Direct detection of grain boundary scattering in damascene Cu wires by nanoscale four-point probe resistance measurements

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(Received 19 May 2009; accepted 21 July 2009; published online 7 August 2009)

Four-terminal conductivity measurements of damascene copper (Cu) wires with various widths have been performed using platinum-coated carbon nanotube (CNT) tips in a four-tip scanning tunneling microscope. Using CNT tips enabled the probe spacing to be reduced to 70 nm, which is the shortest probe spacing in interconnect wire measurements achieved so far. The measured resistivity of Cu increased as the line width decreased and direct evidence of individual grain boundary scattering was observed when the probe spacing was varied on a scale comparable to the grain size of the Cu wires (~200 nm). © *2009 American Institute of Physics*. [DOI: 10.1063/1.3202418]

Nanoelectronics, such as that used in semiconductor devices, requires interconnects to have low and stable electrical resistances to ensure good device performance. Copper (Cu) is currently widely used for interconnects, but as wire dimensions are reduced to submicrometer scales, which is of the order of the mean free path of conduction electrons, an increase in the resistivity is observed. This is conjectured to be due to increased scattering from surfaces and grain boundaries. As the wire width is scaled down, electrons will undergo more reflections at the surfaces, so collisions with surfaces will become a significant fraction of the total number of collisions.[1] In addition, grain boundaries in polycrystalline wires/films may act as partially reflecting planes located perpendicular to the electric field,[2] so they also may contribute to the increase in the resistivity. Previously, the contributions of different scattering mechanisms have been estimated by fitting the observed resistivity versus line width curve to a simple model that takes into account various mechanisms.[3–5] However, there has been no direct observation of the contribution from scattering from individual grain boundaries. This is mainly attributed to the fact that most of the above measurements were performed using a fixed probe spacing of the order of several micrometers or greater.

To investigate these effects directly, it is necessary to perform nanoscale conductivity measurements. In the present letter, we performed nanoscale four-probe conductivity measurements on Cu wires at room temperature using four platinum (Pt)-coated carbon nanotube (CNT) tips[6] on a four-tip scanning tunneling microscope (STM).[7,8] By reducing the probe spacing on a scale comparable to the grain size (<200 nm), we were able to directly detect the influence of *individual* grain boundary scattering. The deduced values were consistent with the *average* contribution obtained by analyzing the resistivity versus line width curve.

Copper interconnects with line widths between 70 nm and 1 μm were prepared by a Cu/low-k damascene process. Figures 1(a) and 1(b) show cross-sectional SEM images of the damascene structure for trenches with linewidths of 70 and 500 nm, respectively. The trench depth was independent of the line width and was ~240 nm. Tantalum was used as a barrier metal for Cu interconnects in this experiment. Cu damascene lines were formed by a conventional Cu process that involved seed Cu deposition, electrochemical plating of Cu to fill the trenches, and chemical-mechanical polishing. Figure 1(c) shows the grain size and its distribution along 70 nm wide damascene lines mapped by electron backscatter diffraction. Each color represents a different crystal orientation. It clearly reveals that there are a significant number of grain boundaries in the damascene lines. Figure 1(d) shows a histogram of the grain size distribution derived from the image in Fig. 1(c). The grain size distribution has a maximum at 120–130 nm, and most grains are smaller than 200 nm.

A four-tip STM enables four-point probe conductivity measurements with various probe arrangements and probe spacings, as shown in Fig. 2(a). By using CNT tips, the probe spacing can be reduced to several tens of nanometers [Figs. 2(b)–2(d)].[6] The minimum probe spacing in the present study was 70 nm [Fig. 2(d)]. A notable advantage of

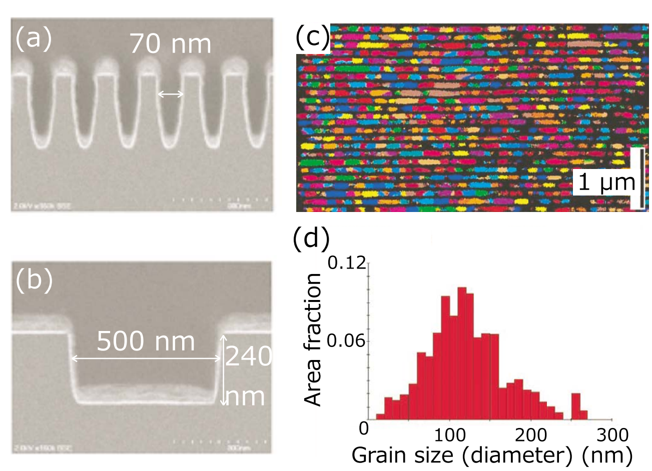

FIG. 1. (Color online) Cross-sectional SEM images of damascene trenches with linewidths of (a) 70 nm and (b) 500 nm. (c) A grain map of the 70 nm Cu wires obtained by electron backscatter diffraction. Each color represents a different crystal orientation. (d) A histogram of the grain size distribution derived from the image in (c).

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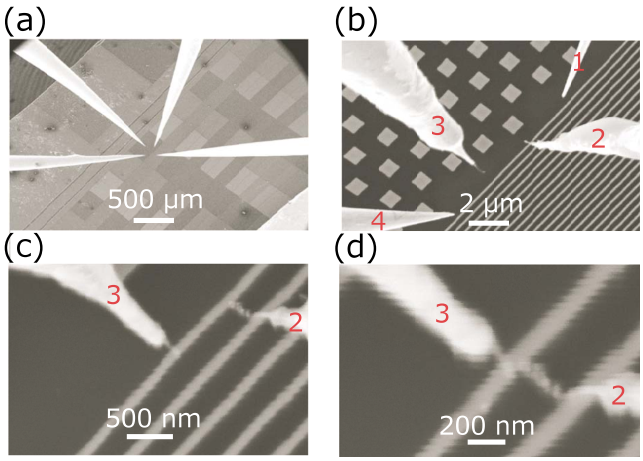

FIG. 2. (Color online) (a) SEM image of the Cu wires being contacted by the four probes. (b) An enlargement of the image in (a). In the four-terminal *I-V* measurements, the current flows between tips 1 and 4, and the voltage drop is measured between tips 2 and 3. [(c) and (d)] Images of the voltage probes 2 and 3 approaching each other. The probe spacings are (c) 750 nm and (d) 70 nm.

CNT tips is that direct electrical contact causes little electrical or mechanical damage either to the sample or the tips, even after repeated contact. On the other hand, despite the CNTs being coated with Pt, the contact resistance between the tip and the sample could not be reduced to less than 50 kΩ due to the very small contact area. This makes it impossible to measure conductive materials whose resistance is less than 50 kΩ by the two-point probe method. Only the four-point probe method is capable of measuring resistances much smaller than the contact resistance (as small as 0.1 Ω in the present case). Therefore, combining CNT tips with a four-tip STM is a very powerful tool for nanoscience and nanotechnology studies.

Four-terminal *I-V* measurements were performed by sweeping the bias voltage between tips 1 and 4 and recording the current flow I and the voltage drop V between tips 2 and 3 [Fig. 2(b)]. The probe spacing between the voltage probes was reduced while measuring the *I-V* characteristics. The two current probes (tips 1 and 4) and one of the two voltage probes (tip 2) were fixed in the same positions during the measurements, and only the other voltage probe (tip 3) was moved between tips 2 and 4 [Figs. 2(c) and 2(d)].

Figures 3(a) and 3(b) show the measured four-terminal resistance as a function of the probe spacing between the contact points of the voltage probes on the Cu wires. There was no change in the measured resistance before and after repeated contact of the CNT tips with the sample. This demonstrates that no significant damage was sustained to the sample by contact with the probe. As mentioned above, for the 70-nm-wide wire [Fig. 3(a)], the minimum probe spacing was 70 nm, which is the shortest probe spacing in interconnect wire measurements achieved so far. The probe-spacing dependence of the resistance is essentially linear down to a spacing of 70 nm, implying classical one-dimensional diffusive transport. Tungsten (W) tips were used for the measurements on wide Cu wires with widths of 500 nm and 1 μm [Figs. 3(b)], so the minimum probe spacing was limited to 500 nm due to the width of the W tips. The solid lines in Figs. 3(a) and 3(b) indicate the linear least-squares fitting results. By taking the product of the gradient of the fitted

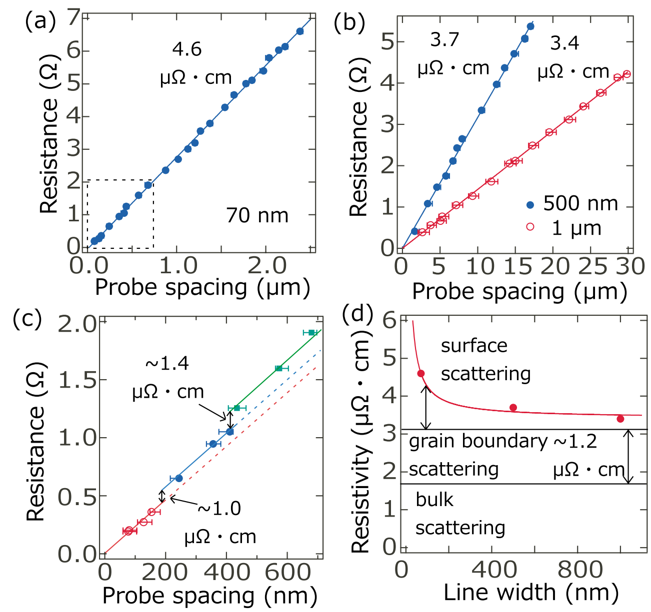

FIG. 3. (Color online) Measured resistances of the 70 nm wide Cu wire (a) 500 nm (filled circles) and (b) 1 μm (open circles) wide wires as a function of probe spacing. The solid lines are the linear least-squares fits. (c) An enlargement of the region contained by the dotted square in (a) for a probe spacing of 700 nm. The solid lines indicate the linear least-squares fits for the data located in intervals of 200 nm, all having nearly the same slope. The jump corresponds to the grain boundary scattering. (d) Plot of the resistivity as a function of the Cu line width. The solid line is the least-squares fit to Eq. (3). The contributions of different scattering mechanisms are also shown.

straight lines and the cross-section of the Cu wires, the three-dimensional resistivity was calculated to be 4.6 $\mu\Omega$ cm, 3.7 $\mu\Omega$ cm, and 3.4 $\mu\Omega$ cm, for the 70 nm, 500 nm, and 1 μm wide Cu wires, respectively. Figure 3(d) shows that the measured resistivity of Cu increases as the line width decreases, which can be ascribed to an increase in surface scattering.

In this measurement, the probe spacing was varied on a scale comparable with the grain sizes in Figs. 1(c) and 1(d). Therefore, the resistance is expected to change when probe 3 crosses a grain boundary. This phenomenon was indeed observed in our measurements. Figures 3(c) shows an enlarged view of the data contained in the dotted square in Fig. 3(a) for data points obtained using a probe spacing smaller than 700 nm. The solid lines show the results of the linear least-squares fit for the data in intervals of 200 nm, which are indicated by different markers. All the lines have nearly the same slope. There are discontinuous jumps in the resistance (0.12 Ω at 200 nm and 0.17 Ω at 400 nm), which we speculate are caused by grain boundary scattering; since grain boundaries are located approximately every 200 nm, these jumps correspond to resistivities of ~1.0 and ~1.4 $\mu\Omega$ cm, respectively.

In order to confirm if the above estimation is reasonable, we performed conventional analyses to estimate the contributions of the grain boundary and surface scatterings. This method utilizes the Fuchs–Sondheimer (FS) theory for the surface effect[9,10] and the Mayadas–Shatzkes (MS) model for the grain effect.[11] The resistivity of the FS theory ρ_{FS} is expressed as

$$\rho_{FS} = \rho_0\left[1 + 2C\lambda_0(1-p)\left(\frac{1}{h}+\frac{1}{w}\right)\right], \qquad (1)$$

where ρ_0 is the bulk resistivity, $C=1.2$ is the form factor for a rectangular cross-section, λ_0 is the mean free path of the carriers, and h and w are the height and width of the nanowire, respectively. Variable p is used to characterize the probability of elastic electron reflection at the surface ($p=0$ indicates purely diffusive scattering, whereas $p=1$ implies total elastic reflection). The resistivity in the MS model ρ_{MS} is described as

$$\rho_{MS} = \frac{\rho_0}{1 - \frac{3\alpha}{2} + 3\alpha^2 - 3\alpha^3 \ln\left(1+\frac{1}{\alpha}\right)}, \qquad (2)$$

where $\alpha=(\lambda_0/d)[R/(1-R)]$ (R is the probability of electrons being reflected at the grain boundary) and d is the mean grain size. We performed least-squares fitting of the three data points in Fig. 3(d) using the following equation:

$$\rho = \rho_{FS} + \rho_{MS} - \rho_0, \qquad (3)$$

where p and R are the fitting parameters.[12] The constants we used are $\rho_0=1.7\ \mu\Omega$ cm, $\lambda_0=40$ nm, $d=200$ nm, and $h \sim 240$ nm. The solid curve in Fig. 3(d) shows the fitted results and the obtained parameters are $p=0.49$ and $R=0.64$. These values are comparable to those reported in the literature.[3–5] The deduced value of ρ_{MS} is $\sim 1.2\ \mu\Omega$ cm, which is the *average* contribution of the grain boundary scattering.[13] It shows good agreement with the values for individual grain boundary scattering in Fig. 3(c). Therefore, we can unambiguously claim that we have directly detected the effect of scattering from individual grain boundaries by varying the probe spacing on a scale comparable to the grain size.

In conclusion, electrical measurements using Pt-coated CNT tips in a four-tip STM have been demonstrated for Cu damascene wires. The minimum probe spacing was reduced to 70 nm, which is similar to or smaller than the Cu grain size. The resistance along the Cu line increased linearly with the measured length, and the resistivity increased as the line width decreased. From the jumps found every 200 nm in the resistance versus probe spacing curve, we have succeeded in directly observing electron scattering from individual grain boundaries.

We would like to thank N. Shimizu and H. Kitada of Fujitsu Laboratory Ltd. for performing sample preparation and grain analysis.

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[12] In Eq. (3), ρ_0 is subtracted because it is considered in both ρ_{FS} and ρ_{MS}.
[13] Because we used $d=200$ nm from Fig. 1(d), ρ_{MS} does not show any dependence on the line width. If we assume that d is the smallest dimension of the wire (see Ref. 3), it will show some dependence. However, the good match between the experimental data and the fitted curve indicates that the assumption that $d=200$ nm is reasonable. Even for wide wires, the height (240 nm) is comparable to $d=200$ nm.