

Beamtime 26-01-945 DUBBLE 30.05.2012 – 03.06.2012

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1. Background and objectives

In this beamtime the real-time growth of lead carboxylates $Pb(C_n)_2$ ($n > 10$) was studied by immersing a lead sample in a fitting solution and using XRD with the Mar CCD 165 camera on the DUBBLE beamline to monitor the growth of the coating. Two different deposition methods were tested: immersion and cyclic voltammetry. A second set of measurements involved the deposition of a droplet of a particular coating solution and monitoring the drying procedure as a function of time. Finally in a third set of measurements the coated samples were exposed to corrosive environments (including oak and acetic acid) whilst the corrosion process was monitored in-situ as a function of time making use again of the eCell and the Mar camera. For all experiments we made use of eCell, of which the prototype has been published by Dowsett and Adriaens [1].

2. Experiments

In the experimental hutch, an aluminum table (specifically made for the set-up) was put into place to position the eCell and the MarCCD detector (see Figure 1). An external shutter was placed in the beam. As the samples evolve with time, X-ray diffractograms need to be taken at regular intervals with strict timing, which means that the camera needed to be triggered. Also this had never done before at DUBBLE and it took the beamline scientists nearly two days to get the entire set-up going.

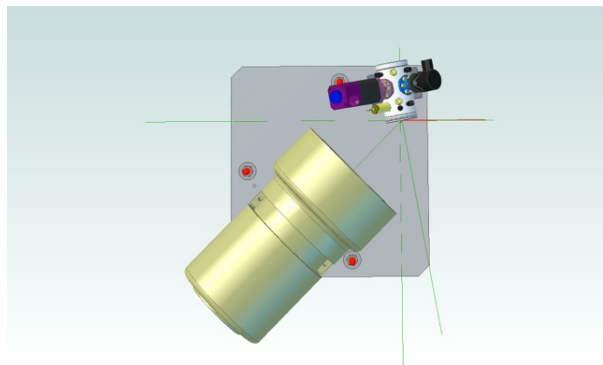


Figure 1

The following experiments were conducted (all data were recorded by XRD):

1. 1st set
 - Immersion of bare lead into a 0.05 M aqueous sodium decanoate solution
 - Cyclic voltammetry of bare lead in a 0.05 M aqueous sodium dodecanoate solution (2 different scan rates were used)
2. 2nd set
 - 0.05 M ethanolic solutions of capric and myristic acid and a 0.05 M aqueous sodium decanoate solution were used in three different experiments
3. 3rd set
 - Lead coated in a 0.05 M solution of myristic acid in ethanol was set to corrode in a aqueous NaCl environment (75 % relative humidity) containing a piece of oak

- A bare lead and an in a 0.05 M myristic acid ethanolic solution coated lead sample were set to corrode in an oak environment
- Linear sweep voltammetry experiments of a dodecanoate coated lead sample and a bare lead sample (as a reference) were performed in an corrosive acetic acid environment

Preliminary results show that:

- Ethanolic solutions produce a lead carboxylate coating upon drying in air. The ethanol evaporates and the carboxylic acid reacts with the lead ions on the sample.
- Oak corrodes lead also at a lower relative humidity than 75 %. The lead carboxylate coating in any case protects this kind of corrosion, i.e. it takes longer for a coated surface to corrode.
- Cyclic voltammetry shows to be a clear and fast deposition method for a lead dodecanoate coating.
- In the linear sweep voltammetry experiments the corrosion products only show to be present upon drying the sample.

[1] M. Dowsett, A. Adriaens, *Analytical Chemistry*, 78(10) (2006) 3360-3365.