliminate temperature related effects caused by Joule heating. Further, the pressure chamber was vented and pumped with inert gas in order to reduce parasitic gas sensing effects.

RESULTS
Graphene membranes with 24 µm cavity diameter were measured in argon (Non-suspended devices have previously been measured in argon, nitrogen, oxygen, CO₂ and air in order to assess the effect of molecular sensing and it was determined that argon was the most inert and stable
environment [1]). Figure 4 shows the voltage output versus time (blue) of a 24 μm cavity diameter device in argon gas. The voltage change in the device follows reasonably the commercial sensor measuring pressure variation in the chamber for several pump and vent cycles, where argon is pumped out of the chamber and then purged back into the chamber.

Figure 1: Fabrication of graphene pressure sensors. First, thermal SiO₂ is grown on Si at a thickness of 1.5 microns. Then cavities are etched into the substrate using Reactive Ion Etching (RIE). Ti/Au contacts are then patterned on the devices followed by graphene transfer and etching. Finally, chips are packaged and wire bonded.

Figure 2: Schematic: contacts made of 150 nm of Ti and 500 nm of Au embedded into the SiO₂.

Figure 3: SEM image of a wire bonded device with a circular cavity of 24 μm diameter to induce biaxial strain. The inset shows a rectangular cavity device for inducing uniaxial strain.

Figure 4: Pressure measurement in argon gas with a 24 μm diameter cavity device. The percent change in resistance was calculated for the cavity region and used with the average strain to extract an average gauge factor of 6.1.

An equivalent resistance model was made in order to estimate the resistance change in the membrane area of the graphene patch. This model was then used in order to calculate the gauge factor of the devices in a similar method to [1]. The model simplifies the circular area into a square of equivalent area as shown in Figure 5, where the resistance $R_2$ is the resistance of the cavity region. $R_2$ is calculated by taking the total change in resistance and assuming that the resistance of the non-cavity regions remains constant.
In order to estimate the resulting strain in the system, a COMSOL model was used. This model has previously been compared to literature data by Bunch et al. [7] and Koenig et al. [8] and found to be very accurate [1]. Figure 6 shows the simulated deflection of a membrane with a diameter of 24 μm. For a pressure difference of 0.8 bar, the maximum estimated deflection is 0.92 μm. For a given pressure, the average strain is then calculated. Gauge factors are then extracted by taking the percent change in resistance and dividing it by the average change in strain over the membrane. Figure 7 plots gauge factors of circular cavity devices (biaxially strained, red open circles). It further compares them with rectangular cavity devices from [1] (uniaxially strained, blue open squares). An average gauge factor of 6.1 has been derived for biaxial strain, which varies within a reasonable range depending on the pressure difference. Solid squares and circles represent our simulations for uniaxial and biaxial strains, respectively. Finally, we have added simulation data from Huang et al [9], which is in the same range.

We note that in contrast to experiments on uniaxial strain, not all circular devices showed similar behavior to the 24 μm cavity case. We found an 18 μm diameter membrane device with a gauge factor of 89. The comparison of the voltage output and commercial pressure sensor output for this device is shown in Figure 8. Several
things are noteworthy about this device. First, it has a much higher gauge factor than typical. Second, there is less noise in the data compared to all other devices tested. While this is an encouraging result, we are currently lacking a clear explanation for this discrepancy and further investigations are ongoing.

CONCLUSION

We have experimentally investigated the influence of the cavity shape on suspended graphene membrane pressure sensors for the first time. Our results confirm earlier simulations, which predicted gauge factors in graphene that are nearly independent of crystallographic orientation and strain direction. The experimental comparison yields higher gauge factors for the 24 µm devices as in previously reported data. A smaller cavity size of 18 µm yields a gauge factor of 89.

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CONTACT
M.C. Lemme; max.lemme@uni-siegen.de