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Monitoring the Conductivity of Thin Metal Layers During the Processes of Grain-growth and Dewetting, Using a Desktop FEG-SEM

AC Bouman[1], Jacob Jansen[2], M Griebling[2], L Lammers[2], VM Santos Costa[2], JJA Vermeulen[2], JGA Vervloat[2], AFJ Hammen[1] and HW Zandbergen[2]

[1]Thermo Fisher Scientific, Eindhoven, Noord-Brabant, Netherlands, [2]TU-Delft, Delft, Zuid-Holland, Netherlands

The electrical resistance of materials depends on the micro-structure. If a material is heated, its microstructure can change, which results in a change in resistance. For thin layers with nanometer-size grains (as in the circuits of computer chips) the correlation between the microstructure and the resistance can be determined using an electron microscope. In this work we show that with a Phenom-Pharos desktop FEG-SEM one can image in-situ microstructural changes while measuring the resistance at the same time.

The experiments were performed using a Thermo Fisher Scientific Phenom-Pharos FEG-SEM, equipped with a back-scatter detector, using an accelerating voltage of 15 kV. For these experiments, a dedicated sample holder was developed (figure 1a) and a commercially available MEMS based heater was used. The MEMS heater consisted of a Molybdenum coil covered with a layer of SiN with four contact points at the top side of the chip (figure 1b). At the side of the chip four contact points are located (figure 1b), allowing a four-point measurement to obtain the electrical resistance across the sample. The SiN layer on top of the heater coil ensures that the electrical systems of the coil and the sample are independent. A 50-75 nm thick patterned layer of Au or Pt was deposited by metal evaporation, using a mask to form a line of about 120 µm wide on top of the heater and large contact points on the sides (see figure 1b) Since the resistance is temperature dependent, we measured it at the same sample temperature each time. This is possible because the change in temperature is quite fast (eg a few seconds) and this quenching to a low temperature was not influencing the thermal changes in the sample (which was checked by heating without quenching). The recording speed was 30 images per minute.

Since during grain growth several thousands of images are recorded an algorithm was developed to determine the size of the grains automatically. We based our algorithm on the one described by ASTM international[2]. This method is based on a few line scans taken from the image and counting the number of intersections of the line scan with a grain boundary visual in the image. We automated this method by adding some image processing steps and determining the number of grain boundary crossings by the high peaks in the derivative of the line scans.

The temperature is increased in steps of 50K. Very small grains were observed before heating the sample. For the Au samples grains are observed to grow at 430 K and for Pt at 500 K. We observed grains to grow rapidly in the first few minutes. Later, the grains grew much slower and we see clearly that they grow by incorporating neighboring grains. This is in agreement with the model of Thompson[1]. A graph of the resistance of the thin gold line as function of the grain size is plotted in figure 1c. As expected the resistance decreases with increasing grain sizes, as the electrons have to pass less grain boundaries.

Upon further increase of the temperature of the Au film at about 800 K dewetting occurs. This is due to the fact that a thin layer is in a meta-stable phase, whereby compared to round particles, the total energy of a thin film is high due to the high surface and interface energies [3]. In the SEM images the dewetted areas appear dark, because the SiN substrate reflects less electrons then the metal. In figure 2ab some examples of a partial dewetted 50nm thick metal layer are shown. While the 50 nm gold layer shows “finger-like” shapes as described by Thomson[3], the 50 nm layer of Platinum and the 50nm gold layer with a 10nm Chromium “sticking-layer” show a more spherical expansion of the dewetted areas. We took the percentage of pixels below a certain threshold as the fraction of dewetted area. The dependence of the resistance on the dewetted area (figure 2c) shows two features. Up to 30-40% dewetted area hardly any influence on the resistance is

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observed. As expected the resistance increases fast when the more than 50% of the area is dewetted. In figure 2c it is seen clearly that at certain points abruptly paths that electrons can travel through the metal are blocked. Although the dewetting proceeds visually quite different (figure 2abc) for the other two metal layers the relation between dewetted area and resistance showed the same shape of which figure 2c is an example.

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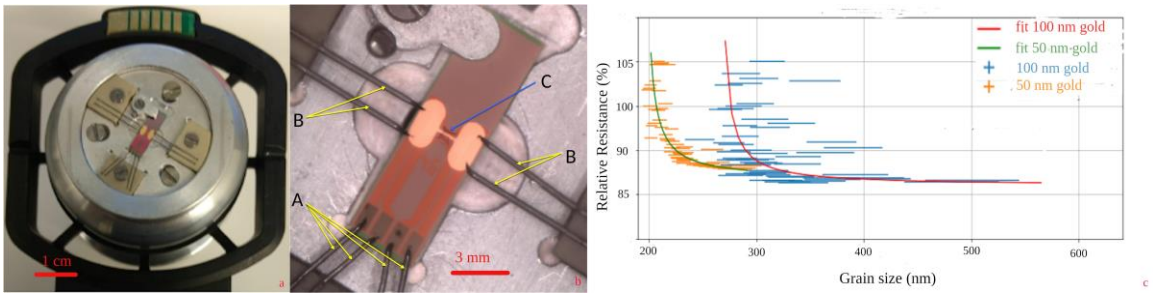

Figure 1. a) Top view of the modified sample holder. b) Optical image of the heater chip clamped in the holder (the middle area of figure1a). At the bottom, four pins (marked A) provide the electrical connections to the heater. At the side of the chip four pins (marked B) are present to measure resistance over the sample. The gold is seen in the middle of the chip: two big contact points are connected by a thin line. The heater coil is underneath the middle area of the gold line. c) The resistance (relative to the resistance at the start of the experiment) as function of the grain sizes for a gold line of 120 µm and 50 nm and 75 nm thick. An exponential curve is fitted to the data.

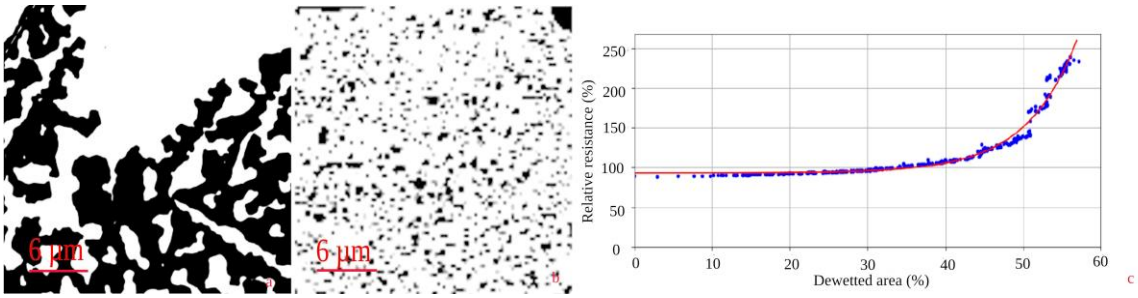

Figure 2. a) partial dewetted (at 800K) 50nm gold layer, showing finger-like paterns. b) partial dewetted (at 800K) 50 nm Pt-layer, showing dewetting progressing in a spherical way and from much more locations than in (a). c) Typical measurement of the relative resistance (100% is the resistance when no dewetting is present) as function of the dewetted area.

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