Project name:	**Investigation of steel surfaces treated with phosphate-free conversion coating processes**

Travel/Event Report

11.02.2013 - 12.02.2013 *(Date of the report to be added)*

General information

Name of the rapporteur	**Name of the rapporteur's organisation**
Dr. Carsten Schellbach	Enthone GmbH
Type of research (nanotechnology/health care/chemistry etc.)	**Name of the research facility**
Nanotechnology/chemistry	Deutsches Elektronen-Synchrotron (DESY)
Date of the measurement, duration	**Location of the event**
11/02/13 – 12/02/13	Deutsches Elektronen-Synchrotron (DESY)
National Industrial Liaison Officer from rapporteur's country participating in the measurement	
Dr. Graham Appleby	

Description of the project

Research description (short summary as written in the application)
EnDURE™ PF is a non-phosphate conversion coating process for industrial metal treatment systems. This process is designed to remove soils, oils and oxides residues and convert the surface of various virgin steel and aluminum base metals for subsequent seal and topcoat processing. EnDURE™ PF conversion coating includes a cleaner, a pretreatment and a sealer process step. The pretreatment stage contains an intrinsic conducting polymer (ICP) dispersion. Due to the redox behavior of the ICP the iron surface is oxidized and a thin film of iron oxides is formed. By using the combination of pretreatment step and sealing step the corrosion resistance is increased. The reason for this behavior is the formation of different iron oxide compositions on the metal surface. The composition of the iron oxide films formed at the different stages and the influence of the pretreatment and the sealer on this formation process is still unclear. The aim of this project is the investigation of the formed iron oxide layers at the different stages of the process and their role in the corrosion mechanism. By using techniques for the characterization of thin layers information about the iron oxide composition and structures can be obtained and associated with other already measured properties.

Summary of activities (experiments performed, beam-time used, preliminary overview of results, next steps and other relevant information)

The experiments were performed at the beam line P08 from February 11th until February 12th. The samples were characterized with high-resolution x-ray diffraction under grazing incidence conditions. For this purpose standard test steel panels were treated with the anticorrosion process at Enthone before the experiments as well as on site during the measurements.
Figure 1 shows the XRD pattern measured at an untreated steel panel. In addition to the expected strong iron reflexes, several very small reflexes could be observed. These reflexes were caused most likely by the normal oxide layer on the steel surfaces. It was tried to identify the oxides with known patterns of several oxides from literature but in all cases one or more characteristical reflexes were absent. Because of this was an identification of the oxides on the steel surface not possible.

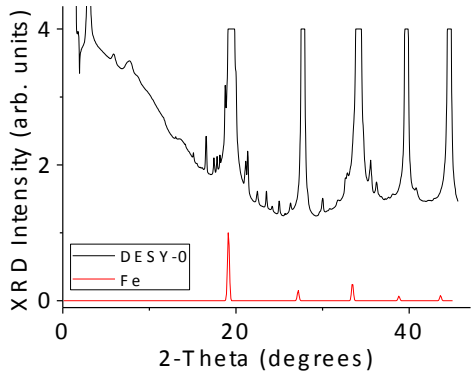

Figure 1: XRD pattern of an untreated steel panel. Also shown is the pattern of pure iron for comparison.

In the following experiments changes in the oxide layer during the anticorrosion treatment were investigated. In figure 2 XRD patterns of an untreated and a treated steel panel are shown. It could be seen that the patterns were identical and no changes caused by the treatment process could be observed.

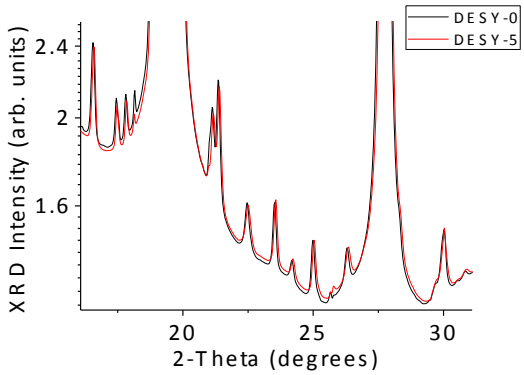

Figure 2: XRD patterns on an untreated and a treated steel panel.

In the following, several panels were treated in different ways and under different conditions to see if any differences were observable. In all cases the patterns were

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similar to the previously measured panels. Only very small differences could be observed (shown in figure 3) but these differences were too small as an evidence of newly formed oxide species on the surface.

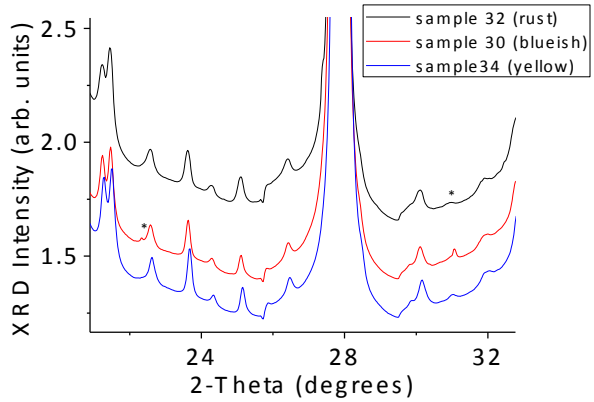

Figure 3: XRD patterns of three different samples. Marked with * are differences in the scattering curves of these three samples.

Furthermore at DESY polished steel samples were investigated because of the high roughness of the panel surface. But the XRD pattern of these samples (figure 4) showed also nearly differences compared to the unpolished samples. During these experiments it was not possible to determine if the polished panels were still too rough for this measurement method.

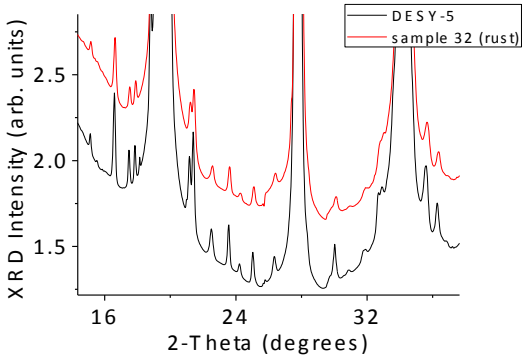

Figure 4: XRD patterns of a polished and an unpolished steel panel.

In summary, it could be seen in this project that small differences between the different patterns were observable but they were too small to attribute them to newly formed oxide species during the anticorrosion treatment. Most likely the investigated steel samples were even after polishing too rough for grazing incidence methods and the surface roughness disturbed the measurements. Another reason for the poor results could be textures and unknown crystallites on the steel surface caused by the production process of the panel. This poor measurability of the samples could also be the reason that the measured patterns do not match with the XRD data of the pure oxides.
During these experiments it could be shown that this method is not applicable for the investigation of the thin oxide layers on steel panel surfaces.

How would you describe cooperation and assistance from national contact points while preparing and carrying out the research at large scale facilities?
The cooperation with Graham Appleby our local contact at DESY was very good. He helped us with identifying the most suitable measurement method and organized the meetings with the groups at DESY as well as the whole beamline time. Also very helpful were his activities to prepare polished steel samples and the result discussion after the beamline time. Despite the poor results we had a great time and learned a lot of things about our process and the possibilities to analyse such surfaces. Many thanks for that.
Other personal remarks

Annexes

Annexes (list of annexes; meeting minutes, graphical illustrations, tables and other supplementary data)

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