Design method of sensors for quantitative mechanical testing of nanomaterials in transmission electron microscopy

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Abstract. Material structure at atomic scale determines its macroscopic physical and chemical properties. Transmission electron microscopy, as an effective and important technique for analyzing the microstructure of materials at atomic scale, has become an indispensable tool for studying the relationship between material properties and microstructure. According to the structure characteristics and working mode of transmission electron microscope, a design method of mechanical sensor based on piezoresistors is presented in this paper. The sensor can be used for in-situ quantitative tensile test in transmission electron microscope.

1. Introduction
The in-situ external field technology of Electron microscopy is to apply a single or coupled external physical and chemical field (such as mechanical, thermal, optical, electrical field or environmental atmosphere, etc.) to the measured nano-materials, and with the help of scanning electron microscope(SEM) or transmission Electron Microscope (TEM) with high spatial and information resolution, in-situ real-time observation of the microstructure evolution process of samples under the effect of external field, and establish the microscopic test and analysis method of the correlation between the microstructure and properties of materials. These technologies provide powerful technical means for the discovery of new phenomena, the revelation of new mechanisms, the establishment of new theories and the development of new materials, and promote the development of material physics and chemistry and other fields. As is known to all, material structure in atomic scale determines its macroscopic physical and chemical properties. TEM, as a very effective and important technical means to analyze the microstructure of materials at the atomic scale, has strongly promoted the development of many fields.

Due to the limited sample loading space in TEM, two problems need to be solved to carry out in-situ mechanical experiments in TEM, namely, which way to drive the samples mechanically and how to detect the mechanical signals of micro/nano-Newton Force or nanometer scale.

Since the 1950s, a number of research groups have studied the mechanical properties of such micron-scale crystalline materials as tin, iron, zinc, sulfide, copper and silicon through the development of bending and tensile testing methods. Through these experiments, researchers have obtained good mechanical properties of micron materials. They are the forerunner of the development of micro/nano mechanical testing technology, and also laid a solid foundation for the application of...
micro/nano-materials [1-5]. With the development of semiconductor industry, especially MEMS (Micro-Electro-Mechanical System, MEMS), the demand for mechanical testing of micro/nano-materials is increasing day by day. A considerable number of micromechanical testing techniques are miniaturization of macroscopic mechanics testing techniques. These methods include bending testing, bugling testing, micro-bridge testing, resonance testing, nano-indentation testing and micro-tensile testing [6]. Uniaxial tensile method can easily obtain the mechanical properties of materials such as elastic modulus, yield strength, Poisson's ratio and fracture strength, and has the advantages of reliable data and easy interpretation. However, there are difficulties in sample transfer, axial alignment, fixation and manipulation when conducting tensile tests on micro-nano samples. The general solution is to grow the micro/nano-materials on a substrate that can be moved macroscopically, such as tweezers, and use the nanomanipulator to directionally transfer or randomly distribute the micro/nano-materials on a tensile platform.

Because of its miniaturization, integration and high sensitivity, MEMS is widely used in in-situ quantitative mechanical testing system for machining integrated stress application and stress measurement. The MEMS system was first proposed by Richard Feynman, a Nobel laureate in physics, in a lecture in 1959. Since the 1960s, with the innovation of technology, some silicon-based micro-devices, such as meteorological chromatographic analysis devices, pressure sensors, mechanical sensors, resonators, micro-mirrors and inkjet nozzle devices, have been successfully developed [7-18]. MEMS is a micron-scale mechatronics integrated device manufactured using key processes including photolithography, deposition and etching, all of which have been developed through the microfabrication of integrated circuits. Compared with traditional integrated circuits, MEMS can not only process electrical signals, but also realize the integration and conversion of mechanical, optical, thermal and other physical signals, chemical signals and biological signals. The most prominent advantage of MEMS devices is that they can achieve basic and mass production of multifunctional integration in a small space. Therefore, they can be applied to many space-constrained devices. The uniaxial tensile method can be matched with the MEMS tensile platform, which perfectly solves the problems of sample transfer and tensile axial alignment. The key is to be able to match the narrow working space of TEM [19-20].

To achieve quantitative mechanical testing, mechanical signal detection methods matching TEM are required, as shown in Table 1. Currently, there are mainly optical and electrical methods. Optical method cannot achieve continuous high resolution observation, and inductance method is not suitable for transmission electron microscopy, so in terms of mechanical sensors, there are mainly MEMS based on the cross-finger capacitance sensor and piezoresistive sensor applicable. Among them, the forked finger capacitance sensor is not easy to be affected by temperature, applicable to a large temperature range. The principle of the fingered capacitor is that in the process of deformation, the coupling degree of the side wall of the fixed plate and the movable plate changes, resulting in the change of capacitance. The forked finger capacitance sensor can design flexible support beams with movable side walls with low stiffness through precise processing technology, and realize the mechanical signal detection of nN level. However, the capacitance variation of the cross finger capacitor is usually at the order of fF or even lower. In order to increase the sensitivity of the device, it is necessary to increase the logarithm of the plate, which will increase the area of the device. Moreover, the device noise and crosstalk noise have significant influence, and the corresponding signal detection circuit is more complex. Piezoresistive sensor is a main way of AFM cantilever beam load detection. Its advantages are high sensitivity, small device size and relatively simple signal processing circuit. Its disadvantage is that it is easy to be affected by temperature, which requires temperature compensation. MEMS devices with piezoresistive sensors can be adapted to the narrow workspace of TEM sample holder front-end. Formatting of manuscript components.
2. Piezoresistive sensor design

2.1. Piezoresistor design

When the stress is applied to the conductor or semiconductor material, the deformation of the material will lead to the change of its band structure, resulting in the change of the resistivity of the material, which is called the piezoresistive effect. The strain coefficient caused by the resistivity change of semiconductor material is 50~100 times that caused by the geometrical deformation, so the sensitivity of the strain gauge made of semiconductor material is much higher than that of the common metal strain gauge. Under stress, the resistivity change of semiconductor material can be expressed as [21]:

\[
\frac{\Delta \rho}{\rho} = \pi \sigma
\]

where, \(\Delta \rho\) is the change value of resistivity, \(\sigma\) is the stress, \(\pi\) is the piezoresistive coefficient, and is a fourth-order tensor. In general, the relative change in resistivity can be expressed as:

\[
\frac{\Delta \rho}{\rho} = \pi_l \sigma_l + \pi_t \sigma_t
\]

where, \(\pi_l\) and \(\sigma_l\) are the longitudinal piezoresistive coefficient and stress along the current direction respectively, and \(\pi_t\) and \(\sigma_t\) are the transverse piezoresistive coefficient and stress along the vertical current direction respectively. The values of the longitudinal and transverse piezoresistive coefficients depend on the crystallographic orientation. For (100) crystal plane, p-type silicon has the largest longitudinal and transverse piezoresistive coefficients (71.8×10^{-11} Pa) and transverse piezoresistive coefficients (-66.3×10^{-11} Pa) in <110> crystal direction. N-type silicon has the largest longitudinal and transverse piezoresistivity coefficients (-102×10^{-11} Pa) and (53.4×10^{-11} Pa) in <100> crystal direction. In the design of mechanical sensor based on silicon piezoresistive, the piezoresistive resistor should be arranged on the crystal with high piezoresistive coefficient value to obtain the maximum detection sensitivity [21].

### Table 1. Comparison of detection type for in situ mechanical test in electron microscope.

| Test method  | EM       | \(F_{\text{min}}\)  | \(D_{\text{min}}\)  | Continuous high-resolution observation |
|--------------|----------|----------------------|----------------------|---------------------------------------|
| Optics       | SEM/TEM  | ~nm                  | ~nm                  | \(\times\)                            |
| DIC          | SEM/TEM  | ~nm                  | ~nm                  | \(\times\)                            |
| differential capacitance | SEM/TEM  | ~nN                  | ~nm                  | \(\checkmark\)                       |
| Electricity  | piezoresistor | ~nN                | ~nm                  | \(\checkmark\)                       |
|              | inductance | SEM                  | ~mN                  | \(\checkmark\)                       |

2.2. Mechanical structure of the sensor platform

The uniaxial tensile test method requires that the sample be mounted on two test beams that can move in opposite directions, as shown in Figure 1. The detection beam can be used in cantilever beam, fixed beam at both ends, folding beam and other forms. In order to simplify the structure and avoid the non-axial displacement in the process of tensile testing, the two ends of the fixed beam should be used as the detection beam of the sensors. According to the mechanical analysis of the fixed beam at both ends, the maximum stress distribution of the fixed beam at both ends is similar to that near the fixed beam at both ends when the external force is applied to the center of the beam. Therefore, the piezoresistors should be prepared in the above-mentioned area.
In uniaxial tensile tests, the amount of elongation of the sample can be given by the following formula:

$$\Delta l = x_2 - x_1$$  \hspace{1cm} (3)

In the above formula, $x_1$ and $x_2$ are the axial moving distances of the detection beam 1 and 2 in figure 1, respectively. Assuming that the stiffness of beam 1 and beam 2 are $k_1$ and $k_2$ respectively, the stiffness of the sample is $k_s$, and the stiffness of the driver is $k_A$, when the stiffness of the driver is much higher than that of the sample beam 1 and 2, the driving force is as follows:

$$F_2 = k_A x_2 + k_1 x_1 + k_2 x_2$$  \hspace{1cm} (4)

The force of the sample is:

$$f_s = k_1 x_1$$  \hspace{1cm} (5)

It is assumed that the length, width and thickness of the sample are $l_s$, $w_s$ and $d_s$ respectively. According to Equation (3), the strain of the sample is:

$$\varepsilon_s = \frac{x_2 - x_1}{l_s}$$  \hspace{1cm} (6)

According to Equation (5), the stress of the sample is:

$$\sigma_s = \frac{k_1 x_1}{w_s d_s}$$  \hspace{1cm} (7)

By using the above method, the mechanical signals of samples can be measured directly, and the microstructure evolution of materials can be in situ observed at atomic scale. In addition, the relationship between the stiffness of beam 1 and the stiffness of the sample needs to be fully considered in order to collect as much force data of the sample as possible through the sensor. If the stiffness of beam 1 is much greater than that of the sample, the central deflection caused by the driving force exerted by the left end of the sample on beam 1 is very small, and the displacement of beam 1 is too small to collect the force information. If the stiffness of the beam is too small and the displacement of beam 1 is too long, the sample will be unable to break. The stiffness of beam 1 depends on the geometric size and elastic modulus of the material. Therefore, it is necessary to estimate the geometric size of beam 1 according to the tensile strength and geometric size of micro-nano scale material samples. For beam 2, there is no need to measure the force, so its stiffness only needs to meet the requirements of the driving sample.

2.3. Design basis of piezoresistive sensor

Piezoresistive detection is realized by converting the displacement or stress of the beam into the corresponding output voltage signal through the wheatstone bridge built inside the tensile platform.

**Figure 1.** Principle diagram of quantitative uniaxial tensile tests.
Based on the above analysis, the maximum value of stress along the length direction appears at the root of the beam or the position connected with the mass block, and the closer to the beam surface perpendicular to the tensile force, the higher the stress value along the thickness direction, so the design should be arranged at the root of the beam deformation piezoresistive. If the half-bridge circuit is constructed, the fixed piezoresistors shall be arranged on the substrate of the tensile platform.

Sensitivity is an important index to measure sensor performance. The sensitivity of a mechanical sensor refers to the ratio of the electrical signal output to the displacement or force generated by the sensitive mechanical structure. The displacement sensitivity is inversely proportional to the length of the beam and has nothing to do with its width and thickness. Force sensitivity is proportional to length and inversely proportional to width and thickness. As the stiffness of the beam increases, the displacement sensitivity of the sensor increases, but the force sensitivity decreases. At the same time, increasing the transverse piezoresistive coefficient, bridge bias voltage and the distance between the piezoresistive axis and the neutral surface of the beam are also effective methods to improve the sensitivity of the sensor.

Noise generation is an inevitable problem in any signal detection field. Piezoresistive devices are disturbed by various noises, which will lead to random fluctuation of the output voltage signal of the device. In the final output signal measured by sensor test system, noise mainly comes from the varistor itself, circuit system and environment noise. The former belongs to the inherent noise of the piezoresistive sensor, which can be divided into four cases: thermal noise, granular noise, g-r noise and 1/f noise, among which thermal noise and 1/f noise are the main noise sources in the piezoresistive resistor. For the sensor, the minimum measurable signal it can obtain depends on the intensity of various background noises. The resolution of displacement and force is defined as the ratio of noise to sensitivity. When the output voltage value is equal to the noise signal value, the sensed displacement (or force) is the resolution of displacement (or force). Displacement and force resolution are related to many parameters of the sensor, including not only the geometric dimensions of the beam and piezoresistive, but also the bridge voltage, bandwidth and processing parameters. Therefore, it is necessary to optimize the design of the sensor according to the performance indexes.

According to the above analysis, the design of piezoresistive sensor needs to consider a large number of parameters. Table 2 shows the influence of each parameter on sensor sensitivity and resolution, where $L$, $W$ and $D$ represent the length, width and thickness of the detection beam respectively; $l$, $w$ and $d$ represent the length, width and thickness of the piezoresistors, respectively; $V$ is the bridge voltage; $N$ is the semiconductor doping concentration; ↑, ↓, —, and ~ indicate increase, decrease, irrelevant, and uncertain respectively.

| Parameter | $S_D$ | $S_F$ | $R_D$ | $R_F$ |
|-----------|-------|-------|-------|-------|
| $L$       | ↓     | ↑     | ↑     | ↓     |
| $W$       | —     | ↓     | —     | ↑     |
| $D$       | —     | ↓     | —     | ↑     |
| $l$       | ↓     | ↓     | ~     | ~     |
| $w$       | ↓     | ↓     | ↓     | ↓     |
| $d$       | —     | —     | ↓     | ↓     |
| $V$       | ↑     | ↑     | ↑     | ↑     |
| $N$       | ↓     | ↓     | ↓     | ↓     |

3. Summary
In this paper, the design idea and method of piezoresistive sensor used for testing stress and strain information of samples in TEM is presented. The performance of piezoresistive sensors is affected by many factors, such as the geometry of the detecting beam and the geometry of piezoresistors, the voltage of the bridge and the doping concentration of the semiconductor. The design of piezoresistive
sensor should be optimized according to the above parameters, the technological conditions of the
device, the working environment of TEM, and the stress and strain range and precision of mechanical
test for micro/nano scale samples.

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