XMaS the UK-CRG	**Experiment title:** Embedded Mesoscopic Particles	**Experiment number:** 28-01-28
Beamline: **BM 28**	**Date of experiment:** from: 24th February 1999 to: 2nd March 1999	**Date of report:** 13th October 1999
Shifts: 18	**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, UK

Dr Chris Binns,
Department of Physics and Astronomy, University of Leicester, UK

Dr Kevin Peters*,
ESRF

Report:

Six days of beamtime in February and March 1999 were used for a study of the structure and morphology of iron nanoparticles embedded in a silver matrix. In particular, we wished to identify whether the crystal structure within the particles was face centred cubic or body centred cubic and how it varied with size. The three dimensional morphology of the particles was also to be measured, to determine if the impact of the particles on the surface caused them to become oblate rather than remain spherical. These structures were then to be correlated with the magnetic properties of the samples, measured using a vibrating sample magnetometer at Leicester.

The samples were prepared in the ultrasmall particle source, by depositing mass-selected Fe particles onto polished poly-ether-ether-ketone (PEEK) substrates. PEEK was used since it has a low atomic mass and is the preferred material for the magnetic measurements. A silver vapour source was used for co-deposition to embed the clusters in a neutral (non magnetic) matrix. A total of 24 samples were prepared with varying cluster sizes (1-3nm) and volume filling fractions. A typical structure is shown in Fig 1.

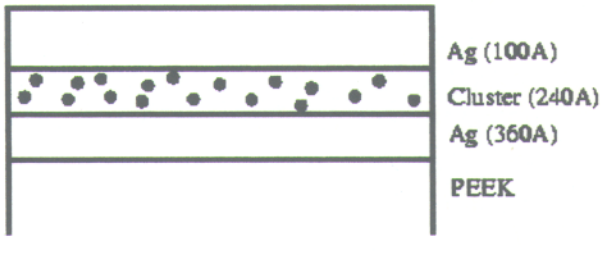

Fig1: The embedded cluster layer is supported on a thick polycrystalline silver buffer layer and capped with a thinner silver layer to prevent oxidation.

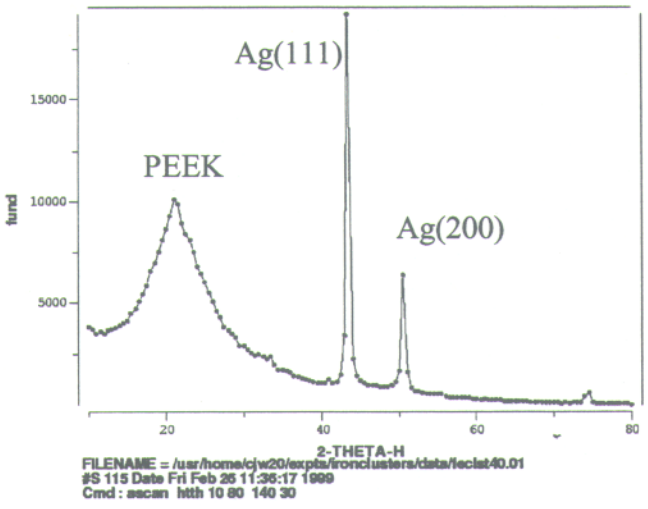

Fig 2: Scattering from the polycrystalline silver matrix, and diffuse scattering from the polymer (PEEK) substrate.

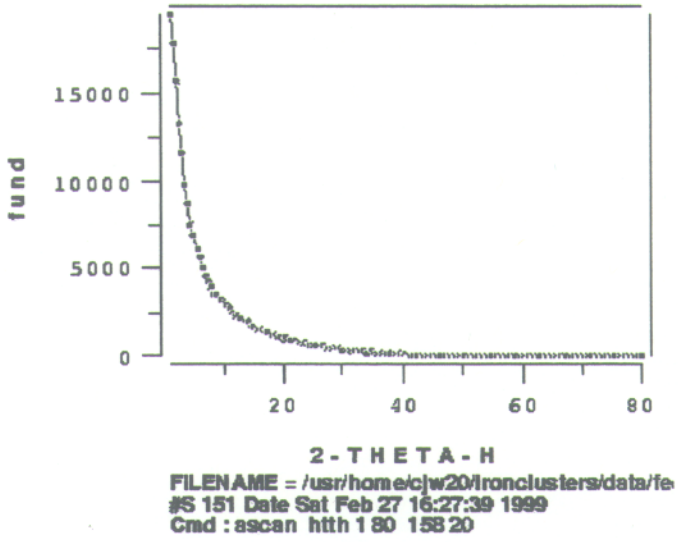

Fig 3: Background due to air scatter. This curve was measured with no. sample in the beam

Fig 2 shows a typical horizontal 2θ scan, with the incidence grazing angle above the critical angle for silver. The dominant features correspond to the PEEK substrate and the silver layers. No features can be attributed to scattering from the Fe. The same negative result was repeated for all samples measured, irrespective of the particle size or X-ray wavelength. Measurements at low angle were dominated by a steeply rising background which we identified as air scattering from the region near the sample (Fig 3).

Conclusions

- We were pleased with our first run on the beamline. All the equipment worked well. Some changes to the experimental setup and the sample configuration need to be made.
- PEEK is a poor choice for a substrate since although it is light, it is highly disordered and will generate a strong diffuse scattering feature. In future highly ordered, single crystal substrates must be used.
- Air scattering could be removed by either enclosing the sample in a vacuum or helium enclosure, or by using slits on the detector arm immediately after the sample. Together with the end of beamline slits, they define (and reduce) the scattering volume without affecting the large angular acceptance of the detector.
- A thinner non-metallic capping layer should be used to protect the sample. Even with the nitrogen stream, surface beam damage was observed.