

## DIAGNOSING ANODE QUALITY PROBLEMS USING OPTICAL MACROSCOPY

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### Abstract

Anode quality assessment has become largely focused on laboratory tests undertaken on anode core samples. This is important, giving valuable insight to many anode quality problems and their potential causes. Anode "structural integrity" is, however, a critical anode property that is not directly assessed by these traditional anode core tests. Structural integrity is best described as how well a baked anode has been made, as evidenced by the visual appearance of the anode structure. Several methods have been used previously to visually assess anode structures, including optical image analysis. While these methods can highlight individual structural detail and features, they are not well suited to assessing overall anode structural integrity. This paper will outline a simple method for assessing baked anode structural integrity that uses USB macroscopy to evaluate anode quality and diagnose problems.

### Introduction

Most smelters have well established baked anode core testing programs based on standardised tests to facilitate the comparison and benchmarking of data between plants. While this has been a major step forward in facilitating anode quality improvement, and ignoring for the moment that further work is required to resolve outstanding issues regarding how baked anodes are cored (i.e. core sampling location on the anode, core sample length, and the protocol used to sample anode production for testing[1]), Structural Integrity (SI) is a critical anode property that is not always measured adequately using these laboratory tests. In this paper, baked anode Structural ("pertaining to structure - the way parts are put together") Integrity ("the condition of being free from defects or flaws") will be defined as how well the baked anode has been formulated and fabricated as reflected in the visual appearance of the cut surface of an anode core.

Optical microscopy has been used previously to examine anode structural features, however these studies have focused on using relatively high magnifications and image analysis to study anode samples that have been impregnated with mounting resins and highly polished[2,3]. Porosity in anodes can be quantified in detail using these methods, but the sample preparation, image acquisition, and image analysis times can extend into several hours, the sample preparation equipment required is extensive, and the microscopes used are quite expensive. In the current work, the objective was to develop a rapid, low cost method of assessing baked anode Structural Integrity suitable for use by plant Process Engineers. Automated image analysis was tested in this application, however the need to examine all of the sample cut surface (to avoid bias from butt particles in the cut surface), and the requirement to assess a range of anode structural features concurrently to make up a composite "image", meant that this was not successful. In effect, the human eye assisted by a simple microscope and the brain were found to be the most practical and

efficient tools for assessing baked anode SI. Further work is required to enable the use of image analysis to quantify the SI of baked anodes using this method.

### Method

A simple optical macroscopy method of assessing baked anode Structural Integrity and diagnosing anode quality problems was developed using a USB microscope and flexible light source. The USB microscope is powered from a laptop computer which is also used to record images of the baked anode core slices using software that is normally supplied with the microscopes. Two different alternatives of the microscope arrangement are shown in Figures 1 and 2. In both cases, the light source is directed at an oblique angle to the anode core sample surface to shade the surface porosity. A similar arrangement using a conventional low magnification binocular microscope has been used to study internal gaseous attack of anode butts[4]. The current arrangement is a much simpler, lower cost, and convenient way to view and record images of the baked anode core slices that that used previously. The details of the USB microscope used is not critical as long as it has reasonable optics (as the magnification required is only x10, this is not a difficult requirement to meet), can be moved or adjusted vertically to focus on the sample, does not have built in vertical illumination that cannot be switched off, and incorporates a digital camera to record the images.

The slice cut from a baked anode core for Air Permeability and Thermal Conductivity testing was found to be well suited for optical macroscopy and is readily available with a minimum of additional preparation. If such slices are prepared for thermal conductivity testing using grinding, no further preparation is required for low magnification optical macroscopy. If only diamond saw cutting is used, the core slices may require further polishing with emery paper to remove saw cut marks.



Figure 1. Baked anode optical macroscopy arrangement using a USB microscope (Traveler model number SU 1071). Note the oblique lighting to shadow surface porosity. As long as it provides

sufficient illumination and the angle to the slice surface is about 30°, the details of the light source is not critical, e.g. relatively small LED torches work well.

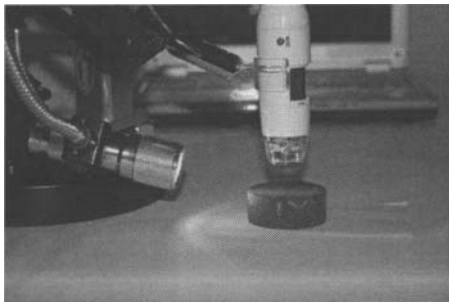


Figure 2. Alternative baked anode optical macroscopy arrangement (Greenpoint USB Digital Microscope)

Using an arrangement such as those shown in Figures 1 or 2, the porosity and defects visible on the surface of the baked anode core slices can be assessed and recorded as digital images. The details of these surface features give insight to the anode formulation and fabrication issues that caused them, and hence help to diagnose quality problems and identify opportunities to improve the SI, overall quality, and performance of baked anodes.

### Results and Discussion

The use of optical macroscopy to diagnose baked anode quality problems will be demonstrated by analysing a series of anode macrographs that show various defects in anodes from several different plants. Experience has shown that there are a number of factors that dominate the characteristics of anode macrostructures, including:

- Green anode forming technology, i.e. vibroformer versus hydraulic press
- Vibroforming technology – vacuum versus no vacuum
- Aggregate particle size distribution, especially butts top size
- Amount of pitch added

The visual appearance of the macrostructures is, then dependent on Paste Plant design and normal operating parameters, as well as abnormal conditions such as poor mixing, over-compaction in the vibroformers, poor control of aggregate particle size, poor pitch level control, poor control of anode forming temperature, and too fast anode baking heat-up rate.

Optical macroscopy gives a quick insight into anode Structural Integrity, with the defects evident in the anode structures helping to narrow down the root cause(s) of any SI problems. This analysis can be combined with normal anode core test results to better diagnose anode quality issues, e.g.:

- Cracking in the macrostructure will increase Electrical Resistivity (ELR), decrease Flexural Strength (FS), and may increase Air Permeability (AP).
- “Compaction faults” will increase ER and decrease FS, usually to a lesser degree than cracking, but unless severe, do not have a strong influence on AP as minor compaction faults are closed porosity.
- Extensive macroporosity increases AP but does not normally have a significant effect on ELR and FS.

The anode macrostructure shown in Figure 3 below has a high degree of Structural Integrity indicating that the green anode was well formulated and fabricated. There are no significant defects present and the structure is relatively consistent. Given this high degree of SI, the electrical resistivity is unexpectedly high at 59 mΩ.mm. Based on these data and observations, this high ELR is quite likely to be due to a relatively low baking temperature and not be related to green anode properties.

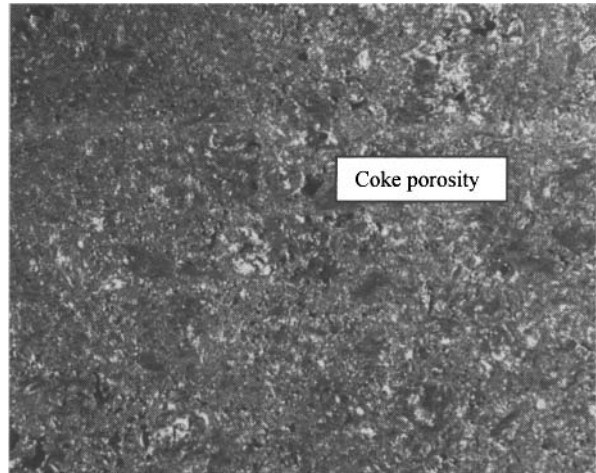


Figure 3. Good quality vibroformed baked anode macrostructure with no significant defects, faults, or cracking, i.e. a high degree of Structural Integrity. Isolated larger pores are from coke particle porosity that was not filled with coke fines/pitch during mixing/forming and do not significantly affect anode quality. Air Permeability (AP) on this sample 0.3 nPm, Electrical Resistivity (ELR) 59 mΩ.mm, and Flexural Strength (FS) 14 MPa (Macrograph x10).

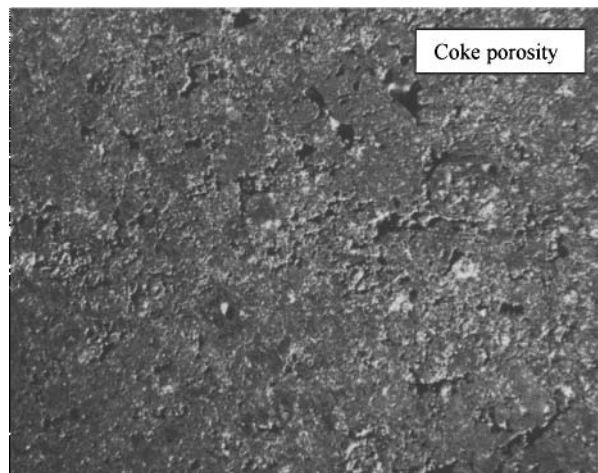


Figure 4. Poor quality vibroformed baked anode macrostructure with extensive small compaction faults and disruption of the binder/fines matrix (AP 0.6 nPm, ELR 57 mΩ.mm, FS 9.5 MPa, Macrograph x10)

The anode macrostructure shown in Figure 4 has low SI due to compaction faults. Experience has shown that this type of structure is most likely due to poor quality mixing (e.g. from excessive mixer wear or too cold mixing temperatures) or cold paste during forming. Anodes with these structures can generate dust as they are consumed in the cells.

A high prevalence of anodes with very low SI macrostructures such as shown in Figure 5 can lead to major anode performance problems with dusting and soft butts. These structures can be caused by the same problems as for Figure 4 when mixing/forming issues are more severe. The anode core data are poor which could be attributed to anode cracking without the macrostructural analysis, however the SI analysis ensures that problem solving can focus on the problem of poor compaction.

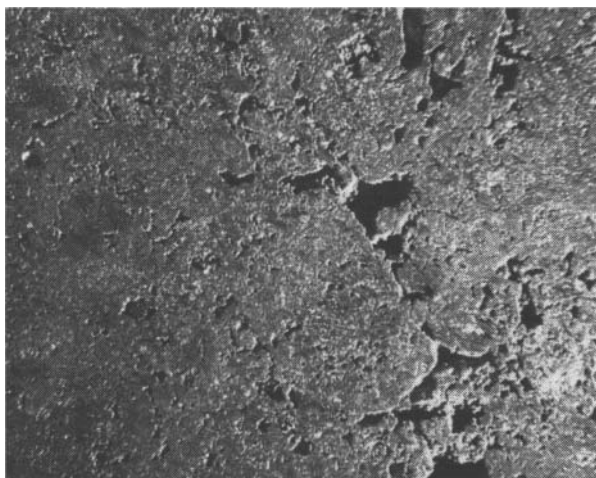


Figure 5. Very poor quality baked anode macrostructure with extensive, large compaction faults and disruption of the binder/fines matrix (AP 2.0 nPm, ELR 62 m $\Omega$ .mm, FS 8 MPa, Macrograph x10)

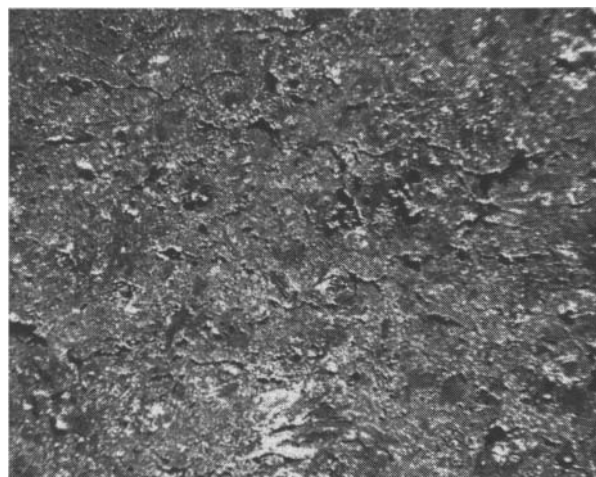


Figure 6. Poor quality vibroformed baked anode macrostructure with extensive cracking of similar orientation. (AP 1.7 nPm, ELR 62 m $\Omega$ .mm, FS 9 MPa, Macrograph x10)

The macrostructure shown in Figure 6 has poor SI due to a prevalence of cracking. There is an orientation to this cracking – this is a characteristic of cracking due to overcompaction during forming. Such overcompaction may be due to too high coverweight pressure, too long vibration time, or too cold paste during compaction. Note that the core test results are similar to those for the anode corresponding to the macrostructure in Figure 5, but optical macroscopy shows that quite different issues have caused the poor SI evident in Figures 5 & 6.

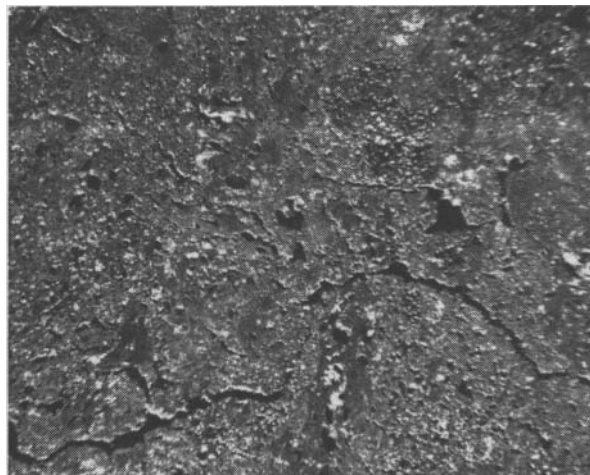


Figure 7. Poor quality vibroformed baked anode macrostructure with a large crack, several smaller cracks, and compaction faults. (AP 1.9 nPm, ELR 62 m $\Omega$ .mm, FS 5 MPa, Macrograph x10)

The macrostructure shown in Figure 7 contains a large crack that has propagated around a butt particle during the forming of the green anode. This suggests that the cause of the cracking is overcompaction and not other potential causes such as too fast heat up rates during baking.

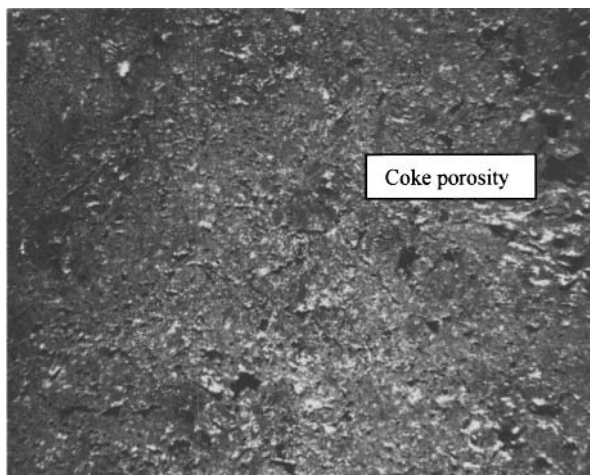


Figure 8. Poor quality vibroformed baked anode macrostructure with a network of relatively coarse macropores and small compaction faults. (AP 0.7 nPm, ELR 52 m $\Omega$ .mm, FS 11 MPa, Macrograph x10)

The macrostructure shown in Figure 8 appears quite consistent but the SI is reduced by a network of coarser porosity and short cracks not evident in a high SI anode structure such as shown in Figure 3. This is characteristic of a slightly over-pitched anode. Such a structure does not normally have a significant effect on anode performance, whereas the macrostructure shown in Figure 9 is characteristic of a greater degree of overpitching – to the point where anode performance is likely to be affected. The SI of the macrostructure in Figure 9 has been degraded by the extensive network of coarser pores that have tended to form “rings” as the pitch volatiles escape from around the coke and butt particles in the green anode structure during baking. The binder/fines matrix has been disrupted by this volatile release porosity; this is likely to lead to anode dusting problems in the cells. This analysis of the anode macrostructures indicates the reason for the relatively high AP, and hence the way to address the anode quality issue.

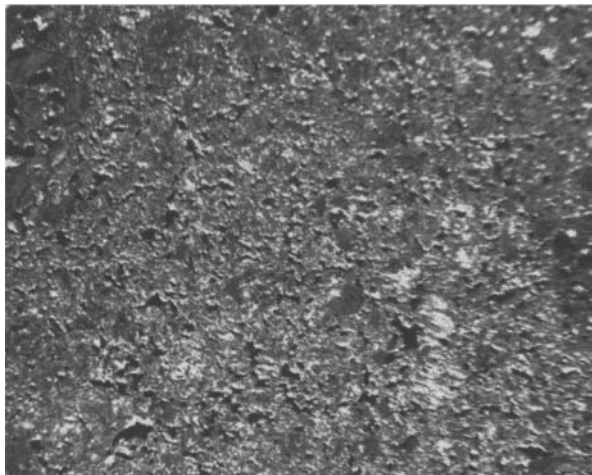


Figure 9. Poor quality vibroformed baked anode macrostructure with extensive coarse macroporosity and small compaction faults. The coarse porosity has formed into a network of rings. (AP 1.7 nPm, ELR 56 mΩ.mm, FS 11 MPa, Macrograph x10)

Optical macroscopy of these anode structures has shown examples of how the analysis of baked anode Structural Integrity can provide insight into the potential causes of anode quality issues. When combined with conventional baked anode core test results, SI observations help to resolve the cause of anode quality defects when more than one option could be deduced from the core data. This gives the ability to respond to anode quality issues faster and maintain better anode performance.

The major characteristics of anode macrostructures such as compaction faults, macrocracking, volatile release porosity, etc are present in anodes from most plants to varying degrees. The detail of these defects and overall texture of macrostructures are, however dependent on individual plant attributes, and hence this assessment technique needs to be “calibrated” to the SI optical macroscopy images from different plants.

USB Optical macroscopy has also been found useful for investigating other Carbon Plant issues such as aggregate problems related to poor packing due to particle shape, and examining green anode cores to show, for example, the relationship between compaction faults and large butt particles.

### Conclusions

1. Low magnification optical macroscopy can be used to rapidly assess the structural integrity of anode samples.
2. Simple, low cost USB microscopes with flexible light sources are sufficient to assess and record anode structural integrity.
3. Structural integrity assessment, either alone or combined with conventional anode core test data, is a powerful means of getting valuable insight into many anode quality problems and their potential causes, thereby allowing improved anode performance.
4. A calibration process is required identify the SI “features” in baked anode optical macroscopy images from different plants.

### References

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