Image Analysis of Porosity in Søderberg Anodes

Stein Rørvik[1)], Kjell R. Kvam [1)], Harald Schreiner[2)], Morten Sørlie[2)] and Harald A. Øye[3)]

[1)] SINTEF Applied Chemistry, Sem Sælandsvei 12, N-7465 Trondheim, Norway
[2)] Elkem ASA Research, P.O.Box. 8040, Vågsbygd, N-4675 Kristiansand, Norway
[3)] Department of Inorganic Chemistry, Norwegian University of Science and Technology Sem Sælandsvei 12, N-7491 Trondheim, Norway

Introduction

A fully automatic method for image analysis of porosity in baked carbon anodes has been developed at Dept. of Inorganic Chemistry during the Expomat / Prosmat research program the last years. The method is based on optical microscopy, and is capable of analysing large sample areas (several cm^2). It provides a logarithmic size distribution of pores the in the range of 1 μm to 10 mm radius. In addition to this size distribution, a relative measure of pore surface area and connectivity is given.

The image analysis has been used to analyse porosity in laboratory baked Søderberg anodes, baked using the procedure described in [1]. The pore size distributions of anodes baked with different methods were compared (see **Figure 4** for an example), finding which anodes had a pore size distribution closest to the industrial anodes. The method is general and has been applied to both Søderberg anodes, prebaked anodes and cathodes.

Method description

Carbon anode samples are impregnated with a fluorescent epoxy[1] under vacuum. The sample surfaces are ground and polished upon curing of the epoxy. Pores filled with fluorescent epoxy light up brightly if viewed with ultraviolet light in a microscope. This reduces the error of creating false pores when cutting and polishing the samples.

The microscope used was a standard reflected light metallurgical microscope, equipped with a motorised XY-stage and auto-focus. The stage movement and focus is controlled by computer software. Digital images were acquired using a video camera and a frame-grabber board. The computer software used was the general image analysis Macintosh™ application NIH image, developed by Wayne Rasband at the National Institute of Health in the US. This software is in the public domain, and is available freely by anonymous FTP from zippy.nimh.nih.gov[2]. The source code has been customised by adding support for the microscope hardware and some extra image analysis procedures. The analysis is controlled by NIH image macros, and the result data is merged using Microsoft™ Excel macros and presented using Microsoft™ Excel templates (**Figure 1**).

The image analysis outputs the porosity values as *sum of the area pores with a specified radius covers, as a percentage of total analysed sample area.* The sum of all porosity values is thus equivalent to the total porosity. The overview image (Figure 1, top), which shows pores down to 50 μm, gives much useful visual information such as cracks and clustering of pores.

A more detailed description of the image analysis method has been published earlier in [2]. The basics are that adjacent frames are captured, the pores entirely inside each frame are measured while the pores cut by the image edges are not. These pores are saved and measured after four frames have been analysed and merged. This process continues recursively, allowing arbitrarily large pores to be measured. Up to 10 mm large pores are present in Søderberg anodes.

The advantage of this method is obvious: There is no upper limit to what pore size that can be measured. Mercury porosimetry stops around 50 μm, while the pore sizes that can be analysed by the present method are only limited by the movement of the stage, which is ± 50 mm.

The image analysis procedure is fully automated, it only requires an operator to place the sample on the microscope and start the procedure using the desired parameters. With sufficient storage space, images of a series of anodes can be acquired during daytime and be analysed in batch by the computer during the night. At low magnification (40x) 16x24 = 384 frames is required to cover a 50 mm sample. It takes about an hour to acquire these frames and 3 hours to analyse them. Usually, 12x16 = 192 frames are sufficient, covering a 28 x 28 mm area which is suitable for a 40 mm sample. See **Figure 2** for an overview of how a 40 mm sample is covered by 192 frames. Increased accuracy for the smallest pores can be achieved by overlapping measurements at high and low magnification (**Figure 3**).

[1] The epoxy is two-component and consist of Bisphenol-A-Diglycidyl-Ether and Tri-Ethylene-Tetramin with Sodium-Fluorescein added as fluorescent dye.

[2] Alternate sources for NIH image are from Library 9 of the MacApp forum on CompuServe, and on floppy disk from NTIS, 5285 Port Royal Rd.,Springfield, VA 22161, part number PB93-504868.

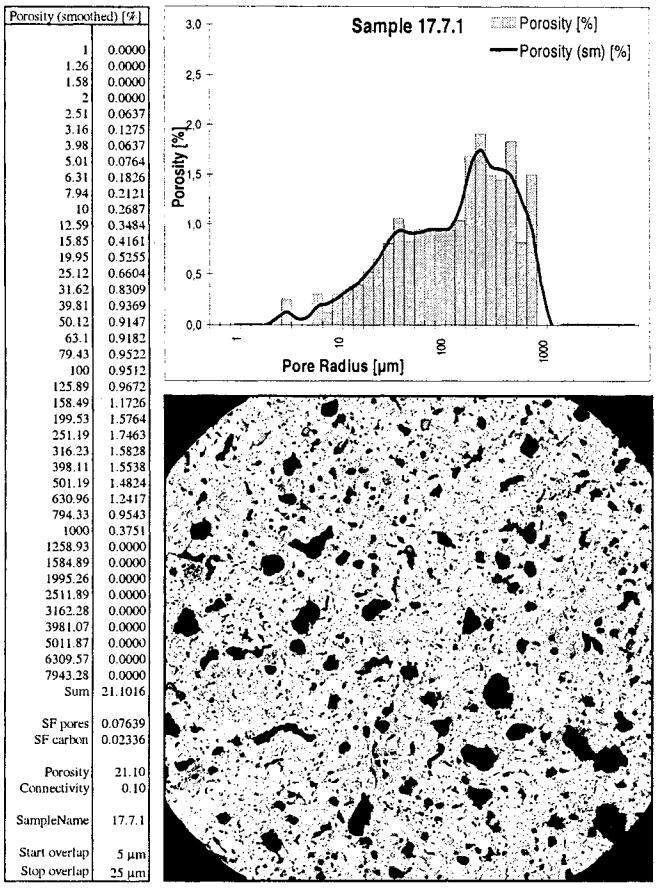

Figure 1: Standard diagram for pore size analysis of a single sample, created by a Microsoft™ Excel template.

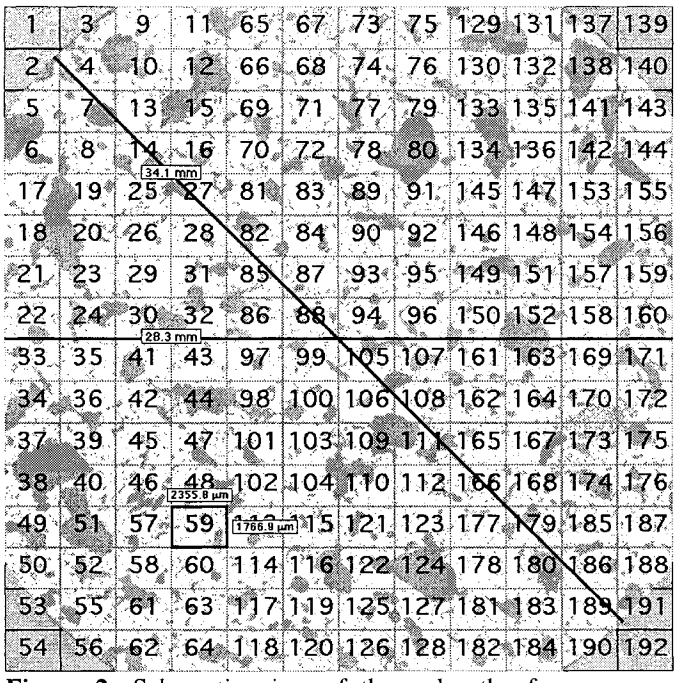

Figure 2: Schematic view of the order the frames are acquired with the sizes involved for a 40 mm sample.

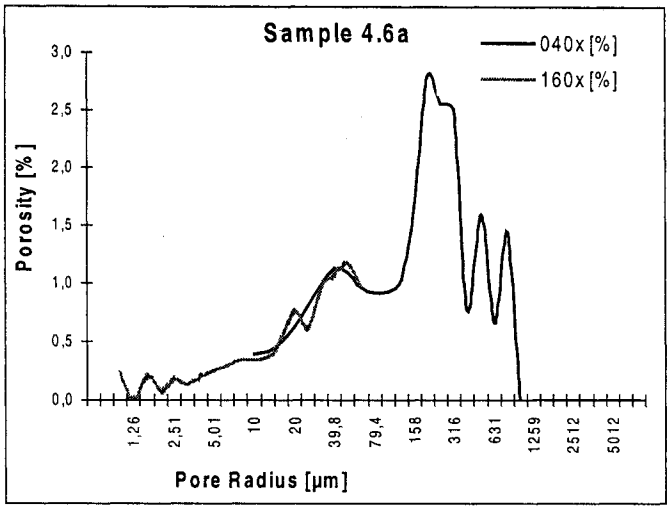

Figure 3: Overlap of pore size distributions acquired at low and high magnification. Overlap interval is from 7 to 70 µm.

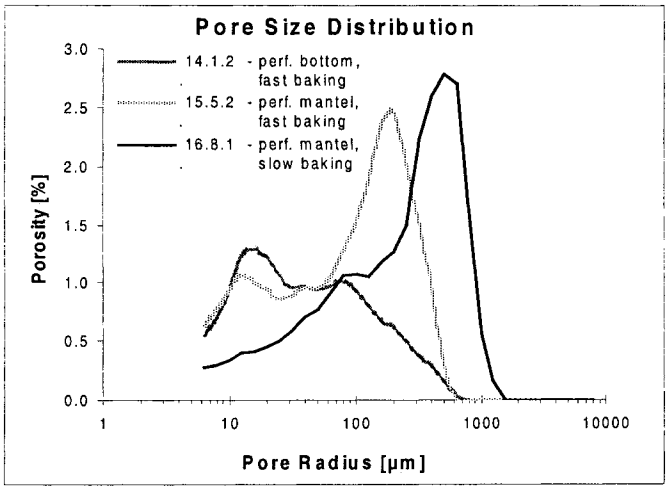

Figure 4: Comparison of pore size distributions of three laboratory Søderberg anodes baked differently [1].

Acknowledgements

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References

[1] Kjell Kvam, Harald Schreiner, Stein Rørvik, M. Sørlie, H. A. Øye: *Porosity Development in Søderberg Anodes – Laboratory Simulation.* Extended Abstract 24th Biennial Conf. on Carbon, Charleston (South Carolina, USA) American Carbon Society 1999.

[2] Stein Rørvik, Harald A. Øye: *A Method for Characterization of Anode Pore Structure by Image Analysis.* The Minerals, Metals and Materials Society (TMS); Light Metals Proceedings 1996, p. 561-568.