Dynamic Current and Conductivity Measurement Using ResiScope

Application Note

Shijie Wu and Theresa Hopkins
Agilent Technologies, Inc., Chandler, Arizona

Introduction

Current Sensing Atomic Force Microscopy (CSAFM™) is an extended SPM mode for simultaneously probing the conductivity and topography of a sample. It takes the combined advantage of scanning tunneling microscopy and force microscopy, making it capable of studying localized electric properties of resistive samples. CSAFM utilizes electrically conductive AFM cantilevers and operates in standard contact mode. By applying a voltage bias between the substrate and the conducting cantilever, a current is generated. This current can be used to construct a spatially resolved conductivity image. It also allows for local current vs. voltage measurements (I/V) with purely topographic feedback and high resolution. It is a measurement useful in a wide variety of material characterization applications; including thin dielectric films, ferroelectric films, nanotubes, conducting polymers, and others.

The current range in CSAFM is usually limited by the particular pre-Amp used in the device. For example, the standard CSAFM modules by Agilent offer three different sensitivity settings of 0.1 nA/V, 1 nA/V, and 10 nA/V, which gives a current range of ±1 nA, ±10 nA, and ±100 nA respectively. However, there are often applications that require CSAFM to measure current in a large range dynamically, such as in the study of conducting polymers and photovoltaic materials. This requirement can be met by using a log amplifier, where the output is proportional to the natural log of the input current or voltage signal, $V_{out} = K \times \log(V_{in}/V_{ref})$. The simplest circuit for a log amplifier is to use a semiconductor diode and an op-Amplifier, as shown in Figure 1. As suggested in the above formula, in order to make accurate measurement with a log amplifier, it has to be carefully calibrated against elements of preset values. This can be quite demanding and often hinders the use of a log amplifier by wide spread users. Here we introduce an advanced dynamic current

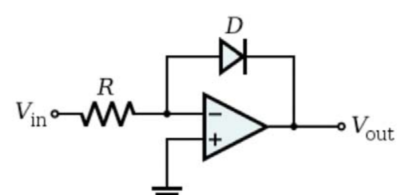

Figure 1. Schematic of a simple log amplifier that uses a semiconductor diode and a operational amplifier, the output is proportional to the log of the input.

measurement module, the ResiScope, which offers accurate current and resistance measurement over a large current range with automatic calibration capability. ResiScope is an add-on module to existing Agilent AFM platforms, the main component of the ResiScope goes in between the AAC mode controller and the head electronic box (HEB), as shown in Figure 2. It offers a wide resistance range measured from 0.1KΩ to 1TΩ, or a current range measured from 1mA to 100fA. The resistance and current output can be in either linear scale or log scale, depending on the convenience of the user application. Figure 3 presents the current vs. voltage curve measured over a 10k resistor in both linear and log scale. The self-calibration function built in the ResiScope module provides a convenient way to ensure the accuracy of the measurement.

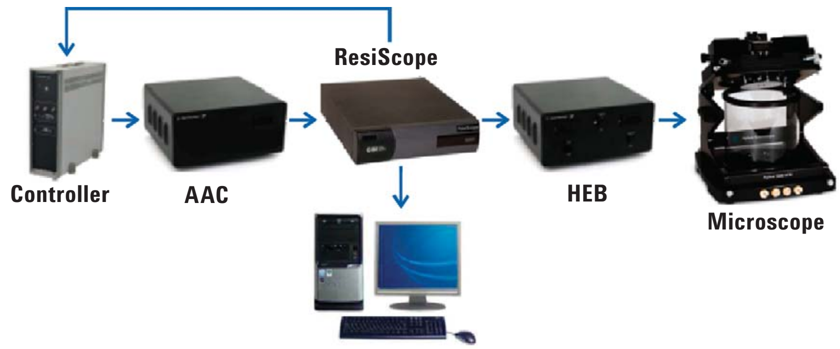

Figure 2. ResiScope is an add-on module to existing Agilent AFM platforms, which plugs in between the AAC controller and the HEB of the system

Application Examples

Conductivity of TiW film

TiW is a common material used for multilevel metallization in VLSI technologies. It provides a good barrier to prevent Si diffusion into Al, the commonly used metal for interconnects in semiconductor devices. It adheres strongly to SiO_2 and Si_3N_4 films and provides a clean and uniform nucleating surface for Al. TiW is an alloy of Titanium and Tungston, usually contains 10 wt% of Ti and 90 wt% of W in the film. The TiW film usually consists of columnar grains of W, while the Ti form solid solution with some W atoms at the grain boundaries. The distribution of Ti is an important factor for the behavior of the TiW film. The resistivity of the TiW film is reported to be in the range of 50-80 μΩ-cm. Figure 4 shows the topography and current images obtained simultaneously on a TiW film deposited on Si. The topography image reveals that TiW film shows a surface roughness of about 6nm in RMS. The current image shows higher conductivity at the areas between the granular domains, corresponding to the Ti/W liquid solution formed at the grain boundaries between columnar W phases.

Resistivity Mapping of SiGe Structures

Silicon-Germanium (SiGe) technology is the driving force behind the explosion in low-cost, lightweight, personal communications devices like digital wireless handsets. The heart of SiGe technology is a SiGe heterojunction bipolar transistor (HBT), which offers advantages over both conventional silicon bipolar and silicon CMOS for implementation of communications circuits. Figure 5 shows the AFM

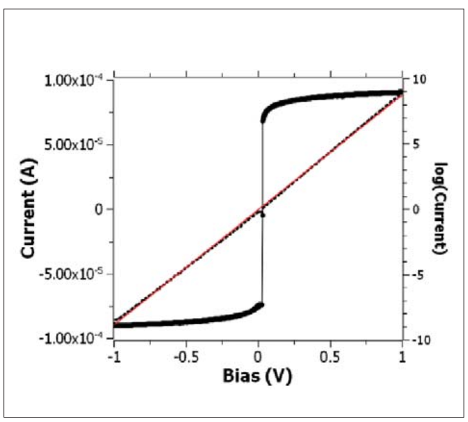

Figure 3. The current (dotted line) with the linear fit of the data measured over a 10 k resistor using ResiScope is presented together with the log(current) (black line with filled circle) in the same graph.

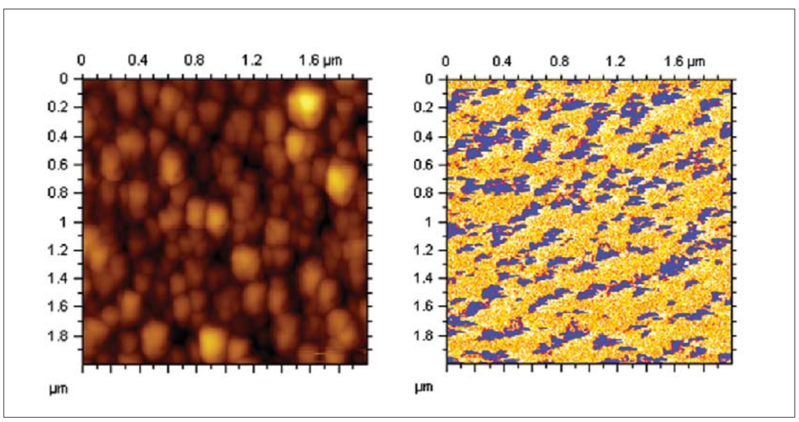

Figure 4. Topography (left) and current (right) images of TiW/Si film using an Agilent 5500 with ResiScope. The surface roughness of the film is about 6nm, and the higher current regions (blue in the current image) correspond to the Ti/W solid solution distributed at the grain boundaries of W domains.

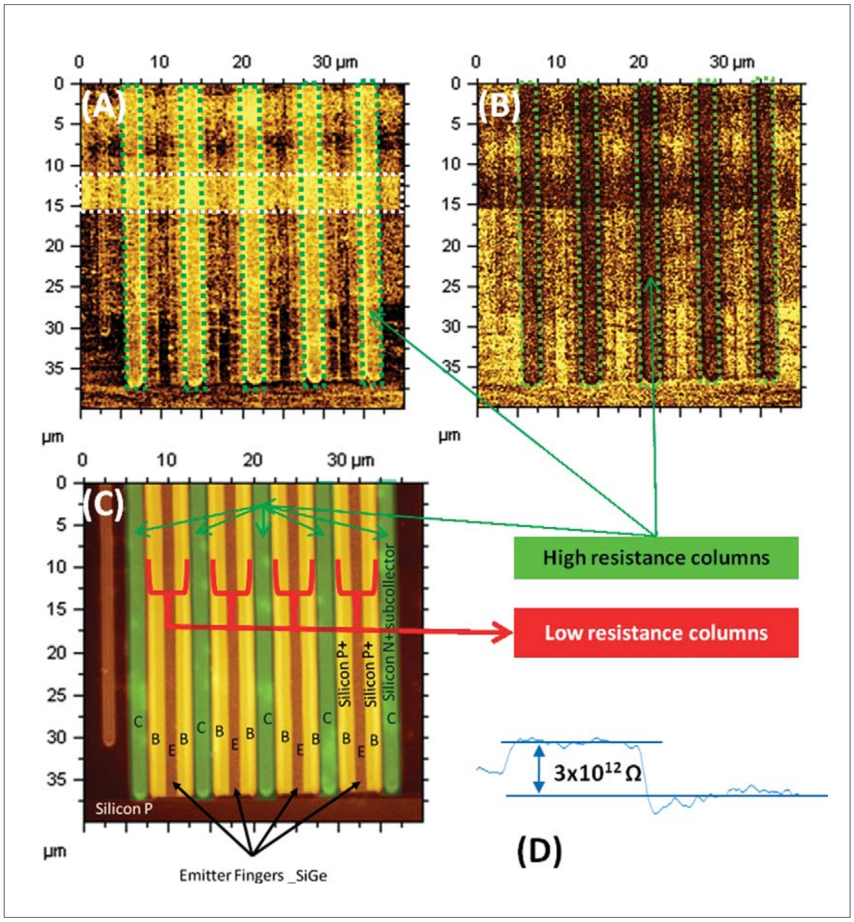

Figure 5. Resistance (A), current (B), and topography (C) images of a SiGe HBT structure. The green dotted frames indicate the regions of higher resistance that corresponds to the N^+ silicon collector. The white dotted frame indicates a region that is less conductive than its adjacent areas, the average difference in resistivity between this region and its neighboring region is estimated about 3 TΩ (D).

an increase in electrical conductivity and magnetic susceptibility as the bonding electrons are "released". Even though the precise origin of this insulator-to-metal transition remains controversial, VO_2 has received great attention because of the large reversible change of electric, magnetic and optical properties at temperatures around 70°C. The transition temperature of vanadium dioxide may be changed by the addition of high-valent transition metals such as niobium, molybdenum, etc. Because of this anomalous behavior, vanadium dioxide has been presented as an attractive thin film material for electrical or optical switches, optical storage, laser protection, and other applications. Because the transition of VO_2 from insulator to metal is accompanied by a relatively large change in conductivity, it becomes convenient to use ResiScope to measure the electric conductance change with temperature. Figure 6 presents the current vs. voltage (I/V) spectroscopy recorded at a series of different temperatures on a thin film sample of VO_2 deposited on ITO. The current is presented in its log value as it was measured by ResiScope. The sudden change in conductivity at temperatures around 60°C for this thin film sample is evident from the measured I/V plot.

images of a SiGe HBT structure. In the topography image (Figure 5C), each of the individual fingers is labeled as B for Base, E for Emitter, and C for Collector. The base is made from P^+ Silicon, collector from N^+ silicon, and emitter from SiGe. The resistance (Figure 5A) and current (Figure 5B) images revealed differences in resistivity over the surface of different material. In particular the collectors of N^+ Silicon shows relatively higher resistivity than the base and emitter, as indicated by the dotted green frames in the resistance and current images. Additionally, the resistance measurement using ResiScope also revealed the variation in resistivity not directly related to the different components of the device. For example, the area enclosed by the white dotted frame in Figure 5A shows a higher resistivity in general comparing to its adjacent neighboring areas. The average difference in resistance is estimated to be about 3 TΩ as shown in Figure 3D, corresponding to a current as small as 10 pA.

Conductivity Change of VO_2 Thin Film with Temperature

Vanadium (IV) oxide is an insulator at room temperature. It is believed to have a distorted rutile structure which results in shorter distances between pairs of vanadium atoms that form metal-metal bonding. At temperatures around 70°C, its structure changes to an undistorted rutile structure and the metal-metal bonds are broken causing

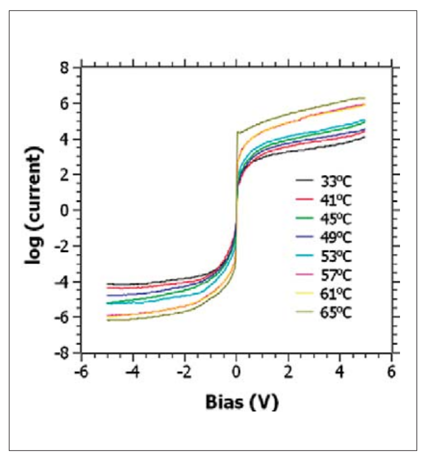

Figure 6. Current vs. voltage spectroscopy recorded at different temperatures on VO_2/ITO thin film. The temperature dependence of conductivity of VO_2 film shows a possible phase transition at about 60°C.

Summary

The examples presented in this note show that ResiScope , with its dynamic range and autocalibration function, is capable of measuring large range current and resistance with high resolution. Consequently, ResiScope can be a powerful tool for charactering electric properties of a wide range of materials, including metals, semiconductors, devices, and thin films, etc.

Note: For the most useful local resistance measurements with the atomic force microscope, the tip-sample contact is very important. We suggest using a new tip that is not contaminated, worn or oxidized. Also, the best results depend on the indent and the nature of the sample surface. Caution must be taken in the running of a CSAFM experiment and the interpretation of the data obtained therein.

AFM Instrumentation from Agilent Technologies

Agilent Technologies offers high-precision, modular AFM solutions for research, industry, and education. Exceptional worldwide support is provided by experienced application scientists and technical service personnel. Agilent's leading-edge R&D laboratories are dedicated to the timely introduction and optimization of innovative and easy-to-use AFM technologies.

www.agilent.com/find/afm

Americas

Canada	(877) 894 4414
Latin America	305 269 7500
United States	(800) 829 4444

Asia Pacific

Australia	1 800 629 485
China	800 810 0189
Hong Kong	800 938 693
India	1 800 112 929
Japan	0120 (421) 345
Korea	080 769 0800
Malaysia	1 800 888 848
Singapore	1 800 375 8100
Taiwan	0800 047 866
Thailand	1 800 226 008

Europe & Middle East

Austria	43 (0) 1 360 277 1571
Belgium	32 (0) 2 404 93 40
Denmark	45 70 13 15 15
Finland	358 (0) 10 855 2100
France	0825 010 700* *0.125 €/minute
Germany	49 (0) 7031 464 6333
Ireland	1890 924 204
Israel	972-3-9288-504/544
Italy	39 02 92 60 8484
Netherlands	31 (0) 20 547 2111
Spain	34 (91) 631 3300
Sweden	0200-88 22 55
Switzerland	0800 80 53 53
United Kingdom	44 (0) 118 9276201

Other European Countries:
www.agilent.com/find/contactus

Printed in USA, July 21, 2011
5990-8413EN

Agilent Technologies