XMaS the UK-CRG	**Experiment title:** Self Assembled Nanoparticles	**Experiment number:** 28-01-47
Beamline: **BM 28**	**Date of experiment:** from: 29th September 1999 to:4th October 1999	**Date of report:** 13 October 1999
Shifts: 15	**Local contact(s):** Simon Brown	*Received at XMaS:*

Names and affiliations of applicants (* indicates experimentalists):

*Prof. Colin Norris,
Department of Physics and Astronomy, University of Leicester, U.K.
*Dr Chris Nicklin,
Department of Physics and Astronomy, University of Leicester, U.K.
Prof. Richard Palmer,
Nanoscale Physics Research Laboratory, University of Birmingham, U.K.

Report:

Five days of beam time were used in October 1999 to determine the structure of self-assembled, surfactant stabilised gold particles on different substrates. Specifically, our aims were to identify:

the packing arrangement of two-dimensional monolayer thick assemblies;
three dimensional ordering in thicker film; and
the influence of the substrate including the substrate morphology on the assembly.

A wide range of samples were prepared at Birmingham by evaporating suspensions of gold particles on different substrates which included silicon(111), stepped silicon(111) graphite and gold(100). A total of 41 samples were prepared with particle sizes ranging from 1.6 to 5nm and thicknesses from 1 to 5 monolayers (ML). Time allowed us to measure 16 of these.

The problem of excessive background encountered in our first run on the CRG beamline in March 1999 was overcome by the use of highly ordered substrates and of post-sample slits positioned at the beginning of the detector arm. With this arrangement the scattering volume could be defined without affecting the resolution. The air scattering was removed and the background was due to the sample. We were thus able to work without using a helium enclosure.

A series of scans were taken for each sample. The high angle scan for one sample confirmed that the structure within the particles was the usual fcc phase.

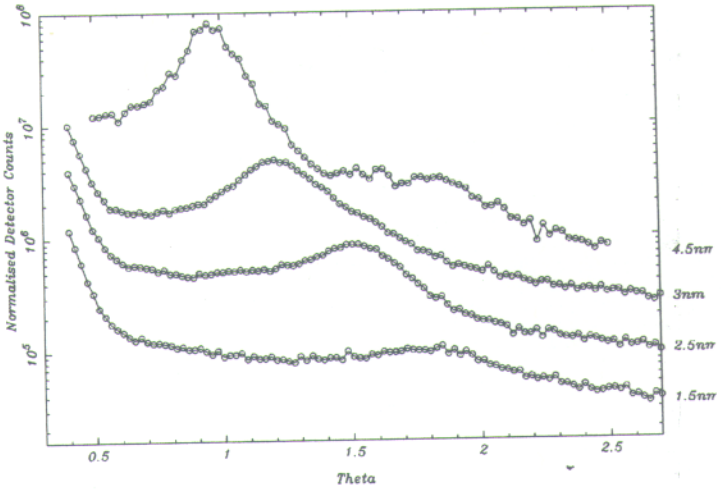

Fig 1: horizontal scans showing small angle scattering for a variety of particle sizes.

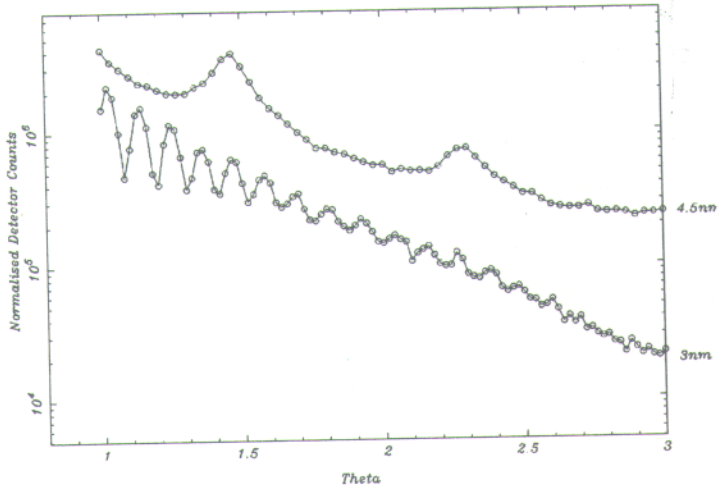

Fig 2: reflectivity curves for two 5ML films of different particle size.

Fig 1 shows a selection of low angle horizontal scans with the incident and exit angle below the critical angle for gold to maximise the relative contribution of the surface. There is a clear correlation between the position of the main feature and the expected size of the particles. For some samples other features could be seen but no clear features could be identified at low angles, which corresponded to a regular close packed arrangement. Scans around the axis of the incoming beam confirmed that the main feature was indeed a ring of scattering consistent with a disordered array.

Fig 2 shows the reflectivity (uncorrected for area of illumination and other factors) for two 5ML films of different particle size. The fine oscillations observed for the 3nm particle film corresponds to the overall film thickness (~25 nm). By contrast, the other plot for the 4.5nm film which was prepared using a different (hexane) solution show oscillations, which correspond to layering of the particles.

In summary, the results confirm that we can detect scattering from planar arrays of gold particles. They form dense but highly disordered assemblies. The measurements show that a critical factor is the conditions of preparation: the temperature, the rate of evaporation, the solvent and the substrate. It indicates that *real time*, *in-situ* measurements could be made of the assembly process using X-ray diffraction and a purpose designed environmental stage. The analysis is continuing.