EUROPEAN SYNCHROTRON RADIATION FACILITY

INSTALLATION EUROPEENNE DE RAYONNEMENT SYNCHROTRON

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office via the User Portal:

https://wwws.esrf.fr/misapps/SMISWebClient/protected/welcome.do

Reports supporting requests for additional beam time

Reports can be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** In situ coherent diffraction imaging studies of metal-coated polymer beads in a curing epoxy adhesive	**Experiment number**: MA-2826
Beamline:	**Date of experiment**: from: 12/11/2015 to: 17/11/2015	**Date of report**: 19.02.2016
Shifts:	**Local contact(s)**: Dr. Yuriy Chushkin	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

The aim of the presented experiment was to investigate the wetting and curing mechanism for metal coated micron sized polymer beads. The polymer beads were made by the Ugelstad technique [1] which is a method for making spherical monodisperse polymer particles. The particles used in this experiment were made of poly(methyl methacrylate) and coated with a ~90 nm thick nickel layer, and a ~20 nm thick gold layer. In addition to the metallic layers, the particles had an outer coating of SiO_2, of thickness in the range from 0 nm to ~100 nm, depending on the particle type. The particles used in this experiment are normally used in the micro-electronics industry for making electrical contact between electrical components [2]. The particles are dispersed in an adhesive and smeared over the contacts. The electronic chip is then placed in an oven so the adhesive can cure at 200° C for 30 minutes.

It has previously been seen particles swell when dispersed in a liquid [3] and that after the curing process has finished the spheres have fractured. It has been theorized by the manufacturer that this can be caused by swelling of the polymer in the adhesive. The main goal of this experiment was to investigate the wetting and curing mechanism, to see if the swelling could be observed and correlated to some of the preexisting defects in the metal coating.

The experimental method chosen for this experiment was 3D X-ray coherent diffraction imaging. This method is suited because it is both sensitive to the metallic coating, and the polymer core. The 3D model retrieved can be used to give accurate information of the curing process. The experimental procedure was to electrostatically stick a series of spheres on a silicon nitrate (Si_3N_4) membrane and choosing a suitable particle for X-ray imaging by a light microscope. A series of scattering images was recorded by a 2 dimensional photon counter detector with the particle rotated from ~ -80° to ~ 80° around a vertical axis. These scattering images were then used as input to an iterative algorithm inverting them to a real space 3D object. The X-ray photon energy was 7.0 keV.

Several particles were imaged before any mixing of the particles with the adhesive. This was done to get a reference of what the particles look like. One of the reconstructions can be seen in Figure 1, along with one of the scattering images, demonstrating how closely the imaged particle is to a perfect sphere. The fact that it was possible to reconstruct the data is an achievement in itself, as practical experience from the beamline tells that reconstructing particles close to a perfect sphere is challenging. The projection of the particle demonstrates that the inner polymer core and the metallic coating is clearly resolved, however the resolution is not adequate to separate the two different metallic coatings. A topic of interest is that the ratio of the density of the polymer core and metal coating is too high; this is likely caused by nanopores in the metallic coating, giving it an effective lower electron density.

Another result from this dataset is that it looks like the polymer core suffers from beam damage. Parts of the polymer core have a smaller density than the rest. By inspecting the 3D model it seems like these areas of smaller density develops around preexisting holes in the metal coating. The effect of radiation damage was also measured on a pure polystyrene sphere. Figure 2 shows how the sphere shrinks as function of exposure time. This gives valuable input to the design of future experiments, where similar sphere will be imaged, as it gives information on how much the spheres can be exposed to the direct beam before the size and shape of the particle changes too much for the reconstruction algorithm to work (which assumes no change of the imaged particle during the beam exposures).

When mixing the particles with the adhesive, the reconstruction algorithm failed to converge to a solution. This is likely caused by a too large sample volume. When mixing the particles in the adhesive, the final object size (particle surrounded by a glue layer) was approximately 8 – 10 µm in diameter.

The final set of measurements performed was raster scanning a metal coated particle in the beam, and measure the displacement of the scattering pattern, which is an indication of the wave front slopes. This lead to the possibility of reconstruction the local wave front, which turned out to be close to perfectly spherical, with a radius of curvature at the sample position equal the distance from the last beam defining slit of the optics and the sample position. This work has recently been submitted for publication to a journal.

Further analysis will be done to acquire quantitative information of the coating porosity, as well as comparing the imaged particles with particles of the same type, but with larger polymer core and metal coating thickness, to see how the length scale affects the particles.

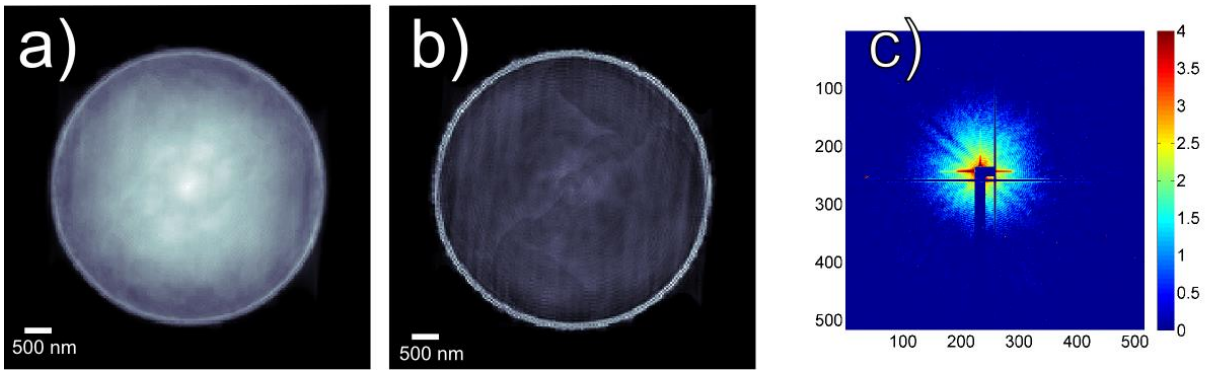

Figure 1: Experimental results from imaging a single particle. a) 2D projection of the reconstructed sphere from the scattering data. b) Cross section of the sphere. c) Scattering pattern created by the sphere before background has been subtracted. The colormap indicates the $\log_{10}$ value of the photon counts. a) and b) indicates the relative (projected) electron density of the material, with a light color indicating high electron density. One can see from both the reconstruction and the scattering image that the particle imaged is close to a perfect sphere. Some regions of the polymer core have lower density, which could suggest beam damage.

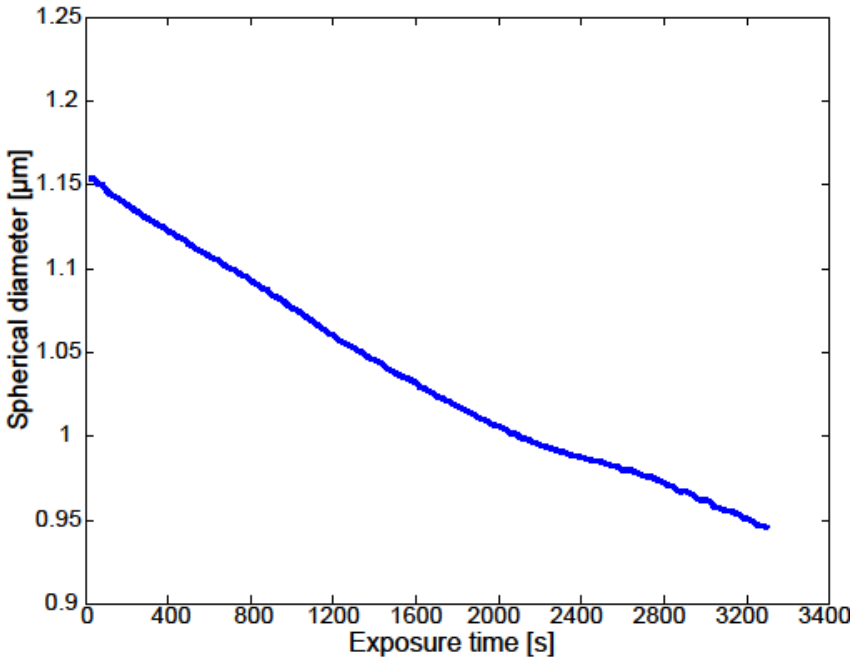

Figure 2: Diameter of pure polystyrene sphere as function of accumulated exposure time. The polymer is seen to suffer from radiation damage.

References

[1] J. Ugelstad, P. Mork, K. H. Kaggerud, T. Ellingsen, and A. Berge, "Swelling of oligomer-polymer particles. New methods of preparation," Advances in Colloid and Interface Science 13, 101-140 (1980).
[2] Kristiansen *et al.*, Characterisation of metal-coated polymer spheres and its use in anisotropic conductive adhesive. High Density Microsystem Design and Packaging and Component Failure Analysis, 2004. HDP'04. Proceeding of the Sixth IEEE CPMT Conference on, IEEE, 2004, pp. 259-263.
[3] Kristiansen *et al.*, Development and Characterisation of Micrometer Sized Polymer Particles with Extremely Narrow Size Distribution. 12th IEEE International Symposium on Advanced Packaging Materials: Processes, Properties and Interfaces (ISAPM 2007), 2007