

# Characterisation of platinum sputter-coatings using scanning tunnelling microscopy

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The surface morphology of platinum sputter-coated films was investigated using scanning tunnelling microscopy. The films were found to be universally very smooth (roughness of around 1nm), but with increasing roughness at higher sputtering pressures. The surfaces were routinely polycrystalline with very small (around 10nm) grains. These results can be linked to expected sputtering behaviour.

The samples were prepared by sputter-coating from a platinum film onto a substrate. In sputter coating, a metal target is mounted in a chamber above the substrate. The chamber is then evacuated, and a nonreactive gas (in our case argon) is backfilled into the chamber at low pressures. This gas is then ionised and accelerated by an electric field into the target. Atoms are knocked off the target by the ion collisions and fall onto the substrate. This technique produces typically very smooth films with a thickness that can be carefully controlled. The structure and characteristics of platinum sputtered films have already been investigated<sup>[1]</sup>, so here we will investigate the morphology of the films.

Scanning Tunnelling Microscopy (henceforth described as STM) is an established technique for imaging surfaces<sup>[2]</sup>. In STM, a bias is applied between an (ideally atomically sharp) metal tip and the conducting sample. The tip and sample are then moved together so that a small current tunnels between the tip and the sample. In the mode of operation used in this experiment, the height of the tip is adjusted so as to maintain a constant current. The current can be considered to a first order approximation to be a function of the distance between the tip and the sample surface. This is especially true for homogenic samples such as the ones used in this experiment.

The images were produced using a Nanosurf Easyscan E-Line STM. Tips were produced by stretching of a very thin platinum iridium wire to breaking point. The tip produced at the fracture point should be extremely sharp (Fig. 1). The platinum samples were mounted onto standard sample

disks for the E-Line STMs using silver conducting glue.

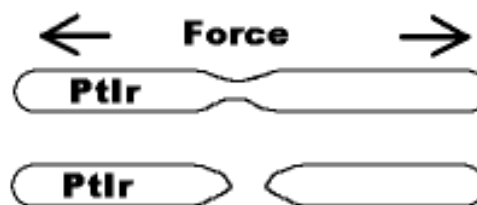


Figure 1 – Formation of tip from stretching wire

There were a total of six different samples of platinum sputter-coatings, prepared at a variety of different sputtering pressures (the pressure of the gas used to knock atoms from the target) between 4 and 25 millitorr.

Before using on the samples, the performance of each STM and tip was tested on highly ordered pyrolytic graphite (HOPG) and confirmed that images such as those generated by Eunkyung Kim<sup>[3]</sup> could be generated.

Each sample was loaded into the STM and imaged in a variety of different locations on the sample surface – this was achieved by rotating the sample holder. Measurements could then be taken from these images and correlated with the differences in sputtering parameters.

Each images was taken over 200nm with a gap voltage of 0.3 V and at constant current of 1 nA. Gains and scan speed were varied so as to produce as low-noise, tip-lag free image as possible, without thermal drift becoming a problem.

Each sample was assessed for average grain size (diameter) by measuring of a

random selection of visible grains, using the Easyscan software's built in measuring tools. In addition, a measurement of the area roughness of each scan was made. The value taken from these measurements was that of the root mean squared roughness – this was considered to better allow for extremities in the image – caused by debris on the sample or by vibrations of the equipment, caused by collisions in the lab.

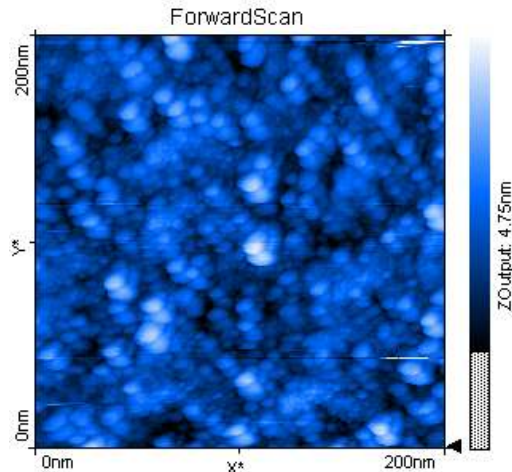


Figure 2 Colour-height map of an area of sputter-coated platinum.

Several of the images showed substantial anisotropy of the grains. Rotation of the image showed that the isotropy was still present, ruling out equipment errors or thermal drift as the cause of the apparent anisotropy. Surfaces proved to be routinely smooth and showing clear signs of grain structure (fig 2), although there were some notable exceptions such as Fig 3 where a hole approximately 2 orders of magnitude larger than the grains can be observed.

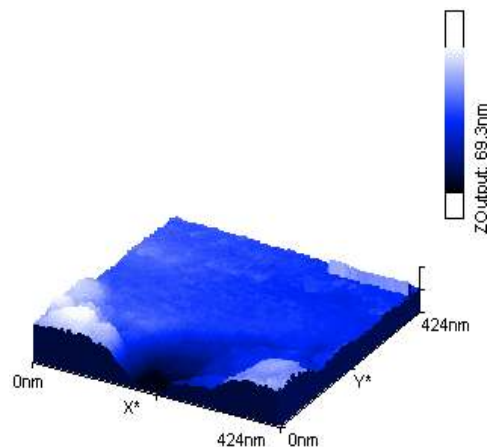


Figure 3 3D map of a hole in the platinum film

Such anomalous images were disregarded when collecting data on surface roughness – very few examples of anything

but a smooth, granular surface were found. The overall results comparing surface roughness and the sputtering pressure can be seen in Fig. 4.

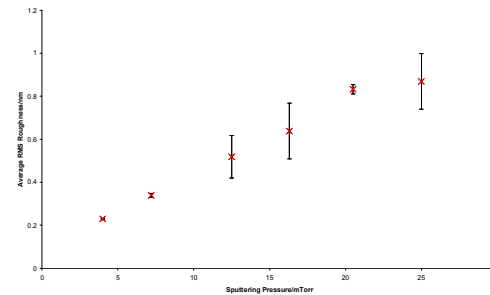


Figure 4 A graph of roughness against sputtering pressure with statistical error bars

The surface roughness is very small, showing the smoothness caused by sputter-coating. There is a clearly defined trend however of increasing roughness with increasing sputtering pressure. The statistical error is plotted along with the data points here – in some cases this is quite large due to the natural variation in morphology across the sample. In addition, gathering a large number of images was often hampered by the frequency of tip crashes, spoiling the tip sharpness and hence the ability to image.

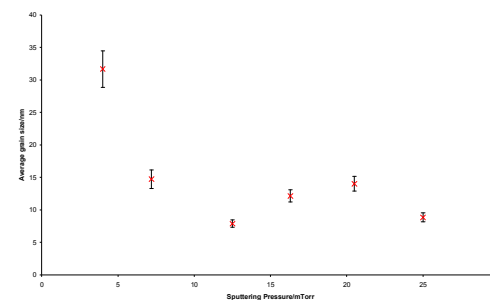


Figure 5 – Grain size against sputtering pressure with statistical error bars

Sputtering pressure and grain size distribution showed no clear simple trend. It seems that control of sputtering pressure is not a suitable method to control the grain size of crystals formed.

The STM images demonstrated that a platinum sputter-coated films are polycrystalline as shown by XRD<sup>[1]</sup> before. It has been possible through the STM images to expand on this data however, providing an idea of grain size being around 10nm and seeing how the grains fit together and form an extremely flat surface. Grain size variation does not appear to be a function of sputtering pressure. This would appear to fit with the fact that amounts of material dislodged from the target per ion would not be expected to vary greatly with pressure.

The observed anisotropy is explained by the angle at which substrates are placed in the sputtering chamber relative to the target. That the degree of anisotropy was not universal across the samples suggests that this depends too on the location on the substrate.

The variation in roughness is a result of the change in mean free path as a result of sputtering pressure. Due to the fewer collisions (longer free path) at lower pressures, the average energy of atoms hitting the substrate is higher, This greater energy results in a wider, flatter spread across the substrate and hence a smoother surface.

### **References**

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2. Scanning Tunnelling Microscopy. G Binnig, H. Rohrer. IBM Journal of Research and Development, Vol 40, No 3, 279-293.
3. Atomic structure of highly ordered pyrolytic graphite doped with Boron. Eunhyung Kim et al Electrochemistry Communications 4 (2001).