ESRF	**Experiment title:** X-ray computed tomography (CT) for ex situ characterization of carbon-polymer composite bipolar plates for vanadium redox flow batteries	**Experiment number**: CH-6642
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Shifts: 9	**Local contact(s)**: Peter Cloetens	*Received at ESRF:*

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Report:

The beamtime aimed at studying the structural changes in bipolar plates (BPPs) due to electrochemical corrosion utilizing phase-contrast computed tomography (CT). For this purpose, a series of ex-situ corroded BPPs were measured at the beamline. These BPPs were composites of electrically conductive carbons (graphite, carbon black and/or carbon xerogels) in a polymeric matrix. ~ 0.5 mm wide and 0.5 mm thick free-standing plates were fixed to Huber pins using epoxy and mounted to the rotation stage. In this study, the pixel sizes used were 60 nm and 20 nm. Measurements were performed on corroded and non-corroded surfaces of the plates. For every tomography scan, 2000 projections were acquired with 0.3 s exposure time. The contrast in the reconstructed images is determined by phase contrast which is governed by the variation in electron density in the material. Even though the constituents of BPPs are carbon-based, slight variations in the density and morphology of the components, viz., graphite, carbon black, carbon xerogel, and polymer allowed enough contrast to identify the different regions and helped to gain information on the structure of BPPs. Figure 1 a and b show the reconstructed slices from composites made of graphite-polymer and graphite-carbon black-polymer, respectively. We observe that in the absence of carbon black, the graphite flakes (dark regions) align almost perfectly with the rolling direction during production. The polymer (gray) matrix binds the flakes together and some space is occupied by air pockets (bright regions). When the relative amount of carbon black to graphite increases, trapped air decreases significantly (Figure 1 b). The carbon black agglomorates (porous, light gray

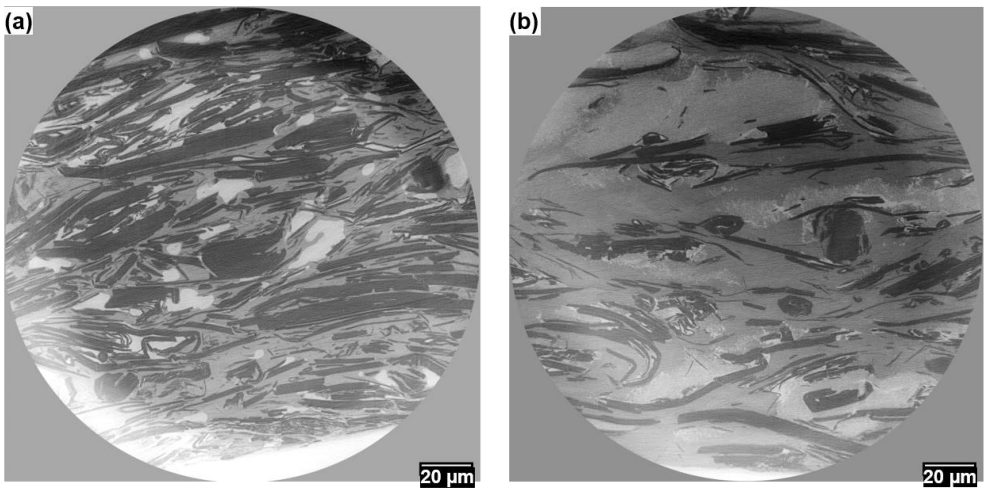

Figure 1: Reconstructed slices with 60 nm pixel size for non-corroded surface from samples containing (a) graphite-polymer and (b) graphite-carbon black-polymer.

regions) tend to provide conductivity pathways between graphite flakes. The latter two structural features enhance the electrical conductivity in the composite plates which was confirmed from the through-plane electrical conductivity measurements.

Figure 2 represents the corroded surfaces from the samples discussed above. The surface contains an open structure with the polymer binding the expanded graphitic flakes in Figure 2 a. The corroded surface also seems highly porous when the sample composition is changed as seen in Figure 2 b but the appearance of carbon black agglomerates (in comparison with polymer for the same weight percent) decreases. This could be related to the loss of carbon black as CO / CO_2 during corrosion. This decrease was later confirmed with scanning electron microscopy (SEM) investigations of the cross-section.

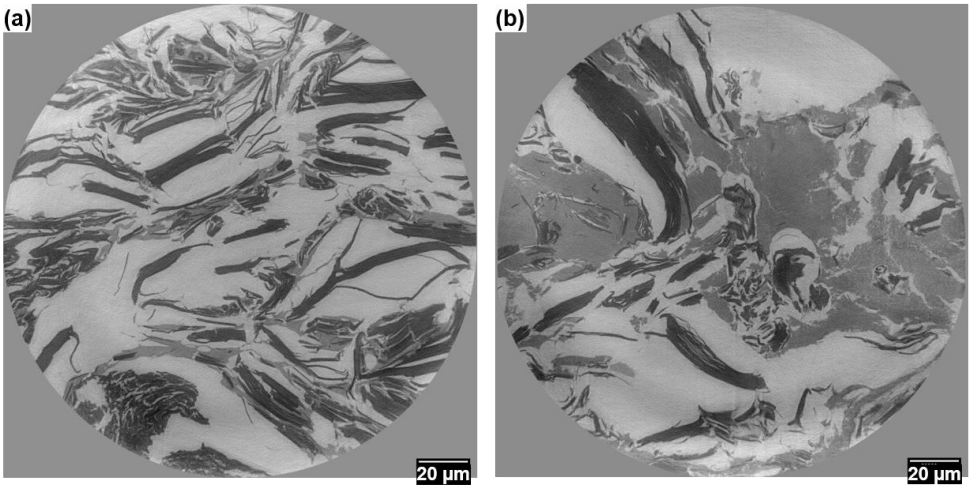

Figure 2: Reconstructed slices with 60 nm pixel size for corroded surface from samples containing (a) graphite-polymer and (b) graphite-carbon black-polymer.

Additional samples with a different fraction of carbon black to graphite or a different carbon filler, i.e., carbon xerogel were also measured. In the end, a total of nine sample surfaces with 20 and 60 nm pixel sizes were measured. The reconstructed volume for one sample displayed abnormal features (Figure 3), the origin of which could probably be due to the glue used for sample preparation.

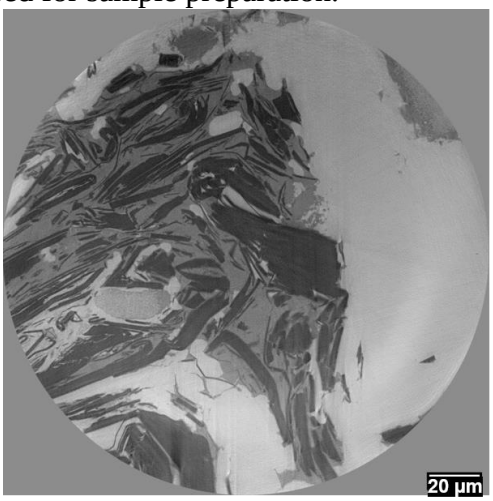

Figure 3: Reconstructed slice with 60 nm pixel size for non-corroded surface from the sample containing graphite-carbon xerogel-polymer.

In conclusion, the results from nano-tomography coupled with in-house analytics as well as synchrotron X-ray diffraction CT have helped in establishing relationships between the composition of BPPs and electrical conductivity as well as the corrosion process.