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Laser Ultrasonic Characterization of Membranes for use as Micro-Electronic Mechanical Systems (MEMS)

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Abstract. Germanium (Ge) on Silicon (Si) has the potential to produce a wide variety of devices, including sensors, solar cells and transistors. Modification of these materials so that a suspended membrane layer is formed, through removing regions of the Si substrate, offers the potential for sensors with a more rapid response and higher sensitivity. Such membranes are a very simple micro-electronic mechanical system (MEMS). It is essential to ensure that the membranes are robust against shock and vibration, with well-characterised resonant frequencies, prior to any practical application. We present work using laser interferometry to characterise the resonant modes of membranes produced from Ge or silicon carbide (SiC) on a Si substrate, with the membranes typically having around 1 mm lateral dimensions. Two dimensional scanning of the sample enables visualisation of each mode. The stress measured from the resonant frequencies agrees well with that calculated from the growth conditions. SiC provides a more robust platform for electronics, while Ge offers better resonant properties. This offers a potential technique for characterising production quality or lifetime testing for the MEMS produced.

INTRODUCTION

Germanium (Ge) and Silicon Carbide (SiC) have been suggested as being suitable for producing devices, such as sensors, avalanche photodiode detectors, and solar cells [1-3]. These materials offer the potential for producing devices on a single chip, taking the technology beyond the current state of the art for silicon (Si) devices [4,5]. By producing a suspended membrane of the material, thus decoupling the Ge or SiC from a silicon substrate, a more rapid and higher sensitivity sensor can be produced, and devices can be built which have a good level of thermal isolation from the environment [4-6]. Such membranes may be fragile, and hence a testing technique is required which will ensure that the devices produced are suitable for the environment in which they are to be used.

A small-scale NDE measurement will ensure that the devices are robust enough. For example, a sensor used for detection of particular vibration frequencies will require a high quality (Q-) factor, offering a narrow bandwidth for detection of vibrations. In contrast, for use in high vibration environments, a high robustness to shock (and hence a lower Q-factor) may be more important. Measurement of the elastic properties of the membrane can deliver such information [7]. To measure the elastic properties, traditionally a static load vs. deflection technique has been used [8]. However, as membranes are made thinner, or with more complicated structures, in order to improve thermal isolation and give a larger vibrational response, such methods can be destructive. A non-contact method will have significant benefits; the alternative is to measure the vibrational properties, identifying resonances of the membrane [5,9].

The use of laser ultrasound to measure the vibrational properties offers the potential for a non-contact, non-destructive measurement of the membrane stress, Q-factor, and any non-linear effects due to large vibration amplitudes. Optical techniques can also be optimised to ensure that spatial resolution is sufficient for measuring membranes with sub-mm lateral dimensions. We have recently published work using laser ultrasound to perform

linear scans of membranes, showing that the properties can be inferred from the displacement profiles obtained [5]. This paper takes the work further, giving full two-dimensional scans of the membranes to ensure that resonant modes are correctly identified and account for mode-mixing. The heating effect due to the use of laser ultrasonics is identified, and the effect on the modes of a vibration displacement amplitude which is too large, leading to non-linear effects, is presented.

THEORETICAL BACKGROUND

The resonances of plates and membranes have been well studied theoretically [10,11]. For resonances governed by the elastic properties of the material, resonant frequencies will be within the range of 10s of kHz for the sizes of sample studied here. For resonances which are stress-governed, the frequencies of the resonant modes are given by

$$f_{vac} = \frac{1}{2} \sqrt{\frac{\sigma}{\rho} \left[\left(\frac{n}{a} \right)^2 + \left(\frac{m}{b} \right)^2 \right]} \quad (1)$$

where f is the resonant frequency in vacuum, ρ is the density of the membrane material, a and b are the membrane lateral dimensions, σ is the residual tensile stress within the membrane, and the mode is described by integers n and m . For the membrane dimensions used here, the fundamental resonance is predicted to be in the region of 50-200 kHz. Previous research has confirmed that the membranes resonate in the stress-governed regime [5]. The stress can be measured by performing a sweep of frequency and identifying resonances from the increased vibrational amplitudes. Resonances are then identified by one- or two-dimensional scans, and the stress can be calculated using equation 1. It is essential to also consider the effect of atmospheric damping, which will shift the resonance by the factor given in equation 2,

$$f_{atm} = \frac{f_{vac}}{\sqrt{1 + 1.34(a\rho_{air} / \rho d)}} \quad (2)$$

where d is the membrane thickness [12]. Note that equation 2 was calculated for circular membranes. The presence of dirt or macroscopic defects such as cracks or changes in boundary conditions will be observed as changes in the expected resonance patterns and / or frequencies [10,11].

EXPERIMENTAL DETAILS

Ge membranes were produced by initially using reduced pressure chemical vapour deposition to form a layer of Ge onto a Si substrate [13]. Due to the different lattice sizes, this leads to compressively strained Ge. However, for high quality, ripple-free membranes, tensile strain is required [14]. This is produced using the two temperature growth method [15]. A freely suspended membrane is then produced using etching of the Si substrate in a carefully chosen pattern using standard lithography techniques. Using this method, membranes with different lateral dimensions and thicknesses can be produced. For this research, Ge on Si membranes were produced with thicknesses between 700 nm and 4 μ m, and side lengths of 1 to 2.5 mm. Further membranes were produced using 3C-SiC on a Si substrate, with thicknesses between 100 nm and 10 μ m, and side lengths of 1.4-2.5 mm. The thickness is measured using white light reflectometry, with a 12% error. Results from selected membranes are presented here, with more detailed results to be published in a forthcoming paper [16].

Figure 1(a) shows an optical microscope image of a typical Ge membrane prior to testing. The membrane was square, with the rounded edges due to the presence of photoresist from the production process; the small black features are dirt on the membrane. These were removed prior to testing to ensure a clean, square membrane for testing. Figure 1(b) shows the experimental set-up for ultrasonic testing. The membrane sample was mounted horizontally on a ring-shaped piezoelectric transducer with a nominal frequency of 700 kHz, but operated over a range of frequencies. This shape of transducer was chosen to enable free-movement of the membrane, and to reduce reflections of the probe laser from the transducer itself. The membrane and transducer set-up were placed within a small vacuum chamber with optical windows, to enable testing at atmospheric and reduced pressures. An Intelligent Optical Systems (IOS) two-wave mixer interferometer with a laser wavelength of 1550 nm was used for detection of the vibration displacements.

This system is optimised for use on unpolished samples; for these samples, measurements were performed at very low laser powers to reduce heating and ensure the optimal level of reflected light. A pair of linear stages were used to produce a two-dimensional scanning system, onto which the IOS laser head was mounted. Frequency scans at fixed positions were used to find resonant frequencies, at positions chosen in regions where high displacements are expected for certain modes, for example at the centre and along the diagonal of the membrane [5]. Following this, positional scans were performed using a raster-scan pattern at fixed frequencies to identify the modes.

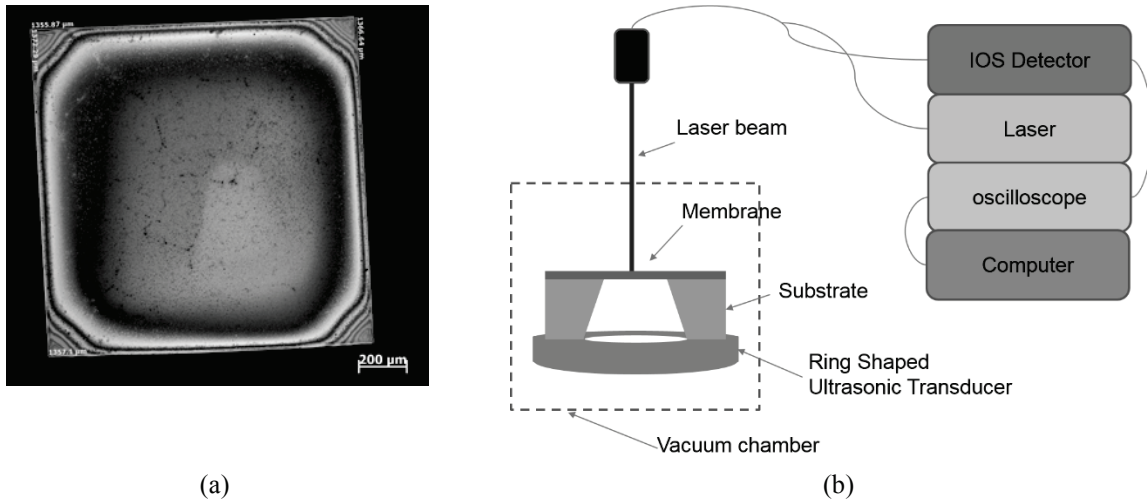


FIGURE 1. Experimental details. (a) Optical microscope image of a Ge on Si membrane, prior to vibrational testing. (b) Measurement set-up used for the scans.

The interferometer gives a measurement of the displacement of the membrane at each position. A 200 μm diameter beam was used; this gives sufficient resolution during scanning to identify the modes [5]. The transducer was operated with a continuous sine wave at the chosen frequency, and the displacement measured in a controlled window around this frequency using digital filtering in a LabVIEW program. From this data the peak-to-peak displacement value was obtained, giving a measure of the magnitude of the vibration amplitude at each position / frequency.

RESULTS

Figure 2(a) shows the calculated magnitude of vibration over the membrane dimensions for a typical mode, in this case the mode-mixed 2:3 and 3:2 mode [5,11]. Regions of higher and lower amplitude are predicted across the membrane in predictable patterns. Figure 2(b) shows the experimentally measured mode for a Ge membrane of thickness 700 μm and lateral dimensions of 860 μm , measured at atmospheric pressure at a frequency of 410 kHz. The straight lines indicate the edges of the membrane, with some slippage observed in one of the scanning stages. This shows excellent agreement with the predicted mode shape, allowing confirmation of the resonant mode. On certain membranes, modes were separated, for example 1:3 and 3:1 modes appearing at slightly different frequencies, indicating that the membrane was not perfectly square and/or that there was some inhomogeneity in the stress.

For the membrane presented in Figure 2, the resonances were measured over a wide range of frequencies, and the stress was found to be 0.231 GPa, in good agreement with predictions from the growth procedure. Quality factors of between 15 and 75 were found at atmospheric pressure. On reducing the pressure, resonances become sharper with a higher Q-factor. This gives easier identification of resonances compared to the noise, however, it also brings experimental issues in that the mechanisms for heat loss are reduced. Calculations show a rise of around 13 K when using the guide (red) laser [5], but the measurement laser also has a heating effect which must be quantified.

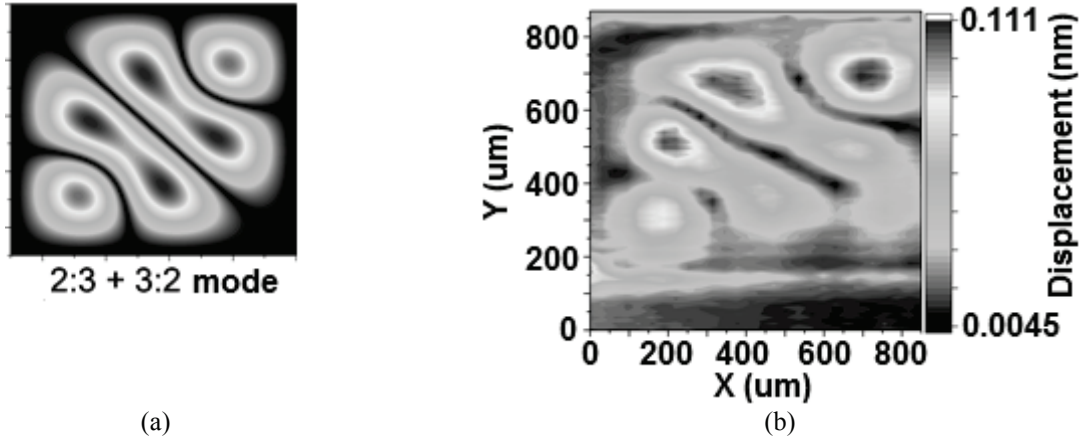


FIGURE 2. (a) Predicted vibration magnitudes for a 2:3 & 3:2 mode-mixing resonance. (b) Scan results for a Ge membrane, 700 nm thick, 860 μm dimensions, at atmospheric pressure and a frequency of 410 kHz.

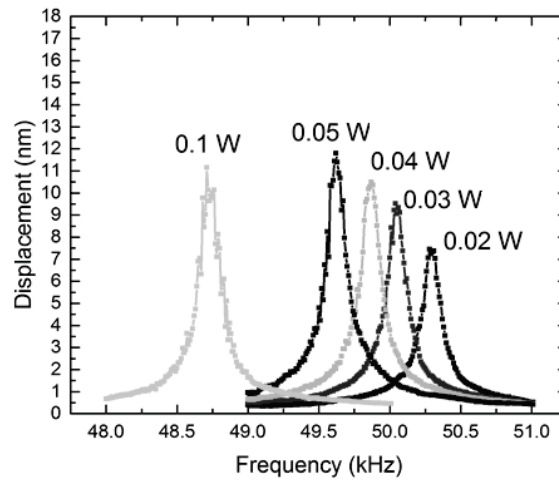


FIGURE 3. Ge membrane, 4 μm thick, 2850 μm dimensions, 50 kHz resonance, at 6×10^{-2} mbar, for different laser powers.

Figure 3 shows the behaviour of the fundamental resonance of a larger Ge membrane as the power of the probe laser is increased from 0.02 to 0.1 W, when the membrane is at a pressure of around 6×10^{-2} mbar, allowing time for thermal equilibrium to be reached. The effect of the heating is obvious; it causes a reduction in the frequency of the resonance, introducing an error into the stress measurement if the heating is not taken into account. The length of time to reach thermal equilibrium also depends on the laser power, with lower power leading to faster settling of the temperature; this was around 20 minutes for a power of 0.02 W, and around 2 hours for 0.1 W. A thermal imaging camera was used to measure the temperature shift at atmospheric pressure, showing an increase in average temperature on the membrane of around 3 K on application of the measurement laser [16]; measurements were not possible at reduced pressures due to the presence of an optical window which absorbed signals at the relevant thermal wavelengths. Similar effects are observed in SiC membranes, with smaller shifts in resonant frequency (of the order of 1% over 2 hours, as compared to over 5% for Ge), due to the different absorptions at the laser wavelength for the two materials.

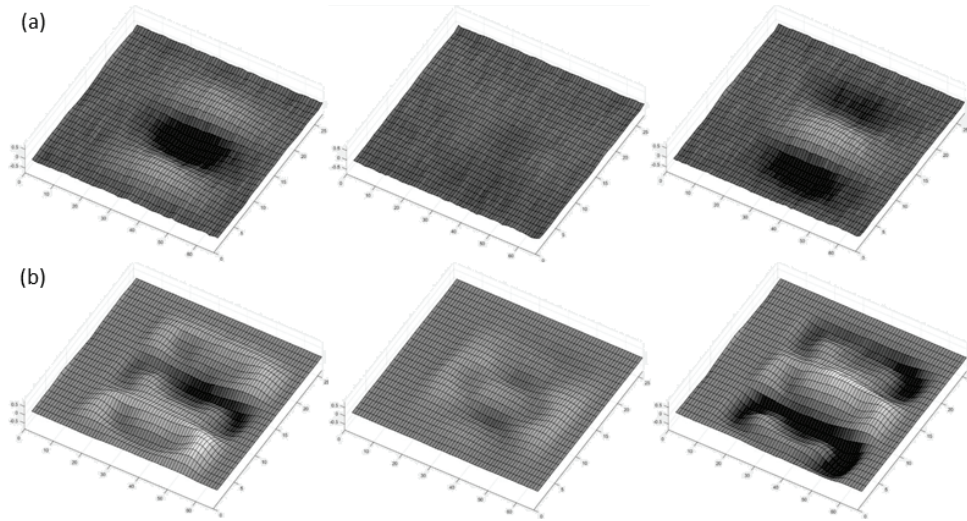


FIGURE 4. SiC membrane, 600nm thick, 206.2 kHz resonance, at 6×10^{-2} mbar. Stills from animation of the motion, taken at (a) 0.1 V excitation voltage, (b) 8.3 V excitation voltage.

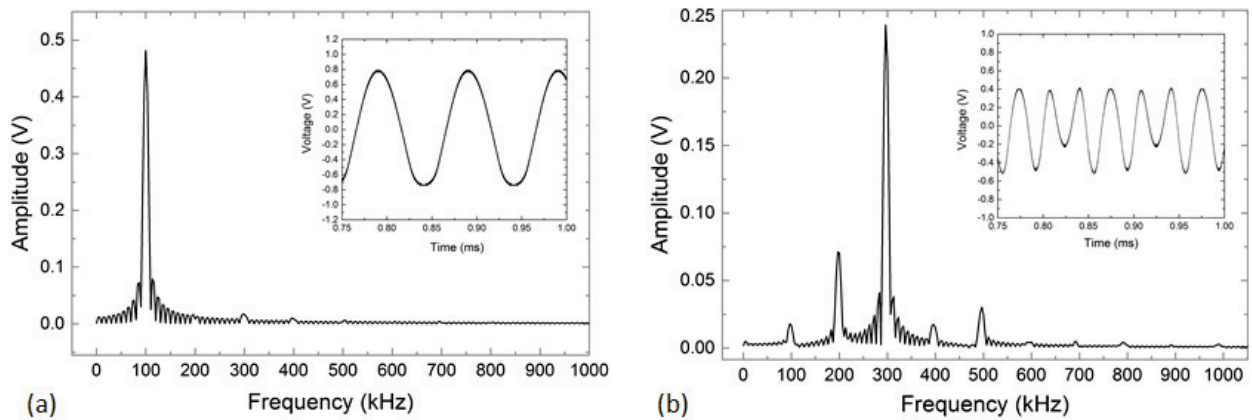


FIGURE 5. Ge membrane, 2 μm thick, 1370 x 1356 μm dimensions, 99 kHz resonance, at 10^{-3} mbar, for different vibration amplitudes. Raw data shown in insets.

On increasing the voltage on the piezoelectric transducer, at atmospheric pressure the expected linear increase in vibration displacement on the membrane was observed, with a maximum displacement of around 30 nm observed for a 3V excitation voltage. At low pressures this is also true for low excitation voltages, with higher vibration displacements due to the reduction in atmospheric damping. Figure 4(a) shows the measured displacements for the 1:3 resonant mode at an excitation voltage of 0.1 V taken at three different times, showing the oscillatory behaviour pattern of the membrane. However, as the excitation voltage is increased, the measurements give the resonant behaviour shown in Figure 4(b) for 8.3 V. This now shows a similar resonance pattern, but with some extra peaks and troughs observed. The raw measured data for two different excitation voltages is shown in the insets to figure 5, along with fast Fourier transforms (FFTs) of the data, for a resonance at a frequency of 99 kHz. This shows the appearance of higher harmonics at higher excitation voltages, with the harmonics dominating the signal at the highest voltages measured. With the reduction in pressure, and hence damping, higher vibration amplitudes are possible. Once a maximum displacement is reached (around 55 nm for Ge, and 70 nm for SiC, with the exact values also depending on the membrane dimensions), the higher harmonics appear as the vibration behaviour becomes non-linear [17].

CONCLUSIONS

The results presented here show the potential for an optical ultrasonic technique for performing NDE on small membrane samples. The measurements can be used to identify the quality of the resonances and robustness to shock, and measure the stress within the membrane, for membranes with sub-mm lateral dimensions. The use of an optical technique has the disadvantage of causing heating of the membrane and associated variations in the measured frequencies of resonances, but preliminary research shows that the frequency shift is likely to be predictable if thermal equilibrium is reached prior to testing [16].

Typical stresses of around 0.23 GPa are found for Ge membranes, while for SiC membranes typical stresses are around 0.38 GPa. Both materials show a relatively low Q-factor at atmospheric pressure, indicating a high robustness to shock. Both materials are prone to non-linear effects if vibration amplitudes are too large. This is an important issue as non-linear oscillations may be detrimental to the long-term operation of devices mounted onto freely supported membranes. Further study is required to identify the link between membrane dimensions, stress, and maximum vibration displacement which should be used, to ensure that materials can be used as sensors without a high rate of failure.

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REFERENCES

1. J. Michel, J.F. Liu, and L.C. Kimerling, *Nature Photonics* **4** 527-534 (2010)
2. Y-H. Kuo, Y.K. Lee, Y. Ge, S. Ren, J.E. Roth, D.A.B. Miller, T.I. Kamins and J.S. Harris, *Nature* **437** 1334-1336 (2005)
3. J. Liu, X. Sun, R. Camacho-Aguilera, L.C. Kimerling and J. Michel, *Optics Letters* **35(5)** 679-681 (2010)
4. V.A. Shah, S.D. Rhead, J. Finch, M. Myronov, J.S. Reparaz, R.J. Morris, N.R. Wilson, V. Kachkanov, I.P. Dolbnya, J.E. Halpin, D. Pratchett, P. Allred, G. Colston, K.J.S. Sawhney, C.M. Sotomayor Torres, and D.R. Leadley, *Solid State Electronics* **108** 13-15 (2015)
5. O. Trushkevych, V. Shah, M. Myronov, J.E. Halpin, S.D. Rhead, M.J. Prest, D.R. Leadley and R.S. Edwards, *Science and Technology of Advanced Materials*, **15** 025004 (2014)
6. V.A. Shah, M. Myronov, C. Wongwanitwatana, L. Bawden, M.J. Prest, J.S. Richardson-Bullock, S. Rhead, E.H.C. Parker, T.E. Whall and D.R. Leadley, *ECS Transactions* **50(9)** 737-746 (2013)
7. D.W. Burns and H. Guckel, *Journal of Vacuum Science and Technology A* **8(4)** 3606 (1990)
8. O. Tabata, K. Kawahata, S. Sugiyama and I. Igarashi, *Sensors and Actuators* **20(12)** 135-141 (1989)
9. A. Biswas, T. Weller and L.P.B. Katehi, *Review of Scientific Instruments* **67(5)** 1965-1969 (1996)
10. A.W. Leissa, *Vibration of Plates* (US Government Printing Office, Washington DC, 1969)
11. L.E. Kinsler and A.R. Frey, *Fundamentals of Acoustics* (John Wiley, New York, 1962)
12. M. Karnezos, *Journal of Vacuum Science and Technology B*, **4(1)** 226 (1986)
13. V.A. Shah, M. Myronov, S.D. Rhead, J.E. Halpin, A. Shchepetov, M.J. Prest, M. Prunnila, T.E. Whall, E.H.C. Parker and D.R. Leadley, *Solid State Electronics* **98**, 93-98 (2014)
14. S.R. Elliott, *The physics and chemistry of solids* (Wiley, Chichester, 1998)
15. V.A. Shah, A. Dobbie, M. Myronov and D.R. Leadley, *Thin Solid Films* **519(22)**, 7911-7917 (2011)
16. L.Q. Zhou, M.J. Pearce, R.G. Prince, M. Myronov, G. Colston, D.R. Leadley, O. Trushkevych and R.S. Edwards, in preparation (to be submitted to Applied Physics Letters)
17. J. Zhu, S. Cai and Z. Suo, *International Journal of Solids and Structures* **47**, 3254-3262 (2010).