Nano Sensors for Strain Measurement by Micro-Raman Spectroscopy

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Summary In this work, a new mathematical model for the nano sensor was proposed. This model provides a spectral-mechanical relationship between the in-plane strain components to be measured and the experimental data from Raman tests. Then, a novel noncontact technique of strain measurement is developed. A series of experiments using this technique confirmed that the nano sensor described in this paper is appropriate for measuring in-plane strain components at microscale by using polarized Raman spectroscopy.

INTRODUCTION

Strain is expected to have an important role in future devices based on micro-systems. Different methods have been used to engineer strain in devices, leading to complex strain distributions. However, it has proved elusive in practice to measure the strain, especially both normal and shear components, directly and wireless with micro/submicro solutions. Owing to the investigating progresses of nano materials, it is possible to nondestructively detect the strain at microscale by using micro-Raman and nano sensors.[1,2] Because it is well known that some numbers of the nano family, such as carbon nanotube (CNT) and graphene, have outstanding mechanical characteristics and their Raman spectra are very sensitive to deformation.[1,3] In this work, a mathematical and methodological study of the nano strain sensor is presented. The spectral-mechanical model of the in-plane strain components with the Raman data is deduced and a new method of strain measurement named as Raman Strain Rosette is developed.

MATHEMATICAL MODEL OF NANO STRAIN SENSOR

Suppose numerous nano particles, such as carbon nanotubes (CNTs), uniformly dispersed on the surface of (or inside) the measured body (Fig.1). The nanotubes deform with measured body and act as sensors. The data detected directly by Micro-Raman spectroscope is the summation of the total spectra from all the nanotubes inside the laser sampling spot. As for any individual, its axial deformation is directly proportional to the increment of G' band (viz. peak shift) of its Raman spectrum. Therefore, the ones with different axial directions would have different peak shifts as their dissimilar axial deformation. Meanwhole, duo to the antenna effect of polarized Raman[4,5], the nanotubes with different axial to polarizing angles have different Raman intensity. By calculating the synthetic contributions from individuals in random directions to the entire spectrum, the mathematical model of strain sensor is deduced as Eq.1, where $\Delta\Omega^{(\varphi)}$ is detected by Raman spectroscope and with polarizing direction in φ, and the sensor coefficient Ψ_{Sensor} can be calibrated by basic tests.

$$\Delta\Omega^{(\varphi)} = \int_{-\pi/2}^{\pi/2} \left[\Psi_{Sensor} \cdot \left(\varepsilon_X \cos^2\theta + \varepsilon_Y \sin^2\theta - \gamma_{xy}\cos\theta\sin\theta\right)\right] \cdot A_L(\theta-\varphi)\cdot\rho(\theta)\,d\theta \Big/ \int_{-\pi/2}^{\pi/2} A_L(\theta-\varphi)\cdot\rho(\theta)\,d\theta \qquad (1)$$

Eq.1 gives the generalized spectral-mechanical relationships of nano sensor. If taking single-wall carbon nanotubes as sensors and using co-polarized Raman spectroscope with He-Ne laser, Eq.1 is simplified into Eq.2.

$$\Delta\Omega^{(\varphi)} = \frac{1}{6}\Psi_{Sensor} \cdot \left[\left(3+2\cos 2\varphi\right)\varepsilon_X + \left(3-2\cos 2\varphi\right)\varepsilon_Y - 2\sin 2\varphi \cdot \gamma_{XY}\right] \qquad (2)$$

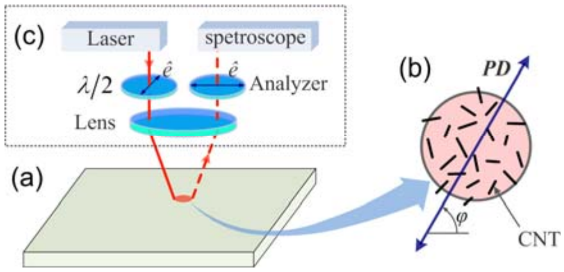

Fig.1. Diagrammatic sketches of nano strain sensors by Raman spectroscopy. (a) Measured body, (b) Carbon nanotubes dispersed uniformly on the surface of the measured body, where ***PD*** is the laser polarizing direction. (c) Polarized Raman spectroscope.

And if grapheme or fullerene, which have no the antenna effect of polarized Raman, $\Delta\Omega = \Psi_{\text{Sensor}}\cdot[\varepsilon_X+\varepsilon_Y]/2$. And if the admixture of kinds of nano materials, the relationship would be insoluble since the sensor coefficient of different nano

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materials are not similar. From the view of phenomenology, an equivalent sensor coefficient can be achieved by calibrating experiments and the nano sensor relationships can be expressed as Eq.3, where T is named as antenna ratio.

$$\Delta\Omega^{(\varphi)} = \Psi_{Sensor\_equ} \cdot \left[T \cdot \varepsilon_X + (1-T) \cdot \varepsilon_Y + (1-2T) \cdot (\varepsilon_X - \varepsilon_Y) \cos^2 \varphi - (1-2T) \cdot \gamma_{xy} \cos\varphi \sin\varphi \right] \quad (3)$$

METHODOLOGY AND EXPERIMENTS OF NANO STRAIN SENSOR

Based on this model, we introduce a novel noncontact technique of strain measurement named Raman Strain Rosette, which detects the Raman peak shift from a same sampling spot with three different polarized directions (shown in Fig.2), and then substitute them into the corresponding relationship (such as Eq.2) to compose a simultaneous equations set. By solving this equations set, the strain components are achieved.

This proposed technique was applied in several experiments such as the following one. The sample is a fiber-reinforced (parallel to the x-direction) epoxy bar. A slot normal to x-direction is prepared at the middle of the bar (Fig.3a) and a single-wall CNT film is attached on the sample. The bar was four-point bend loaded and an area as Fig.3b shows was scanned by Raman and using Raman strain rosette at each spots. The results show that non-uniform deformations exist near the vicinity of the slop tip (Fig3c-e). Particularly, the shear zone (Fig.6e) is parallel to the fiber direction, neither the slop direction nor the 45° direction, which shows the specific mechanical behaviors of fiber-reinforced materials.

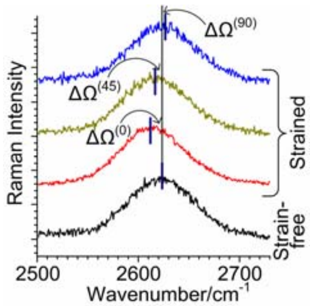

Fig.2. G' band Raman peak with different loading and polarizing directions.

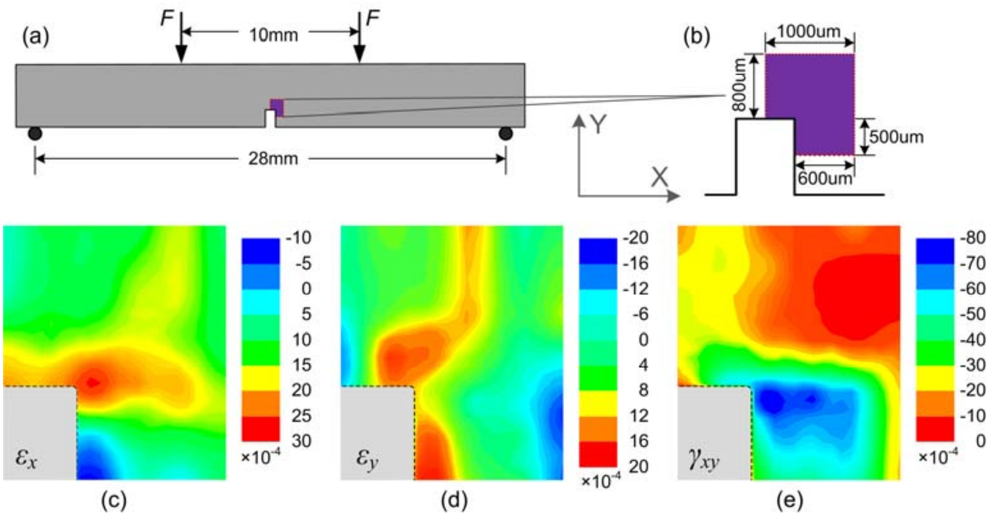

Fig.3. The experiments on the fiber-reinforced epoxy bar under four-point bend loading. (a) The geometrical shape, dimensions and loading type, (b) Raman mapping region, (c)~(e) the distributions of ε_x, ε_y and γ_{xy} , respectively, near the vicinity of the slop tip achieved by Raman Strain Rosette.

CONCLUSIONS

The model of nano strain sensor propounded in this work gives a spectral-mechanical relationship between the Micro-Raman data and the in-plane strain components. Based on this model, a method named Raman Strain Rosette is introduced. The experimental application verifies that Raman Strain Rosette is practicable to quantitative measuring the in-plane deformation, including both normal and shear strains at microscale.

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