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Chinese Academy of Sciences State Key Laboratory of Tranducer Technology Shanghai 200050, China Abstract. Graphene, one of the recently discovered carbon nanostructures, has shown good piezoresistive properties. One of the most important areas of research for graphene sheets, in terms of basic science and application in strain or stress sensors, is the measurement of gauge factors. The gauge factors of various layers of graphene sheets are measured based on the equivalent stress beam. The measurement is carried out using a beam-bending method to detect the change in resistance of graphene sheets in different bending states. The gauge factor ranges from 10 to 15, depending on the number of layers in the graphene sheet. These results reveal the piezoresistance effect of single- and multi-layer graphene sheets, which will be of benefit in the fabrication of microsensors. The resistance of graphene sheets decreases as temperature increases from 20°C to 60°C, and the gauge factor is not very sensitive to changes in environmental temperature. © 2013 Society of Photo-Optical Instrumentation Engineers (SPIE) [DOI: 10.1117/1.JMM.12.1.013009]

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1 Introduction

The development of materials that outperform silicon and are more compatible with nanoelectromechanical systems (NEMS) fabrication technology gives rise to intriguing possibilities. Among these new materials, the family of carbon materials has proven to be attractive as piezoresistor candidates. It is no surprise, therefore, that carbon compounds and allotropes are the most studied and researched materials in the world. Even elemental carbon shows complicated bonding, forming a number of allotropic structures. As a twodimensional (2-D) sp² hybridized form of carbon, graphene is a conceptually new class of material that is only one atom thick. On this basis, it offers new insight into low-dimensional physics. 1-3 A single 2-D sheet of graphene is a hexagonal structure, with each atom forming three bonds with each of its nearest neighbors. These bonds, known as σ bonds, are oriented toward these neighboring atoms and are formed from three of the valence electrons. These covalent carbon—carbon bonds are nearly equivalent to the bonds holding diamond together, giving graphene special mechanical, electrical, optical, and thermal properties.^{4,5} In contrast, graphite is a layered material formed by stacks of graphene sheets separated by 0.3 nm and held together by weak van der Waals forces. Bulk graphite has been studied for decades, but until recently there had been no experiments on graphene. This was due to the difficulty in separating, transferring, and isolating single or multiple layers of graphene for study.6,7

The mechanical and electrical properties of graphene have been investigated using experimental and theoretical approaches. On the experimental side, Frank et al. 8 recently measured the Young's modulus of a stack of graphene sheets (less than five layers) to be 0.5 TPa. More recently, by nanoindenting the center of a free-standing monolayer graphene

membrane with an atomic force microscope. Lee et al. measured the Young's modulus as 1.0 TPa, assuming the thickness of graphene to be 0.335 nm. They also reported the intrinsic breaking strength of graphene as 130 GPa. Metallic conductors subjected to mechanical strain exhibit a change in their electrical resistance. Strain may be compressive or tensile and is typically measured by strain gauges. Frazier and Allen¹⁰ investigated graphite films and found that the maximum gauge factor was 16.8. Kim et al.¹¹ found that the gauge factor of carbon fiber ranged from 100 to 400, which is much higher than for silicon-based piezoresistors. Inspired by this unique property, much research has been devoted to the theoretical and experimental study of the piezoresistance of carbon family materials in order to develop a new generation of piezoresistive sensors. However, there are few reports on the gauge factor of graphene sheet. Nair et al. investigated the piezoresistive effect of graphene films obtained by ultrasound cleavage of graphite in organic solvents and deposition on flexible plastic substrates. 12,13 Fully reversible changes in the resistance were observed as a function of strain that could exceed 8% before the films started to lose their continuity. The piezoresistive gauge factor was up to 30. Lee⁶ reported that the gauge factor of graphene on Ni and Cu films produced by chemical vapor deposition under ambient pressure was 6.1 with an applied strain of up to 1%. The gauge factor of the graphene is different due to the various fabrication methods.

The bandgap of a graphene sheet will change under strain. 14 It is expected that a mechanically excited graphene sheet will undergo some change in resistivity. In this research, the electrical conductance and gauge factor of single- and multilayer graphene sheets were studied using a bending method. The aim was to reveal the resistance effect of tension and/or compression on graphene and to apply the findings to the engineering field.

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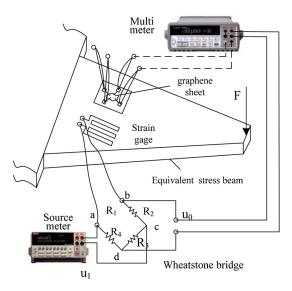


Fig. 1 Schematic representation of the measurement.

2 Experiment

2.1 Method

Figure 1(a) shows a schematic illustration of the measurement method. An equivalent stress beam was used as the bending apparatus. A strain gauge and a silicon wafer deposited with a graphene sheet of a few layers were fixed on the beam's surface. The stress on the surface of the cantilever was equivalent. That means that the single-/multilayer graphene sheet and the strain gauge were subject to the same strain when the cantilever bent downward. The strain could be precisely controlled with the micrometers of the apparatus.

When a concentrated force was applied at the end of the cantilever, the graphene sheet and the strain gauge were in a tension or compression state, leading to a variation in the sample's resistance. According to the relation between the strain and the change rate of the electrical resistance, the gauge factor of the multilayer graphene sheet could be measured with a Wheatstone bridge and calculated according to this definition of the gauge factor:

$$\frac{\Delta R_g}{R_g} = \frac{1}{4} G F_g \cdot \varepsilon, \tag{1}$$

$$\frac{\Delta R_s}{R_s} = \frac{1}{4} G F_s \cdot \varepsilon,\tag{2}$$

where R_g and R_s are the resistances of the graphene sample and strain gauge, respectively; ΔR_g and ΔR_s are the variations of the graphene sample resistance and strain gauge, respectively; ε is the strain of the graphene sheet and the strain gauge on the equivalent stress beam; and GF_g and GF_s are the gauge factors of the graphene sheet and strain gauge, respectively.

Substituting Eq. (1) into Eq. (2), the gauge factor of the graphene sheet could be expressed as

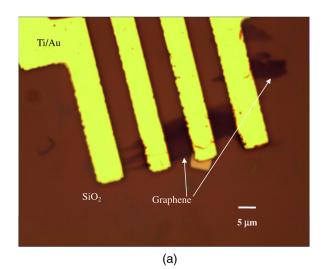
$$GF_g = \frac{\Delta R_g / R_g}{\Delta R_s / R_s} \cdot GF_s. \tag{3}$$

The strain gauge was made of constantan alloy, and the value of the resistance could be established according to the strain gauge parameters.

2.2 Graphene Sample Preparation

The fabrication process began with a silicon wafer with a $300-\mu m$ silicon dioxide layer. First, a 20-nm chromium layer was deposited by electron-beam evaporation on a wafer to fabricate a marker pattern for deposition of the graphene sheet on the wafer surface.

Then the graphene sheets were deposited on the wafer with chromium markers on its surface using a mechanical cleavage method with highly oriented pyrolytic graphite. Next, photoresist was spun coated on its surface, again to define the electrode pattern. After exposure and developing, a Ti/Au metal bilayer was deposited on the top layer. Finally, a liftoff process was carried out. After the photoresist was cleaned in deionized water, the final fabricated graphene sheet contacted with Ti/Au electrodes was obtained. Figure 2 shows the optical microscope and Raman



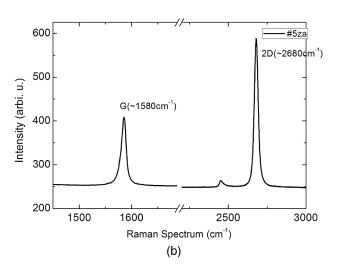


Fig. 2 (a) Optical microscope and (b) Raman spectrometer image of the monolayer graphene sheet.

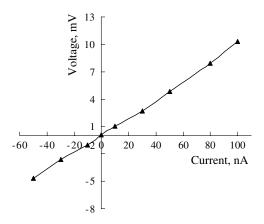


Fig. 3 Current-voltage characteristic of the graphene sheet at room temperature.

spectrometer images of the graphene sheet. In accordance with Gruneis et al., ¹⁵ the number of layers of graphene was investigated using a micro-Raman spectrometer. Figure 2(b) is a Raman spectrometer image of the monolayer graphene sheet. The 2-D band is symmetric and can be fitted into only one Lorentzian peak, and the full width at half-maximum of the 2-D band is 25.6 cm⁻¹. In bilayer graphene, the 2-D band is dispersive and fitted into four Lorentzian bands with different frequencies. The full width at half-maximum of the 2-D band is about 51.2 cm⁻¹, or twice that of monolayer graphene in the Raman spectrum. The full width at half-maximum of the 2-D peak increased as layers were added to the graphene.

3 Results and Discussion

3.1 Current-Voltage Characteristic of the Graphene Sheet

Figure 3 presents a typical current-voltage (I/V) relationship for a multilayer graphene sheet at room temperature. The resistance of the multilayer graphene is about 102 k Ω . From the current-voltage graph, the current was found not to be zero at zero voltage. This may be because a contact resistance and capacitance existed between the metal electrodes and the graphene sample. The metal electrodes were deposited with a liftoff process. Sometimes the fabrication process may have a detrimental effect on the contact between the Ti/Au electrodes and the graphene sample. This may also be a result of random noise during the measurement. During the test, the electrical current ranged from 10 nA to more than 1 μ A, depending on the width of the graphene layer and the separation of the contacts. When a concentrated force was applied on the end of the cantilever and the strain changed from 0% to 0.084%, the graphene sheet's resistance increased from 102 to 114 k Ω . The change was clear and reproducible even after tens of repetitions.

3.2 Effect of the Layer Number

To investigate the effect of the number of graphene layers on the gauge factor, more than 10 graphene samples with different numbers of layers were measured under different bending states. The relationship between the gauge factor and the number of layers of the graphene sheet is given in Fig. 4. It is clear that the gauge factor increased from about 10

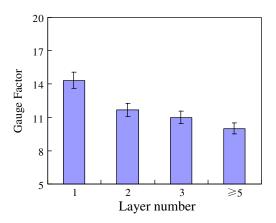


Fig. 4 The relationship between the gauge factor and the number of layers in the graphene sheet.

to 15 as the number of layers in the graphene sheet decreased. The graphene sheet's resistance increased from about 100 to 350 k Ω .

3.3 Effect of the Stress and Temperature

After tensile testing, the force direction applied on the end of the cantilever was changed in order to measure the graphene sheet's resistance under compressive conditions. The results indicated that the graphene's resistance decreased linearly with the increase in the strain. Also, the gauge factor was almost the same as that in the tensile state, considering the measurement errors. Figure 5 shows the results for a single-layer graphene sheet, and the corresponding gauge factor was about 14.3.

Temperature is another important factor that may affect the resistance of the graphene sheet, so the experimental temperature was changed during the measurement. The relationship between the single-layer graphene's resistance and strain at different temperatures is shown in Fig. 6. In contrast with most metals and alloys, the resistance of the graphene sheet decreased slightly as the temperature was increased from 20°C to 60°C. But a change in the gauge factor was not detected. So the gauge factor of the graphene sheet appears not to be sensitive to the change of the temperature, when the environmental temperature was less than 60°C. That would be of benefit in the application of a graphene sensor based on the resistance change.

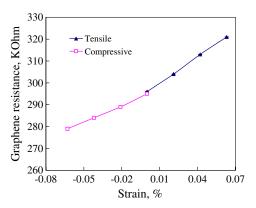


Fig. 5 The resistance of the tensile and compressive single-layer graphene sheet under strain.

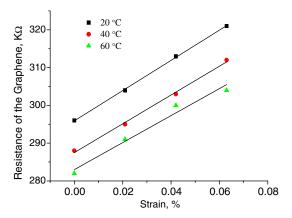


Fig. 6 The relationship between the resistance and the temperature of the single-layer graphene sheet.

4 Conclusions

The electrical resistance and gauge factor of single- and multi-layer graphene sheets were measured with an equivalent stress beam. Nanometer-thick graphene sheets with Ti/ Au electrodes were prepared by exfoliating thin layers of graphite in silicon dioxide films on a silicon substrate. This method is a simple, direct, and nondestructive approach for obtaining the gauge factor of thin membranes using the bending method. The corresponding gauge factor ranged from 10 to 15. Generally, the gauge factor is about 2 for metals and about 20 to 150 for semiconductor materials. 16,17 The graphene sheet had a gauge factor that was higher than those of many metals and smaller than those of semiconductors. But it was not sensitive to change in environmental temperature. Graphene is a transparent material with a high Young's modulus. That would be helpful in the application of the graphene-based sensors. As per other NEMS devices based on molecules such as carbon nanotubes, graphene sheets may be used as piezoresistive sensors to detect strain, force, ¹⁸ or gas concentration in different environments. ^{19–21} Further efforts may yield special applications in the fields of biosensing, microorganisms, and microchemistry.

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