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In-situ Heating Experiments Within a Tabletop SEM: Grain Growth and Dewetting of Thin Layers of Au and AuPd With and Without an Al_2O_3 Coating

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Nowadays electron microscopes are employed not only in the study of samples in static conditions, but also for the investigation of dynamic effect, i.e. phase changes or chemical reactions. In this work we will show that this is possible also with a *low-end tabletop* SEM. As an example of its capability, the behavior of a thin metal film is studied while it is heated up to around 1000 K.

The experiments were performed with a Phenom-Pro SEM equipped with a back-scatter detector, using an accelerating voltage of 10 kV. For this experiment, a dedicated sample holder with a commercially available heater-chip was developed (figure 1a and 1b). The heating element consisted of a Platinum coil covered with a layer of SiN with four contacts. In the middle of the chip, a 50 nm layer of Au was deposited by sputtering on top of the heating coil. The SiN layer between the coil and the Au film ensured that these two were electrically not connected. The resistance of the coil was determined by a four-point measurement. The temperature dependence of the resistance was calibrated using Raman-spectroscopy. The speed of recording depends on the size and quality of the images and is limited by the dwell time of the SEM. In this work, we used images of 1024x1024 pixels and the best quality, which is internally in the Phenom-PRO done by frame averaging. This allowed taking one image per 2 seconds.

During a step-wise increase of the temperature, at 430 K grains are observed to grow. By using a flood-fill algorithm with a chosen threshold, the average grains size was monitored, as shown in figure 1c. Although it looks like the grains are formed during this heating, we know from previous TEM-experiments [1] that after sputtering already grains with diameters of a few nanometer are present. Using the Phenom-PRO SEM we did not observe these grains due to a combination of lack of resolution and the fact that several grains are on top of each other in the viewing direction leading to image blurring. We observed grains to grow rapidly at first (steep part of figure 1c). Later the grains did grow by incorporating neighboring grains. Thompson [2] has a model, which describes a primary growth - the forming of the grains at a temperature of about 0.2 times the melting temperature (in our case 430 K) - and a secondary growth – the growth of grains by incorporating neighboring grains. Thompson also predicts that the grain diameters will be of the order of the film thickness. In our case the diameter of the grains was already few times this number just after the first rapid growth in figure 1c.

When increasing the temperature to 800 K dewetting occurred. This is due to the fact that the mobility of the atoms is high enough to minimize the total energy, whereby compared to round particles, the total energy of a thin film is high due to the high surface and interface energies. The dewetting started mainly where the film was weakest, i.e. the areas where the gold is deposited on a non-flat surface, such as the edges of the heating coil. The process evolved from there with a fingerlike shape (figure 2a), as predicted by Thompson [3]. As shown in figure 2d, dewetting followed the grain boundaries.

In a second experiment, the Au film was covered with a 10nm layer of Al_2O_3. In this case, the dewetting at 800 K appeared to be completely different, as shown in figure 2b, and progressed much slower at the

same Temperature. Moreover, no fingers were observed, and the growth appeared to be spherical. Also, in the case where the Au layer was covered with Al_2O_3, no grains were observed. Possibly, the size of the grains is too small compared to the microscope resolution or the blur in the images was induced by the diffuse scattering in the Al_2O_3 layer. Fingers may not be visible because of the small grain size.

The different contrast in the SEM images taken at room temperature and at 800 K shown in figure 3 was caused by the infrared radiation on the back-scatter detector, which was positioned relative close to the sample. The reason that we still see something was that the area heated is very small. To improve the contrast, the heating coil can be further reduced in lateral size.

[1] T.Kozlova, In-situ transmission electron microscopy investigations of electromigration in metals, Thesis, TU-delft (2015)
[2] CV Thompson, *Annual reviews of Materials Science* **20** (1990), p. 245.
[3] CV Thompson, *Annual reviews of Materials Science* **42** (2012), p. 399.

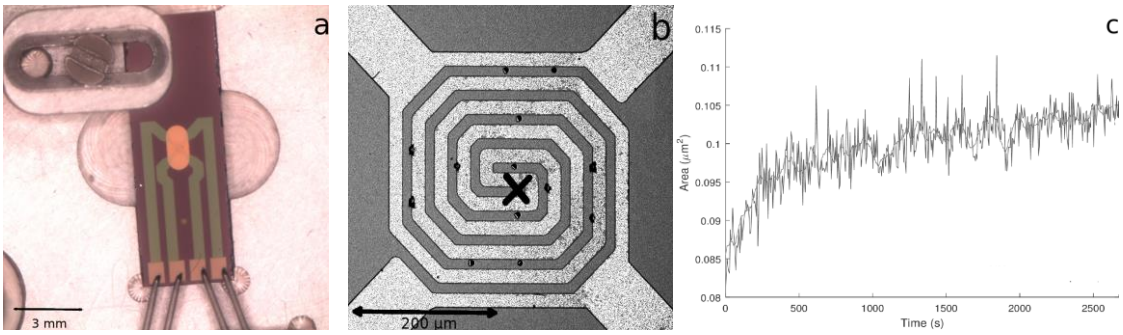

Figure 1. a) Optical image of the heater chip clamped in the holder. At the bottom, four pins provide the electrical connections. Underneath the gold (the oval in the center) the heating coil is located. b) SEM image of the heating coil with a marker positioned at the location that is always taken as the center of all images in this paper. c) Average grain size for a 50 nm layer of gold as function of time when heating at 430 K.

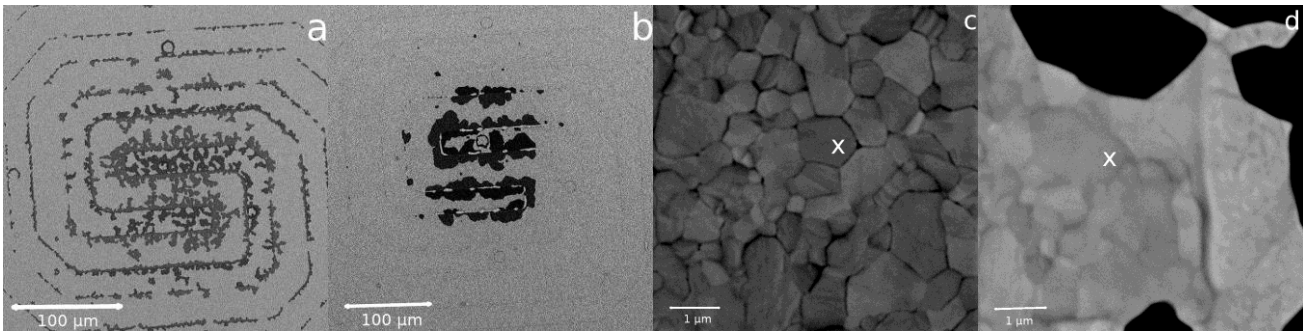

Figure 2. Four SEM images of the coil after several minutes heated at 800 K (a, b and d) and at room temperature (c). a) 50 nm gold layer only, showing finger-like dewetting. b) 50 nm gold layer with 10 nm Al_2O_3 on top showing dewetting in a sphere-like way. c) after the grains have grown at 430K. d) after several minutes heated at 800 K. The sharp corners of the dewetted areas coincide with the grain boundaries. The marker in c and d indicates the same location. It is clear that also the grains grow when other parts of the sample are dewetting. The difference in contrast of the 2 images is due to the infrared radiation of the sample hampering the detector.

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