Precise Resistivity Measurement of Submicrometer-Sized Materials by Using TEM with Microprobes

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Precise electric resistivity measurements of submicrometer-sized materials have been demonstrated by using the piezodriving mechanics of two microprobes in a transmission electron microscope. By introducing two supplemental copper cables connected to a specimen, an electric circuit similar to that used in the four-terminal method was realized in a specimen holder with two microprobes. By using the proposed method, we determined the resistivity of a needle-shaped Pt-Ir specimen, whose resistance is only of the order of 0.1 Ω, with a satisfactory precision of <2 × 10⁻⁴ Ω. This method can be employed in microscopy studies on many submicrometer-sized and/or nanometer-sized materials.


(Received January 26, 2009; Accepted March 31, 2009; Published May 20, 2009)

Keywords: carbon nanotube, conductivity measurement, four-terminal method, transmission electron microscopy

1. Introduction

The fabrication of low-dimensional materials (wires, rods, tubes, etc.), which are submicrometer-sized or even smaller, is one of the fascinating research topics in nanoscience and nanotechnology. For example, researchers have synthesized many types of functional wires that can be used in several applications for nanoelectronic devices,¹ electron emitters,² magnets,³ piezoelectric tools,⁴ etc. Using the metallic wires in the interconnection and/or packaging will be another important way of the applications. Resistivity measurements are one of the most important measurements for characterizing the specimens. For example, with respect to the application of Cu wires to interconnections, we need to measure the grain boundary resistivity with a precision of 10⁻¹ Ω, while the diameter of the wire is only 50 nm or even smaller than that.⁵⁻⁷ Thus, it is particularly important to develop a method that allows a precise resistivity measurement for a local area.

Until now, scanning tunneling microscopy (STM) has been widely used in the evaluation of I–V (current vs. voltage) characteristics.⁸ Another advanced experiment is to manipulate a probe inside a transmission electron microscope (TEM); this experiment helps in obtaining correlations between the conductivity and the crystallographic structures.⁹⁻¹¹ Recently, some of the authors¹² developed a special equipment (double-probe piezodriving holder) in which two microprobes can be independently moved in three dimensions. In fact, this tool has been employed in (1) the studies on the local conduction path in an Ag-based conductive adhesive¹³ and (2) the identification of electric and magnetic phases in hole-doped manganites,¹² wherein the resistivity measurement was combined with other established TEM techniques (i.e., bright-field/dark-field imaging, Lorentz microscopy, electron holography, etc.). However, in the previous studies, the resistivity was measured by a “two-terminal method” using two microprobes, wherein the contact resistance was not negligible. Another problem was a mechanical drift of the probe tip during the resistivity measurement, which caused uncertainties (fluctuations) in the observations. As a result, it was difficult to examine a metallic specimen having a small resistivity (e.g., <3 Ω). Furthermore, since the sharpened microprobes (50 nm in diameter) were plastically deformed after they were brought into contact with the specimen, this experiment could not be performed for some cases.

Here, we report on improving a precision of local resistivity measurement in TEM. An electric circuit similar to that used in the four-terminal method was realized in a specimen holder with two microprobes by introducing two supplemental copper cables connected to a specimen. By using the proposed method, we determined the resistivity of a needle-shaped Pt-Ir specimen, whose resistance is only of the order of 0.1 Ω, with a satisfactory precision of <2 × 10⁻⁴ Ω. Furthermore, to prevent the probe tip from plastic deformation, we have fabricated a microprobe with a dual structure. This dual-structured microprobe consisted of a multiwall carbon nanotube (MWCNT) attached to a commercial Pt-Ir probe.

2. Experiments

In order to investigate the accuracy of the resistivity measurements, a needle-shaped Pt₀.⁰⁹Br₀.₁⁰ tip was used. Note that this Pt₀.⁰⁹Br₀.₁⁰ tip is widely used in STM studies. TEM observations were performed by using a JEM-3000F transmission electron microscope (accelerating voltage: 300 kV) at room temperature about 293 K. As described later in detail, we will also discuss the usefulness of a dual-structured microprobe. In order to fabricate a dual-structured microprobe, the Pt-Ir probe was brought into contact with a high-purity (>95%) MWCNT synthesized by chemical vapor deposition (Materials Technologies Research Ltd.) in the chamber of either TEM (JEM-3000F) equipped with the double-probe piezodriving holder¹² or SEM (Technex Lab...
Tinny-SEM 1710L) with a similar manipulator system. And then the residual hydrocarbon in the microscope chamber was decomposed to form an amorphous-like carbon deposit by continual electron beam irradiation. The MWCNT in contact with the Pt-Ir probe could be fixed by this carbon deposit, taking care that the deposition occurred selectively at the vicinity of the contact point and there was virtually no carbon deposit at the other portions of the MWCNT. Resistivity measurements were carried out by using equipments Keithley 6220 DC current source-meter and Keithley 2182A nanovoltmeter. The effect of thermo electromotive force was reduced by using a function ‘delta mode’, which was supplied by the latter equipment. For measuring I–V curves, 10–100 nA and 100 μA DC currents were supplied to MWCNT and Pt−90Ir−10 specimens, respectively.

3. Results and Discussion

In order to design the typical electric circuit used in the four-terminal method, we have introduced two additional polyester-coated copper cables (Cables 1 and 2) in the double-probe piezodriving holder (refer Fig. 1(a)). These additional cables (diameter: ~200 μm) were used along with the two microprobes (Probes 1 and 2) for realizing the electric circuit, as shown in Fig. 1(b). The specimen is a needle-shaped Pt-Ir (Pt0.90Ir0.10) alloy. Probes 1 and 2 were brought into contact with positions “1” and “2” in the specimen, respectively (refer Fig. 1(b)). Cables 1 and 2 were connected to positions “A” and “B” in the specimen, respectively. Cable 1 was connected to the specimen by using a thin plate of metallic terminal (Terminals A). While, Cable 2 was connected directly to the specimen (Terminal B). When an electric current is passed between positions 1 and A, the electric potential drop in the specimen can be measured by using Probe 2 and Terminal B. As a result, the electric resistance between positions 2 and B can be determined by using the four-terminal method in which the effect of contact resistance can be ignored.

By using this experimental setup (Fig. 1), the accuracy of the resistivity measurement is evaluated. Figure 2 shows a TEM image of the needle-shaped Pt0.90Ir0.10 specimen. Probe 1 (for applying an electric current) and Probe 2 (for measuring an electric potential) are in contact with the specimen. The inset shows a model of the needle-shaped specimen that was used in calculation of the resistivity. The distances d-a, d-b, and d-c and parameter γ can be estimated from TEM observations.

Fig. 1 (a) Top of a double-probe piezodriving TEM holder. A four-terminal circuit is constructed by using two probes (1 and 2) and the two additional cables (1 and 2). (b) Schematic illustration of the four-terminal circuit. The viewing field corresponds to the dotted square shown in (a). The electric potential drop between positions B and 2 can be measured by applying an electric current between positions A and 1.

Fig. 2 TEM image of needle-shaped Pt0.90Ir0.10 specimen. Probe 1 (for applying an electric current) and Probe 2 (for measuring an electric potential) are in contact with the specimen. The distances d-a, d-b, and d-c and parameter γ can be estimated from TEM observations.
the electric resistance between position c and position d ($R_{c-d} = R_{c-b} - R_{b-d}$). In a similar manner, by moving Probe 2 to other positions such as “b” and “a”, the local resistances $R_{b-d}$ (between b and d) and $R_{a-d}$ (between a and d) are determined. The results were plotted as a function of the distance $D$ between the probe tip (Probe 2) and original position d (refer Fig. 3). Errors in the resistivity measurements were smaller than $2 \times 10^{-4}$ $\Omega$ when an electric current of 100 $\mu$A was supplied, i.e., the error bar is smaller than the diameter of the closed circles. In the previous experiments based on the two-terminal method, the errors could not be reduced below 3 $\Omega$. Therefore, improvement in the precision is significant.

The broken line in Fig. 3 represents the electric resistance calculated for a model specimen (Pt$_{0.6}$Ir$_{0.4}$), whose shape is similar to the experimental specimen. As shown in the inset of Fig. 2, the theoretical resistance $R$ is calculated by the following equation:

$$R = \frac{\rho}{\pi} \left( \frac{\gamma}{\alpha} \right)^2 \int_0^{\infty} \frac{1}{(\gamma - z)^2} dz$$  \hspace{1cm} (1)

where $\rho = 24.5 \times 10^{-8}$ $\Omega \cdot m$ is the specific resistivity of the Pt$_{0.6}$Ir$_{0.4}$ alloy, $\alpha = 0.295 \times 10^{-6}$ m is the radius of cross section at position d, and $\gamma = -2.981 \times 10^{-6}$ m represents the spacing between the cone’s top (assuming a perfect conical shape) and position d. $\alpha$ and $\gamma$ were determined by TEM observation. As shown in Fig. 3, the calculated resistivity is in excellent agreement with the observed resistivity, and the deviation from the observed value is smaller than $2 \times 10^{-2}$ $\Omega$. Thus, by using the experimental setup shown in Fig. 1(b), we can obtain precise values of resistivity that are comparable to the calculated values.

Although the results in Figs. 1–3 were obtained by using the metallic probes made of a Pt-Ir alloy, they are subjected to plastic deformation during the experiments. In fact, the physical contact between the probe tip and the specimen is made unstable by the plastic deformation. In order to inhibit the plastic deformation in the probe, we propose to use a dual-structured microprobe, in which a Pt-Ir tip is connected to a MWCNT. Figure 4 shows the performance of the dual-structured microprobe. Due to the excellent elasticity of the MWCNT, it could be deformed considerably, as shown in Figs. 4(a) and 4(b). Despite this large deformation, the MWCNT recovered its original form when it was separated from the specimen. Another advantage of this dual structure is that the physical contact between the MWCNT and the specimen is relatively stable due to the restoring force of the deformed nanotube. Figure 4(c) shows an I–V curve that was obtained by using the dual-structured microprobe. Since the tip of the MWCNT was in contact with the metallic cathode during the measurement, as shown in the inset, the I–V curve is mainly controlled by the resistivity of MWCNT. In fact, the electric resistance determined from the slope is $15.0 \pm 0.5$ k$\Omega$, which agrees well with the values obtained using MWCNTs in the previous studies.$^{[16,17]}$ Thus, the dual-structured microprobes possess excellent elasticity and metallic characteristics, i.e., these microprobes can be used in the resistivity measurements for microfabricated-metallic specimens in nanometer size, although their internal resistance ($15.0$ k$\Omega$) is relatively large. It appears that the uncertainty in the measurements ($\pm 0.5$ k$\Omega$) is, for example, due to the current fluctuations at the contact areas, where substantial Joule heat is generated because of contact resistance. Therefore, it is necessary to reduce the effects of contact resistance when high-precision resistivity measurements have to be carried out.

4. Conclusion

In summary, we have proposed a method for conducting accurate electric resistivity measurements of submicrometer-sized specimens by using TEM observations and the piezodriving mechanics of microprobes. In order to eliminate the effect of contact resistance, we have introduced two supplemental cables that should be used along with the two piezodriving probes. As a result, the resistivity of a submicrometer-sized metallic specimen (Pt-Ir) could be evaluated within a precision of $2 \times 10^{-4}$ $\Omega$. The dual-structured microprobes, consisting of a MWCNT and a Pt-Ir needle, exhibit excellent elasticity and ohmic characteristics. It is expected that these methods can be used for conducting resistivity measurements of many submicrometer-sized and/or nanometer-sized specimens of nanowires, metals, and semiconductors.

Acknowledgements

This study was supported by Grant-in-Aid for JSPS Fellows (N. K.), Scientific Research (S), and Scientific Research (B) from the Japan Society for Promotion of Science. This study was also supported by the program “Post-Silicon Materials and Devices Research Alliance” from MEXT. The authors also thank the support from NEDO grant for the project “Development of Alternatives to High-Temperature Lead Solders.”
Fig. 4 (a) Dual-structured microprobe comprising a MWCNT and a Pt-Ir probe. (b) MWCNT is in contact with the specimen, and its bending elasticity is shown. (c) I–V curve obtained with the setup shown in the inset.

REFERENCES