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Experimental and simulation studies of resistivity in nanoscale copper films

A. Emre Yarimbiyik [a,b], Harry A. Schafft [b], Richard A. Allen [b,*], Mark D. Vaudin [c], Mona E. Zaghloul [a]

[a] The Department of Electric and Computer Engineering, The George Washington University, Washington, DC 20052, United States
[b] Semiconductor Electronics Division, National Institute of Standards and Technology, Gaithersburg, MD 20899-8120, United States
[c] Ceramics Division, National Institute of Standards and Technology, Gaithersburg, MD 20899-8520, United States

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ABSTRACT

The effect of film thickness on the resistivity of thin, evaporated copper films (approximately 10–150 nm thick) was determined from sheet resistance, film thickness, and mean grain-size measurements by using four-point probe, profilometer, and electron backscatter diffraction (EBSD) and X-ray diffraction (XRD) methods, respectively. The resistivity of these films increased with decreasing film thickness in a manner that agreed well with the dependence given by a versatile simulation program, published earlier, using the measured values for the mean grain size and fitting parameters for surface and grain boundary scattering. Measurements of the change in sheet resistance with temperature of these films and the known change in resistivity with temperature for pure, bulk copper were used to calculate the thickness of these films electrically by using Matthiessen's rule (this is often referred to as an "electrical thickness"). These values agreed to within 3 nm of those obtained physically with the profilometer. Hence, Matthiessen's rule can continue to be used to measure the thickness of a copper film and, by inference, the cross-sectional area of a copper line for dimensions well below the mean free path of electrons in copper at room temperature (39 nm).

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1. Introduction

The increase in the effective resistivity of copper interconnects as physical dimensions approach the bulk mean free path of electrons (approximately 39 nm at 25 °C [1]) is a serious concern because of the impact it has on reducing circuit speed. A model and a highly versatile simulation program, published earlier [2,3], were used to examine and predict size effects on the resistivity of thin metal films and lines [2,3]. The experimental effort reported here is intended to characterize the resistivity behavior of actual films and compare the results with the predictions of the previously introduced simulation program [3]. This program uses three input parameters. One is the experimentally determined mean grain size for a given film thickness. The other two are fitting parameters that characterize the scattering of electrons from the surfaces and from the grain boundaries within the film.

To a good approximation, metals such as aluminum and copper obey Matthiessen's rule [4], which states that the resistivity of a metal is equal to the sum of the temperature dependent resistivity of the pure, bulk form of the metal plus a temperature independent residual resistivity. This means that the change in resistivity with temperature of the metal is not affected by impurities or other sources of scattering that contribute to the increase in resistivity above that of the pure, bulk form of the metal. Hence, $(\mathrm{d}\rho/\mathrm{d}T)_{\mathrm{pure,bulk}} = \mathrm{d}\rho/\mathrm{d}T$ of the metal as long as the metal obeys Matthiessen's rule. This is a powerful result because, with the value for $(\mathrm{d}\rho/\mathrm{d}T)_{\mathrm{pure,bulk}}$, available in the literature [4], the thickness of a metal film and the cross-sectional area of a metal line can be calculated, respectively, from sheet resistance measurements of the film and from line resistance measurements made at two temperatures, respectively (the thickness of the films measured with this method will be regarded as "electrical thickness" throughout the paper). For films, $t = (\mathrm{d}\rho/\mathrm{d}T)_{\mathrm{pure,bulk}}/(\mathrm{d}R_S/\mathrm{d}T)$, where R_S is the sheet resistance and t is the film thickness. It is therefore important to determine if and how Matthiessen's rule is impacted by size effects.

To obtain experimentally the dependence of copper resistivity on film thickness (size effect), copper films of varying thicknesses were evaporated on Pyrex and silicon wafers, and the sheet resistance and physical thickness of the films were measured. To look for a size effect on Matthiessen's rule, the change in sheet resistance with temperature, $\mathrm{d}R_S/\mathrm{d}T$, was measured for each film thickness. The value for $(\mathrm{d}\rho/\mathrm{d}T)_{\mathrm{pure,bulk}}$ used in this paper is 0.0067 μΩ cm/°C [4].

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* Corresponding author. Tel.: +1 301 975 5026; fax: +1 301 975 5668.
E-mail address: richard.allen@nist.gov (R.A. Allen).

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2. Experimental

2.1. Deposition

A Denton Infinity 22[1] e-beam and resistance-heated evaporator[2] was used to deposit a very thin film of chromium (3 nm as indicated by the crystal sensor of the evaporator) and copper films from approximately 10 nm to 150 nm thick in order to study size effects on the effective resistivity of copper films. The wafers were nominally at room temperature during deposition. The chromium film serves to make the copper adhere to the Pyrex and Si wafers. Three-inch Pyrex wafers and 1-in. Si wafers were used as substrates. Films were deposited simultaneously on the two different types of substrate when the different measurements described below had contradictory requirements for the substrate. When the films were so deposited, the thicknesses of the films on the two substrates were assumed to be identical. In addition, a mask was used to provide a step for thickness measurements as described in Section 2.2. The manufacturer claims that the Pyrex wafers have a surface roughness of 1.5 nm. Copper pellets, 99.999% pure, were the source of the copper films. Chromium rods were used to deposit the chromium.

2.2. Thickness measurement

A Dektak profilometer was used to make physical thickness measurements of the film. There is approximately a 10 nm peak-to-peak noise in the output of the Dektak data, which may be partially caused by the surface roughness of the film. In order to obtain superior precision, the height levels were averaged over a distance of the scan on both sides of the transition using the Dektak software.

To be able to make a satisfactory measurement of the thickness of a film of copper with the Dektak profilometer required a well-defined, abrupt transition between the metal film and the substrate. This was obtained by the use of a special mask held in place by Kapton tape. Many mask materials were tried. By far the best was formed by breaking in two a thin (≈170 μm), 1 in. diameter (100) silicon wafer in such a manner that the fracture is along a (111) plane, which makes a 54.74° angle with the surface of the wafer and provides a knife edge at the bottom of the wafer (facing the Pyrex wafer). With such a sharp, straight edge in contact to the substrate, an abrupt edge to the copper film could be created.

2.3. Sheet resistance measurements

The sheet resistance is commonly measured by a four-point probe method (the four-point method is the preferred method of measuring sheet resistance, but is only applicable to non-patterned films; for patterned films, the Greek Cross variant of the van der Pauw structure provides optimal results). Four in-line, equally-spaced probes are placed in contact with the specimen surface and a current is passed between the two outer probes. The magnitude of this current is measured via the voltage across a standard resistor. The voltage is measured between the two inner probes. The current and voltage values are measured for forward and reverse current directions. The sheet resistance, R_S, is proportional to $R = V/I$, where V is the mean of the absolute values of the measured voltages. The proportionality factor is a function of the geometry. For an infinite sheet with thickness much smaller than the probe spacing (which defines the samples measured in this paper), the sheet resistance is given by [5]

$$R_S = \frac{\pi}{\ln 2}\frac{V}{I}. \tag{1}$$

This method has some important limitations, one of them being the necessity to locate the probes in high-symmetry positions in limited-area samples. To ensure that the measured R_s was not corrupted by Joule heating, the currents were scaled with the film thicknesses and select measurements were repeated at twice the current to ensure that R_s has not changed, which would indicate the presence of Joule heating.

A significant improvement for the four-point probe sheet resistance measurement is achieved with the dual configuration method [6], which makes it possible to make dependable sheet resistance measurements quite close to the rim of the wafer or near any area in the wafer clear of copper. In this method, measurements with the traditional configuration, explained above, are made. Then, current is conducted between one outer and the more distant inner probe, while the voltage is measured across the remaining two probes. In this second configuration, the voltage and current values in the forward and reverse directions are measured. The scaling factor K is determined from a transcendental equation [6]. A highly accurate value for this K factor can be obtained [6] using the following quadratic fit:

$$K = -14.696 + 25.173\left(\frac{R_1}{R_2}\right) - 7.872\left(\frac{R_1}{R_2}\right)^2 \tag{2}$$

where R_1 and R_2 are the voltage-to-current ratios in the first and second electrical configurations, respectively. This simplification is valid if the value for R_1/R_2 is within the interval [1.18, 1.3] [6]. Values obtained for this ratio were usually 1.24. The sheet resistance in the dual configuration method is

$$R_S = KR_1. \tag{3}$$

The standard deviation of sheet resistance measurements made by use of the dual configuration method was at most 0.3%, when repeated measurements were made by raising the probe head between measurements. An in-house built measurement system[3] and probe heads[4] were used to make the sheet resistance measurements.

2.3.1. Location of thickness and sheet resistance measurements to calculate resistivity

A mask, as described in Section 2.2, was placed close to the rim of each Pyrex substrate. Hence, the physical measurement of film thickness with the profilometer was made away from the center of the wafer. This was done so that a large area of the copper film in the central part of the wafer would be available for the four-point probe measurement of sheet resistance. The resistivity was calculated from the sheet resistance measurement near the center of the wafer and the value of the physical thickness obtained from measurements near the rim of the wafer. This could be done because previous measurements of the sheet resistance along the diameter of wafers consistently indicated that the film thickness was constant over the wafer. That this is, in fact, true is substantiated by the observation (shown later in Fig. 5) that the physical

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[1] Certain commercial equipment, instruments, or materials are identified in this paper in order to specify the experimental procedure adequately. Such identification is not intended to imply recommendation or endorsement by the National Institute of Standards and Technology, nor is it intended to imply that the materials or equipment identified are necessarily the best available for the purpose.

[2] This work was performed in part at the NIST Center for Nanoscale Science and Technology's Nanofab facility in Gaithersburg, MD.

[3] The electronics and the probe heads in this system meet, or exceed, all performance aspects of ASTM Method F-84 for measurement of bulk silicon resisitivity by four-point probe, and ASTM Method F-374 for measurement of sheet resistance of thin conducting films on silicon wafers. This system has been used for a number of years to certify silicon resistivity standards (NIST Standard Reference Materials) that are used world-wide in industry, academia, government labs, and other national standards laboratories.

[4] The separation of the probes in the probe head used is 0.635 mm (25 mils).

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