Non-contact local conductivity measurement of metallic nanowires based on semi-near-field reflection of microwave atomic force microscopy

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In this study, a non-contact and quantitative evaluation method was developed to measure the conductivity of metallic nanowires at nanometer-scale resolution. Using a coaxial probe, microwave images and topographical images were simultaneously obtained for three nanowires via microwave atomic force microscopy (M-AFM). A semi-near-field model was established to describe the distribution of the electric field between the probe and the sample. Based on this model, the local conductivities of metallic nanowires on the nanometer scale were quantitatively evaluated in a single scan, using a metal strip substrate to calibrate the reflected signal.

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Metallic nanowires continue to be promising functional materials for various applications, such as magnetic data storage media,1–3 giant magnetoresistive nanowires,4,5 metamaterials,6–8 thermoelectric power conversion,9,10 chemical or biological sensors,11,12 and transparent electrodes.13 Conductivity is one of the most valuable properties for applications that use metallic nanowires. The traditional evaluation method for metallic nanowires is based on a four-probe method,14–16 which is inefficient, as it requires the fabrication of tiny electrodes. Alternatively, atomic force microscopy (AFM)17–19, a non-destructive metrology tool, has been demonstrated to be effective in characterizing nanowires as in recent decades, due to its high spatial resolution and free environmental vacuum degree. Functionalized AFM, electrostatic force microscopy (EFM), Kelvin probe force microscopy (KFM) and conductive AFM have been used to investigate the surface charge distribution, surface potential variation, and tip–sample I–V properties of metal and metal oxide nanowires.20–22 However, it is difficult to measure the conductivity of nanowires using these techniques due to their working mechanisms. In addition, AFM has been combined with a microwave system to image and quantitatively characterize the local surface electrical properties of nanomaterials and nanodevices. Scanning microwave microscopy (SMM), has been developed to determine the dielectric constant of thin SiO2 film and the resistivity of a Si sample, based on capacitance and impedance measurements.23 The relative permittivity of dielectric materials and the sheet conductance of MoS2 film on a SiO/Si substrate have been measured by microwave impedance microscopy (MIM).24,25 For local conductivity measurement, microwave AFM (M-AFM) composed of a w-band microwave system and AFM has been developed, and a slit probe which can propagate and emit a microwave by itself has been designed and fabricated.26 This probe helps the simultaneous scanning of topography and microwave images to be realized; the quantitative evaluation of the conductivities of metallic films in a non-contact mode has been achieved.27,28

Nevertheless, it is still a challenge to quantitatively measure the conductivity of metallic nanowires. As opposed to a scan on a flat surface, the measurement of nanowires also depends on their surface shape because, the tip aperture of the probe is comparable with the diameter of the nanowires. Moreover, the change of local resistance between different metallic nanowires is too small. To the best of our knowledge, so far, there is still no technology that can measure the conductivity of individual metallic nanowires in situ. This study demonstrates the first quantitative determination of the conductivity of metallic nanowires using M-AFM. A novel M-AFM probe with a coaxial tip structure was fabricated to increase measurement sensitivity. To describe the impacts of the nanowire’s diameter and the tip–sample distance on the reflected signal, a semi-near-field theoretical model was established. Using this model, the conductivities of Al, Ag, and Cu nanowires were quantitatively determined from the microwave image.

Figure 1(a) shows the schematics of the coaxial M-AFM probe. The SEM images are shown in Figs. 1(b) and 1(c). A cantilever with a thickness of approximately 15 μm and a tip with a height of approximately 5 μm was fabricated by wet etching from a piece of undoped GaAs wafer. A pyramidal hole with an approximate depth of 18 μm was excavated on the reverse surface of the cantilever by focused ion beam etching (FIB) etching at the position of the tip. To confine microwaves inside the probe, the top and bottom surfaces of the probe were coated with 50 nm Au films by electron beam evaporation, so that the Au films on the tip and in the pyramid-shaped hole served as the outer and inner conductors of the coaxial structure, respectively. Finally, using FIB etching, a small hole with an approximate depth of 1 μm was fabricated at the end of the tip, which served as an aperture for emitting microwaves. The size of the tip aperture is approximately 250 nm, according to an SEM image. The spatial resolution of the coaxial probe was estimated to be 700 nm, according to the topography scanning profile, which is lower than that of the slit probe described in Ref. 28, which was reported to be 170 nm. However, since the coaxial-structured tip works in the transverse electromagnetic mode without a cutoff frequency, and can deliver more microwave power to the aperture, thereby resulting in an increase in sensitivity, the coaxial probe may still be a powerful tool for the quantitative measurement of nanomaterials.

In the quantitative evaluation experiment, Al, Ag, and Cu nanowires were obtained by the stress-induced migration method,29 commercial purchase, and the template electro-deposition method,30 with diameters of 660 nm, 240 nm, and 720 nm, respectively. Each sample was scanned in non-contact mode on a Pt substrate with Au strips which were used for calibration. Both the width and interval of the strips

M

A

F

P

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were 5 μm. The scanning speed was 3 μm s⁻¹, and the scanning area was 10 × 10 μm² for the Ag nanowire and 20 × 20 μm² for the Al and Cu nanowires, based on their sizes. A 94 GHz microwave signal with 10 dBm of source power was applied to the probe. The reflected microwave was received by a square-law detector which provided a direct-current output voltage according to

\[ V = k_0 |E|^2 + b_0. \]

Here, \( E \) is the intensity of the reflected microwave, and \( k_0 \) and \( b_0 \) are the performance parameters of the detector. The measurement was conducted in air, and the temperature and relative humidity of the environment were 20.0 °C and 50%, respectively.

Fig. 1. (Color online) (a) Schematic of the coaxial M-AFM probe and the calibration substrate. The gray and gold parts in the profile of the probe represent GaAs and Au, respectively. (b), (c) SEM images of the tip side (b) and back side (c) of a coaxial M-AFM probe. The inset in figure (b) depicts the aperture fabricated on the tip. (d), (e) Schematics of the image charge when the coaxial probe is over a plane surface (d) and a spherical surface (e).
Considering that the wavelength of the microwave (∼3 mm) is much longer than the tip–sample distance (∼5 nm), the measurement result needs to be analyzed by a near-field model to improve accuracy. At every instant, the electric fields of the incident and reflected microwaves can be approximated as quasi-static fields produced by the original charge on the probe and the polarization charge on the sample. According to the principles of electrostatics, the polarization charge can be represented by an image charge for simplification. When the probe is located over the substrate or the strips, which can be regarded as infinite and isotropic planes compared to the size of the tip, the image charge is mirror-symmetrical to its original charge on the tip. Therefore, the image charge should exhibit an identical trumpet-like distribution, as shown in Fig. 1(e). In this case, the tip can be approximated as a spherical surface with the whose diameter is 2\(r\), the sample’s surface within the size of the tip can be approximated as a spherical surface with the same diameter. Thus, the image charges form a double-trumpet-like distribution, as shown in Fig. 1(c). In this case, if a single point charge \(q_1\) is located above this surface at a distance \(z_1\), the image charge \(q_2\) and its position can be determined by

\[
q_2 = -\frac{r}{z_1 + r} q_1, \quad (2)
\]

and

\[
z_2 = \frac{z_1 r}{z_1 + r}, \quad (3)
\]

respectively. Here, \(z_2\) is the distance of the image charge \(q_2\) from the surface of the sphere.

However, this thorough near-field model only describes the spatial distribution of the electric field and is independent of the sample’s conductivity. Therefore, the reflection coefficient \(\Gamma\) of the sample is introduced to establish a semi-near-field model. On the surfaces of metallic materials, the reflected wave \(E\) and the incident wave \(E_0\) obey the relation given by

\[
\Gamma = \frac{E}{E_0} = \frac{1 - \sqrt{\sigma/j\varepsilon_0\omega}}{1 + \sqrt{\sigma/j\varepsilon_0\omega}}, \quad (4)
\]

where \(\sigma, j, \varepsilon_0\), and \(\omega\) are the conductivity of the sample, an imaginary unit, the permittivity of free space, and the frequency of the microwave, respectively. Combining Eqs. (2) and (4), the image charge that depends on both the tip–sample distance and the conductivity of material can be represented by

\[
q_2 = -\frac{r}{z_1 + r} \Gamma q_1. \quad (5)
\]

Therefore, considering \(q_1\) as a charge element on the tip, the reflected microwave can be calculated as the electric field integral of \(q_2\), using Coulomb’s law. If \(\varepsilon_1, \rho_1\), and \(w_1\) express the vertical distance of a charge element on the inner conductor from a nanowire surface, the linear charge density along the inner conductor, and the diameter of the inner conductor, respectively, the corresponding quantities of its image charge element in the nanowire sample, which are represented as \(z_2, \rho_2\), and \(w_2\), can be calculated as

\[
z_2 = r - \frac{r^2 (r + z_1)}{(w_1/2)^2 + (r + z_1)^2}, \quad (6)
\]

\[
\rho_2 = \frac{r}{\sqrt{(w_1/2)^2 + (r + z_1)^2}} \rho_1, \quad (7)
\]

and

\[
w_2 = \frac{r^2}{(w_1/2)^2 + (r + z_1)^2} w_1. \quad (8)
\]

The corresponding quantities for the outer conductor, \(z_0, \rho_0, \) and \(w_0\), also have the same relationship with \(z_1, \rho_1, \) and \(w_1\). Therefore, the wave reflected by a nanowire sample has the following form:

\[
E = \frac{|\Gamma|}{4\pi \varepsilon_0} \int_{z_a}^{z_i} \rho_2 (z_0 + z_2) dz_2 + \int_{z_c}^{z_i} \rho_2 (z_0 + z_2) dz_2. \quad (9)
\]

Here, \(z_0\) is the vertical distance of the tip apex from the substrate’s surface. \(z_a\), \(z_b\), \(z_c\), and \(z_d\) represent the boundary region from the apex of the inner conductor and the outer conductor, as shown in Fig. 1(d). The calculated electric field is the reflected wave at the center of the tip, which represents the signal received by the probe and propagated to the detector. Although \(z_0\) varies in the experiment because of the tip vibration in non-contact mode, it can be considered to be its constant average value, since the measured signal simply corresponds to the microwave signal obtained at the average tip vibration position. Based on the conservation of charge, the charge density on the boundary area of the tip follows \(\rho_1 = -\rho_0\). In the calculation, the depth of the boundary area is considered to be the same as the width of the aperture, which implies that \(z_b - z_a = z_d - z_c = w_0\). For the reflections from the substrate and the Au strips, the reflected microwave can be easily obtained using Eqs. (6)–(9), for \(r = \infty\).

Finally, by substituting Eq. (9) into Eq. (1), the relation between the output voltage of the detector and the reflection coefficient of the sample can be expressed as

\[
V = k_1 k_2 |\Gamma|^2 + b_0. \quad (10)
\]

Here, \(k_1\) represents a performance parameter of M-AFM related to \(k_0\) and the charge on the tip; \(k_2\), however, represents the near-field effect on the electric field distribution and can be calculated by
If the substrate and strip contain two reference materials with known conductivities, using Eq. (10), the parameters $k_1$ and $b_0$ can be determined. Then, $|\Gamma|^2$ of the samples can be evaluated based on their measured voltages, and thereby, the conductivities of these materials can be obtained from Eq. (4).

A scanned topographical image and a microwave image of three metallic nanowire samples, Al, Ag, and Cu, were simultaneously measured by M-AFM, as shown in Fig. 2. It can be observed that the microwave image corresponds to the topographical image, which confirms that this coaxial probe can emit microwaves. The average output voltages along the center line of each strip or nanowire were extracted, thereby avoiding measurement errors near the boundaries of the materials. The output voltage versus the standard conductivity of each material is shown in Figs. 3(a)–3(c).

Here, since the thicknesses of the film and strips on the substrate are 300 nm and the diameters of all the nanowires are more than 200 nm, the size effect of conductivity is not considered. The standard conductivities are obtained from the values of the corresponding bulk materials.33,34) Since the microwave signal strongly depends on the tip-sample distance, the error bars of the average output voltages mainly originate from small changes in the standoff distance in different scans. One abnormal phenomenon to be noted is the discordance of the Al nanowire. According to Eqs. (4) and (10), the output voltage monotonously depends on the conductivity of the material. However, although the true conductivity of Al is lower than that of Au, the measured voltage of the Al nanowire is higher than that of the Au strip, as shown in Fig. 3(a). This is because the curved surface of the nanowire generates a trumpet-like image charge which is more concentrated under the tip, as shown in Fig. 1(e), and thus generates a stronger reflected wave than the strip does. Using the standard conductivities and the reflected voltage signal of the Pt substrate and the Au strip, the two undetermined parameters $k_1$ and $b_0$ of each measurement were calibrated using Eqs. (10) and (11). Subsequently, $|\Gamma|^2$ of the three nanowires can be determined from the output voltage. Here, using Eq. (11), $w_{i1}$ was determined to be 5 nm according to the beam size used for FIB etching; $w_{o1}$ was determined to be 250 nm according to the aperture size of the tip; $z_0$ was determined to be 5 nm based on the force curve.

![Fig. 2.](Color online) Scanning topography image (left) and microwave signal image (right) of the Al (a, b), Ag (c), (d), and Cu (e), (f) nanowires on the Pt substrate with Au strips.

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measurement. The results for the nanowires, the Pt substrate, and the Au strip versus their standard conductivities are shown in Figs. 3(d)–3(f), respectively. From these results, it is noted that the $|G|^2$ determined for the surface of the sample, substrate, or strip monotonously depends on the conductivity of the material. Therefore, the semi-near-field model is demonstrated to be appropriate for the description of the electric field distribution of the M-AFM probe. Furthermore, the conductivities of the three nanowire samples were calculated from $|G|^2$ using Eq. (4). As shown in Fig. 4, the deviations of the evaluated conductivities from the standard values for Al, Ag, and Cu nanowires are 14.21%, 10.26%, and 8.57%, respectively. The deviations can be attributed to both theoretical and experimental reasons. First, in the proposed quasi-static charge model, the real distribution of the charges on the tip was approximated to the ideal case of a standard coaxial line, and the effect of the reflected wave on it was omitted. Secondly, the experimental reason is that the shape of the fabricated tip is not as regular and symmetric as the design because of the resolution limit of FIB etching, which also affects the true distribution of the microwaves. Moreover, it should be noted that here, two reference materials are needed to realize the quantitative evaluation demonstrated in this study, since the microwaves emitted from the tip are affected by the measurement environment, and change with the setup of the probe. Therefore, the proposed substrate with strips could be a useful tool to use as a sample holder, to realize the calibration during the same scan as the actual sample measurement.

In this study, by using a coaxial probe, topographical images and reflected microwave images of Al, Ag, and Cu nanowires were simultaneously obtained using the non-contact mode of M-AFM. The measurement was conducted on a Pt substrate with Au strips for calibration. A semi-near-
field model based on the image charge method and reflection coefficient was established to describe the spatial distribution and conductivity dependence of a microwave between the tip and the sample. This is essential for non-contact quantitative evaluation of metallic nanowires was quantitatively achieved in a single scan. This indicates that M-AFM is a promising in situ method for the characterization of one-dimensional nanomaterials.

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Fig. 4. (Color online) Evaluated conductivities of the Al, Ag, and Cu nanowires compared with their standard values.

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