



# Characterisation of an industrial Søderberg electrode

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**It does not matter how slowly you go as long as  
you do not stop**

**~Confucius~**

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# Abstract

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Søderberg electrode management (together with the initial design of the electrode system) is commonly considered as one of the most important aspects of profitable and safe submerged arc furnace operation, with some even considering it as the most important aspect. Notwithstanding the importance of electrode management, very little research on Søderberg electrodes has been published in the journal peer-reviewed public domain.

The overall main aim of this study was to obtain, sample and characterise an industrial Søderberg electrode. The case study electrode (that was obtained from a South African ferrochrome smelter) was cut off approximately 0.5 m below the contact shoes of a submerged arc furnace. The operational history (slipping rate, current, paste levels, etc.) proved that the case study electrode was a representative specimen from a stable ferrochrome submerged arc furnace.

Fit-for-purpose sampling procedures and apparatus were developed to obtain samples that were manageable in size and representative of the full-size industrial Søderberg electrode, which was used for the characterisation experiments. These experiments included electrical resistivity (measured with a specially designed two-probe system), degree of graphitisation (calculated from X-ray diffraction), bulk density determinations (measured with a standard American Society for Testing and Materials method and gas pycnometry), porosity determination (with computerised tomography and associated image processing, as well as scanning electron microscopy), compressive breaking strength (with a breaking strength instrument), and thermal shock (with a method developed during this study).

The characterisation results indicated no significant electrical resistivity, degree of graphitisation, and bulk density changes from 0.7 to 2.7 m on the non-delta side (outward-facing). In contrast, these characteristics changed relatively significantly on the delta side (inward-facing) of the electrode. The delta-facing tip of the electrode had the lowest resistivity, as well as the highest DOG and bulk density.

No significant difference in porosity as a function of length below the contact shoes was observed; however, slight increases occurred near the perimeters. It was postulated that oxidation of carbon resulted in increased pore volumes near the electrode perimeter. This was confirmed by the results obtained from the scanning electron microscopy data, which showed that the pore sizes were significantly larger on the electrode perimeter than on the inner part of the electrode. No significant difference in compressive breaking strength was observed over the electrode area investigated. The thermal shock results showed a very slight decrease in strength at the non-delta side of the electrode compared to the delta side. This characterisation data can be used to verify whether Söderberg electrode models accurately predict important electrode characteristics.

**Keywords:** Söderberg electrodes; submerged arc furnace (SAF); ferroalloy production; ferrochrome; electrical resistivity; degree of graphitisation; bulk density; porosity; compressive breaking strength; thermal shock

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## List of Abbreviations

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|                                      |           |                                 |            |
|--------------------------------------|-----------|---------------------------------|------------|
| 3-dimensional                        | 3D        | Maximum slipping rate           | $S_{\max}$ |
| Area                                 | A         | Afternoon shift                 | A/S        |
| Baking power                         | P         | Morning shift                   | M/S        |
| Bulk density                         | $\rho_D$  | Night shift                     | N/S        |
| Capacitance                          | C         | Polyvinyl chloride              | PVC        |
| Computerized tomography              | CT        | Pressure 1                      | $P_1$      |
| Current                              | I         | Pressure 2                      | $P_2$      |
| Degree of graphitisation             | DOG       | Resistance                      | R          |
| Density of air                       | $\rho_L$  | Resistivity                     | $\rho$     |
| Density of water                     | $\rho_o$  | Sample volume                   | $V_s$      |
| Electrodes 1                         | E1        | Saturated weight                | S          |
| Electrodes 2                         | E2        | Scanning electron microscopy    | SEM        |
| Electrodes 3                         | E3        | Slipping index                  | K          |
| Empty sample chamber volume          | $V_c$     | Submerged Arc Furnaces          | SAF        |
| Energy dispersive x-ray spectroscopy | EDS       | Temperature 1                   | $T_1$      |
| Ferrochrome                          | FeCr      | Temperature 2                   | $T_2$      |
| Furnace 1                            | F1        | Voltage                         | V          |
| Furnace 3                            | F3        | Volume 1                        | $V_1$      |
| Hight                                | H         | Volume 2                        | $V_2$      |
| Inductance                           | L         | Volume of the reference chamber | $V_r$      |
| Interlayer spacing                   | $d_{002}$ | Weight                          | D          |
| Interquartile range                  | IQR       | X-Ray Diffraction               | XRD        |

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# Chapter 1

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## Background, motivation and objectives

In this chapter, a brief background of the Söderberg electrode system will be given; this includes a brief history of the electrode, basics in its construction and usage, possible dangers, its management (Section 1-1), as well as current gaps in literature. In Section 1-2, the general aim and specific objectives of the study will be presented.

### 1-1 Background and motivation

In the early 1900s, an engineer, Carl Wilhelm Söderberg, a photo of whom is presented in Figure 1-1, from Christiania (currently known as Oslo), Norway, designed a new type of electrode to be used in pyro-metallurgical smelting processes, i.e. the self-backing continuous electrode. This type of electrode would later become known as the Söderberg electrode (Richards, 1920).



**Figure 1.1** Carl Wilhelm Söderberg, 1876-1955 (Larsen, 2012).

After building his first electrode in 1909, he experienced many difficulties in the development of his idea. In 1915, he actively started pushing the company (Elkem), where he was an engineer, to start developing this electrode. In November 1918, after considerable development work, they started and operated a 400 mm electrode in a ferromanganese furnace without furnace disturbances or electrode fractures (Richards, 1920). Since then, the Söderberg electrode system has been applied widely and is still used extensively today. Even though the process of making Söderberg electrodes is virtually the same as 100 years ago, the maximum current load (150 kA) and electrode size (2 000 mm) have increased considerably (Innvær *et al.*, 1995; Larsen, 2012).

To make a Söderberg electrode, tubular steel casings of the appropriate diameter are joined/welded together (Bruff *et al.*, 1985; Habashi, 2004; Nelson & Prins, 2004; Visser, 2006). Raw electrode paste in the form of large cylindrical blocks and/or smaller cubical blocks or briquettes is placed into the casing on a semi-continuous basis to form the electrode. The electrode paste consists of a mixture of coke (derived from coal heated in the absence of air to remove volatile components leaving mostly carbon (Stansberry *et al.*, 1999)) and/or other solid fillers, and coal tar pitch as a binder. At room temperature, the electrode paste is a solid material that starts to soften at approximately 60 °C, and melts into a liquid at around 80 °C (Innvær, 1992). At temperatures between 450 and 475 °C, this liquid paste mixture hardens to a solid carbonaceous material (Shoko *et al.*, 2013). The temperature at which the transition from liquid to solid occurs is commonly referred to as the baking isotherm (Shoko *et al.*, 2013). The aforementioned temperature is typically achieved just above the contact shoes, i.e. the copper or copper-alloy components that transfer electricity into the electrode casing (Beukes *et al.*, 2013; Innvær, 1992). The baked portion of the electrode is lengthened by ‘slipping’, which describes the controlled upward movement of the contact shoes on the electrode casing. Slipping length depends on the electrode condition and can vary from a few millimetres per eight-hour working shift to over a meter if an electrode break occurred. The baking procedure and duration thereof depend on the length of the slip. From the baking isotherm to the tip of the electrode baking continues, with the carbonaceous material being transformed to graphite – this is typically only achieved close to the tip of the electrode where temperatures could be 2 500 to 3 000 °C (Jonsson & Ingason, 1998; Matsumoto, 1985).

Although many types of smelters exist, wherein Söderberg electrodes are used, this study focused on ferroalloy smelting. This industry typically uses a three-electrode system, with the three electrodes submerged deep in the furnace burden (material to be smelted), hence the term submerged arc furnace (SAF). The three electrodes are typically arranged in a triangle, where the current being passed through them arcs together and forms what is commonly known as the delta (Kelly, 1958; King, 1975).

In SAFs in which Söderberg electrodes are used, very harsh conditions lead to severe mechanical and thermal stresses. These stresses lead to unavoidable continuous wear of the electrode tip, and may cause electrode breaks that can vary from inconsequential small/short hard electrode breaks, to disastrous soft/green breaks (exposing liquid electrode paste) (Beukes *et al.*, 2013; Innvær, 1992; Innvær *et al.*, 1984; Nelson & Prins, 2004; Shoko *et al.*, 2013). Furnace shutdowns (stoppage) that cause a temperature differential between the outside and the inside of the electrode enhance thermal stresses, which can contribute to electrode breaks. Broken electrodes cause unstable furnace operations that negatively impact productivity and/or personnel occupational safety (e.g. can cause

explosions) (Bailey, 1950). It is therefore essential that electrodes be designed and operated in such a way that they can withstand unavoidable mechanical and thermal stresses during normal operation, and resist breakages due to unforeseen/planned shutdowns.

Søderberg electrode management (together with the initial design) is commonly considered one of the most important aspects of profitable and safe SAF operation, with some authors even considering it as the most important aspect (Arnesen *et al.*, 1979). This term, i.e. Søderberg electrode management, entails many aspects that have to be monitored and controlled continuously or on a shift basis (e.g. during SAF shutdown) (Ray *et al.*, 2007; Sem, 1954).

Notwithstanding the importance of electrode management, relatively little research has been published in the peer-reviewed public domain. As an example, according to Scopus (the scientific search engine with the broadest coverage), in the period 2013 to 2020, only six papers published in the peer-reviewed scientific journal domain specifically considered Søderberg electrodes (Beukes *et al.*, 2013; Friedrich *et al.*, 2017; Karalis *et al.*, 2016; Shoko *et al.*, 2013; Shoko *et al.*, 2015; White *et al.*, 2020). Several conference proceeding papers have also been published during this time, e.g. (Jordan & Hockaday, 2013; Larsen, 2012; Larsen *et al.*, 2013; Skjeldestad *et al.*, 2015). These conference proceedings papers also gave valuable topic-specific insights. However, many such contributions were made principally to demonstrate the capability of commercial companies to serve their clients instead of divulging all relevant information as is common in academic journal papers. In the more than 100 years that Søderberg electrodes existed no detailed characterising of an operational electrode was published in the peer reviewed public domain. To partially improve the knowledge base on Søderberg electrodes available in the peer-reviewed public domain, this study focused on characterising an industrial Søderberg electrode.

## 1-2 Aims and objectives

The general aim of this study was to characterise an industrial Søderberg electrode. In order to achieve this general aim, the specific objectives were:

- (i) Obtain a full-size Søderberg electrode from a ferroalloy producer to serve as a case study electrode, together with a reasonable period of operational history of this electrode, prior to it being removed from the SAF. Obtaining sample material would usually not be considered as an objective for a PhD study, since such materials are usually readily available. However, finding and convincing a ferroalloy producer to donate a full-size Søderberg electrode and supplying the operational history thereof for research is not easy.

- (ii) Sampling of the case study S derberg electrode, to yield representative samples that can be handled conveniently in the laboratory. Due to the size of typical industrial S derberg electrodes, sampling will be challenging.
- (iii) To characterise the case study S derberg electrode in terms of electrical resistivity, degree of graphitisation, bulk density, porosity, compressive breaking strength and thermal shock resistance. Changes in these parameters will be investigated as a function of electrode length below the contact shoes, as well as from the delta to the non-delta side.
- (iv) Contextualise the above-mentioned characterisation results within the context of previous findings and/or modelling studies.
- (v) Indicate topics that should receive research attention to advance knowledge in this under-investigated study field.

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# Chapter 2

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## Literature survey

In this chapter, the candidate will discuss relevant literature concerning Söderberg electrodes. Firstly, the history (Section 2-1) and basic principles (Section 2-2) of the Söderberg electrode will be discussed. Next, the applications where Söderberg electrodes are used (Section 2-3), and the components' design (Sections 2-4, 2-5 and 2-6) will be considered. Lastly, electrode management (Section 2-7), important characteristics (Section 2-8) and electrode breaks (Section 2-9) will be discussed.

### 2-1 Historical perspective

#### 2-1-1 *Carl Wilhelm Söderberg*

Carl Wilhelm Söderberg (as shown in Figure 1.1) was born 30 August 1876 in Gothenburg, Sweden. When he was one year old, he moved to Christiania (Christiania changed its name to Oslo in 1925) with his parents. After finishing secondary school (high school), he moved to Hanover, Germany, to study electricity and electrochemistry at the Technical University. In 1910, he moved back to Norway and worked for Hiorth Steel Factory in Jøssingfjord for two years. In 1912, he started working for Norway's largest chemical and metallurgical enterprise, Det Norske Aktieselskap for Elektrokemisk Industri (Elektrokemisk), or as it is known today: ELKEM. It was here, in 1918, that he succeeded in building his first working self-baking continuous electrode (Richards, 1920). This type of electrode would later be known as the Söderberg electrode (Toromanoff & Habashi, 1989).

As a side note, the name of the inventor (Söderberg) and that of the electrode (Søderberg) are spelt differently. It is common practice to use the "Søderberg" spelling for the electrode, and the candidate will continue to do so. When referring to the person, "Söderberg" will be used.

Söderberg continued his work on the electrode until he retired in 1931 at the age of 61. As he was not a very sociable man, he spent the rest of his life in seclusion at his home in Nordby, Norway. He died on 7 March 1955, after a short illness, at the age of 79 (Toromanoff & Habashi, 1989).

#### 2-1-2 *Idea, development and building of the Søderberg electrode*

During his time working at Hiorth, Söderberg attempted to employ an electrode made of unprepared coal but was unsuccessful in getting it to arc and bake continuously. He would not give up on the idea

and took it with him when he started working for Elektrokemisk. As World War 1 broke out, it became more challenging to obtain pre-baked electrodes. It was then that Söderberg's idea was further researched and put to the test (Toromanoff & Habashi, 1989). In November 1918, a 400 mm electrode was successfully operated on a ferromanganese furnace (Richards, 1920). Also, in November 1918, a 600 mm Söderberg electrode was installed by the A/S.Fiskaa Verk per Kristianssand S. in Norway for a ferrosilicon furnace (Söderberg & Sem, 1922). Between 1919 and 1921, commercial installations of Söderberg electrodes took place in Norway, Sweden, South Africa, Italy and Germany (with a 950 mm electrode being the largest at the time) (Toromanoff & Habashi, 1989).

The photos in Figure 2.1 shows some original Söderberg electrode systems from around 1920. Figure 2.1a is a more general view of the entire electrode with the chamber above where the electrode was fed with its raw components. Figure 2.1b shows a close-up view of the electrode with its accompanying components.

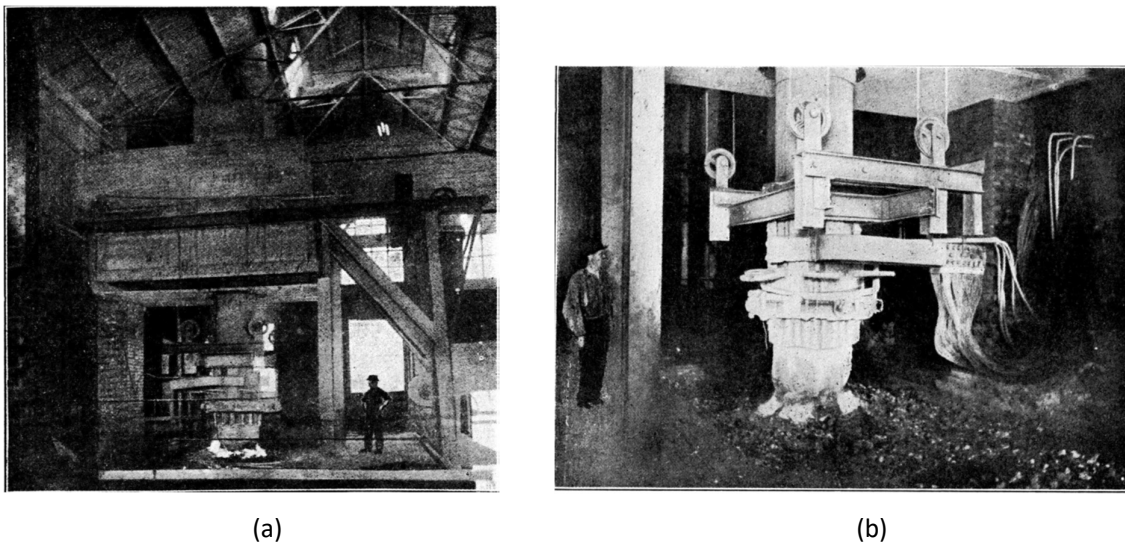


**Figure 2.1** (a) A general view of the electrode and the filling chamber above. (b) A close-up view of the electrode and its components (Richards, 1920).

In 1920, preliminary experiments were started to determine whether this technology could be adapted for the aluminium industry. In 1923, commercial small-scale installations were adopted in Germany, France, Switzerland and the United States (Toromanoff & Habashi, 1989).

Today, after more than 100 years, the Söderberg electrode system is still the most commonly used electrode in submerged-arc furnaces for ferroalloys, platinum smelting and slag cleaning operations. Over the years, the sizes of both furnaces and electrodes have increased significantly, and electrodes with diameters up to 2 000 mm are in use today (Larsen, 2012). The management of these large

electrodes is more demanding than the original smaller ones, and it puts a great deal of strain on their operators.

## 2-2 Basic principles of formation and use of Søderberg electrodes

### 2-2-1 *Purpose of electrodes*

The primary purpose of electrodes is to conduct electrical energy into the heart of a submerged-arc furnace (SAF). This electrical energy leaves the electrode's tip and is transformed into heat for a chemical reaction (a reduction reaction) (Andreae, 1950b; Fitt & Aitchison, 1993; Innvær *et al.*, 1986). The chemical reaction is usually between metal ores, reductants and fluxes, and produces metal. Essential properties that all electrodes need include good electrical conductivity, mechanical strength and thermal conductivity (Chung, 2002; Karagoz *et al.*, 1994) as per requirements for the specific furnace.

### 2-2-2 *Types of electrodes*

There are mainly two types of electrodes used in SAFs. They are pre-baked electrodes and Søderberg electrodes (Habashi, 2004; Tucker, 1981). A combination design, with a pre-baked core and an outer Søderberg section, has relatively recently also been implemented. As the name suggests, pre-baked electrodes are made in a separate manufacturing facility than the SAF it is used in. The Søderberg electrode is baked *in situ*, meaning it is baked in the furnace while it is running.

### 2-2-3 *Manufacture*

The basic manufacturing of the two electrode types (i.e. pre-baked and Søderberg) starts pretty much the same. The only difference is where this takes place and the finishing of the electrodes. Both start with the same raw materials, i.e. a mixture of a binder (usually coal-tar pitch) and a carbonaceous material/aggregate (usually calcined anthracite which is a high-grade coal and/or coke), termed electrode paste. Each manufacturer has their own requirements related to carbon content and impurities. In pre-baked electrodes, raw materials are pressed into a mould (that is larger than the end electrode) with a hydraulic press. This moulded paste is then baked at 1 000 °C and then graphitised between 2 500 and 3 000 °C (Sem, 1954; Victor, 1953). These graphitised cylinders are then lathed to the right dimensions, and threads are cut into both ends (male on one end and female on the other). Figure 2.2 shows the male threaded end of a pre-baked electrode. These threads are used to attach the new electrode to the one already installed in the furnace. All the electrode sections

need to be made to exact specifications. If the electrode's dimensions or the threads are not exactly correct, the electrode will either not fit in its housing or the threads will not catch.



**Figure 2.2** The male threaded end of a pre-baked electrode  
([https://www.rsrefractory.com/refractory\\_brick/graphite-electrode/](https://www.rsrefractory.com/refractory_brick/graphite-electrode/) accessed on 06/01/2021).

In Söderberg electrodes, the electrode paste comes in cylinders, blocks or briquettes (see Section 2-6 for more detailed information). These 'blocks' are loaded into a steel casing above the SAF where they start to melt into the finished dimensions. It is baked with help from the electrical current running through the electrode and heat from the SAF (Richards, 1920). When the electrode gets too short, a new casing segment is welded to the top of the column, and new paste 'blocks' are added (Fitt & Aitchison, 1993; Hodson, 1923; Innvær *et al.*, 1986; Richards, 1920; Toromanoff & Habashi, 1989; Vatland & Bruff, 1995).

Some of the most significant advantages of the Söderberg electrode are:

- The electrodes can be larger than those of the pre-baked electrodes (Hodson, 1923).
- As the casing serves as the mould and the electrode paste is loaded semi-continuously, there is no need for threads to connect one electrode section to another.
- Furnace downtime, required to thread pre-baked electrodes together in some operations, is not required.
- There is no need to transport electrode pieces around. As graphite is relatively brittle, *in situ* baking of a Söderberg electrode is very advantageous.
- Pre-baked electrodes are very expensive, since the process requires a separate manufacturing facility, separate baking at a very high temperature and the machining of the electrodes.

## 2-3 Applications of Søderberg electrodes

As Kempken, Degel and Schmieden (2006) said: “The submerged arc furnace (SAF) has been one of the most amazing metallurgically diversified melting units”. It is one of the most popular systems for producing ferroalloys, including ferrochrome, ferronickel, ferromanganese and ferrosilicon. However, it is also used to produce iron, silicon metal, silicomanganese, copper, lead, zinc, titanium oxide, calcium carbide and phosphorus, as well as used in materials recycling (Fitt & Aitchison, 1993; Innvær, 1992; Kempken, Degel & Schmieden, 2006; Kunze & Degel, 2004; Skjeldestad *et al.*, 2015; Wilkinson *et al.*, 2001).

The Søderberg electrode system has some limitations. The most significant one of these is the metal from the steel casing that forms the electrode’s shell. If the steel has no impact on the final product (as for the ferroalloys), it is not a limitation. However, if the product needs to be pure, such as for silicon metal (Boisvert, 2001), the Søderberg electrode system cannot be used. As the case study Søderberg electrode considered in this study (see Section 3-1) came from a ferrochrome SAF, this process is considered further as an example of the application of a Søderberg electrode system.

### 2-3-1 Example application (ferrochrome industry)

Figure 2.3 shows a flow diagram of the process steps utilised by the ferrochrome (FeCr) producer, from which the case study Søderberg electrode was obtained. The specific SAF was an open/semi-closed SAF, which was fed with oxidative sintered pellets and a small fraction of chromite ore. The oxidative sintered process for chromite is also known as the Outotec process (Basson & Daavittila, 2013). Although this type of FeCr production operation is usually associated with closed SAFs (Outotec, 2011), it is also common practice to add such a sintering plant to existing facilities that might include open/semi-closed SAFs.

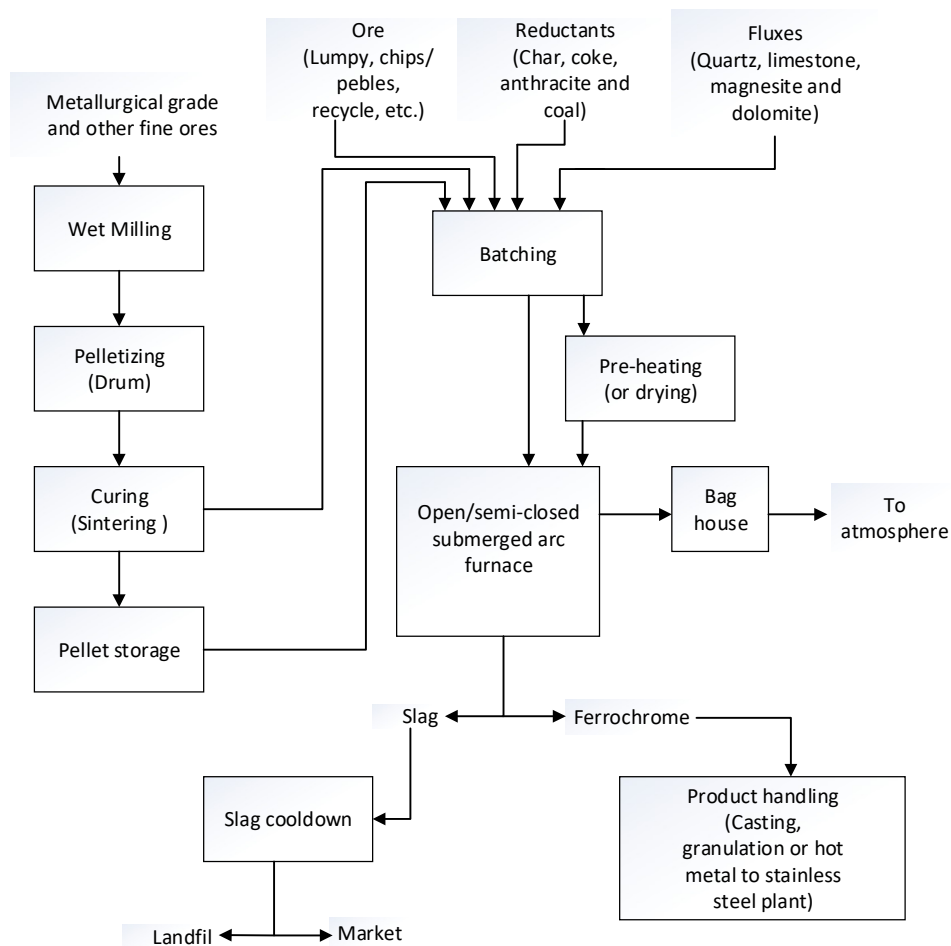


**Figure 2.3** A flow diagram, adapted from Beukes *et al.* (2010), indicating the process steps utilised by South African FeCr producers using the oxidative sintered process.

The process flow of the FeCr producer, from which the case study electrode was received, starts with the wet milling of chromite ore fines (0 to 25 mm) until the desired size is reached. The wet ore is then de-watered to an appropriate moisture level. Next, it is mixed with coke or anthracite (that provides a heat source during sintering) and bentonite (that acts as a binder for the pellets) to obtain a ratio of 97 wt% ore, 2 wt% anthracite and 1 wt% bentonite (Glastonbury *et al.*, 2015). The wet mixture is then pelletised in a pelletising drum. The over- and undersized green (newly formed) pellets are removed and recycled, while the appropriate-sized green pellets are layered on a sintering belt. The sintering belt is protected by a layer of already sintered pellets and moves through a grate furnace for the sintering process (drying, preheating, sintering and cooling). The sintering process implies that the carbon in the pellets is consumed in order to solidify the pellet into a hardened pellet (this happens in oxidising conditions). This peak temperature is approximately 1 300 °C. This partially fuses the pellets and creates a porous, nearly uniform-sized and mechanically strong SAF feed material. At the exit of the furnace, the pellets are cooled by inflowing air (Beukes *et al.*, 2010; Singh & Rao, 2008; Zhau &

Hayes, 2010). The sintered pellets are then stored for later use. Upon feeding, the sintered pellets are batched with ore, reductants (char, coke, anthracite and/or coal) and fluxes (quartz, limestone, magnesite or dolomite) in the proper fractions, i.e. 2400-3000 kg/t ore, 550-700 kg/t reductants, and 100-400 kg/t fluxes (Holappa, 2013). These batches are charged (fed) into the furnace through chutes from overhead bins (Andreae, 1950a). The charge in the SAF is smelted with the assistance of three Söderberg electrodes. The off-gas handling system consisted of a dropbox and cyclone to remove the larger particles, as well as a bag filter plant to remove the fine particles. The tapped metal is cast into sand moulds, while the slag is water-cooled.

## 2-4 Basic components of a submerged arc furnace and associated equipment

### 2-4-1 The SAF

As the design of a SAF depends on its application, this section focuses on components common to most. Figure 2.4 illustrates a SAF showing the furnace's main components, whereas Figure 2.5 is a rough illustration showing more of the electrical components.

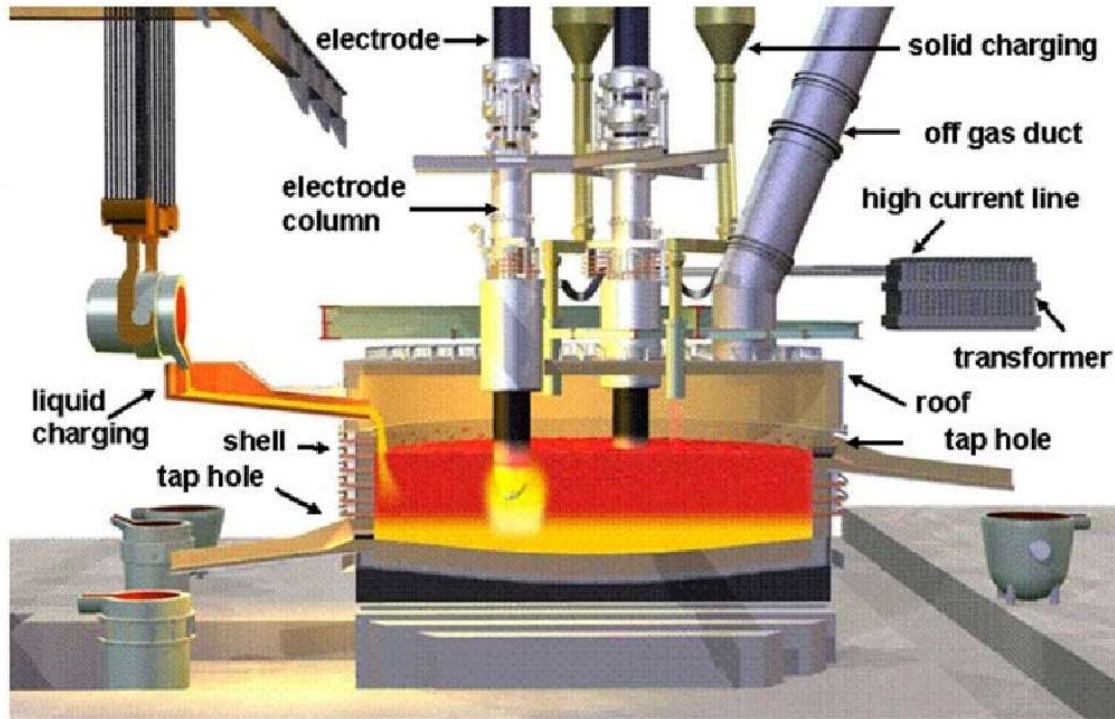


**Figure 2.4** The main components of a submerged arc furnace (adapted from Degel & Kunze, 2003).

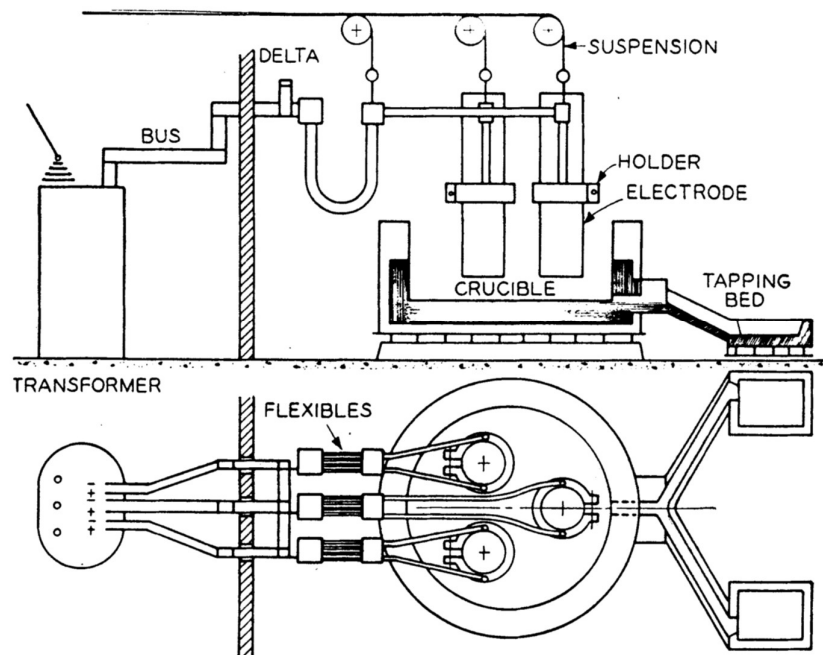


**Figure 2.5** The side and top view of a furnace showing the main electrical components (Andreae, 1950a).

#### 2-4-1-1 The furnace

The furnace is simply a large crucible. It is generally made of a steel casing, lined carbon or refractory bricks. Some designs have a freeze lining on the inside. The basic idea of this freeze lining is that the slag produced in the furnace ‘freezes’; in other words, solidifies, on the inside of the furnace. This helps insulate the furnace and protects the outer walls from wear (Andreae, 1950a; Hearn *et al.*, 1998; Kunze & Degel, 2004). The reason for insulating the furnace is to keep as much heat inside as possible, to reduce the specific electricity consumption (MWh/ton FeCr produced). Depending on the design, the insulation may be simple or complex, such as sidewall copper stripe cooling (as illustrated in Figure 2.6).

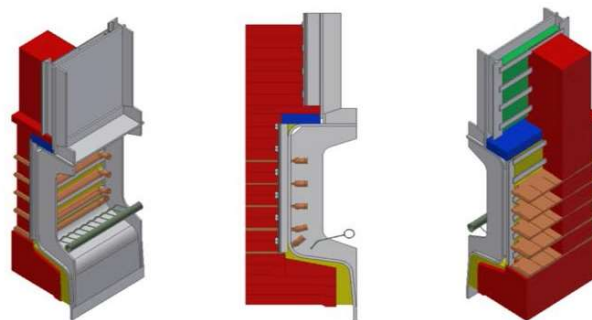


**Figure 2.6** Sidewall copper stripe cooling (Kempken, Degel, Schreiter, *et al.*, 2006).

The other major part of the furnace itself is the tap hole(s). It is the place(s) in a furnace where the slag, matte and/or metal are drained (Friedrich *et al.*, 2017; Riekkola-Vanhanen, 1999). The tap hole is a specially designed refractory inset in the sidewall of the furnace.

#### **2-4-1-2    *Post-tap hole***

For most SAFs, the main product that it produces is the metal that was melted. However, as in most chemical reactions, the main product is not the only one. In smelting, there usually are three or four products, namely: the molten metal product, gases, slag (usually a mixture between metal oxides and silicon dioxide) and/or matte (sulphur-containing by-products). The molten metal, slag and/or matte usually have different densities and can be either tapped separately or separated in the tapping runner (furrow in which metal/slag/matte are tapped), depending on the design (Andreae, 1950a). SAFs need to be tapped/drained of its contents at regular intervals. The tap hole(s) is(are) usually opened by utilising hydraulic or pneumatic drilling, and if necessary, with additional oxygen lancing. For this, pure oxygen is used to burn the tap hole open using a long, thin metal pipe (Riekkola-Vanhanen, 1999). After the furnace is drained, the tap hole(s) is(are) again plugged with what the industry terms a mud gun. With this, a type of clay called taphole clay is pushed into the tap hole and plugs it, so that the regular smelting operation can continue (van Beek *et al.*, 2016).

In open/semi-closed SAFs, the gases are extracted through the off-gas duct, filtered and cleaned in a baghouse and released into the atmosphere. In closed SAFs, the CO-rich off-gas is cleaned with a venturi (usually a wet venturi) system and the cleaned-off gas is used as an energy source on-site while the excess is flared (du Preez *et al.*, 2015).

#### **2-4-1-3    *Transformer(s)***

The duty of transformers (see Figures 2.5 and 2.6) is to convert the primary voltage derived from the power supplier to a lower secondary voltage with a much higher current. As all SAFs have different current requirements, transformers are specially designed for each SAF. With low secondary voltage, the conductors must be able to handle very heavy currents. This results in tremendous forces acting between conductors. Therefore, the coils and leads need to be braced carefully (Andreae, 1950a).

#### **2-4-1-4    *Bus bars***

The bus bars (see Figure 2.5) are the stationary (made from copper or a copper alloy) connection between the transformer and electrodes. The outgoing and incoming conductors of the same phase are laid close together to carry the heavy current with as little voltage drop as possible. This lowers the reactance to the lowest possible level (Andreae, 1950a).

As the currents coming from the transformer are very high, the circuits need to be divided into several parallel branches. The branches are bundled together and are known as 'interlaced' busses. These interlaced busses stop at the flexibles, which are placed as close to the furnace as possible (Andreae, 1950a).

#### **2-4-1-5    *Flexibles***

As seen in the top illustration of Figure 2.5 (the side view), the flexibles (also made from copper or a copper alloy) hang between the bus bars and the electrodes. This is because the furnace loads are controlled by moving the electrodes up and down, and the flexibles (as the name would suggest) can flex while this is happening. If the bus bars were connected directly to the electrode, this would not be possible. Most frequently, copper cables are used for the flexibles. They are usually water-cooled, enabling them to be connected directly to the electrode holder (Andreae, 1950a).

#### **2-4-2    *Söderberg electrode column and vital components***

Figure 2.7 shows a detailed schematic of an electrode column with all of its main components. Numbers 1 to 4 are the carbonaceous components that make up the electrode itself and are discussed in Sections 2-2. Number 5 is the smelting zone in the furnace. Lastly, numbers 6 to 12 are the different non-carbonaceous components that make up the electrode column itself.

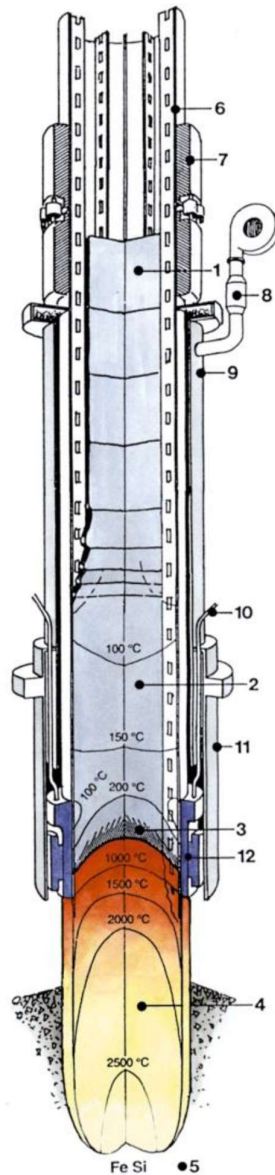


**Figure 2.7** Illustration of the Søderberg electrode, from Schei *et al.* (1998). Electrode zones and equipment are numbered in the figure: 1 – paste cylinders, 2 – melted paste, 3 – baking zone and 4 – baked electrode are the components that make up the carbonaceous electrode itself. 5 is the smelting zone of the furnace. 6 – casing with fins, 7 – slipping rings, 8 – fans for heating air, 9 – suspension mantle, 10 – current tubes, 11 – heat shield, 12 – electrode holder and contact shoes.

#### 2-4-2-1 Casing and fins

The cylindrical steel casing serves as a mould for the electrode paste while it melts and bakes. The electrode holders (see Section 2-4-2-7) and the slipping equipment (see Section 2-4-2-2) press against this casing, holding the whole electrode upright and in position. Consequently, the casing must be rigid enough to withstand the radial pressures without buckling or collapsing in on itself. Another important function of the casing is to conduct the current from the contact shoes (see Section 2-4-2-7) to the baked part of the electrode (see number 4 in Figure 2.7) (Asphaug & Innvær, 1997).

The fins are welded onto the inside of this casing and help to conduct the current to the electrode itself. It also transfers heat to the electrode's interior to help it bake more evenly (Asphaug & Innvær, 1997; Innvær *et al.*, 1995). The casing and fins will be discussed in more detail in Section 2-5.

#### 2-4-2-2 *Slipping rings*

With the large electrode dimensions and weights, the holder's grip alone is usually not sufficient to carry the weight of the electrode. The extra holding pressure is provided from the slipping equipment. This prevents the electrode from sliding down. That is why the controlled displacement of the electrode down through the holder is termed 'slipping' (Andreae, 1950b; Asphaug & Innvær, 1997).

The slipping device works in the following way. Two rings (upper and lower slipping rings) are mounted, one just above the other, quite high up on the electrode, an example of which can be seen in Figure 2.8. The two rings press against the electrode casing by means of air or water pressure over a rubber diaphragm (Asphaug & Innvær, 1997).

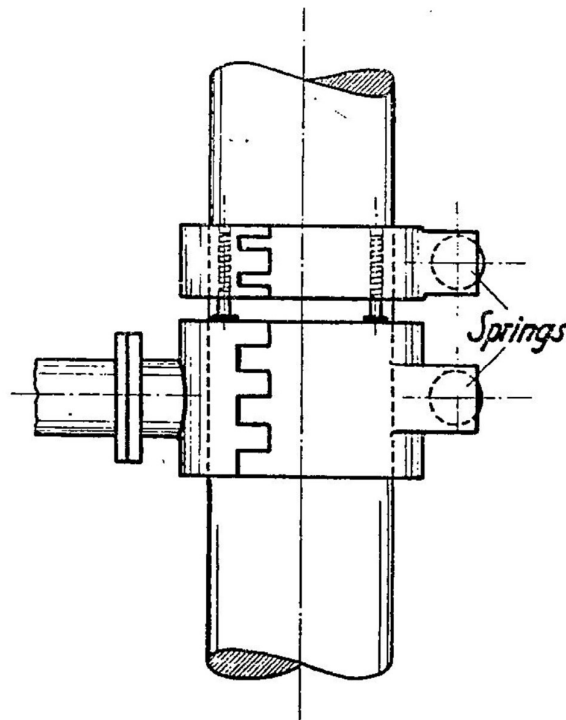


**Figure 2.8** An example of an automatic slipping device (Sem, 1954).

The lower ring is typically fixed to the electrode suspension gear, whereas the upper ring may be moved vertically in relation to the lower ring by means of hydraulic cylinders. When the electrode is about to be slipped, the upper ring's grip is loosened, the ring is lifted, and the grip fastened again. Then, the lower ring grip is loosened, and the electrode is 'slipped' down through the holder by

lowering the upper ring to its original position. Finally, the lower ring grip is fastened again, and the slipping sequence has been completed (Asphaug & Innvær, 1997).

#### **2-4-2-3 Fans for heating air**

The paste above the holder area (the area between the paste cylinders, 1 in Figure 2.7, and melted paste, 2 in Figure 2.7) softens and liquefies so that the entire cross-section area is filled without any segregation occurring. Paste segregation happens when easy-flowing components (such as binder and anthracite fines) flow out between the fins, while the more coarse parts stay in the central part of the electrode (Asphaug & Innvær, 1997).

To control the temperature conditions in the area where the electrode paste softens and liquefies, air (heated or unheated, depending on the furnace design) is blown down through the annulus (the area between two cylinders) between the suspension mantle (see section 2-4-2-4) and the electrode casing. Around 6 000 m<sup>3</sup> per hour of air is typically required for this process (Asphaug & Innvær, 1997; Ord *et al.*, 1995).

#### **2-4-2-4 Suspension mantle**

The suspension mantle is a cylindrical shield that surrounds the electrode's upper part from the heat shields (Section 2-4-2-6) to the slipping rings (Section 2-4-2-2). This mantle helps to keep the casing clean and helps control the temperature (Andersen *et al.*, 2001; Asphaug & Innvær, 1997).

#### **2-4-2-5 Current tubes**

The current tubes (made from copper or a copper alloy) are the connection between the flexibles (Section 2-4-1-5) and the contact shoes (Section 2-4-2-7).

#### **2-4-2-6 Heatshield**

The heatshields are there to protect the electrode holder (Section 2-4-1-6), current tubes (Section 2-4-2-5) and the contact shoes (Section 2-4-2-7) from the heat coming up from the furnace bed. They are usually water-cooled, but each designer has their own design and preferences. This is probably the part of the installation where most ingenuity is displayed.

#### **2-4-2-7 Electrode holder and contact shoes**

The Söderberg electrode is held upright in the SAF by means of an electrode holder. It is constructed as a clasp holder and can easily be moved along the electrode during slipping (see Section 2-7) (Söderberg & Sem, 1922). The electrode holder grips around the electrode with pressure. The friction force counteracts the electrode's weight and 'holds' it, which explains the term 'holder'. Often, the holder is slightly tapered in the downward direction to allow for paste shrinkage during baking. In this

way, the best possible grip is obtained. The holder is suspended in the furnace building through the hoisting system, which allows the electrode to be moved up and down (Asphaug & Innvær, 1997). The holder transmits the current from the copper tubes through what the industry calls contact shoes to the electrode (Andreae, 1950a).

There are four types of holders, i.e.:

*I. The conventional holder*

This is the original type of holder and is not used anymore these days. Here, the contact shoes are pressed against the electrode casing through spring-loaded bolts mounted to a water-cooled pressure ring. By tightening or loosening the bolts (in those days, it was done by hand), the spring pressure could be adjusted (Asphaug & Innvær, 1997).

*II. The cylindrical spring-loaded holder*

This term refers to the outer form of the holder assembly. The conventional holder's pressure ring is replaced by several segments linked together to form a closed cylinder. This covers the clamps almost completely. As with the conventional holder, the cylindrical spring-loaded holder uses mechanical springs to provide pressure on the contact shoes. However, the design allows the shoes to be unloaded by means of pneumatic pressure, which counteracts the spring force (Asphaug & Innvær, 1997).

*III. The membrane holder*

This type of holder is also a cylindrical holder and resembles the cylindrical spring-loaded. However, the spring is replaced with hydraulic pressure, which transfers through a rubber diaphragm or membrane. This is why it is called the membrane holder. This is the most common type of holder in recent times (Asphaug & Innvær, 1997)

*IV. The modular holder*

The modular holder is a relatively new design (last couple of decades). It needs a different type of casing with fin extensions on the outside. All connections on the electrode column itself will connect to these fin extensions. This type of holder gives very thorough mechanical and electrical contact and applies no radial force on the electrode casing. Therefore, there is no risk of buckling or collapsing of the casing (Asphaug & Innvær, 1997).

## 2-5 Casing design and material

Electrode casings can be called the backbone of the Söderberg electrode as it is the rigid form around what will later be the electrode itself. It provides strength as the electrode forms and bakes, as well as provides a conductive shell for the contact clamps. This helps to conduct the current to the baked part of the electrode and the furnace itself. The casing quality depends on only two factors, i.e. the design and the fabrication (Andersen *et al.*, 2001). One often-overlooked part of fabrication is the proper connection of the fins onto the existing electrode column.

### 2-5-1 *Design and fabrication*

The first casings were made of iron. A casing section was made by taking several longitudinal segments of iron sheets and bending a part of it at a right angle. One part of this bend became the outer wall, and the other became the fins on the inside. Enough of these segments were then welded together, with the walls on the outside and the fins protruding inward, to produce a casing section of the right size for the electrode. The fin part of the casings also had windows/holes pierced into them. This was done to keep the electrode paste mixture united through the fins and helped the baked electrode to not slip out of the casing. The casing's outer shell was also pierced with numerous smaller holes for the tar vapours to escape. All the casing sections for a specific electrode were made the same way (Richards, 1920).

Although many of the components that make up the electrode casing are the same as it was originally, the construction has changed. For starters, iron is no longer used to make the casings. Mild steel is the most commonly used material in the construction of casings, but stainless steel is also used on some plants (Mc Dougall, 2007). Next, the casings are no longer constructed with the segment method. Round steel tubes of the right diameter and thickness are used for the body of the casing. The diameter and thickness will change depending on electrode size and design specifications. The fins are made separately and welded onto the inside of the casing. The fins still have windows punched into them for the same reason as mentioned above. Different fin configurations are shown in Figure 2.9. The fin design, spacing and length, and the fin window design and spacing depend on the specific furnace design. All casing sections for a specific electrode are all made the same way. The uniformity assures that there are no unforeseen casing-specific problems. Lastly, holes are no longer pierced into the casing exterior of the casing. As will be explained in Section 2-6, the electrode paste quality was improved since the first casing designs were used. Vapours are no longer produced at lower temperatures. Consequently, the holes are no longer necessary.

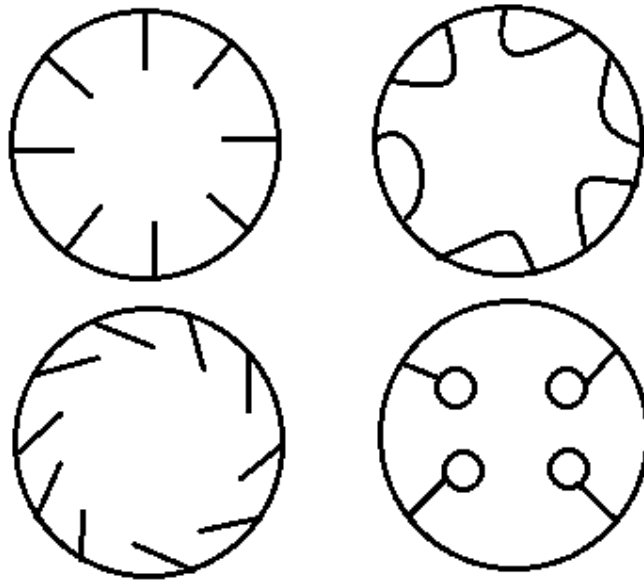


**Figure 2.9** Different fin designs (adapted from Bartlett et al., 2015).

### 2-5-2 Installation

The most significant advantage of the casing segment concept is that, as the electrode begins to get shorter, a new casing segment can be welded to the top of the column (Søderberg & Sem, 1922). It is of utmost importance that the new casing segment is welded solidly to the existing electrode column; this includes welding the fins in joints without overlapping them (Andersen *et al.*, 2001; Innvær *et al.*, 1986). In Section 2-9, the candidate will discuss what happens when the casing and fins are not welded together properly. Figure 2.10 shows an example of a cylindrical steel Søderberg electrode casing with the fins and their windows. It is clear from the photo that all the casings and the fins are properly welded (and/or bolted) together with no unwanted holes or separation of the fins.

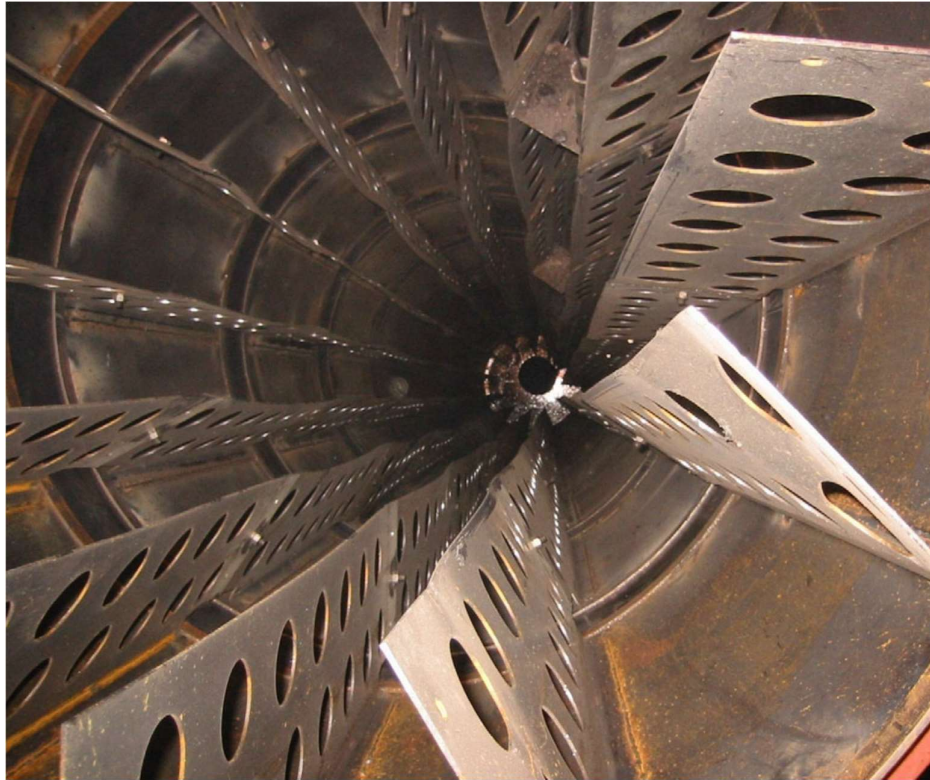


**Figure 2.10** A cylindrical steel Søderberg electrode casing (Nelson & Prins, 2004).

## 2-6 Søderberg electrode paste

### 2-6-1 *Production*

The making of electrode paste has been fundamentally unchanged for years (Innvær, 1989). The electrode paste is fundamentally a mixture of a binder (usually coal-tar pitch) and a carbon aggregate such as calcined anthracite, coke or scrap graphite (Asphaug & Innvær, 1997; Fitt & Aitchison, 1993; Fitt & Howell, 1998; Innvær, 1992; Larsen *et al.*, 2004; Richards, 1920; Shkoller & Proshunin, 2008; Skjeldestad *et al.*, 2015). Although this is the basic building blocks for electrode paste, other raw materials can be added for more specialised pastes (Innvær, 1992). All raw materials are tested to make sure they comply with the exact specifications of the manufacturer before these materials are prepared for mixing (Innvær, 1992). This is done to ensure the best possible electrode paste is produced.

#### 2-6-1-1 *Coal-tar pitch*

Probably the most important raw material in the making of electrode paste is the coal-tar pitch. Coal-tar is produced as a by-product of the coking process. In this process, coal is heated to between 1 000 and 1 200 °C and held at this temperature for 14 to 20 hours. The yield of coal-tar is only 3 to 4 %.

After the water and chlorides are removed and the tar is distilled, the final product is coal-tar pitch. Between 15 and 20 kg of coal-tar pitch is produced for every metric ton of coal coked (Madshus, 2005; Menéndez *et al.*, 2000; Skjeldestad *et al.*, 2015). The coal tar pitch is distilled specifically for electrode paste to remove all or most of the volatiles that evaporates at a temperature range between 270 and 360 °C. If these volatiles are not removed, they can evaporate at the bottom of the electrode column and condense higher up. These condensed compounds will then mix with the liquid electrode paste and make it more fluid (Skjeldestad *et al.*, 2015). As the fluidity of the paste level is closely monitored (to have it at the desired level), it could cause the operator to lower the column's temperature. This will influence the melting of the solid paste and may result in an electrode break.

In Shoko *et al.* (2013), it was demonstrated that irrespective of chemical compositional differences of coal tar pitches (measured with Fourier Transform Infra-Red spectroscopy (FT-IR) analysis), the baking isotherm temperature stayed consistent. This indicates that as long as the coal tar pitch was distilled correctly, they all function similarly in a furnace, atleast with regard of baking isotherm temperature.

#### **2-6-1-2    *Aggregate used to manufacture Søderberg electrode paste***

The aggregate usually comprises either coke or calcined anthracite (Shoko *et al.*, 2013). Similarly to the coal-tar paste, the aggregate should be free of any volatile compounds. The coke usually has an ash content of about 0.25 %, volatile matter content of 1.7% and a 88% fixed carbon content (Prabhakaram & Murthy, 1961). Removal of volatile compounds is achieved by either using coke or by the calcination of the anthracite at temperatures above 1300°C (Asphaug & Innvær, 1997; Fitt & Aitchison, 1993; Innvær, 1989, 1992; Kashlev & Strakhov, 2018; Sem, 1954). The other reason to do this is to complete the shrinking of the anthracite associated with carbon reordering (Beukes *et al.*, 2013); otherwise, the particles will shrink in the electrode. This will cause stresses that could break the electrode. To calcine the aggregate, it is heated from 1 200 to 3 000 °C, typically in a shaft furnace (Asphaug & Innvær, 1997; Innvær, 1989; Sem, 1954). After calcination, the aggregate is crushed and screened into sizes ranging between 125 µm and 15 mm (Asphaug & Innvær, 1997; Fitt & Aitchison, 1993).

#### **2-6-1-3    *Mixing of raw materials and making the electrode paste***

According to Richards (1920), during the first usage of Søderberg electrodes, the electrode paste components were manually mixed on-site and fed into the electrode column at 80 °C. Today, the paste is mixed off-site, still at approximately 80 °C. Typically, 70 to 80 % aggregate is used with the coal tar pitch, but this can be changed according to the furnace design's specifics (Larsen *et al.*, 2004; Sem, 1954), although a minimum of 18 % of the mixture needs to be a binder (coal-tar pitch).

Each Smelter has its own specifications for the properties of the electrode paste. An example of these kinds of specifications can be found in Table 2.1. The table gives the minimum and maximum requirements that both the green paste and the baked paste needs to have for the Tasmanian Electro Metallurgical Company Pty. Ltd. (TEMCO) smelters (Wilkinson *et al.*, 2001).

| <b>Green Paste</b>     |                             |
|------------------------|-----------------------------|
| Apparent density       | 1.55-1.60 g/cm <sup>3</sup> |
| Plasticity             | 15-25%                      |
| Ash                    | 5-7%                        |
| Volatiles              | 10-13%                      |
| Fixed Carbon           | 80-85%                      |
| Binder Softening point | 62 °C                       |
| <b>Baked Paste</b>     |                             |
| Apparent density       | 1.36-1.41 g/cm <sup>3</sup> |
| Electrical resistivity | 68 Ohm.mm <sup>2</sup> /m   |
| Thermal conductivity   | 8.0W/rnK                    |

**Table 2.1** TEMCO specifications for electrode paste.

After mixing the paste, it is either cast in moulds to form cylinders (Figure 2.11a) or blocks, or it is extruded into briquettes (Figure 2.11b) (Amaro *et al.*, 2001; Asphaug & Innv  r, 1997; Innv  r, 1992; Larsen *et al.*, 2004; Nelson & Prins, 2004). The type and size of these paste ‘blocks’ depend on the size and design of the electrode it is to be used in.



(a)



(b)

**Figure 2.11 (a)** Electrode paste cylinders, **(b)** Electrode paste blocks (Dall, 2008).

All three types of paste configurations have their own advantages and disadvantages. The advantages of cylinders and blocks are that they are very easy to count and know how much paste is loaded into the electrode column. Because of the design of the electrode casing, the cylinders fit easily in between the ribs. As seen in Figure 2.11a, the cylinders can be stored very easily and cleanly. The disadvantage

of cylinders is that specialised lifting equipment needs to be used to load the heavy cylinders. This cannot be automated and needs to be done manually or at least semi-manually. Another potential problem was described in Nelson and Prince (2004), i.e. that the heavy cylinders can be dropped into the electrode column, which can cause damage and/or electrode breaks. The blocks have all the same disadvantages as the cylinders. Another disadvantage that the blocks have is that their shape can make it difficult to load them into the column, as the edges could potentially hit the casing fins damaging them.

Briquettes have the advantages of lower production costs (no moulds); they are easy to transport, and the electrode column's charging can be automated. There is also data suggesting that less electrode segregation occurs with briquettes (Amaro *et al.*, 2001). The main problem with briquettes is the formation of bridges in the electrode column. As the name suggests, this is when the briquettes form a row across the electrode column stopping new briquettes going lower. This makes a hollow under the bridge that can produce a weak place in the electrode and cause an electrode break.

### 2-6-2 *Properties*

The binder in the electrode paste may behave like a Newtonian fluid, but as soon as the aggregate is added, the flow of the electrode paste exhibits a more complicated rheological characteristic. The behaviour of the electrode paste is highly temperature-dependent (Fitt & Aitchison, 1993). It is solid at room temperature and will soften at approximately 50 °C and flow at approximately 80 °C (Fitt & Aitchison, 1993; Fitt & Howell, 1998; Innvær, 1992; Skjeldestad *et al.*, 2015). It reaches its minimum effective viscosity at around 200 to 250 °C (Fitt & Howell, 1998). At 475 °C, the electrode is fully baked into a solid carbon material (Beukes *et al.*, 2013), but it is then not yet graphitised. Even though different pastes' melting and baking characteristics will change somewhat, this baking isotherm (transformation temperature from liquid to solid paste) stays relatively constant (Shoko *et al.*, 2013).

Plasticity is defined as the percentage by which the diameter of a small paste cylinder increases when heated under standardised conditions. This is considered as one of the most important variables with regard to the quality of the electrode paste. To get the electrode paste to flow easily, the binder content needs to be high (around 30 to 40 %). As previously mentioned, according to Sem (1954), a minimum of 18 % binder needs to be used for the electrode paste to be efficient. A low binder percentage gives a dry paste with very low plasticity (Asphaug & Innvær, 1997). Each electrode paste batch (cylinder, block or briquette) is marked with its plasticity. This plasticity is used as quality control during operation. Figure 2.12 shows a paste cylinder with its plasticity indicated on it.



**Figure 2.12** Paste cylinder with its plasticity indicated on it (Dall, 2008).

The shape in which the electrode paste comes in will change its melting characteristics. When the electrode paste cylinders are loaded into the electrode column, they are stacked one on top of the other. This weight will compress the paste cylinder as it enters the softening zone (the area where the temperature is around 50 °C) of the column. This will increase the surface area of the paste cylinder that is submerged in the already melted electrode paste. This will help the paste cylinder to melt faster. Depending on circumstances, it usually takes between 6 and 24 hours for a paste cylinder to fully melt and cover the entire column.

As the paste briquettes are much smaller than the paste cylinders, they will mix more evenly in the liquid paste, which will help them melt more evenly and create a more stable homogeneous layer of high-viscosity paste. This, in turn, will minimise the possibility of segregation.

Shoko *et al.* (2013), presented the dimensional changes of coal tar pitch that is heated to 1300 °C in three consecutive cycles. The results show that when the coal tar pitch is heated to around 1200 °C in the first cycle the dimensional changes are around 12 to 14 %. During cycles two and three those dimensional changes are just 0.5 to 2 %. This shows that as the coal tar pitch is heated to around 1200 °C for the first time reordering of the carbon matrix takes place (not yet graphite, but become a more structurally arranged carbon matrix). These structural changes are then set and does not change during subsequent heating cycles.

## 2-7 Operational Söderberg electrode management

Electrode management can be considered as a process within the process. 'Electrode management' comprises many different aspects (Asphaug & Innvær, 1997). The production of the electrode is an 'almost' continuous process. By 'almost', it refers to the fact that the electrode slipping (and therefore the baking of the new electrode) is done in steps. Other parts of the process that also go on intermittently are joining new casings (see Section 2-5) and the charging of electrode paste (see Section 2-6) (Asphaug & Innvær, 1997).

Besides the electrode itself, the operation depends on surrounding electrode equipment, such as the electrode holder with contact clamps, current supply components comprising water-cooled flexible cables, slipping device, electrode hoists, fans, heating elements and other control equipment. All these components require maintenance to keep the process functional (Asphaug & Innvær, 1997).

When all the above is taken care of, the basis is laid for good electrode operation. However, another important condition remains – the actual furnace operation and its effect on the electrode condition itself. Four areas, in particular, need attention (Asphaug & Innvær, 1997; Innvær *et al.*, 1984):

- The conditions for softening and liquefaction of the electrode paste.
- The current load on the electrode, which is limited, dependent on the electrode diameter and casing thickness.
- The electrode consumption, which determines the need for slipping and baking of new electrodes. High consumption in relation to the baking capacity may cause the baking zone to get too low, with the risk of breakage in the un-baked part of the electrode (also known as a green break) as a consequence.
- Changes in operating conditions (reduced load level, shutdown, recovery), which cause the temperature distribution to change and 'thermal' stresses to develop, with the risk of hard breakages to occur.

### 2-7-1 Paste management

Electrode paste management is another important part of electrode management. Figure 2.13 is a visual representation of a partial Söderberg electrode column with the baking isotherm area enlarged (indicated by the red square). This figure will be used to help explain the different aspects of paste management.

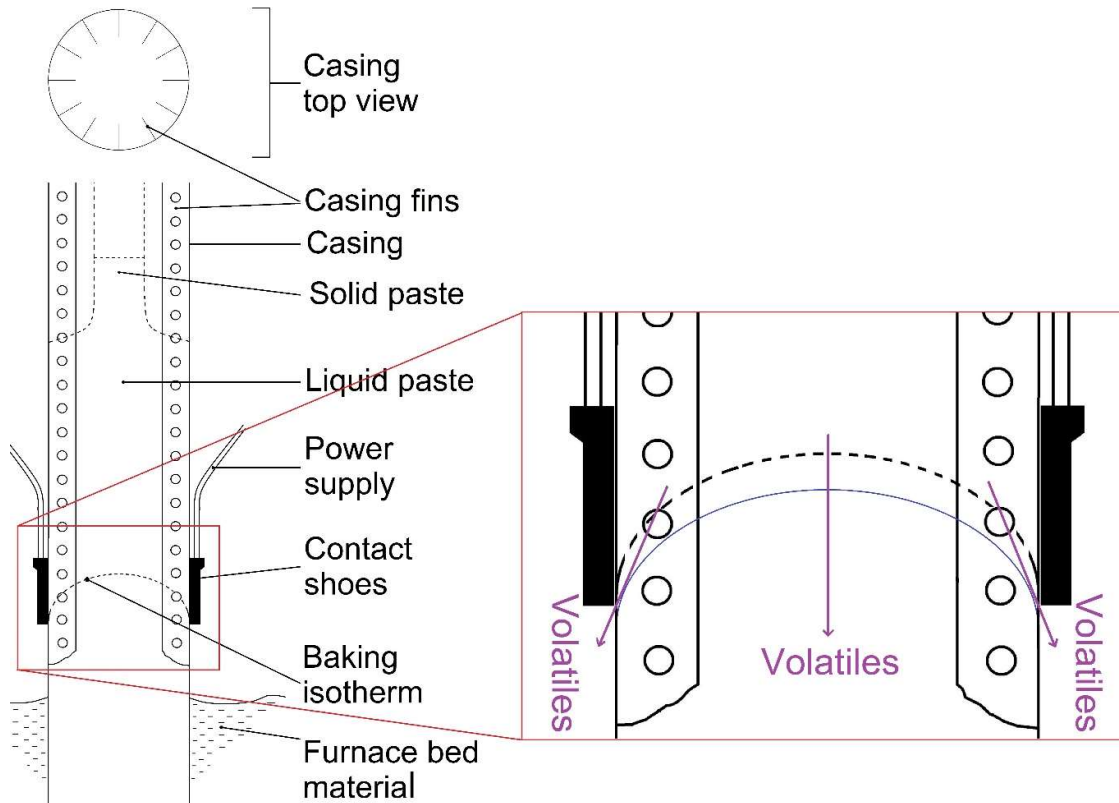


**Figure 2.13** Representation of a partial Søderberg electrode column, adapted from Beukes *et al.* (2013), with the baking zone area enlarged (indicated by the red square).

#### 2-7-1-1 Paste softening and melting conditions

At a temperature of 80 to 100 °C (Arnesen *et al.*, 1979; Richards, 1920; Skjeldestad *et al.*, 2015), the paste is soft enough to flow out under the weight of the solid paste column above and fill the entire cross-section of the casing. This liquid paste level (see Figure 2.3.1) should be 2 to 3 m (Arnesen *et al.*, 1979; Larsen *et al.*, 2004) above the top of the contact shoes.

If this liquid paste level becomes too low, volatile waste gases from the baking process may escape upwards and represent a fire hazard. These volatile waste gases have to escape down through the pores of the already baked electrode (purple arrows in Figure 2.13). Here, these volatile gases crack (as a result of the high temperature in the baked electrode) and leave a considerable amount of carbon in the electrode, thereby improving its quality. The liquid paste level must be watched carefully after a long slip (see Section 2-7-3-2.) (Asphaug & Innvær, 1997; Sem, 1954).

As previously pointed out, the paste must not segregate (see Section 2-4-2-3) as it softens and flows out. Paste segregating happens when the paste melts so that the area in-between the fins is enriched by anthracite fines and binder agent. Simultaneously, the central region of the electrode will be depleted from the binder and enriched by coarser anthracite particles. This will cause a weak electrode

with an interior of loosely bound anthracite gravel. Segregation may occur if the temperature distribution is unfavourable, and the liquid paste level is not properly controlled (Asphaug & Innvær, 1997; Innvær *et al.*, 1986; Larsen, 2012).

#### 2-7-1-2 *Baking conditions*

The baking zone (the area between the contact shoes in Figure 2.13) is where the electrode is 'produced', i.e. where the liquid paste is baked into the solid electrode. Volatile compounds must be driven off in this area, since its occurrence in the electrode will cause cracking that might cause an electrode break while in operation. This area is also known as the baking isotherm (the black dotted line in Figure 2.13). The management of the baking isotherm position is considered one of the most important aspects of electrode management (Innvær *et al.*, 1986; Larsen, 2012). Shoko *et al.* (2013) proved that the baking isotherm for most electrode pastes is around 475 °C, so it is important to keep this temperature isotherm above the contact shoes (Innvær *et al.*, 1986; Larsen, 2012). Once the temperature is around 1200 °C the electrode's dimensions will be set. (see Section 2-6-2).

#### 2-7-2 *Electrode current*

The baked part of the electrode is a good conductor compared to the un-baked electrode paste. However, a considerable amount of resistance heating ( $R \cdot I^2$ ) is developed by the electrode current passing through the paste. This heat generation and the subsequent temperature increase are what mainly (heat rising up into the column from the furnace also contribute) bake the electrode paste. However, there is a limit to the temperature that the electrode can withstand, and therefore there is a limit to the current (Asphaug & Innvær, 1997).

A guiding number for the current density is 7 ampere per square centimetres ( $A/cm^2$ ) of the sectional area of the electrode. The total current of a 1 m diameter electrode can be calculated to approximately 55 kA. For an electrode with double that diameter (2 m), the cross-sectional area is increased four times. Then, one should expect that the electrode current could be increased proportionally, i.e. to 220 kA. However, this is not the case. The normal load for a 2 m diameter electrode is, in practice, only 130-150 kA, which corresponds to an average current density of only 4.5  $A/cm^2$  (Asphaug & Innvær, 1997).

The reason for this is, under normal operating conditions, the temperature in the electrode will be highest in the centre and will lessen towards the outer surface. This peak temperature in the centre of the electrode restricts the current load that the electrode can withstand. Therefore, to prevent the

temperature in the electrode centre from getting too high (and possibly damaging the electrode), the current has to be lowered to less than  $7 \text{ A/cm}^2$  (Asphaug & Innvær, 1997).

The other problem with a higher temperature in the centre of the electrode is that the electrical conductivity of carbon increases with temperature (Powell & Schofield, 1939). This means that the current paths will be pulled towards the centre of the electrode, and contribute to an even higher temperature in this region (Asphaug & Innvær, 1997).

Another phenomenon, which has the opposite effect, and tends to pull the current paths and the heat development towards the electrode's surface, is the skin effect. Balancing these two effects will optimise the electrode's current load (Asphaug & Innvær, 1997).

#### **2-7-2-1    *Skin effect***

Skin effect is when an alternating current tends to concentrate in the outer layer of a conductor (Innvær, 1992). It is caused by the self-induction of the conductor and results in increased resistance. The skin effect is more pronounced in higher frequencies. A measure of the skin effect is the skin depth: the distance from the surface at which the current density is reduced to approximately 37 % of the current density close to the surface. This effect is usually only seen in electrodes with diameters greater than 1 m (Asphaug & Innvær, 1997).

A similar effect as the skin effect is caused by the mutual induction between the electrodes, and is called the proximity effect. The proximity effect causes the current in one electrode to be displaced towards one of the neighbouring electrodes (Innvær, 1992).

The skin and proximity effects both increase the apparent resistance of the electrode. The increase can be expressed as the ratio  $R_{AC}/R_{DC}$ , where  $R_{AC}$  is the resistance to alternating current and  $R_{DC}$  is the resistance to direct current (Asphaug & Innvær, 1997).

#### **2-7-2-2    *Regulating the electrode tip height***

Dhainaut (2004) showed that the electrode tip position influences whether the electrical arc goes through the metal bath or the slag. This arc produces the heat (through Ohmic heating) needed to smelt the ore and keep the metal and slag melted – therefore, the tip position is important.

#### **2-7-3    *Electrode slipping***

As previously indicated, electrode slipping is the process of feeding a new electrode through the holder to compensate for the tip's consumption. It was previously explained in Section 2-4-2-2 how

the slipping equipment works, and how the slipping during normal operation should be performed evenly, in small increments (Asphaug & Innvær, 1997).

For a new electrode to be sufficiently baked, the slipping rate must not be too high in relation to the electrode current. The electrode current provides the heat generation required for baking (Innvær *et al.*, 1995). The baking power (P) may be expressed in terms of the resistance (R) and the current (I), as:

$$P = R \cdot I^2 \quad (2-1)$$

R (which means the electrode's resistance itself and has nothing to do with the operating resistance of the furnace) is considered to be constant. Consequently, the baking power becomes proportional to the square of the electrode current (Asphaug & Innvær, 1997).

As every unit length of the electrode requires a certain minimum of baking energy, the maximum slipping rate that can be allowed is proportional to the baking power. This maximum slipping rate ( $S_{\max}$ ) may then be expressed in terms of a 'slipping index' (K) and the electrode current (I), as:

$$S_{\max} = K \cdot I^2 \quad (2-2)$$

Each electrode should have its own slipping index value and has to be kept as constant as possible. If this slipping index is exceeded by too much, the baking isotherm will move below the contact shoes and can cause a soft break (see Section 2-9-1) (Asphaug & Innvær, 1997).

The electrode consumption rate depends on the electrode paste quality and the current in the furnace. The slipping lengths are kept as constant as possible with the time between the slippings varying according to the consumption. Some processes have higher currents than others, and therefore, higher electrode consumption. Additionally, any furnace may, from time to time, have considerably higher electrode consumption than normal (due to breaks or mechanical wear) and, as a result, a higher slipping rate (Asphaug & Innvær, 1997; Innvær, 1992).

If more slipping than the maximum allowed rate is required to maintain the electrode length, special measures must be taken to prevent soft breakage. The electrode current has to be reduced according to certain rules (each furnace has its own rules) so that the baking zone can be allowed to go below the contact shoes when the electrode is slipped. The reduced electrode current must then be maintained until the baking zone has resumed its normal level (Asphaug & Innvær, 1997). There are two different procedures for this special kind of electrode slipping: forced slipping and long slipping.

### 2-7-3-1 *Forced slipping*

For forced slipping, the electrode current should be reduced to the level where the steel in the casing alone will carry the whole current. This corresponds to a current density in the steel of  $2.5 \text{ A/mm}^2$  and is considered a safe limit. In such a situation, the casing alone can carry the electrode's weight with little risk of a soft breakage, even though the baking zone gets below the contact shoes (Asphaug & Innvær, 1997).

The 'maximum allowed slipping rate' (calculated in the previous section) can now be disregarded and slipped at three to four times the normal rate until the desired electrode length has been reached. The baking zone is probably considerably lower than it usually is. After finishing this forced slip, the current should be maintained at this lower level for typically one hour, and then gradually increased at a maximum rate of  $5 \text{ kA/h}$  (Asphaug & Innvær, 1997).

The baking process's continuity will be maintained fairly well if this forced slipping is performed at an even rate. The baking gases will still be allowed to escape downwards through the baked and porous carbon electrode. If paste segregation can be prevented from entering the baking region, a good quality electrode can be maintained, and the operating conditions will not be influenced that much, except for the reduced current in the period (Asphaug & Innvær, 1997).

It can be impossible to perform a forced slip with a relatively smaller diameter electrode. This is because small electrodes have a smaller steel cross-section in the casing in relation to the maximum electrode current compared with larger electrodes. To reduce the electrode current to the level where the casing alone can carry the whole current (with a maximum current density of  $2.5 \text{ A/mm}^2$ ), therefore, a much more drastic reduction for a small electrode than it is for a large one is needed. It is recommended then that smaller electrodes perform long slipping (Asphaug & Innvær, 1997).

### 2-7-3-2 *Long slipping*

With so-called long slipping, the total elongation of the electrode is done in one operation. As much as 1 to 2 m slipping may be required, with a corresponding lowering of the baking zone. In this case, it is necessary to reduce the electrode current even more than during forced slipping (Asphaug & Innvær, 1997).

For large electrodes, long slipping should be avoided, if possible. The strain on the electrode during a long slip is considerable. Furthermore, the baking gases have limited possibility to escape downwards through the baked and porous electrode. The gases can build up in the electrode casing itself and may be ignited by the furnace's heat. This can result in an explosion. With a slip length of 1 m or more, it is preferable (if possible) to perforate the casing by drilling holes, to allow the gases to escape. 3 mm

holes at 200 mm spacing are recommended (Asphaug & Innvær, 1997). As the baking gases escape from these holes, they usually ignite. These flame tongues can be used to determine how much the electrode current must be reduced. If the flame tongues are more than 15 cm long, the current must be reduced correspondingly. A reduction to below 40% of the normal current may be required. With guidance from the intensity of these flames, the current is again gradually increased. As an example, after a long slip of 1.5 m on a 1.55 m diameter electrode, the required recovery time is typically 24 hours (Asphaug & Innvær, 1997).

#### **2-7-4     *Shutdown routines***

A stable operation at a constant load is always best for the furnace process and the health of the electrodes. On the other hand, during transient conditions, as experienced during furnace shutdown and recovery periods, the electrodes are exposed to thermal stresses. These stresses may cause electrode breakages if one does not follow certain prescribed procedures and rules (Asphaug & Innvær, 1997; Larsen *et al.*, 2013).

Before going into the specific procedures, the candidate will explain the phenomena that occur during these condition changes.

##### **2-7-4-1     *Shrinkage and thermal stresses***

During the baking process, the electrode paste will shrink somewhat, and the temperature radiates outward from the centre of the electrode to the outside surface. Consequently, the baking does not take place simultaneously over the entire cross-section of the electrode. Therefore, the shrinkage in different sections causes stresses, and small cracks can develop. These small cracks (micro-cracks) are arbitrarily distributed in the electrode and cause reduced mechanical strength (Asphaug & Innvær, 1997).

During normal operation of the electrode, the temperature distribution is the same as during baking, i.e. the temperature is highest in the central region, and falls towards the outside surface. During this normal operation, the latent stresses (caused by the shrinkage) will be relieved, and no further micro-cracks will form. This means that we may consider the electrode 'free' from stresses during normal operation (Asphaug & Innvær, 1997; Larsen *et al.*, 2013).

When the load is reduced drastically, e.g. during shutdown periods, it is no longer considered a normal operation. The electrode's temperature will then change, and at different rates at its centre compared with the outside surface. Because thermal expansion varies with temperature, new mechanical stresses will develop (Asphaug & Innvær, 1997). These thermal changes cause stresses and are usually

referred to as thermal stresses. As the electrode materials have low elasticity, these thermal stresses can be severe and are a common cause of electrode breakages (Asphaug & Innvær, 1997).

The immediate cooling effect on the surface (when the furnace is shut off) will temporarily cause tensile stresses at the surface. In particular, the tensile stresses in the longitudinal direction may be considerable. After some time, however, the temperature profile will flatten out. This means that the material in the central region will try to shrink more than the outside surface will allow, and tensile stresses will develop in the middle. Simultaneously, the original tensile stresses at the surface will shift into compressive stresses (Asphaug & Innvær, 1997). If these stresses exceed the strength of the electrode, the electrode will break. The danger of this happening increases with increasing electrode diameter and with the duration of the shutdown. Therefore, thermal stresses are a limiting factor concerning the size of electrodes.

#### **2-7-4-2    *Short shutdowns***

Because of the large dimensions of some electrodes, the thermal inertia (the time it takes an object to start to cool down) of an electrode is considerable. Therefore, it takes some time after a temperature change before thermal stresses of any significance start to develop in the central part of the electrode.

For short shutdowns (up to about 4 hours for larger electrodes (Innvær *et al.*, 1986; Larsen, 2012)), it is generally advised to decrease and increase the current at the fastest possible rate. Shutting off the furnace is done directly, and the recovery period should preferably be less than one hour. In this way, the cooling down of the electrode is minimised, and so are the thermal stresses.

#### **2-7-4-3    *Medium duration shutdowns***

From 4 to 12 hours is considered a period of medium duration for a shutdown. If such a shutdown is scheduled and prepared in advance (as it preferably should be), it is recommended to reduce the electrode current by 10% for the 48 hours prior to the shutdown, to reduce the 'shock' on the electrode. Slipping should be performed as normal during this period (Asphaug & Innvær, 1997; Innvær *et al.*, 1986).

During the shutdown period, it is recommended to cover the exposed part of the electrode with some kind of thermal isolation, usually the charge (feed material) of the furnace. After the shutdown, recovery should start at 70% of the normal current. This will give 50% of the normal heat generation ( $R \cdot I^2$ ) in the electrode. After that, the current may be increased gradually for some hours, until the full current is reached (Asphaug & Innvær, 1997).

The larger the electrode diameter is, the longer the recovery time that is required. As a rule of thumb, the current can be increased from 70% to 100% of its full value by a rate of 2.5 kA/h, independent of the electrode diameter (Asphaug & Innvær, 1997).

#### **2-7-4-4    *Long duration shutdowns***

Shutdowns of more than 12 hours are considered ‘long duration’ stops. 48 hours before a shutdown (which is scheduled to last for 12 hours or more), the current should be reduced to 80% of its full value. For the last eight hours prior to the shutdown, a gradual reduction from 80% to 70% is recommended (Asphaug & Innvær, 1997; Innvær *et al.*, 1986).

After the shutdown, recovery should start at 50% of full current, which means 25% of the heat generation. From this level on, the current is increased gradually. The larger the electrode diameter is, the longer recovery time is required. As a hand rule, the current can be increased from 50% to 100% of its full value by 1.5 kA/h, independent of the electrode diameter (Asphaug & Innvær, 1997).

## **2-8        Important characteristics of Söderberg electrodes**

Richards (1920) stated that “Baked carbon taken from the furnace end of a Söderberg electrode is dense, hard and of the ordinary quality of previously backed electrodes. It shows in this respect no difference from ordinary carbon electrodes”. This implies that the characteristics of a fully baked Söderberg electrode are the same as for pre-baked graphite electrodes. The most important of these characteristics are electrical conductivity, mechanical strength and thermal conductivity (Chung, 2002; Karagoz *et al.*, 1994). Additionally, the electrode should be corrosion-resistant/chemically inert.

### **2-8-1        *Mechanical properties***

A Söderberg electrode has to be strong enough to withstand different mechanical forces acting on it. These include compressive, flexural and tensile forces (Kortovich & Micinski, 1985; Miller *et al.*, 2010). When the Söderberg electrodes are in operation, they are subjected to a great deal of stresses, so to minimise the chances of electrode breaks (see Section 2-9), they have to have good mechanical properties i.e. does not break easily (Bullón & Pérez, 2007).

### **2-8-2        *Electrical properties***

The main purpose of Söderberg electrodes is to conduct electricity from the contact shoes to the furnace itself. Therefore, the process of producing the Söderberg electrode was designed to do this as efficiently as possible.

### 2-8-3 *Thermal properties*

#### 2-8-3-1 *Thermal conductivity*

As the temperature of a furnace can be in excess of 2 500 °C, the Söderberg electrode needs to be good at conducting the heat to the electrode's interior. This needs to be as quick as possible to prevent breaks that result from a temperature variation from the outside to the inside of the electrode.

#### 2-8-3-2 *Thermal shock resistance*

During furnace shutdowns, the electrode will cool down. This cooling process will be different for different parts of the Söderberg electrode, which could result in cracking of the electrode (Section 6-4). It is consequently very important that the Söderberg electrode has a very good thermal shock resistance.

### 2-8-4 *Corrosion resistance and chemical inertness*

Chemical and/or electrochemical reactions will take place, especially as the casing and fins burn away. However, that is only the outermost part of the electrode, and it should be designed in such a way that as soon as the casing is gone, this kind of corrosion (e.g. oxidation of the carbon) should be at a minimum. If this is not done, it will damage the Söderberg electrode.

## 2-9 **Electrode breaks**

Even if it is infrequent and/or an isolated occurrence, most smelting furnaces will experience an electrode break of one sort or another at some stage (Innvær *et al.*, 1995; Ord *et al.*, 1995). From an electrode management perspective, this is the most serious problem that can be encountered (Arnesen *et al.*, 1979). If a breakage occurs on one electrode in a three-electrode furnace, it will have the immediate effect of reducing the total furnace power by half (Ord *et al.*, 1995). Generally, there are two main kinds of electrode breaks, i.e. soft/green breaks and hard breaks.

### 2-9-1 *Soft electrode breaks*

The term soft/green electrode breaks refers to those kinds of breaks high up in the electrode where the electrode paste has not fully baked yet. During this kind of electrode break, soft/liquid paste can spill into the furnace and cause an explosion due to the paste being exposed to a very high temperature. For this reason, even though this type of break seldom occurs, it is considered more dangerous than hard breaks (Asphaug & Innvær, 1997; Beukes *et al.*, 2013; Beukes *et al.*, 2012; Innvær, 1989, 1992; Innvær *et al.*, 1995).

### 2-9-1-1 Causes

The first reason for soft breaks is faulty casing construction, as described in Andersen (2001), where window spacing in the fins was inconsistent and, at times, spaced too far apart. This caused the baked part of the electrode to 'fall' out of/through the casing and caused liquid/soft paste to run into the furnace. Furthermore, poor welding of the metal casing and the casing fins, as shown in Figure 2.14, could cause a hole to be arced into this casing, which will also result in liquid/soft paste to run into the furnace (Andersen *et al.*, 2001; Beukes *et al.*, 2013; Innvær *et al.*, 1984).

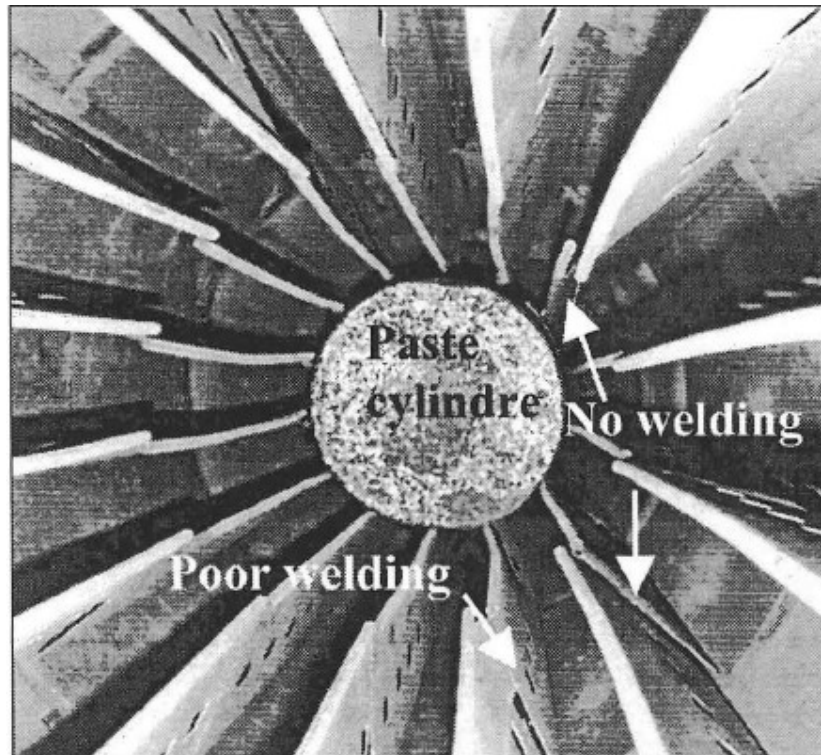


**Figure 2.14** A Søderberg electrode as seen from above. This photo shows some fins that were welded together very poorly, as well as some that were not welded at all (Andersen *et al.*, 2001).

The second possible cause for soft breaks is the inconsistent melting of the electrode paste. If the electrode paste does not fully melt and fill the whole casing, including through the windows of the fins, it will create weak spots that could cause the electrode to break (Innvær, 1989, 1992; Larsen, 2012).

The third, and more common, reason for soft breaks, is when the baking isotherm goes below the contact shoes (Beukes *et al.*, 2013), and the steel casing cannot carry the applied electrical current. This happens when the slipping rate is too high compared to the current/heat generation of the specific electrode or furnace operation or if long slips (1 to 2 meters) occur (Asphaug & Innvær, 1997; Larsen, 2012). The soft/liquid paste does not contribute to the mechanical strength, and if the baking

isotherm falls below the contact shoes, the whole electrode is held up by only the steel casing (Beukes *et al.*, 2013).

#### 2-9-1-2 *Prevention*

Because soft breaks can be so disastrous, smelters are usually very strict in the management of electrodes. All prevention of soft breaks starts with the casing. If the casing is designed wrong, no amount of management will prevent soft breaks. The correct design should be used for the specific furnace it is used in. This also includes the window spacing in the fins (Andersen *et al.*, 2001). It is also important that the casings are welded together properly and solidly without any holes. This implies that quality control checks should be performed regularly to find these kinds of problems before it is too late.

Next, the melting and baking conditions of the electrode paste need to be addressed. The temperature in the electrode casing needs to be high enough (80 °C) to allow the paste to flow and fill the whole casing and the casing windows (Fitt & Howell, 1998; Innvær, 1989, 1992). If the temperature in the casing is too low, either a fan with heaters should be installed, or, if it is already installed, the temperature should be increased to facilitate the melting process (Andersen *et al.*, 2001).

Lastly, the slipping rate needs to be managed. To manage the slipping rate, the position of the baking zone is needed to be known. This is done through knowing the relationship between the electrode current and the slipping rate (Asphaug & Innvær, 1997; Innvær, 1989, 1992; Innvær *et al.*, 1986). Thermocouples can also be installed to measure the temperature isotherms more accurately (Skjeldestad *et al.*, 2015). The combining of such temperature measurements with mathematical modelling can be used to effectively prevent soft breaks (Innvær, 1989, 1992; Innvær *et al.*, 1995).

#### 2-9-2 *Hard electrode breaks*

Hard electrode breaks or the breakage in the baked electrode part of the electrode are, under normal conditions, the most common problem that electrodes experience (Innvær, 1980; Innvær, 1989, 1992; Innvær *et al.*, 1984; Innvær *et al.*, 1995). Although they are not as dangerous as a soft break, they will cause instability in the furnace that can cause soft breaks or further hard breaks in the future. Figure 2.15 shows an electrode break during furnace operation. The shortened electrode and the electrode stump in the furnace will disrupt the furnace operation until the stump is removed or consumed, and the electrode is regrown.



**Figure 2.15** A hard electrode break during furnace operation (Nelson & Prins, 2004).

#### 2-9-2-1 Causes

Approximately 90% of hard breaks are connected to furnace shutdowns (Innvær *et al.*, 1995), which cause thermal stresses, as explained in Section 2-7-4-1. These shutdowns can be unforeseen or pre-planned ones (Innvær, 1989, 1992; Innvær *et al.*, 1984; Innvær *et al.*, 1986; Innvær *et al.*, 1995; Larsen, 2012). Unforeseen shutdowns can occur due to power supply problems or accidents, and/or furnace problems that force the plant to shut down. Pre-planned shutdowns happen as a result of maintenance. If the furnace has a high current density before shutdowns (planned or unforeseen), it will increase the chances for hard breakages. Furthermore, multiple short shutdowns over the course of a short time will increase the chances for hard breaks, even if these shutdowns do not last longer than 3 to 4 hours (Innvær *et al.*, 1984; Innvær *et al.*, 1986). Power fluctuations will result in compounded thermal stresses in the electrode and will contribute to breakages.

If the shutdowns and/or power fluctuations are not taken into account, it can cause the electrode to be under-baked. If a soft break does not happen, the electrode can break as it reaches the hot furnace bed. Conversely, if the operators overcompensates and over-bakes the electrode, the same may occur.

Other causes that can contribute to hard breakage include running a furnace with a higher current density than it was designed for (Innvær *et al.*, 1984; Innvær *et al.*, 1986). This is usually done to increase the production rate (Innvær *et al.*, 1995). Mechanical wear can cause pieces or sections of the electrode to break off, especially on the tip. The electrode paste can segregate and cause weak

places in the electrode. Figure 2.16 shows two examples of paste segregation between aggregate and paste.



**Figure 2.16 (a)** An electrode stub. There is evidence that larger aggregate segregated from the finer aggregate/pitch binder at the electrodes external boundary (Nelson & Prins, 2004), **(b)** a close-up of another electrode showing significant segregation between aggregates (Nelson & Prins, 2004).

Lastly, if electrode casings are open at the top and dust can fall into the casings or the paste blocks are covered in dust, it can cause a discontinuity layer in the electrode. This layer will be a weak place in the electrode and serve as a place for a hard break to occur (Innvær *et al.*, 1984; Nelson & Prins, 2004).

Figure 2.17 shows the sheer line (how the break looks) of four different kinds of hard breaks. This can help to identify why a break occurred. Although it is not a definitive explanation, it can be a starting point in the search for the cause of a break (Nelson & Prins, 2004).

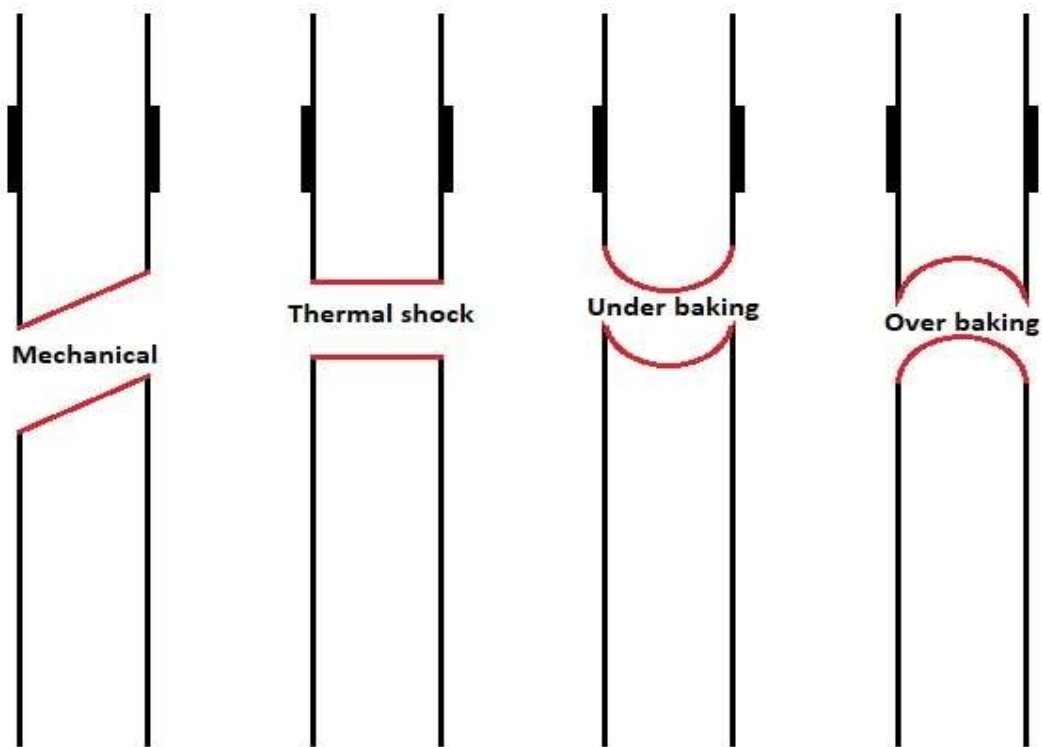


**Figure 2.17** The characteristic hard electrode break edges and their possible main causes (adapted from Nelson & Prince, 2004).

#### 2-9-2-2 Prevention

One cannot prepare for an unforeseen shutdown; therefore, operators can only control how the furnace will start-up after the shutdown. If the shutdown is less than 3 to 4 hours, the furnace can start normally as the stresses in the electrodes are usually at a minimum, and breaks will occur less often (Innvær, 1992; Innvær *et al.*, 1986). If the shutdown lasts longer than 4 hours, the start-up sequence for that specific furnace must be used. It should take between 9 and 12 hours (depending on the design specifications) to get the electrodes to full power. This is done to relax the stresses built up during the shutdown (Andersen *et al.*, 2001; Innvær *et al.*, 1995; Skjeldestad *et al.*, 2015).

For a planned shutdown, the furnace's current should be reduced by 10 to 20 % for 1 to 2 days before the shutdown. This is done to lower the thermal stress due to temperature reduction in the furnace (Innvær *et al.*, 1986). Once the furnace is shut down, the electrode should be slipped 5 to 30 cm, and the electrode should be covered with the hot charge mix to prevent heat loss and reduce stresses (Larsen *et al.*, 2013). However, thermal stresses will occur during shutdowns – there is no preventing it. Effective electrode management and good quality control on materials and equipment can reduce the chances that these stresses turn into hard breaks (Innvær, 1989).

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## Chapter 3

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### Obtaining and sampling the case study Söderberg electrode

In this chapter, it is discussed how the samples for this study were obtained and prepared. In Section 3-1, the process of obtaining the case study Söderberg electrodes and associated samples from the smelter is described. This includes the shutdown of the furnace and the removal of the electrodes from it, as well as the drilling out of core samples from the electrodes. In Section 3-2, the preparation of these core samples is explained. This includes Section 3-2-1 and 3-2-2, wherein the cutting of disks from the core drill samples and drilling of cylindrical sample pellets from these disks are discussed, respectively.

#### 3-1 Obtaining the electrode

In 2013, a large ferrochrome producer decided to rebuild two (named Furnace 1 and Furnace 3) of their operational submerged arc furnaces (SAFs). Both were circular SAFs with three Söderberg electrodes each, placed in a triangular and equally spaced configuration, as indicated in Figure 3.1. This figure also shows the numbering of the electrodes as well as the position of the tap hole that is used to drain (i.e. drain the melted slag and metal) the furnace. The delta marked in the figure refers to the closest spacing between the three electrodes, where they face one another. Furnace 1 (F1), with a 1.3 m electrode casing outer diameter, was shut down on 30 June 2013, and Furnace 3 (F3), with a 1.4 m electrode casing outer diameter, on 31 July 2013. The candidate will refer to both furnaces, although only Electrode 2 from F3 was sampled for this study.

Before the shutdowns, both SAFs were melted down completely (i.e. no new raw materials were loaded for at least 16 hours) and the furnace was drained to remove as much as possible of the liquid metal and slag. After the furnace was drained, the electrodes were picked up to the 500 mm (0.5 m) electrode regulating position (i.e. moving the entire column up or down, without slipping the electrode), where after the furnace was left to cool down for five days. After this period, the furnace was loaded again with the same raw material mixture used during regular operation, to prevent the electrodes from falling over once cut off.

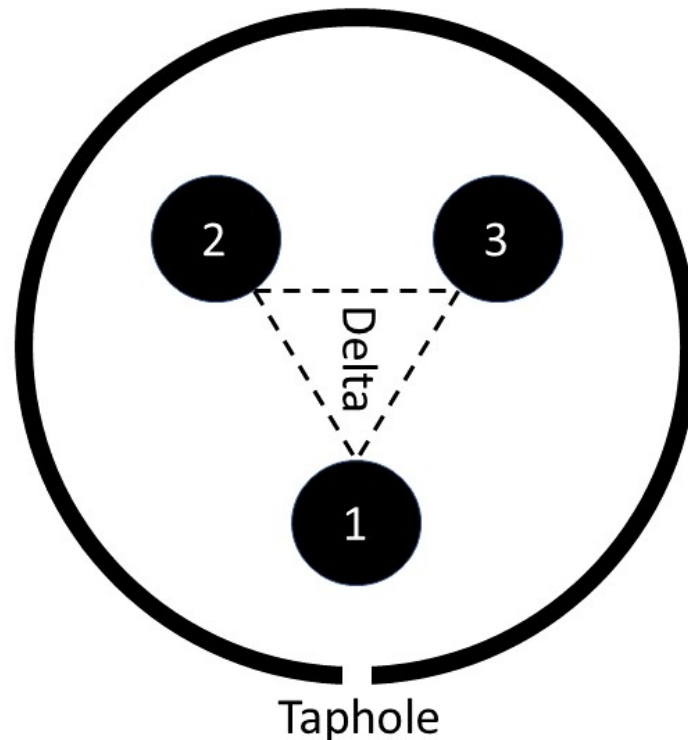


**Figure 3.1** Diagram from a top view indicating the electrode numbering of the SAF from which the case study electrode was removed.

It was decided to cut off all six electrodes (three electrodes for each SAF). Figure 3.2 shows the machine used to cut off the electrodes. It used diamond-coated wire/cable that spins around two pulleys, as shown in Figure 3.2. Water was sprayed onto the diamond-coated wire/cable to protect it from overheating.

For F1, Electrode 1 was cut approximately 1.2 m below the contact shoes, while Electrodes 2 and 3 were cut approximately 1 m below the contact shoes. The electrodes of F3 were cut closer to the contact shoes than those of F1. Electrode 2 (which served as the case study electrode) of F3 was cut approximately 0.5 m below the contact shoes, as indicated in Figure 3.3. Electrodes 1 and 3 were cut approximately 1 m below the contact shoes.



**Figure 3.2** Diamond-coated wire saw used to cut off the electrodes. This specific picture was of Furnace 1.



**Figure 3.3** Photo indicating how Electrode 2 from Furnace 3 was cut off, approximately 0.5 m below the contact shoes with a diamond-coated wire saw.

The cutting time per electrode ranged between 45 and 110 minutes. However, the cutting equipment had to be moved from one electrode to the other. Therefore, the whole process took approximately seven hours per SAF.

Figure 3.4 shows the electrodes of F1 after they had been cut off. They were standing upright thanks to the reloading of the furnace with raw materials before the cutting started. It is noted that Electrode 1 (leftmost electrode in Figure 3.4) of F1 broke apart along the casing fin joints during the cutting process. After the cutting was finished, the electrode stumps still attached to the contact shoes were picked to the top electrode regulating position. This was done to make room to remove the cut-off portions of the electrodes. Figure 3.5 shows the mechanism used to pull the electrode pieces from the piles of raw materials (used to keep them upright) and remove it from the bed of the SAF. A 10-ton air hoist was used for this purpose.



**Figure 3.4** Upright, post-cut electrodes from Furnace 1 that is being held up by the raw material mixture.



**Figure 3.5** A 10-ton air hoist being used to remove an electrode from a furnace.

Figure 3.6 shows the diagonal cross-section of Electrode 3 from F3 that was cut off approximately 1.0 m below the contacts shoes. For each of the cut electrodes, the side facing the delta was marked. The cavities/slots where the fins used to be and the thermal couple pipes can clearly be seen. The cracks running through the whole electrode either formed during operation, or formed as a result of the stresses during the cooling down process, or it happened during the cutting and removal of the electrodes from the SAF. These kinds of cracks were observed in all of the electrodes.

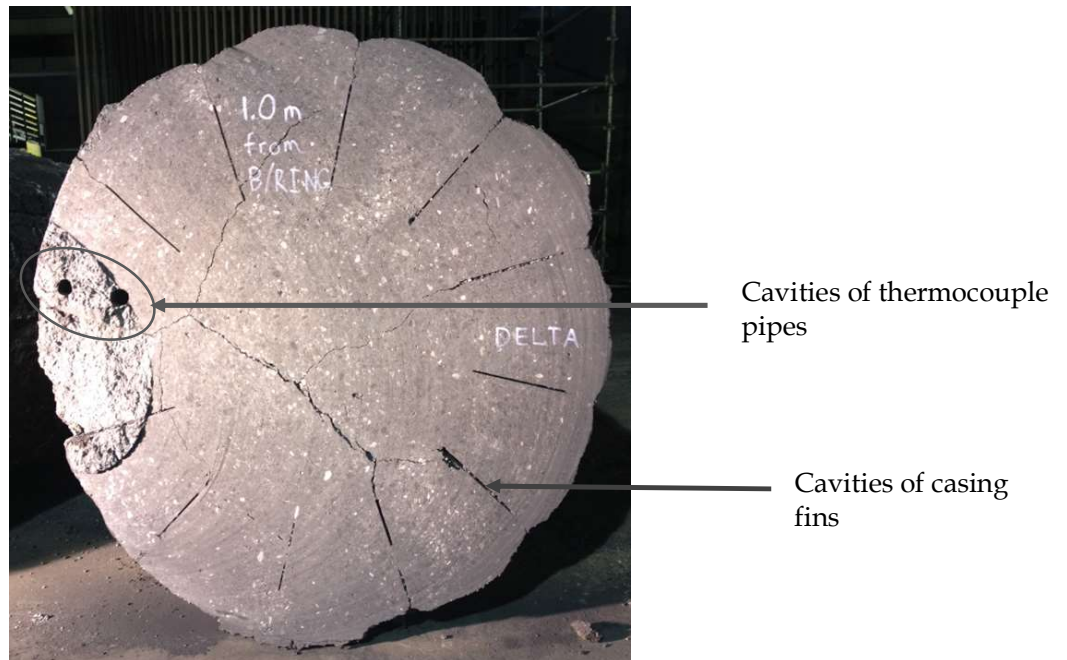


**Figure 3.6** Cross-section of Electrode 3 from Furnace 3 that was cut off with a diamond-coated wire saw approximately 1.0 m below the contact shoes. The arrows indicate cavities where the casing fins and thermocouple pipes used to be.

After Electrode 2 of F3 was removed, 100 mm (outer diameter) core samples were drilled out from it. This core drilling was done from the outside towards the delta side, straight through the electrode. The core samples were drilled out at lengths of 0.20, 0.65, 1.14, 1.64, and 2.20 m below the plane at which the electrode was cut off. Because the electrode was cut off at 0.5 m below the contact shoes for Electrode 2, the middle point of each core sample was 0.70, 1.15, 1.64, 2.16, and 2.70 m below the contact shoes.

Figure 3.7 presents an example of one of the above-mentioned 100 mm (outer diameter) core samples. As is evident, the core sample was broken at a number of places. This could be due to the drilling process and/or the cracks observed in Figure 3.6.



**Figure 3.7** An example of a 100 mm outer diameter core drill sample, collected by core drilling through the entire diameter of the electrode, from the outside towards the delta side of the electrode.

## 3-2 Processing the core samples

### 3-2-1 Obtaining sample disks

The core samples (Section 3.1) had to be cut into manageable sizes. To do this, a Metabo CS23355L 355 mm 2300 W cut-off grinder was modified. Figure 3.8 shows the new, prior to any modifications, cut-off grinder. This grinder was designed to hold and cut metal, and not relatively brittle carbon composite samples, such as core drilled samples of a Söderberg electrode.



Original metal clamps

**Figure 3.8** Unmodified Metabo CS23355L 355 mm 2300 W cut-off grinder. The arrow indicates the original metal clamps.

The first required modification was to find a way to securely hold a core sample in place during cutting. The standard metal clamps, indicated in Figure 3.8, would not work, because it would require too much clamping force on the rounded sides of the core sample to keep it secure. Therefore, thick (30 mm) polyvinyl chloride (PVC) was cut into triangles, which were fitted onto the adjustable side of

the original metal clamping plate. The original stationary metal clamping plate was covered by a flat 30 mm PVC piece. This configuration (i.e. triangles on one side and flat on the other), as presented in Figure 3.9a, was used to clamp down the rounded core sample. Secondly, a mechanism needed to be installed for the cutting of sample disks, with consistent thickness (10 mm), from the rounded core samples. This was achieved by installing an adjustable bolt on a metal plate, fitted to the base of the cut-off grinder, as indicated in Figure 3.9b. The adjustable bolt acted as a stopper for the core sample to press against. This ensured that 10 mm thick sample disks could be cut relatively accurately and consistently from the core samples.

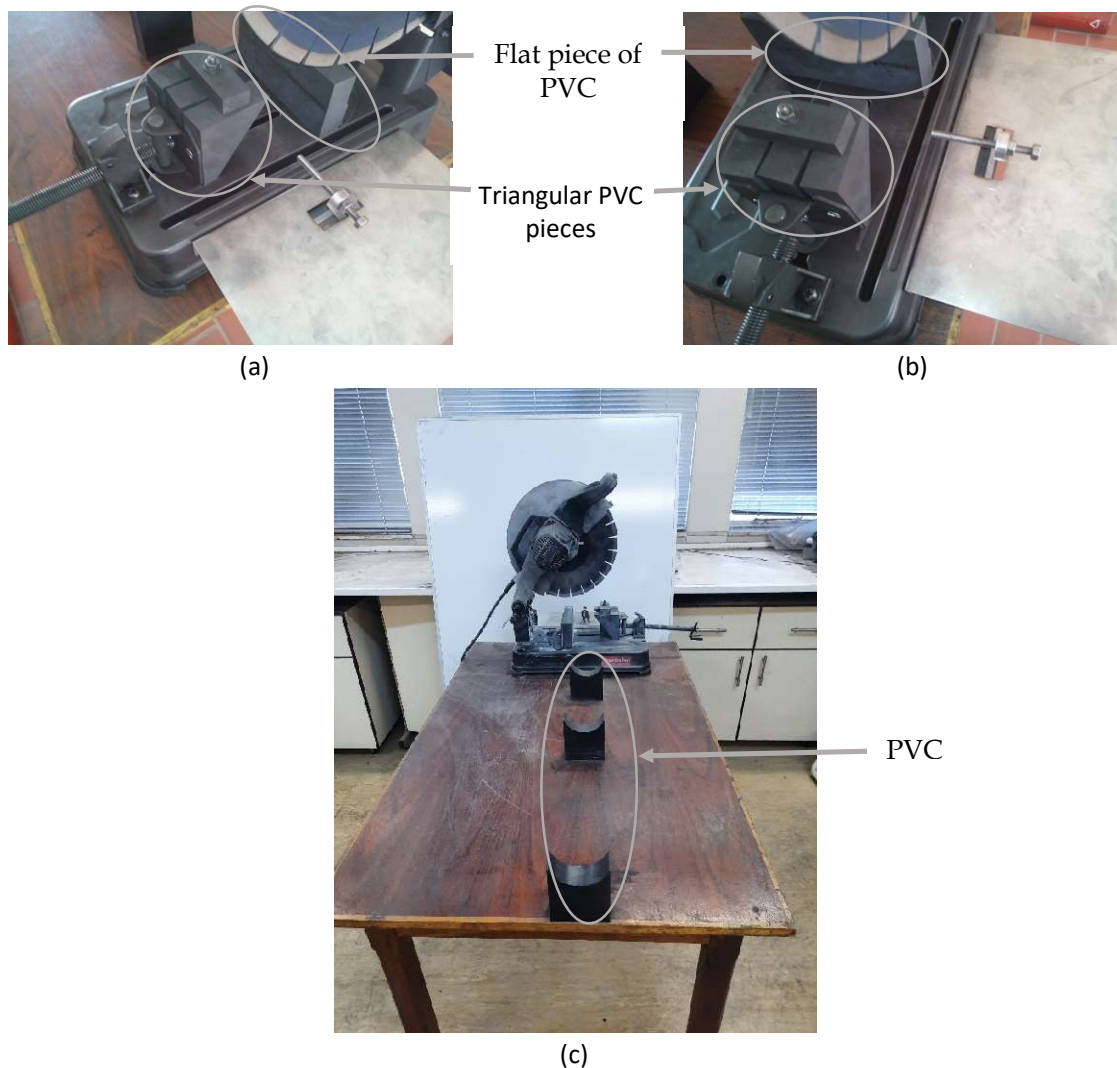


**Figure 3.9** (a) Clamp modifications to hold the round core samples. The top arrows indicate the flat 30 mm PVC piece fitted to the original stationary metal clamping plate. The bottom arrows indicate the triangular PVC pieces fitted onto the adjustable side of the original metal clamping plate; (b) Body modifications to adjust the thickness of the cut. (c) The table on which the cut-off grinder was mounted, onto which cradles were fitted to hold the core samples.

The third modification to the standard cut-off grinder intended only for cutting metal, was that a masonry cut-off blade needed to be fitted. A Tork Craft 350 mm Professional Series, wet/dry, diamond blade ([http://www.torkcraft.com/index.php?route=product/product&path=1019\\_1326\\_1328&product\\_id=11010](http://www.torkcraft.com/index.php?route=product/product&path=1019_1326_1328&product_id=11010), accessed 06/11/2020) was used to cut the core samples. The original bushings, onto which metal cut-off grinding blades could be fitted, were replaced with fit-for-purpose bushings to accommodate the aforementioned masonry blade. The fourth modification was that precise lathe turned; tight-fitting bushings were installed in the pivot point of the cut-off grinder to ensure accurate cutting. Lastly, the cut-off grinder was mounted on a wooden table to hold it steady, while the cutting was being done. Cradles made from PVC, as indicated in Figure 3.9c and Figure 3.10a, were fitted on the table. These cradles were attached in-line and the same height as the grinder table, to hold the core samples perpendicular to the cut-off blade.

No water was used to cool the cutting disk and core sample during cutting, because the candidate did not want to contaminate and/or alter the S  derberg electrode matrix. For example, oxidation of the remnants of the casing fins that melted away below the contact shoes would be enhanced by water. Because the cutting process generated heat, it was stopped from time to time for the blade and core sample to cool down. With no water used during this cutting process, a great deal of dust was produced. Therefore, the cutting was done outside, and the candidate wore safety goggles and a respiratory mask.



**Figure 3.10** (a) An example core sample sitting on the cradles, while being cut; (b) Broken core sample being held together with the modified clamp from the cut-off grinder.

Because some core samples were broken, it was challenging to keep all the pieces together, as shown in Figures 3.10a and b. The broken pieces were, for instance, clamped together as shown in Figure 3.10b, to ensure that the matching parts of sections stayed together.

The photos in Figure 3.11 (where a is the 0.70, b the 1.64, c the 2.16, and d the 2.70 m below the contact shoes core samples) are all the delta-sided edge of the respective core samples. Each of these

edge pieces had a cavity/slot in them that was part of the cavities leftover from the casing fins that partially melted away (Figure 3.6). No such cavity was observed on the 1.15 m below the contact shoes core sample. The aforementioned cavities contained solidified metal (likely remnants from the fins that melted) as indicated in Figure 3.12a (core sample 0.7 m below contact shoes), and Figure 3.12b (core sample 1.64 m below the contact shoes). It was therefore impossible to cut sample disks from the core samples closer to the delta side of the electrode). What was possible was to cut two small rectangular samples from the 1.64 m core sample with a hand-held angle grinder that had a masonry disk installed. One sample, on the direct edge of the core sample, was 35 x 35 mm in size, whereas the other sample, 90 mm from the same edge as the first sample, was 22 x 35 mm in size.

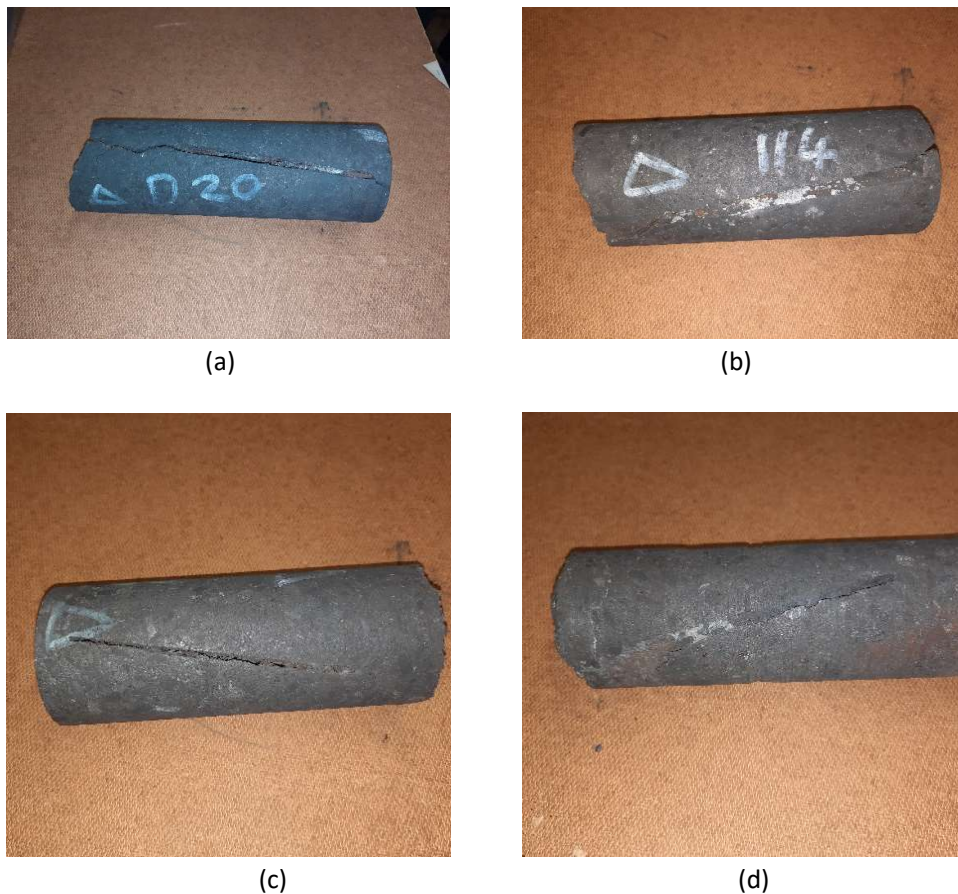


**Figure 3.11** The delta edge of core samples at (a) 0.7 m, (b) 1.64 m, (c) 2.16 m, and (d) 2.70 m below the contact shoes indicating cavities with embedded metal (likely remnants of the melted casing fins).

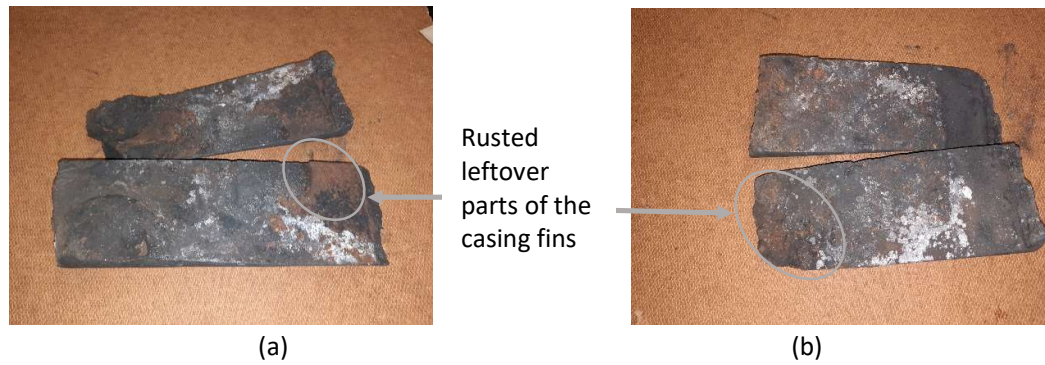


**Figure 3.12** The delta edge of core samples at **(a)** 0.7 m, **(b)** 1.64 m below the contact shoes broken open to show leftover rusted part of the casing fins.

In Table 3.1, the total lengths of each core sample are presented. The table also shows the distance of the last cut from both edges (delta and non-delta sides). As is evident from this data, the last cuts on the non-delta side of the core samples were much closer to the core sample edges than those of the delta side of the core samples. This was due to the partially-metal-filled cavities discussed in the previous paragraph.

| Core sample length below the contact shoes (m) | Core sample length (m) | Last cut closest to the delta side (m) | Last cut closest to the non-delta side (m) |
|------------------------------------------------|------------------------|----------------------------------------|--------------------------------------------|
| 0.70                                           | 1.316                  | 0.285                                  | 0.007                                      |
| 1.15                                           | 1.33                   | 0.045                                  | 0.03                                       |
| 1.64                                           | 1.292                  | 0.265                                  | 0.016                                      |
| 2.16                                           | 1.25                   | 0.245                                  | 0.02                                       |
| 2.70                                           | 1.258                  | 0.541                                  | 0.015                                      |

**Table 3.1** Total core sample lengths, as well as distances of the last cut from both edges.

Figure 3.13 shows examples of the round disks that the candidate obtained after cutting the core samples. These disks were 100 mm in diameter and 10 mm thick. Each disk was sealed in an individual zip lock bag and labelled to keep track of its position within the electrode column.



**Figure 3.13** Example disks, which were 100 mm in diameter and 10mm thick, as obtained from the core samples.

An observation made during the cutting of the sample disks was that it felt as if cutting became easier as one moved closer to the delta side of the electrode. It almost felt as if a self-lubricating process was occurring, which indicated that the delta side of the electrode may have been more graphitised than the non-delta side of the electrode. It is well known that graphite is a dry lubricant (Shaji & Radhakrishnan, 2002). Although this observation cannot be quantified or considered as a scientific characterisation of the case study electrode, it was noted in order to correlate/compare with the more detailed results presented later.

### 3-2-2 *Obtaining cylindrical sample pellets*

The sample disks described in Section 3-2-1 were easy to work with within a laboratory environment, which is a prerequisite for a sample (Skoog *et al.*, 2004). However, if characterisation experiments were conducted on these disks, only a limited number of tests would have been possible, due to a finite amount of core drilled material. Most characterisation experiments also did not require large pieces of the electrode. Therefore, smaller cylindrical sample pellets were drilled out of each disk.

In order to obtain cylindrical sample pellets, a 12 mm outer diameter tile core drill bit from Tork Craft, as presented in Figure 3.14, ([http://www.torkcraft.com/index.php?route=product/product&path=794\\_1829\\_1830&product\\_id=9574](http://www.torkcraft.com/index.php?route=product/product&path=794_1829_1830&product_id=9574), accessed 06/11/2020), was fitted to a drill press. This enables

8 x 10 mm (8 mm diameter and 10 mm high) cylindrical sample pellets to be drilled out of the disks, as indicated in Figure 3.15a. The condition of each disk determined how many pellets were drilled out of that specific disk. For example, the top part of the disk shown in Figure 3.15a was broken off. This 'defect' limited the number of cylindrical sample pellets obtained for it. Typical 8 x 10 mm cylindrical sample pellets are presented in Figure 3.15b.



**Figure 3.14** 12 mm tile core drill bit used to obtain 8 x 10 mm cylindrical sample pellets ([http://www.torkcraft.com/index.php?route=product/product&path=794\\_1829\\_1830&product\\_id=9574](http://www.torkcraft.com/index.php?route=product/product&path=794_1829_1830&product_id=9574) (visited 06/11/2020)).

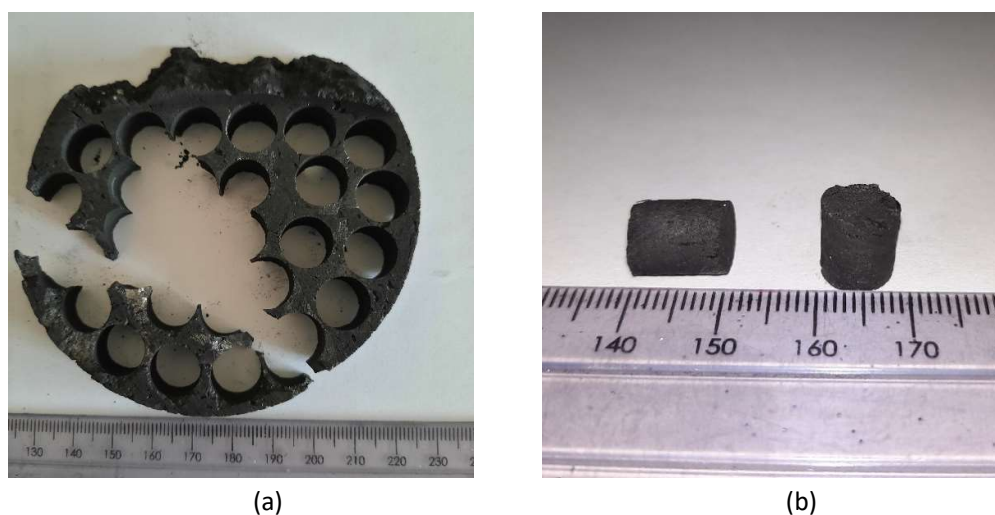


**Figure 3.15** (a) Example disk from which 8 x 10 mm cylindrical sample pellets were drilled out; (b) Example 8 x 10 mm cylindrical sample pellets.

One of the density determination methods (Section 4-3-1) required larger samples than the 8 x 10 mm cylindrical sample pellets described thus far. Therefore, from some of the disks, 18 x 10 mm cylindrical sample pellets were drilled out, in a similar manner as described for the 8 x 10 mm cylindrical sample pellets. The only difference between the two methods was that a larger tile core drill was used.

For the same reason as indicated for the cutting procedure (Section 3-2-1), no water was used as a coolant during the drilling of the 8 x 10 or 18 x 10 mm cylindrical sample pellets. This prevented possible contamination and/or alteration of the Söderberg electrode matrix. Multiple core drill bits

were used during the drilling. They were swapped at regular intervals to keep them and the disks from overheating. The powder left over as a result of the drilling procedure on a specific sample disk was collected in a glass polytop and labelled, for later use in XRD analysis (Section 4-2). The cylindrical sample pellets, leftover pieces of the disk from which the pellets were drilled, as well as the glass polytops containing the associated powder were each sealed in an individual Ziploc bag. The individual Ziploc bags were then placed in a larger Ziploc bag, to ensure that these related materials (all from the same sample disk) remained together.

Similar to the observation made during the cutting of the sample disks, it also felt as if drilling of the cylindrical sample pellets became easier as one moved closer to the delta side of the electrode. Again, this indicated possible increased graphitisation on the delta side of the electrode.

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# Chapter 4

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## Experimental methods

In this chapter, the experimental methods are discussed for the different samples prepared in Chapter 3. The primary function of Söderberg electrodes is to conduct electrical current into the smelting zone of the furnace. It is, therefore, fitting that electrical resistivity is the first experimental method that is discussed (Section 4-1). The degree of graphitisation is discussed next (Section 4-2), because it will be compared to that of the electrical resistivity. Next, the bulk density (Section 4-3) and porosity experimental methods are explained. Then, in Sections 4-5 and 4-6, the compressive strength test and thermal shock experimental methods will be discussed. In Section 4-7, scanning electron microscopy and energy dispersive x-ray spectroscopy methodologies are discussed, while the final section (Section 4-8) discusses the statistical handling of data. It was decided to use these techniques since the characteristics they test are considered the most important aspects related to the operation of a Söderberg electrode.

### 4-1 Electrical resistivity measurements

The electrical resistivities of the 8 x 10 mm cylindrical sample pellets (described in Section 3-2-2) were measured at room temperature using a GW Instek resistance precision meter (Model LCR-6020 Series). The meter can be used to measure inductance (L), capacitance (C) and resistance (R) of samples over a wide frequency (10 Hz-20 kHz), resistance (0.99-99.9 MΩ) and voltage range (10.00 mV-2.00 V). As indicated in Figure 4.1a, a two-probe device was manufactured (by the North-West University Instrument Makers) similar to the design presented by (Heikkilä *et al.*, 2015) and connected to the LCR meter. This two-probe device had a bottom and a top probe, which were both turned from solid copper. The stainless steel tightening wheel mechanism (Figure 4.1a) was used to gently tighten a cylindrical sample pellet in-between these copper probes prior to a measurement. A torque wrench was then used to fasten the hex/Allen key located on top of the stainless steel tightening wheel mechanism further. This ensured that each sample was set under the same torque pressure (i.e. 0.5 Nm). The crocodile clamps of the LCR meter were clamped into fit-for-purpose holes drilled in the copper probes. These holes ensured consistent placement of the clamps on the probes, as well as consistent contact area between the clamps and the probes. The frame and connectors that attached the probes to the frame were made of polyvinyl chloride (PVC) plastic, to isolate the frame from the sample. As illustrated in Figure 4.1b, the face of the bottom copper probe on which the cylindrical sample pellet sat had a small rim on the outside, while the corresponding face of the top copper probe

was flat. These shapes were used to ensure that the cylindrical sample pellets were held in-between the two copper probes in a repeatable manner. Both copper probes were also set on a 360° articulating spring mechanism. This kept the ends of the cylindrical sample pellet perpendicular to the probe faces, even if the cylindrical sample pellet ends were not perfectly perpendicular to the cylindrical sample pellet sides.

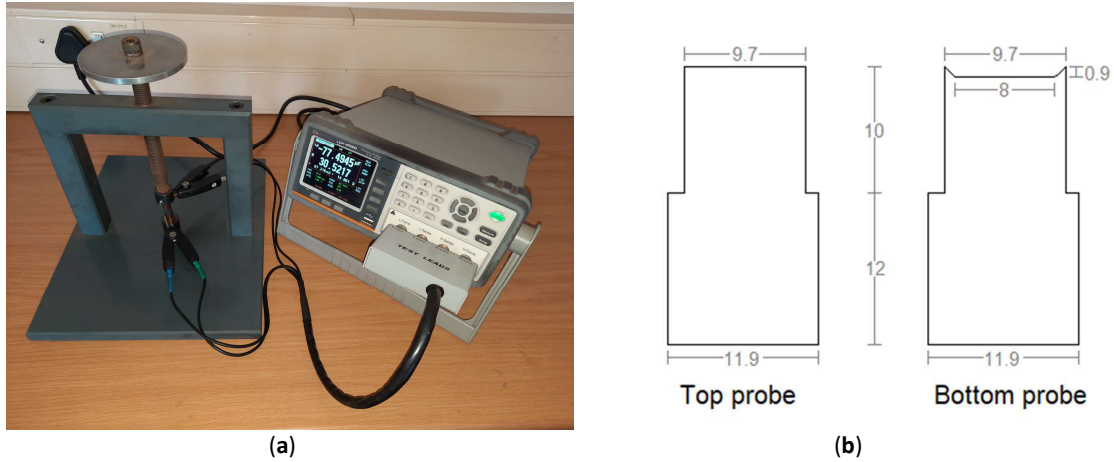


**Figure 4.1** (a) Photo indicating the GW Instek resistance precision meter (Model LCR-6020 Series) and two-probe device, similar to the design presented by (Heikkilä *et al.*, 2015), used to measure the electrical resistivity of the cylindrical sample pellets; (b) Illustration indicating the shapes of the copper probes, in-between which the cylindrical sample pellets were held.

To ensure precision and accuracy, the LCR meter was switched on for 30 minutes and a daily calibration was conducted before measurements commenced. Cylindrical copper, aluminium and stainless steel samples, which had dimensions similar to the cylindrical sample pellets, were measured during calibration. The calibration values were stored by the LCR meter to correct for errors caused by impedance of the experimental setup. All the probes and the calibration samples were cleaned with tartaric acid (0.002 mol/L) before measuring commenced. This was done to remove any oxidation that may have occurred in-between sampling periods. The resistance values of the measured cylindrical sample pellets were calculated from Ohm's law:

$$R = V/I, \quad (4-1)$$

where  $R$  is the resistance ( $\Omega$ ),  $V$  is the voltage across the sample (V), and  $I$  is the measured current (A). Assuming uniform current distribution, the resistivity was calculated by Equation (4-2):

$$\rho = (V.A)/(I.H), \quad (4-2)$$

where  $\rho$  is the material resistivity ( $\Omega.m$ ),  $V$  is the voltage across the sample,  $I$  is the measured current (A),  $A$  is the cross-sectional area of the cylindrical sample ( $m^2$ ), and  $H$  is the height of the sample (m).

## 4-2 Degree of graphitisation

In this study, X-ray diffraction (XRD) was used to determine the degree of graphitisation (DOG). This was done with XRD analysis (Zou *et al.*, 2003). The DOG was calculated with Equation (4-3) (Zou *et al.*, 2003):

$$\text{DOG} = (3.440 - d_{002}) / (3.440 - 3.354) = (3.440 - d_{002}) / 0.086, \quad (4-3)$$

where DOG is the degree of graphitisation (a unit less number ranging from 0 to 1, with 1 indicating fully graphitised and 0 fully un-graphitised), 3.440 is the interlayer spacing of fully un-graphitised carbon (nm), 3.345 is the interlayer distance for pure graphite (nm), and  $d_{002}$  is the interlayer spacing derived from XRD analysis (nm).

The powder generated during drilling of the 8 x 10 mm cylindrical sample pellets (as explained in Section 3-2-2) was used for the XRD. A small amount of silicon metal powder, used as a reference material, was added to the powdered S  derberg electrode material and mixed thoroughly, prior to analysis.

The samples containing the reference material were prepared for XRD analysis with the frontloading method. The samples were packed tight into the sample holders, such as those empty holders presented in Figure 4.2. Figure 4.2a shows the top view, and Figure 4.2b shows the side view of the sample holders. The circular space wherein the sample was packed is 40 mm in diameter and 5 mm deep. The excess sample was removed with a straight-edge glass slide to ensure the sample was level with the top of the sample holder.

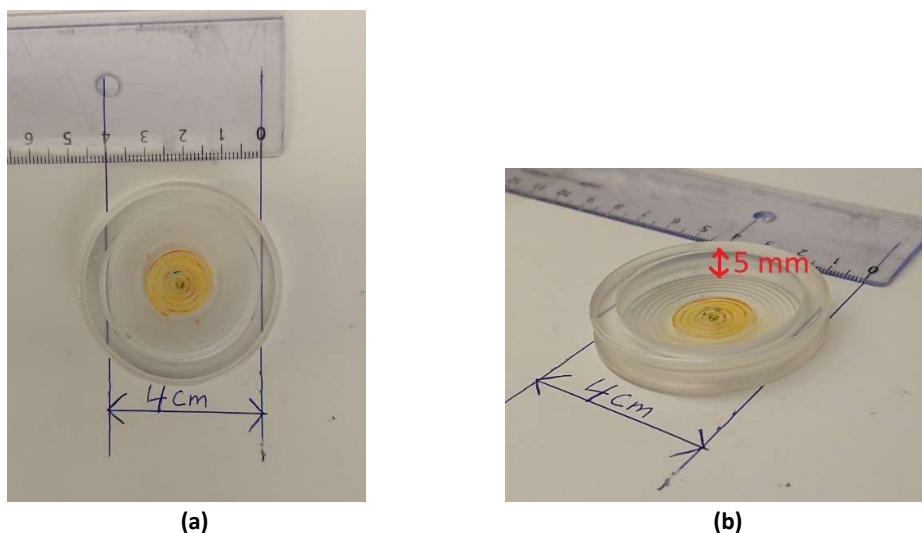


**Figure 4.2 (a)** Top view of the deep front loading sample holder showing the diameter of the sampling space; **(b)** Side view of the deep front loading sample holder showing the depth of the sampling space (<https://www.ntnu.no/wiki/display/imtlab/Frequently+asked+questions+%28FAQ%29+for+the+XRD-lab>, accessed 2020/11/10).

The prepared sample holders were analysed on a Bruker D8 A25 DaVinci X-ray Diffractometer, and LynxEye™ SuperSpeed Detector with xyz-stage mounted and using Cu-K radiation. Figure 4.3 presents a photo of the DaVinci X-ray Diffractometer.

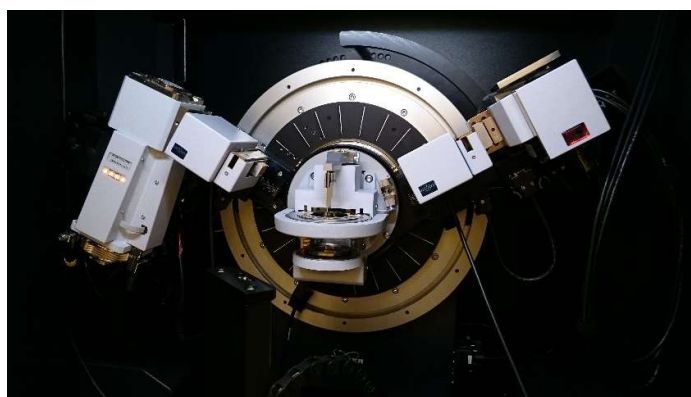


**Figure 4.3** Bruker D8 A25 DaVinci X-ray Diffractometer and LynxEye™ SuperSpeed Detector with xyz-stage mounted and using Cu-K radiation (<https://www.ntnu.no/wiki/pages/viewpage.action?pageId=86728099>, accessed 2020/11/10).

### 4-3 Bulk density determinations

Bulk densities were determined with two different methods, which are subsequently explained.

#### 4-3-1 *ASTM C20-00 method*

Cylindrical sample pellets with 18 x 10 mm dimensions (as prepared in section 3-3-2) were used in these determinations. The samples were first air-dried at 105°C for 3 hours in an oven. After cooling in a desiccator, the samples were weighted (D). They were then saturated by boiling them in distilled water for 2 hours. Thereafter, the samples were weighed in water to get the saturated weight (S). The bulk density was calculated with the following formula:

$$\rho_D = [D/(D-S)(\rho_o - \rho_L)] + \rho_L, \quad (4-4)$$

where  $\rho_D$  is the bulk density of the sample ( $\text{g.cm}^{-3}$ ), D the sample weight after drying, S the saturated sample weight,  $\rho_o$  the density of water (taken as  $0.9982 \text{ g.cm}^{-3}$ ) and  $\rho_L$  the density of air (taken as  $0.0012 \text{ g.cm}^{-3}$ ).

#### 4-3-2 *Gas pycnometer*

A gas pycnometer uses the gas law to calculate the volume of an object. The combined gas law equation is:

$$(P_1 \cdot V_1)/T_1 = (P_2 \cdot V_2)/T_2 \quad (4-5)$$

where P is the pressure, V is the volume, and T is the temperature. It shows the relationship between pressure, volume and temperature for a fixed quantity of gas. Therefore, if the temperature stays the same and the volume, to where the gas is transferred, changes, the pressure will also change.

For a gas pycnometer, the temperature is kept constant ( $T_1 = T_2$ ) so it (T) can be removed from Equation (4-5) and then modified to:

$$V_s = V_c + V_r/[1 - (P_1/P_2)] \quad (4-6)$$

where  $V_s$  is the sample volume,  $V_c$  is the known volume of the empty sample chamber,  $V_r$  is the volume of the reference chamber (known from a prior calibration step),  $P_1$  is the first pressure of the sample chamber (with the sample being tested) and  $P_2$  is the second (lower) pressure (after the expansion of the gas into the sample chamber) of the combined volumes of sample and reference chambers.

An AccuPyc II 1340 gas pycnometer, of which a photo is presented in Figure 4.4, was used. Sørderberg electrode pieces remaining after the 8 x 10 mm cylindrical sample pellets were drilled out of the 10 mm thick disk (Figure 3.13a) were used for this purpose. Each sample was first weighed and then placed in a 35 cm<sup>3</sup> sample holder in the instrument chamber. The chamber was purged five times with 99.99% helium. A known quantity of gas, at a known pressure, was then expanded into the chamber and the new pressure measured. The difference between the initial and final pressures and the known volume of the chamber is then used (with Equation 4-6) to calculate the volume of the sample; and with the sample mass, the sample bulk density could be calculated.



**Figure 4.4** AccuPyc II 1340 gas pycnometer (<https://www.ntnu.no/wiki/display/imtlab/AccuPyc+II+1340>, accessed 2020/11/10).

#### 4-4 Porosity determination with computerised tomography (CT)

The term computerised tomography stems from computerised (with a computer), tomo (to cut), and graph(y) (pictures) (Caldemeyer & Buckwalter, 1999). There are two category types of CT scanners, namely medical CT scanners and industrial CT scanners (du Plessis *et al.*, 2016). Medical CT scanners are used for medical diagnosis of everything from bone and joint problems, to cancer and internal injury detection (<https://www.webmd.com/cancer/what-is-a-ct-scan#1>, accessed 2020/11/24). Industrial CT scanners are used for a wide range of applications. For instance, they are used in the manufacturing and electronic industries for quality control purposes and in analytical applications (De Chiffre *et al.*, 2014). Medical CT scanners usually have a voltage range of around 80-130 kV, whereas industrial CT scanners have a voltage range of around 225 kV (du Plessis *et al.*, 2016; Raman *et al.*, 2013). The reason for the lower voltages for medical CT scans is to lessen the radiation dose (higher radiation dosages can be dangerous) that patients are exposed to (Raman *et al.*, 2013). This is not a problem with industrial CT scanners that only operate on inanimate objects. This higher voltage results

in better quality images, as well as greater penetration power into denser substances (du Plessis *et al.*, 2016).

In a medical CT scanner, the X-ray unit (which carries the X-ray source and the electronic detector) rotates continuously around a stationary patient (De Chiffre *et al.*, 2014). An industrial CT scanner works in a similar, but slightly different manner. In addition to the higher voltages, the other big difference is that, in an industrial CT scanner, the sample rotates while the source and electronic detector stay stationary. Figure 4.5 shows the setup for a Nikon XT H 225 ST. The X-rays travel from the X-ray source (on the left) through the rotating sample (middle) to the detector (right).

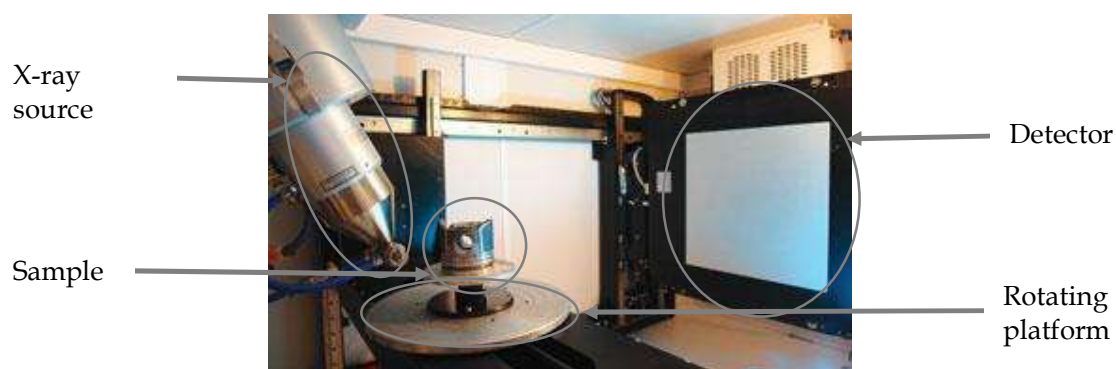


**Figure 4.5** The inside of a Nikon XT H 225 ST. It shows the X-ray source on the left, the sample in the middle on the rotating platform, and the detector on the right (<https://www.nikonmetrology.com/en-gb/product/xt-h-225-st>, accessed 2020/11/10).

During this study, a Nikon XT H 225 ST with a 225kV reflection target source and a Perkin Elmer 1620 detector CT instrument was used. Figure 4.6 shows an example of the outside of one of these instruments, while, as previously stated, Figure 4.5 presents the inside.



**Figure 4.6** A Nikon XT H 225 ST CT instrument (<https://www.nikonmetrology.com/en-gb/product/xt-h-225-st>, accessed 2020/11/10).

Similar to the gas pycnometer density determinations (described in Section 4-3-2), S  derberg electrode pieces remaining after the 8x10 mm cylindrical sample pellets, drilled out of the 10 mm thick disk (Figure 3.13a), were used. The samples were placed in glass polytops stacked six high and wrapped in cellophane to keep them aligned and easy to handle. Each polytop was scanned individually, with 2 000 sample image slices being generated per polytop. From these 2 000 image slices per sample, 10 images, which were evenly spaced (every 160 images between images 200 and 1 800), were used for the actual porosity determinations. An example of a CT image slice is presented in Figure 4.7a. ImageJ (<https://imagej.nih.gov/ij/>, accessed 20/10/2020) was used to analyse the CT image slices. This was done by adjusting the threshold value so that the pores and background were red in colour (Figure 4.7b) in all images. In each image, the boundaries of the solid particle(s) visible in the CT image slice were then manually marked (indicated by the yellow line and white markers in Figure 4.7b). Thereafter, the measurement tool of ImageJ was used to determine the surface area of red pixels within the selected area(s), as a percentage of all the pixels within the selected area(s). This corresponded to the percentage pores in the particular CT image slice.

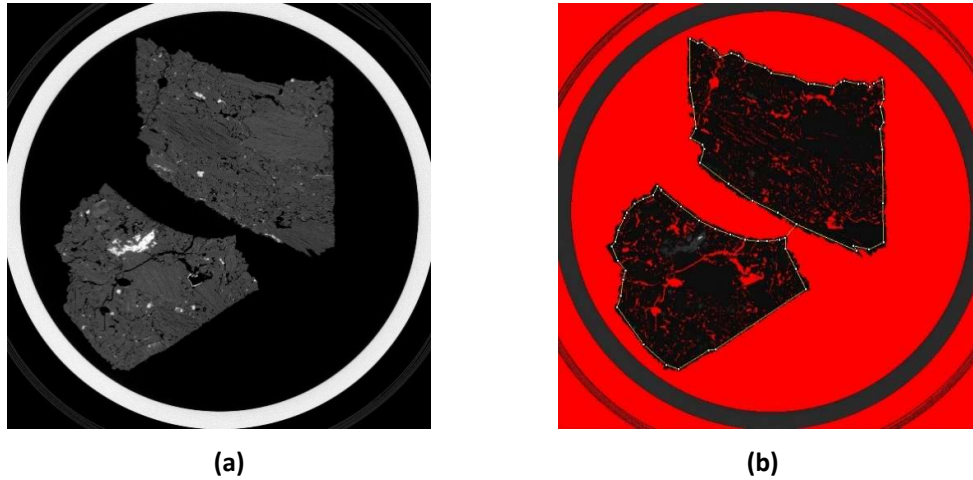


**Figure 4.7** (a) An example of a CT image slice; (b) ImageJ (<https://imagej.nih.gov/ij/>, accessed 20/10/2020) was used to analyse the CT image slices. The threshold value was adjusted so that the pores and background were red in colour. The boundaries of the solid particle(s) visible in the CT image slice were manually marked (indicated by the yellow line and white markers).

## 4-5 Compressive strength test

Compressive breaking strength is defined as the compressive load required to fracture a sample. The compressive breaking strengths of 8 x 10 mm cylindrical sample pellets were determined using an Ametek Lloyd Instruments LRXplus 5 kN strength tester (Figure 4.8). A NEXYGENPlus material test and data analysis software were used to control the instrument and capture the generated data. The speed of the compression plates moving towards one another was kept constant at 10 mm.min<sup>-1</sup> during all compressive strength tests. The 8 x 10 mm cylindrical sample pellets were placed upright in the breaking strength machine (flat sample faces on compression plates as shown in Figure 4.9), and the maximum load to induce breakage of each sample pellet was recorded.



**Figure 4.8** Ametek Lloyd Instruments LRXplus 5 kN strength tester.

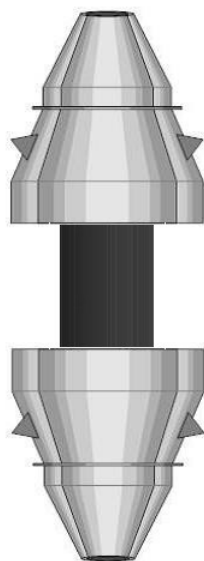


**Figure 4.9** Graphic representation of compression strength test of an 8 x 10 mm cylindrical sample pellet.

## 4-6 Thermal shock

The candidate wanted to determine what the impact of thermal shock (i.e. heating a sample to a very high temperature and rapidly cooling it) would be on the compression strength of the 8 x 10 mm cylindrical sample pellets. This is important because if there is damage caused by the thermal shock, it could help explain how and where electrode breaks (see Section 2-9) occur. An 18kW Lenton Elite (model TSH 18/75/300) tube furnace with a programmable temperature controller (as shown in Figure 4.10) was used for this experiment. Figure 4.11 shows a simple schematic illustration of the furnace setup. Ceramic heat shields were inserted at both ends of the furnace tube to isolate the stable temperature zone. This was done to maintain a stable working temperature. These heat shields also protect the stainless steel caps fitted to the ceramic tubes to seal their respective ends. The stainless steel caps have a gas inlet on the one side of the furnace tube, and a gas outlet on the other. The thermocouple is located at the centre of the stable temperature zone. The furnace is heated by 12 electrodes, which are located adjacent to the furnace tube.



**Figure 4.10** 18kW Lenton Elite (model TSH 18/75/300) tube furnace with a programmable temperature controller.

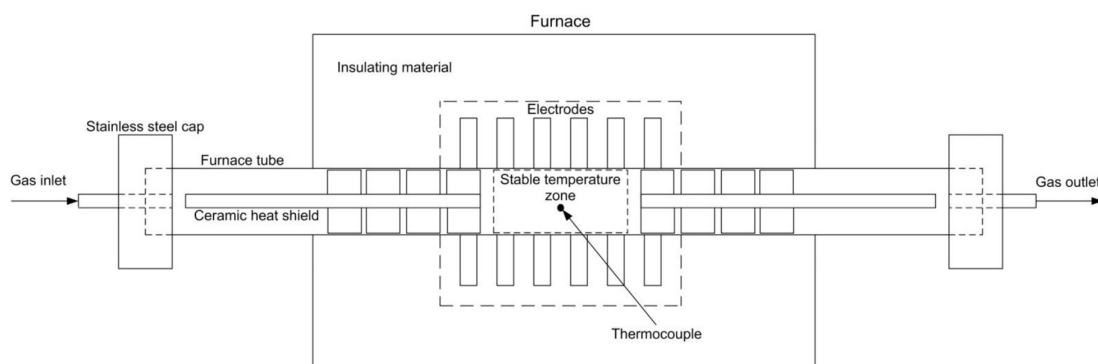


**Figure 4.11** Simple schematic illustration of the furnace setup (du Preez *et al.*, 2018).

Because the electrode was carbon-based and would oxidise (i.e. burn) in the presence of oxygen and high temperature, this experiment had to be conducted in an inert atmosphere. The 8 x 10 mm cylindrical sample pellets were placed in a ceramic boat and positioned in the stable temperature zone. Then the tube (and boat therein) was flushed with N<sub>2</sub> gas (purity and supplier name) at a flow rate of 2 NL per minute for 30 minutes to replace all the oxygen. After the 30 minutes, the gas flow rate was reduced to 1 NL per minute for the remainder of the heating process. The furnace was then heated to 1 500 °C at a rate of 10 °C per minute. The furnace was left to stabilise for 10 minutes at this temperature, where after the boat (with the 8 x 10 mm cylindrical sample pellets) was removed from the furnace and left to air cool until it reached room temperature. Although oxidation of the pellets occurred when removed from the furnace, it was done in this manner to ensure that all pellets reacted and cooled down homogenously. If pellets were removed and placed in an inert atmosphere, the transfer of pellets could potentially result in different levels of oxidation of some pellets. The furnace was turned off and left to cool down to room temperature before the next experiment was conducted.

The compressive breaking strength of these thermally shocked 8 x 10 mm cylindrical sample pellets was then tested, as indicated in Section 4-5.

## 4-7 Scanning electron microscopy (SEM)

### 4-7-1 *Sample preparation for energy dispersive x-ray spectroscopy (EDS)*

Electrode samples obtained from 0.01, 0.6, and 1.28 m from the delta side of the 1.64 m core sample were subjected to energy dispersive x-ray spectroscopy (EDS) in order to determine the chemical composition and the degree of oxidation. Samples were also retrieved from the fin leftovers from the delta side (see Figures 3.11 and 3.12). These samples were set in resin (Technoresin), and left to cure and harden according to the specifications of the manufacturer.

### 4-7-2 *Sample polishing*

The square Söderberg electrode samples (described in Section 3-2-1) and resin-set samples (Section 4-7-1) were polished using a LECO SS20 Spectrum System Grinder polisher. It operated at an average speed of 300 rpm for grinding and 150 rpm for polishing. Two 250 mm Struers MD Piano (one was 220 grit and the other was 1200 grit) magnetic discs were first used for the rough grinding and levelling. A continuous flow of water was used as a lubricant, as well as for washing away the grind particles. After that, the Struers MD Allegro, Dac, and Nap magnetic discs with the Struers DiaDuo-2 9, 3, and 1 µm polishing compounds were used for the polishing steps.

### 4-7-3 *SEM imaging and EDS analysis*

Backscattered and secondary electron scanning electron microscope (SEM) micrographs of the polished samples were taken with an FEI Quanta 200 SEM. An Oxford EDS detector running with INCA software was used to analyse the samples.

## 4-8 Statistical handling of data

For electrical resistivity, between 10 and 30 of the 8x10 mm cylindrical sample pellets from a single 10 mm thick sample disk were measured, and each such pellet was measured 15 times. DOG determinations were only done once on each sample considered. In the ASTM C20-00 density experiments, two of the 18x10 mm sample pellets from a single 10 mm thick sample disk were considered, and each such pellet was used only once. In the gas pycnometer analyses, cut-off samples were used, and each analysis was repeated five times. Porosities based on CT scanning and image

processing were conducted as indicated in Section 4-4. For compressing breaking strength of both standard and thermally shocked experiments, between 10 and 30 of the 8x10 mm cylindrical sample pellets were tested from each considered 10 mm thick sample disk.

Where multiple repetitions were done, outliers were identified with a commonly applied method (e.g. <https://www.mathworks.com/help/stats/box-plots.html>, accessed 30/10/2020). This was done by multiplying the interquartile range (IQR) with 1.5, and data points that were above the 75<sup>th</sup> quartile plus the IQR and below the 25<sup>th</sup> percentile minus the IQR were disregarded in further calculations. Furthermore, where applicable, standard deviations were presented with average values to indicate data precision.

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# Chapter 5

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## Operational history

In this chapter, the operational history of the case study submerged arc furnace (SAF) will be discussed. The characterisation results presented in Chapter 6 of this study will only be relevant to Söderberg electrodes used in SAF operations, if the case study electrode (Electrode 2 from Furnace 3, Section 3-1) represented normal/stable operation. To explore this (i.e. if the case study electrode represented normal/stable operation), the following was considered for the month prior to the furnace shutdown during which the case study electrode was removed:

- the total daily consumable consumption (Section 5-1),
- the daily slipping (Section 5-2),
- the daily average current (Section 5-3),
- and the daily average solid and liquid paste levels (Section 5-4)

of all three Söderberg electrodes (Electrodes 1, 2 and 3).

### 5-1 Electrode consumables

The most apparent consumables for a SAF are the raw materials that go into the SAF itself. For the SAF from which the case study electrode was removed, these raw materials included chromite ore and oxidative sintered pellets made with the Outotec process (Section 2-3-1); (Basson & Daavittila, 2013), reductants (coke, char, anthracite and coal) and fluxes (which were mostly quartz). However, two other consumables are used in SAFs, specifically for the Söderberg electrodes itself, namely electrode paste blocks (see Section 2-6) and the casings (see Section 2-5). These consumables need to be continuously added as the electrode is consumed in the SAF.

The graph in Figure 5.1 shows the average number of electrode paste blocks loaded each day into the casings of Electrodes 1 (E1), 2 (E2) and 3 (E3) of Furnace 3 (F3) during July 2013. The trend lines for the number of electrode paste blocks loaded per day shows that the consumption stayed approximately the same, at between 1.8 and 2.5 blocks per day per electrode. The only 'outliers' in this data series are the five electrode paste blocks loaded into E1 on 8 July 2013 and the zero electrode paste blocks loaded into E1 and E2 on 19 July. For July 2013, a total of 206 electrode paste blocks were

consumed by F3. Each block weighs approximately 295 kg, which means that 60 770 kg of paste was used. For this month, F3 also produced 8 302.94 tons of ferrochrome. Therefore, for every ton of ferrochrome produced, 7.32 kg of paste was used.

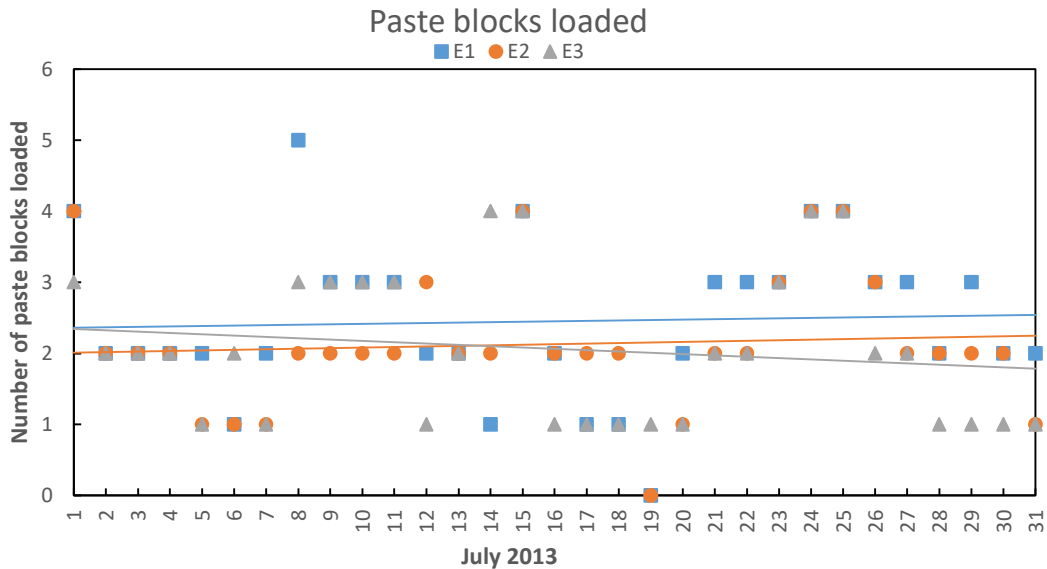


**Figure 5.1** The daily number of electrode paste blocks loaded into each of the three electrodes (Electrodes 1, 2 (case study electrode) and 3) of Furnace 3 during July 2013. The straight lines are trend lines for the daily paste block consumption of each electrode.

Figure 5.2 shows the total daily number of casings installed onto each of the three electrodes (E1, E2 and E3) of F3 during July 2013. E1 (closest to the tap hole as seen in Figure 3.1) had the most casings added, i.e. five; one every six to eight days. E2 (case study electrode) only had two casings added onto it, one on 1 July and the second on 16 July. E3 had three casings added in the first 13 days and nothing for the rest of the month.

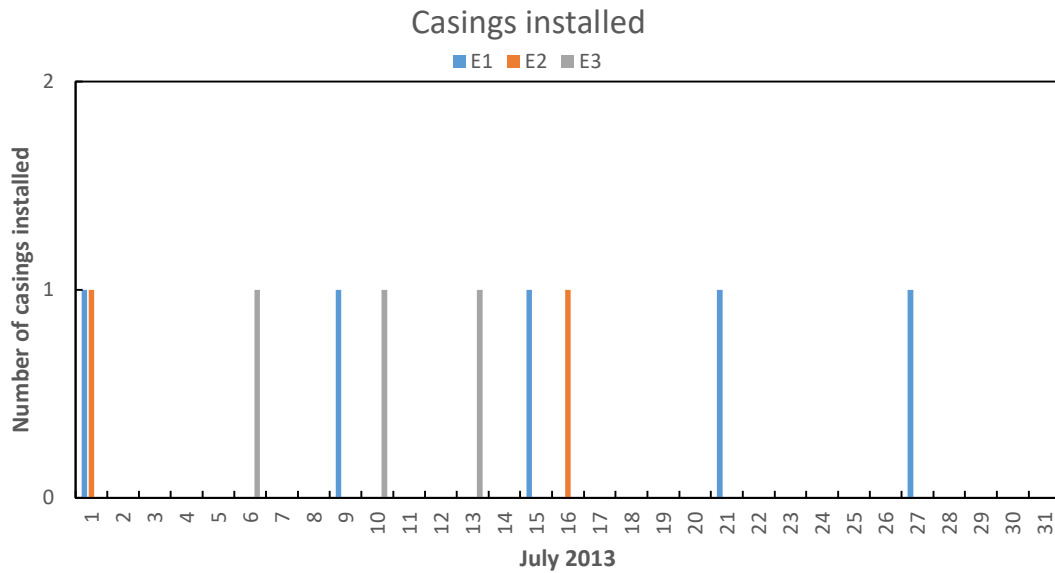


**Figure 5.2** The total daily number of casings installed onto each of the three electrodes (Electrodes 1, 2 (case study electrode) and 3) of Furnace 3.

## 5-2 Electrode slipping

As explained in Section 2-7-3, normal slipping is the process of extending (making it longer) the electrode in small increments on a shift basis to account for consumption thereof. Long slips (usually defined as more than 100 cm) happen as a result of a break of the electrode.

Figures 5-3a, b and c show the total daily slipping taken on E1, E2 and E3 of F3 during each of the three daily shifts. The morning shift (M/S) started at 06:00 and ended at 14:00; the afternoon shift (A/S) from 14:00 to 22:00; and the night shift (N/S) from 22:00 to 06:00 the next morning. As is evident from the data presented in Figures 5-3a to c, the slips taken per shift on E1 varied between 4 and 40 cm; E2s between 4 and 12 cm, and E3s between 4 and 14 cm, whereas the shift average slipping for E1 was 9.55 cm, E2 8.15 cm and E3 8.95 cm over the entire month. This proves that short, normal slips were taken on all three electrodes, with the only 'outlier' being the 40 cm slip taken during the A/S on E1 on 19 July. However, this 40 cm slip is still not considered as a long slip, just a bit longer than a normal slip. Furthermore, such a 40 cm slip would not have required the operators to cut back on power input or current through the electrode.

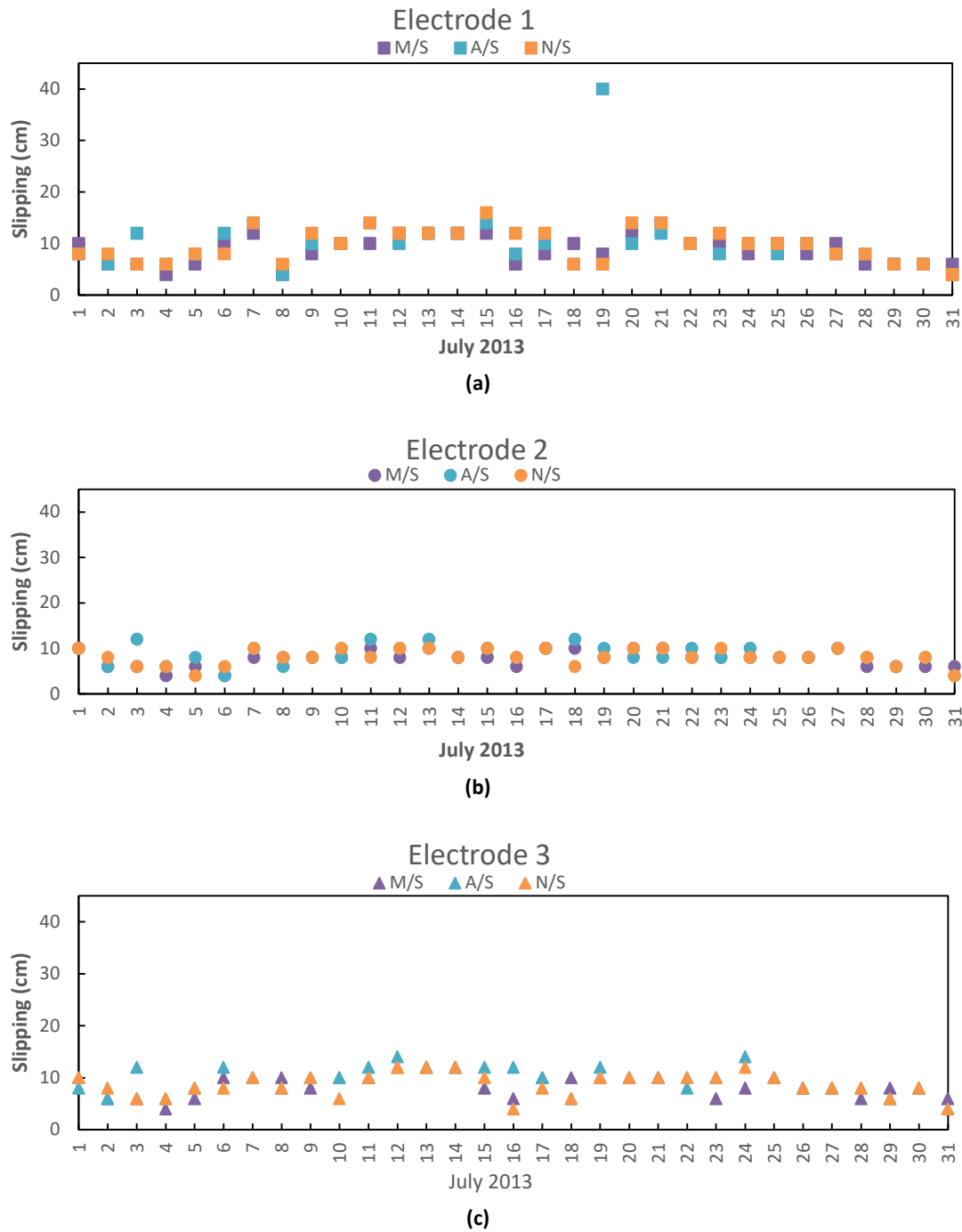


**Figure 5.3** The daily shift average slipping of (a) Electrode 1; (b) Electrode 2(case study electrode); and (c) Electrode 3 of Furnace 3. M/S, A/S and N/S = morning, afternoon and night shifts, respectively.

Figure 5.4 shows the daily average slipping taken on E1, E2 and E3 of F3 for July 2013. The trend lines (dotted lines) show that even with the longer than normal slipping (i.e. 40 cm afternoon shift) on E1 on 19 July, the slipping stayed relatively consistent. E1's slipping varied between 14 and 54 cm, E2's between 14 and 32 cm, and E3's was between 14 and 28 cm per day. The daily average slipping on E1 was 28.65 cm, 24.45 cm on E2, and 26.84 cm on E3 over the entire month.

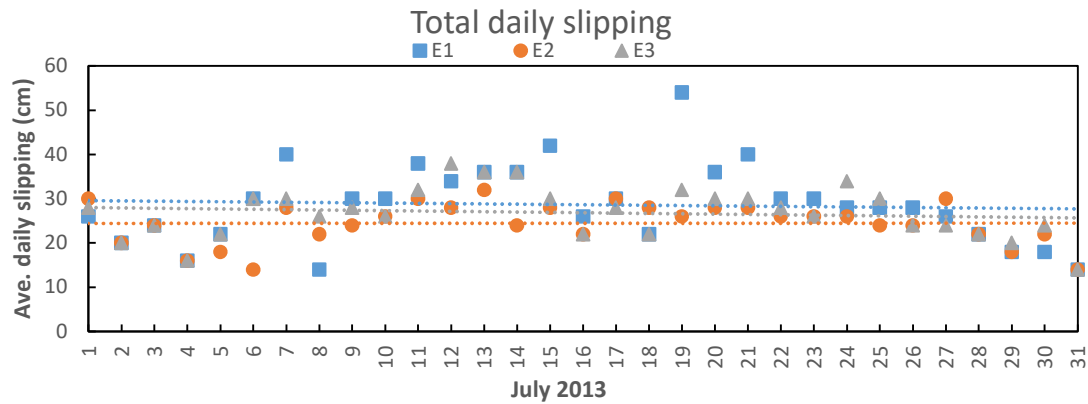


**Figure 5.4** The daily average slipping for each of Electrode 1, Electrode 2 (case study electrode), and Electrode 3 of Furnace 3. The dotted lines show the average slipping on each electrode.

### 5-3 Electrode current

Figure 5.5 shows the daily average current passing through E1, E2 and E3 of F3 for July 2013. This varied between approximately 53 and 76 kA. E1, E2 and E2 had averages of 72.2, 72.2 and 68.4 kA, respectively, in the period between 1 and 28 July 2013. This indicates that no significant operational issues occurred during the month. In the last three days prior to the shutdown (29 to 31 July 2013), the energy input was purposefully reduced for all three electrodes, which resulted in the average current passing through the electrodes on the last operational day being 63, 58 and 60 kA for E1, E2 and E3, respectively. Such a gradual reduction in power input contributes significantly to prevent thermal shock on the electrode (Larsen *et al.*, 2013) and was therefore done purposefully to protect the electrodes for this study.

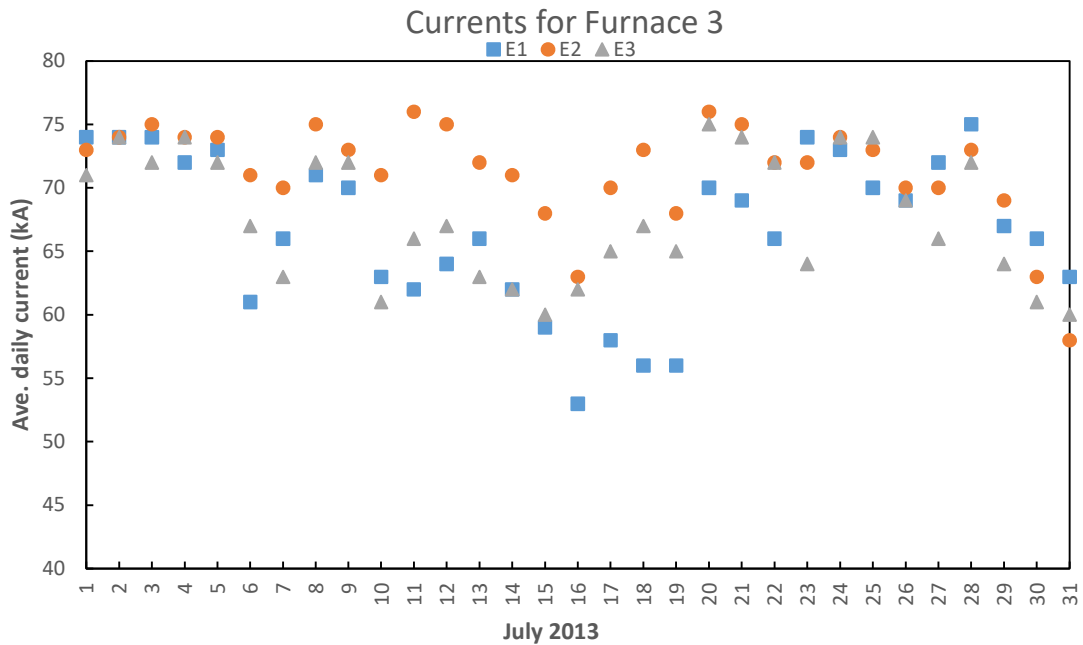
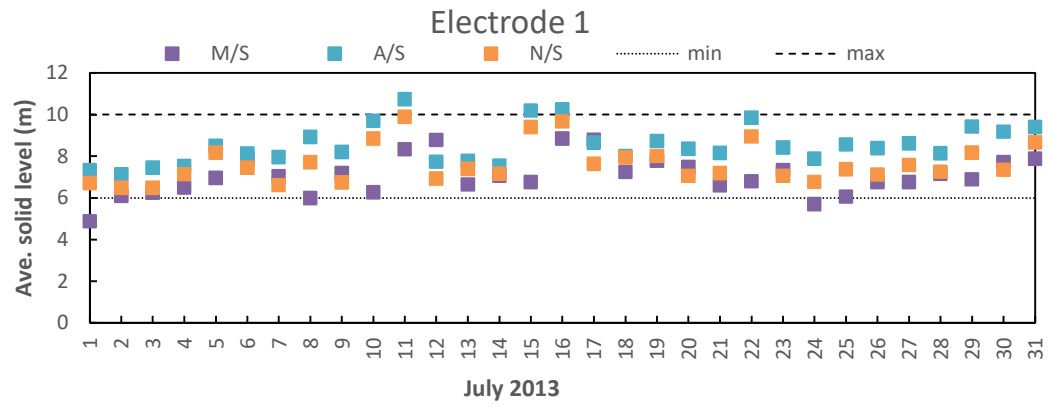


**Figure 5.5** The daily average current for each of the three electrodes of Furnace 3.

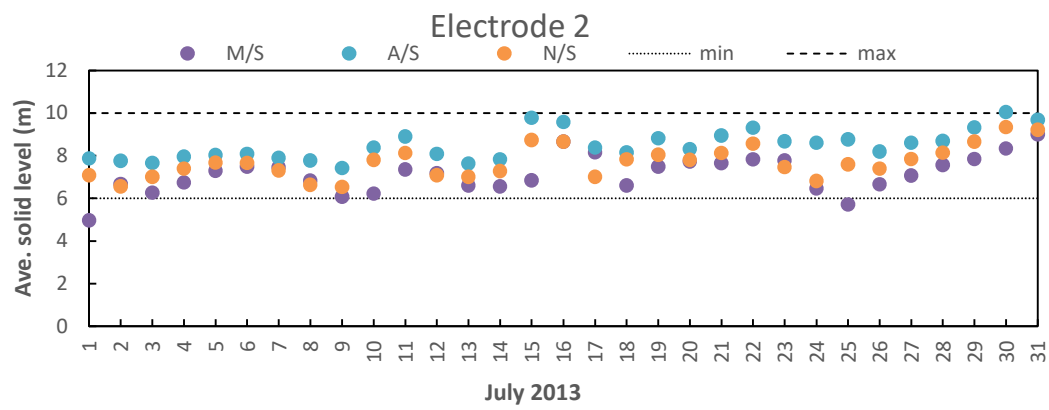
## 5-4 Solid and liquid electrode paste levels

### 5-4-1 Solid paste levels

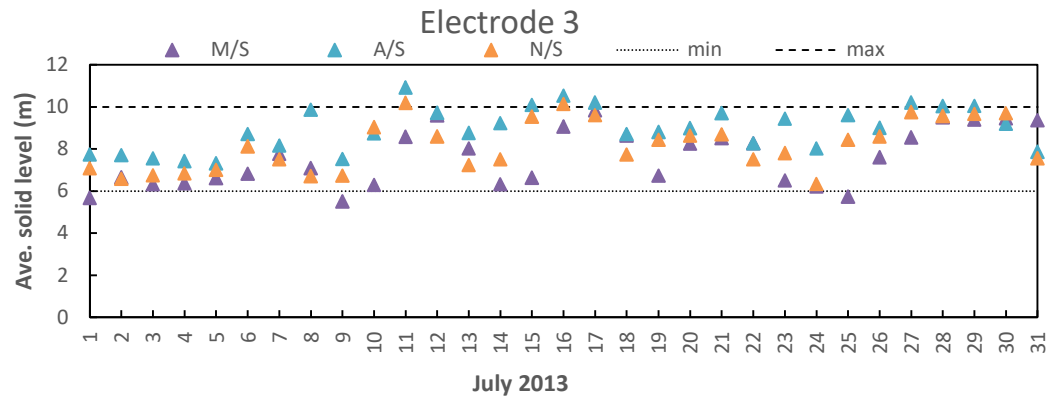
Figures 5.6a, b and c show the daily shift solid paste level (in meters) of E1, E2 (case study electrode) and E3 of F3. The straight bottom line is the minimum (6 m) and the top straight line is the maximum (10 m) value for the desired solid paste level range, as specified by the operational personnel. During a couple of the morning shifts, the solid paste level dipped just below the minimum level, though it was not enough to consider it as a problem. The reason why this only happened during morning shifts, is because an operational plant meeting is conducted early every morning. This results in a bit later than normal (if compared to the other shifts) measurement of solid and liquid levels, and loading of electrode past blocks, if required. The afternoon and night shifts did overload the electrodes slightly on occasion, but again this will not have had any negative impact on the electrodes.



(a)



(b)



(c)

**Figure 5.6** The daily shift solid paste level of **(a)** Electrode 1; **(b)** Electrode 2 (case study electrode); and **(c)** Electrode 3 of Furnace 3. The bottom line is the minimum, and the top line is the maximum desired paste levels.

Figure 5.7 compares all three electrodes' daily average solid paste levels with each other. Again, the two straight lines show the desired minimum and maximum solid paste level range, specified by the

operational personnel. This data also shows that the electrode solid paste levels were relatively consistent and within specifications.

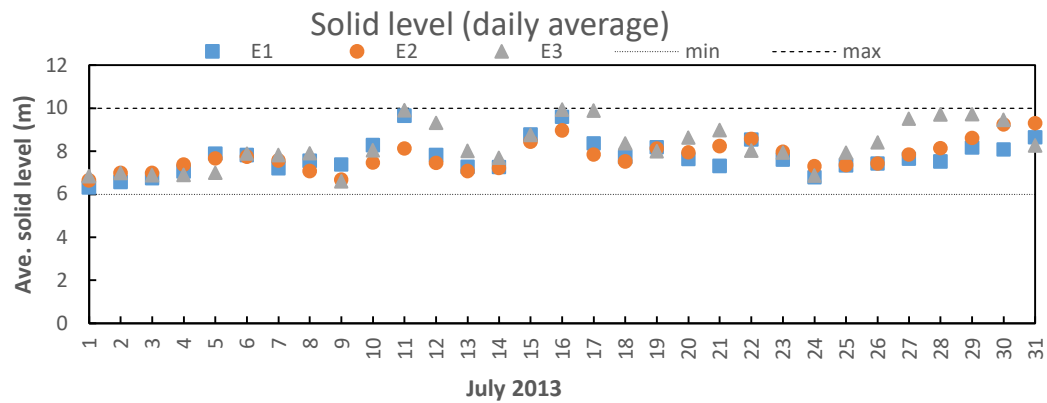
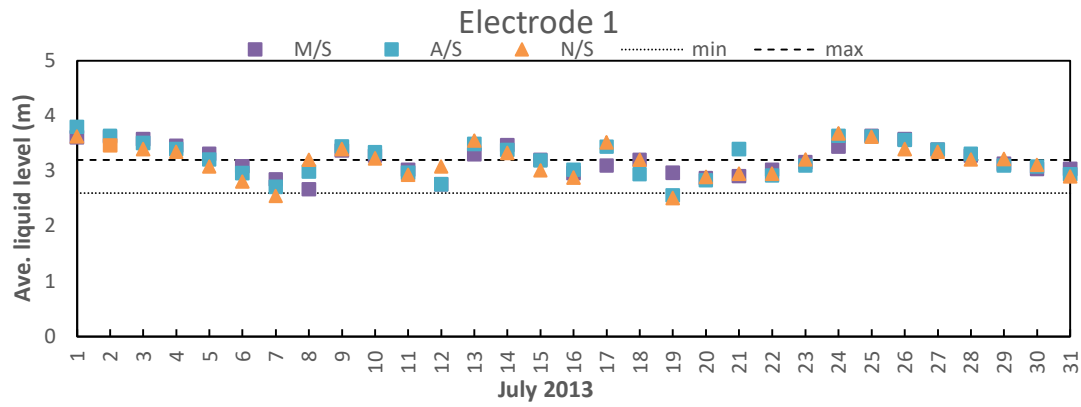


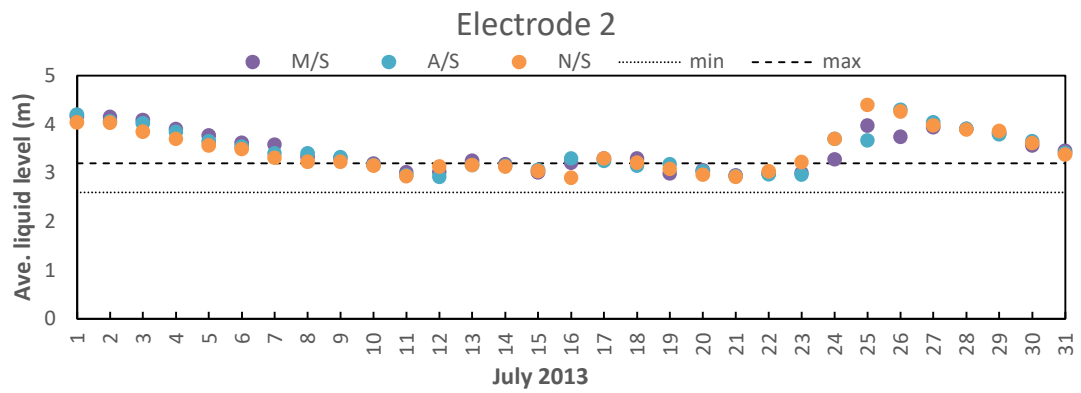
**Figure 5.7** The daily average solid paste levels for all three electrodes of Furnace 3. The bottom line is the minimum and the top line is the maximum levels desired.

#### 5-4-2 *Liquid paste levels*

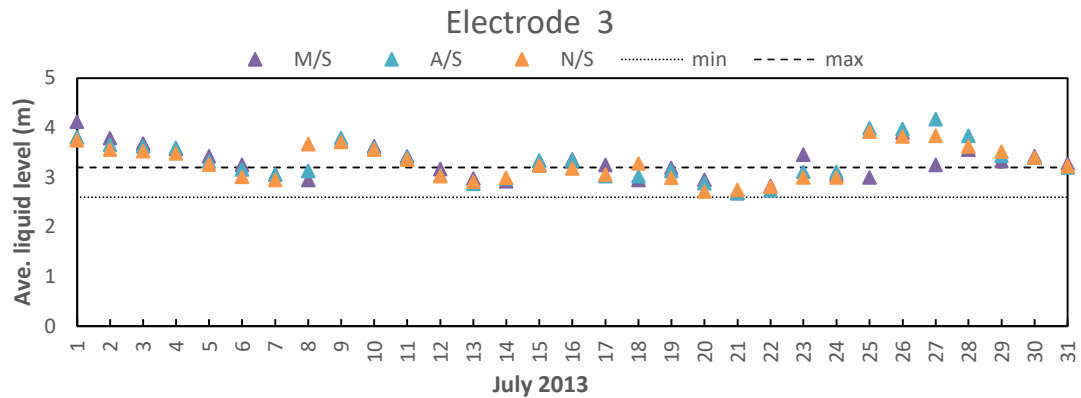
Figures 5.8a, b and c show the daily shift liquid paste levels (in meters) for E1, E2 (case study electrode) and E3 of F3. In these figures, the straight bottom line is the minimum (2.6 m) and the top straight line is the maximum (3.2 m) desired liquid paste level range, specified by the operational personnel. As is evident from the data, the liquid paste levels were well above the minimum level for most of the time, except for E1 on 7 and 19 July. However, even then, the lowest level was 2.51 m (during the night shift on 19 July), which is very close to the minimum value of the specified range. In contrast, the maximum liquid paste level was above the maximum desired level on numerous occasions. However, this is not a serious issue, since the only reason why a maximum level is specified, is because the operational personnel do not want the electrode column to become too heavy. A too-heavy column can contribute to slip-throughs, i.e. unintended movement of the entire column through the electrode clamps that hold it in position.



(a)



(b)



(c)

**Figure 5.8** The daily shift liquid paste levels of **(a)** Electrode 1; **(b)** Electrode 2 (case study electrode); and **(c)** Electrode 3 of Furnace 3. The bottom line is the minimum, and the top line is the maximum levels desired.

Figure 5.9 shows the daily average liquid paste for all three electrodes of F3, with the two straight lines indicating the desired minimum and maximum liquid paste levels, as was done previously. Again, at no point did the daily average go below the minimum liquid paste level, which was the most important criterion here.

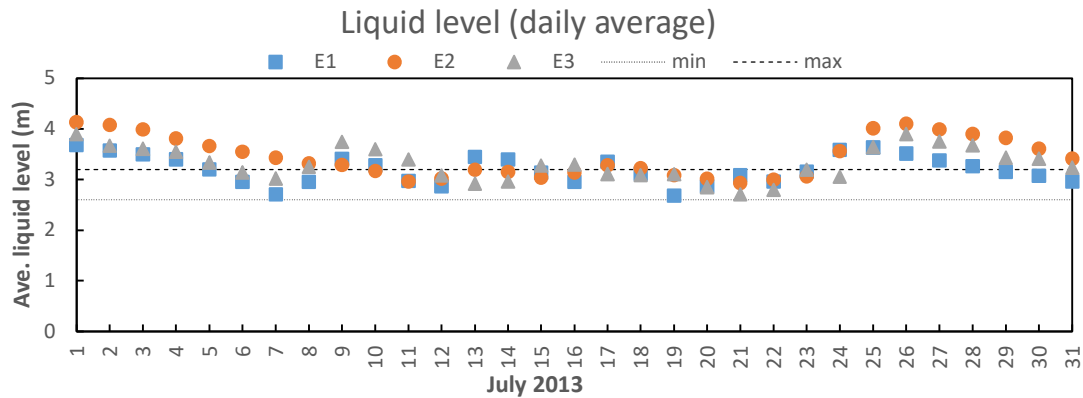


**Figure 5.9** The daily average liquid paste for all three electrodes of Furnace 3. The bottom line is the minimum, and the top line is the maximum levels desired.

## 5-5 Comparisons

### 5-5-1 *Electrode slipping and casing consumption*

Figure 5.10 shows the comparison between the average daily slipping and the casings installed for E1, E2 and E3 of F3. This figure, along with the trend lines presented in Figure 5.4, clearly proves that the electrode with the most slipping (E1) also had the most casings installed, which makes sense. Similarly, the electrode with the least slipping (E2) had the least amount of casings installed. The most likely reason why E1 had the most slipping (and associated casings installed) is because it is the electrode closest to the tap hole (see Figure 3.1). The so-called tap-hole electrode experiences a great deal more mechanical stresses than the other two electrodes, since the flow of liquid materials (metal and slag) pushes the electrode towards the SAF shell, which can also result in uneven solid feed material flowing into the SAF bed around it. In general, the tap-hole electrode is also the most important electrode, since it keeps the tap-hole area hot, without which tapping will be impossible. Therefore, operational personnel prefer the tap-hole electrode rather a bit too long, than too short.

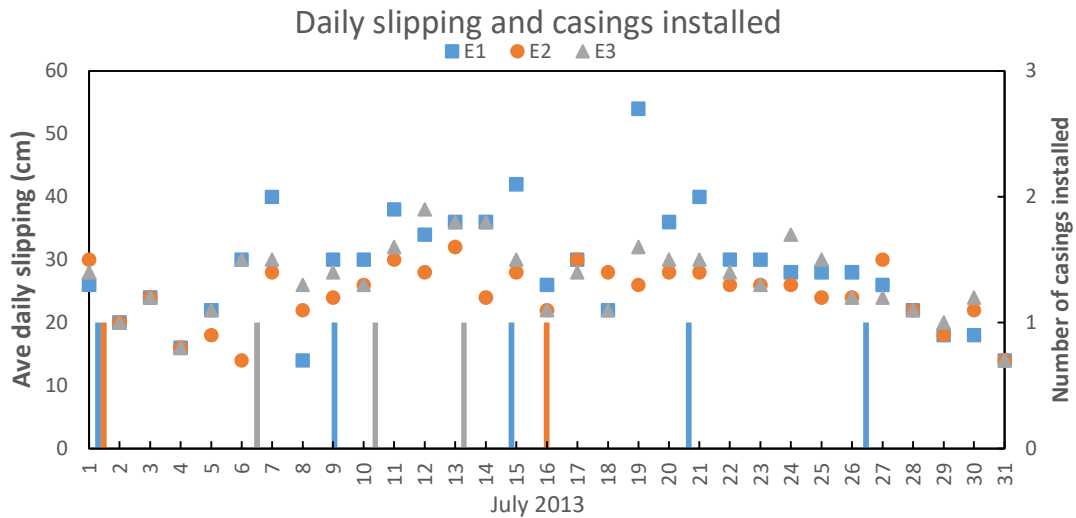


**Figure 5.10** The comparison between the average daily slipping and the casings installed for Electrode 1, Electrode 2 (case study electrode) and Electrode 3 of Furnace 3. The markers show the slipping, and the bars show the number of casings.

#### 5-5-2 Electrode slipping and current

Figure 5.11 shows the daily average slipping (bars) and the average daily current passing through E1, E2 and E3 (markers) of F3. One of the more significant features in it is the period of lower current on E1, which was experienced in the period 16 to 19 July 2013 – indicated by the purple oval. Such a period of sustained lower current indicates that an electrode might be short, hence the bit longer slipping taken on this electrode on 19 July. Thereafter, the electrode current recovered to levels comparable with the other two electrodes.

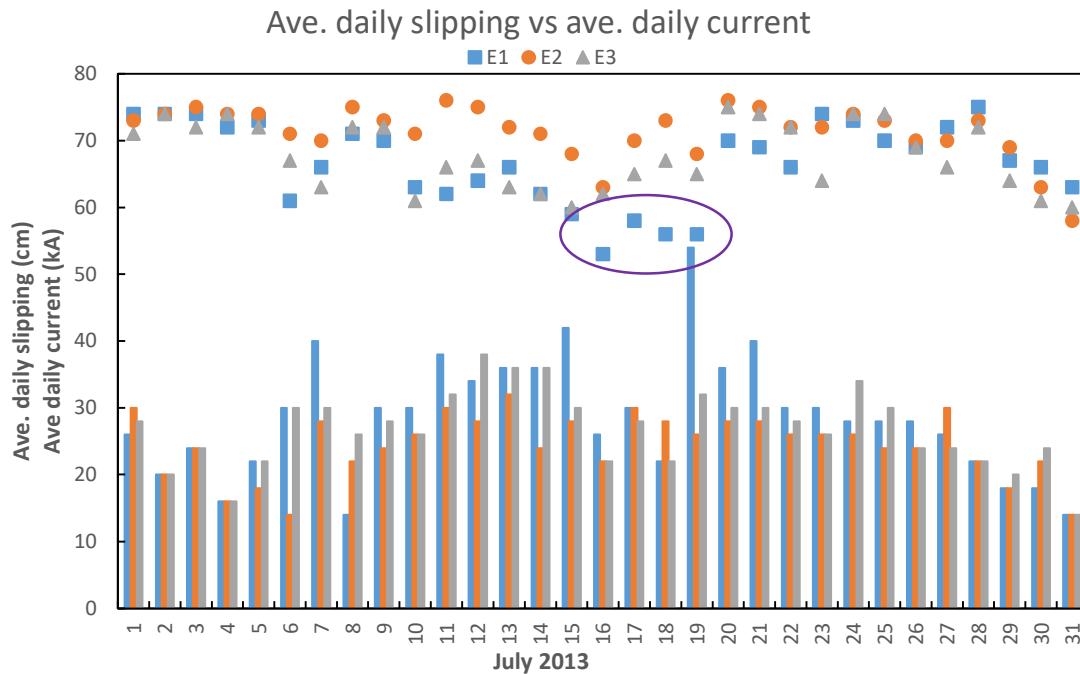


**Figure 5.11** The comparison between the average daily slipping (bars) and the average daily current passing through Electrode 1, Electrode 2 (case study electrode) and Electrode 3 (markers) of Furnace 3.

### 5-5-3 Solid and liquid levels vs paste block consumption

Figures 5.12a, b and c show the daily average solid and liquid paste levels, compared with the number of paste blocks loaded for E1, E2 and E3. These comparisons clearly illustrate how the loading of paste blocks is an instantaneous action (reflecting immediately on the graphs), which also results in immediately higher solid paste levels. The only reason why the loading of paste blocks and increases in solid levels are not perfectly correlated, is since solid levels are not re-measured once blocks are loaded. In contrast to the aforementioned, increased liquid levels are a delayed consequence, as a result of many factors (solid levels, electrode column temperature, etc.), which affect melting of the solid paste.

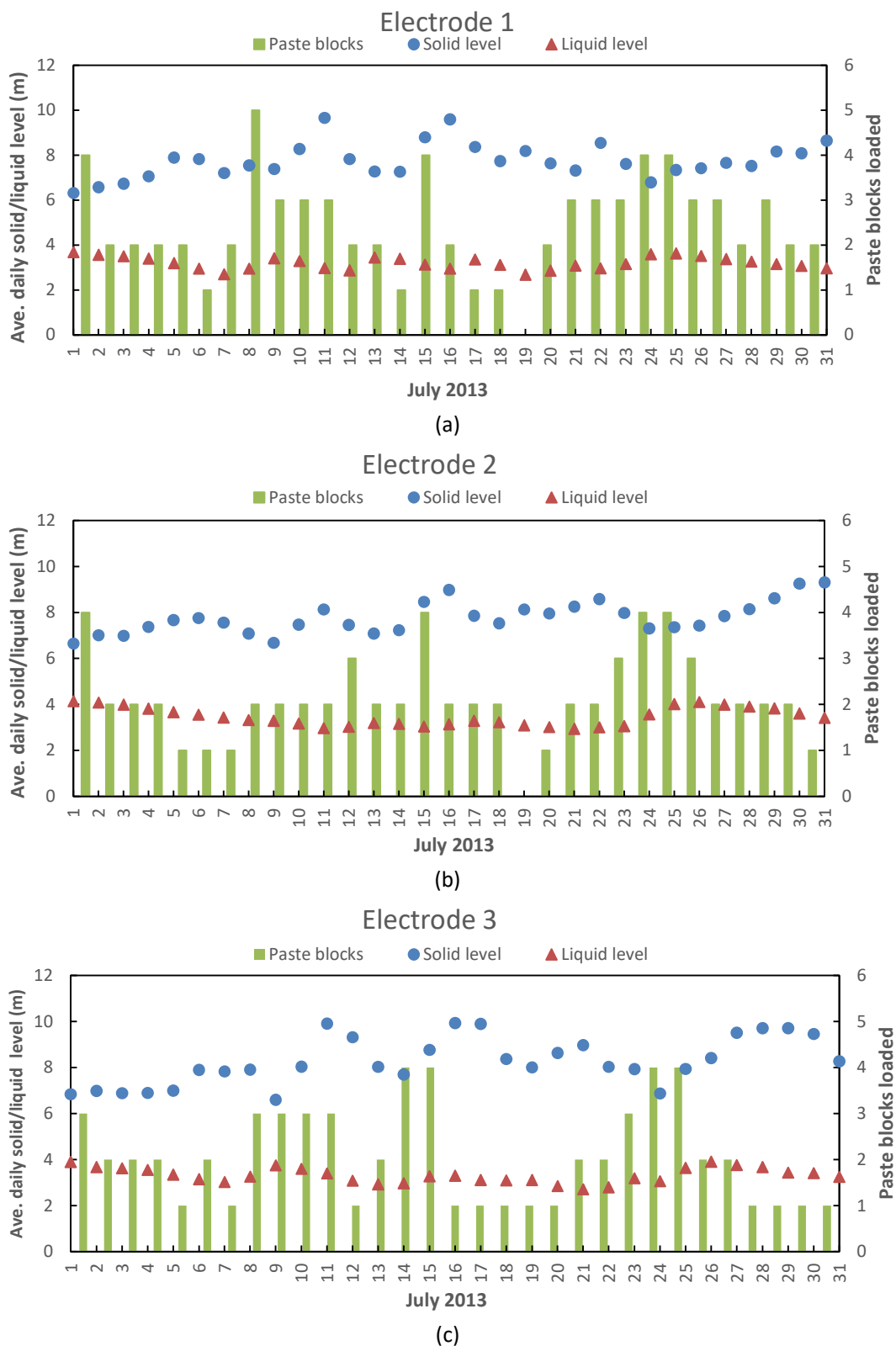


**Figure 5.12** The daily average solid and liquid paste levels compared with the number of paste blocks loaded for (a) Electrode 1, (b) Electrode 2 (case study electrode) and (c) Electrode 3 of Furnace 3.

## 5-6 Conclusion

The Sjøderberg electrode consumables (electrode paste block consumption and casing additions) were stable during the month prior to the shutdown. Except for the little longer than normal slip (i.e. 40 mm) on 19 July 2013, there were no long slips taken on any of the electrodes. The currents passing through the electrodes were relatively stable, with no real problems occurring during the month. Lastly, both the solid and liquid paste levels were stable, with no abnormally low levels during the month. Considering all the aforementioned, in conjunction with a visual inspection of the case study electrode (that occurred during sampling) and its length before being cut off (it was approximately 3 m long), it can be concluded with a very high level of confidence that it was representative of a stable SAF operation. Therefore, characterisation results presented for the case study electrode in Chapter 6 can be accepted as applicable to normal SAF operations.

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## Chapter 6

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### Characterisation of case study Söderberg electrode

In this chapter, the Söderberg electrode characterisation results will be discussed. This includes electrical resistivity (Section 6-1), degree of graphitisation (Section 6-2), bulk density (Section 6-3), porosity (Section 6-4), compressive strength (Section 6-5) and thermal shock (Section 6-6).

#### 6-1 Electrical resistivity measurements

Figure 6.1 presents the average measured resistivity and linear fitting of this data, as a function of length below the contact shoes and distance from the electrode's delta side. It should be noted that no experimental data could be collected on, or very close to 0 and 1.4 m from the electrode delta side, since the 100 mm core drilled samples (Figure 3.7) were typically  $\leq 1.3$  m long. This smaller electrode diameter than the original outer casing diameter of 1.4 m (introduced in Section 3-1), is due to several factors. Firstly, the mild steel casing usually burns off and/or melts within the first approximately 0.5 m below the contact shoes, which is also shown in Figure 3.3 for the specific electrode considered here. The casings used on the case study electrode were 3 mm thick. Therefore, 6 mm (3 mm on each side) can be attributed to the casing loss. Secondly, the electrode paste will shrink at temperatures above the baking isotherm (Larsen *et al.*, 2013). This occurs since the carbonaceous material structure becomes more organised and closer to that of graphite, due to the increased temperature the electrode experiences as it descends. Thirdly, chemical corrosion, i.e. oxidation of the electrode carbon above the bed material, can also contribute, especially since the furnace considered here was an open-SAF. In open-SAFs, atmospheric air containing oxygen can relatively freely enter (determined by off-gas flow rate) and results in a partially oxidising environment above the bed material (Beukes *et al.*, 2017). Lastly, due to frictional and mechanical forces exerted on the electrode, physical erosion can also erode its outer perimeter. Due to all the aforementioned reasons, all result figures wherein the case study electrode diameter is specified will indicate it as 1.3, not 1.4 m.

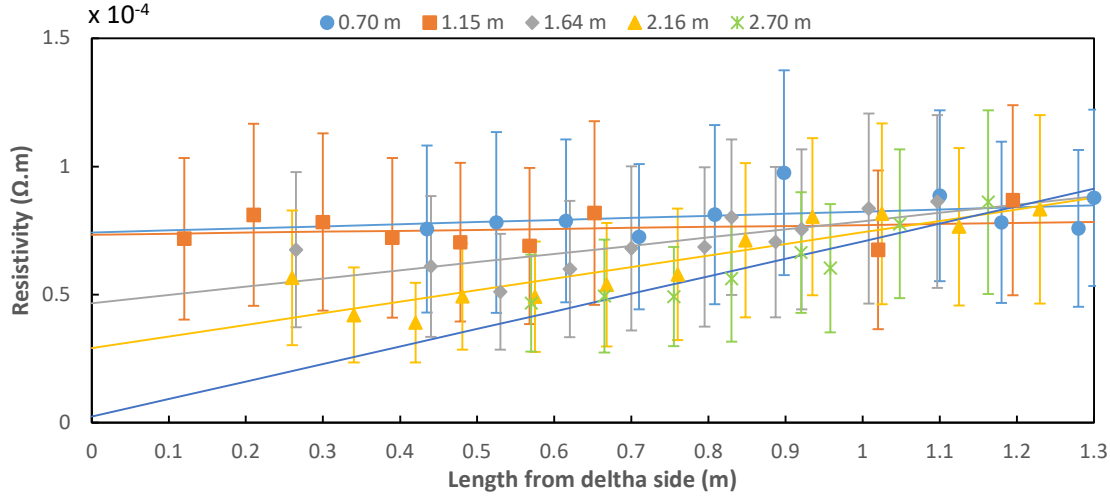


**Figure 6.1** Average measured electrical resistivity (markers) with associated standard deviations (bars) and linear fitting of this data (lines), as a function of length below the contact shoes and distance from the delta side of the electrode.

The standard deviations of the resistivity measurements presented in Figure 6.1 were relatively large. This was mostly due to the actual resistivities being so low (i.e.  $10^{-5}$  to  $10^{-6}$   $\Omega.m$ ). Therefore, to ensure that the average values could be used to identify trends, very large numbers of measurements (i.e.  $N = 150$  to  $450$ ) were conducted. Considering the average values, Figure 6.1 clearly indicates that the electrical resistivity on the outside (non-delta side) of the electrode between 0.7 and 2.7 m below the contact shoes, was essentially consistent. In contrast, the resistivity decreased (implying increased conductivity) significantly from 0.7 to 2.7 m below the contact shoes on the delta side of the electrode. To visualise the electrical resistivity results better, the average data associated with the fitted lines presented in Figure 6.1 at 10 mm intervals across the diameter of the electrode at each of the investigated lengths below the contacts shoes, were spatially interpolated across the entire investigated area (0.7 to 2.7 m below the contacts shoes), as indicated in Figure 6.2. In effect, this representation gives a three-dimensional (3D) cross-section perspective of the investigated electrode, with colour indicating the third dimension. This 3D perspective indicates that resistivity was at its lowest at the bottom end on the delta side of the electrode, where the submerged arc would most likely jump off the electrode.

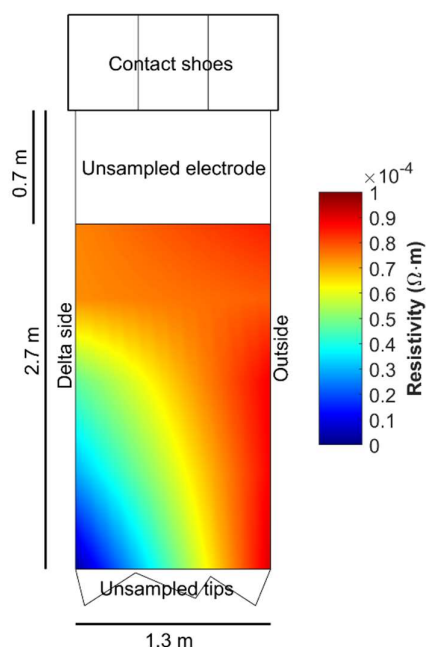


**Figure 6.2** The fitted line data associated with resistivities at 10 mm intervals across the electrode at each of the lengths below the contacts shoes indicated in Figure 6.1 were spatially interpolated across the entire investigated area (0.7 to 2.7 m below the contacts shoes) by using the 'grid data' function in Matlab, with triangulation-based linear interpolation.

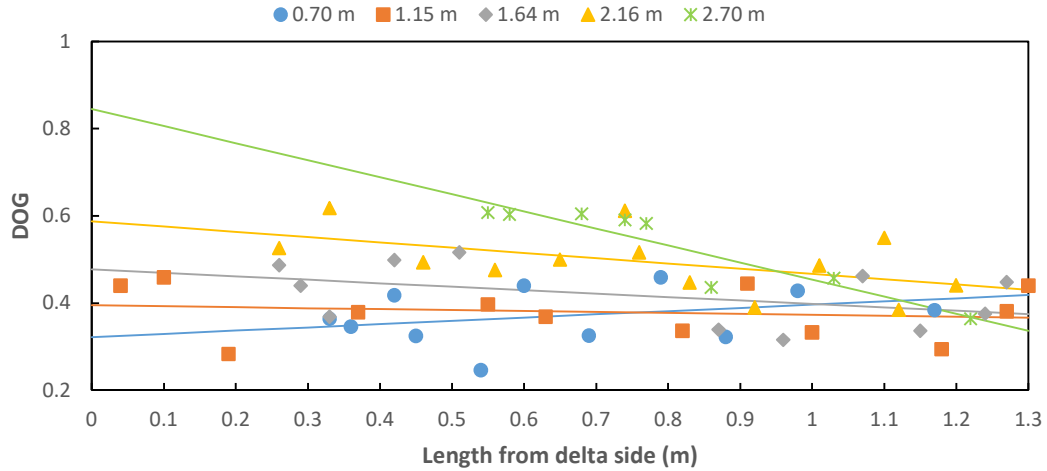
To contextualise the resistivity results presented in Figures 6.1 and 6.2, they were firstly compared to high temperature bulk bed electrical resistivity values reported relatively recently for carbonaceous reductants (i.e. metallurgical coke, semi-coke, coal and charcoal used in SAF smelting) (Surup *et al.*, 2020). Such high temperature bulk bed resistivities simulate the so-called coke bed (area around and below electrode tips in a SAF), where reductants serve as one of the primary electrical conductors (Tangstad *et al.*, 2019). At 1 600 °C, it was found that the reductant bulk bed resistivities ranged from approximately  $2 \times 10^{-2}$  to  $5 \times 10^{-3}$   $\Omega \cdot m$  (Surup *et al.*, 2020). These values were approximately 60 to 250 times higher (therefore less conductive) than the approximate  $8.40 \times 10^{-5}$   $\Omega \cdot m$  resistivity observed from 0.7 to 2.7 m below the contact shoes on the non-delta side of the electrode (reddish area on the right-hand side of Figure 6.2). Correspondingly, the high temperature reductant bulk bed resistivities were approximately 2 000 to 8 000 times higher than the  $2.4 \times 10^{-6}$   $\Omega \cdot m$  obtained at 2.7 m below the contact shoes on the electrode delta side (dark blue area in the bottom left-hand corner of Figure 6.2). Obviously, interparticle contact in a carbonaceous reductant bed is far inferior to the relatively solid carbonaceous mass of a Söderberg electrode, but the aforementioned comparison does indicate, on a relative scale, the conductivity ability of a Söderberg electrode.

To further contextualise the resistivity data presented in Figures 6.1 and 6.2, it was compared to Söderberg electrode paste resistivity values derived from finite element thermo-electrical modelling (McDougall *et al.*, 2004; Tangstad *et al.*, 2019). These studies postulated resistivities to be  $5.88 \times 10^{-6}$

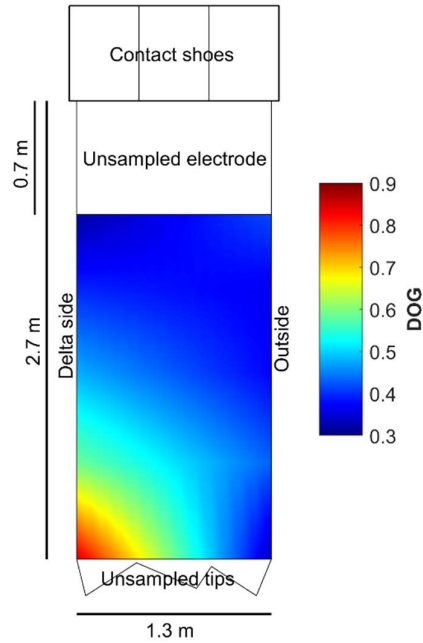
and  $2.63 \times 10^{-6} \Omega \cdot \text{m}$ , in the electrode where the temperatures were 1 000 and 2 600 °C, respectively. Such temperatures correspond roughly to the 0.7 m (both delta and non-delta side) and 2.7 m (delta side only) positions below the contact shoes in Figure 5b, where resistivities were approximately  $8.48 \times 10^{-5}$  and  $2.41 \times 10^{-6} \Omega \cdot \text{m}$ , respectively. Although the modelled values (McDougall *et al.*, 2004; Tangstad *et al.*, 2019) and values presented in the current work are in the same order, the differences in the corresponding values ( $5.88 \times 10^{-6} - 2.63 \times 10^{-6} = 3.25 \times 10^{-6} \Omega \cdot \text{m}$  versus  $8.48 \times 10^{-5} - 2.41 \times 10^{-6} = 8.24 \times 10^{-5} \Omega \cdot \text{m}$ ) suggest that the models do not quickly enough account for lower resistivities as a function of electrode length below the contact shoes. Compared to the aforementioned models (McDougall *et al.*, 2004; Tangstad *et al.*, 2019), a more comprehensive S  derberg electrode model has also previously been introduced (Larsen *et al.*, 2013). This model might contain more detailed S  derberg electrode paste properties and might better account for the variation of such properties as a function of electrode length below the contact shoes. However, this model was essentially introduced as a black box to demonstrate the capabilities of a commercial company (Larsen *et al.*, 2013); therefore, it was impossible to include the material properties specified therein in the current comparison. Furthermore, no evidence could be found that any of the three models (Larsen *et al.*, 2013; McDougall *et al.*, 2004; Tangstad *et al.*, 2019) accounted for the very apparent asymmetrical variation of resistivities observed in the case study electrode (Figure 6.2).

## 6-2 Degree of graphitisation (DOG)

Figure 6.3a presents the average measured DOG and linear fitting of this data, as a function of length below the contact shoes and distance from the delta side of the electrode. Figure 6.3b presents the associated spatially interpolated 3D image. From both these figures, but particularly from Figure 6.3b that visually represent the entire electrode cross-section, it is evident that DOG is inversely related to the electrode resistivity (Figure 6.2). This makes sense, since increased DOG of the electrode paste will result in lower resistivity (i.e. higher conductivity). It is particularly insightful to also note that only the electrode tip on the delta side of the electrode (red area in bottom left-hand corner of Figure 6.3b, which corresponded to a maximum DOG of approximately 0.84) approached full graphitisation. As previously stated (Section 4-2), DOG is unitless, with 1 indicating fully graphitised and 0 fully ungraphitised. The majority of the investigated cross-sectional electrode area had  $\text{DOG} \leq 0.5$ . This could imply that the energy transfer for the majority of the electrode was not enough to fully graphitise the electrode. As far as the candidate could assess, DOG for a real world, or a modelled S  derberg electrode, has not yet been presented in the peer-reviewed public domain; therefore, it was not possible to contextualise the measured DOG with such data.



(a)



(b)

**Figure 6.3** (a) Average measured degree of graphitisation (DOG) (markers) and linear fitting of this data (lines), as a function of length below the contact shoes and distance from the delta side of the electrode; (b) The fitted line data associated with DOG at 10 mm intervals across the electrode at each of the lengths below the contacts shoes indicated in Figure 6a were spatially interpolated across the entire investigated area (0.7 to 2.7 m below the contacts shoes) by using the 'grid data' function in Matlab, with triangulation-based linear interpolation.

As indicated in the previous paragraph, DOG determines to a large extent the electrical resistivity of the Söderberg electrode. However, the DOG of the electrode paste is also related to other important characteristics. For instance, DOG is associated to possibly the most important aspect that causes electrode breaks, i.e. thermal stress. Particularly, green breaks (implying exposure of liquid paste in

the break zone) in the contact shoes area are feared by operational personnel, due to the devastating consequences such breaks could have. In the most authoritative work on Søderberg electrode thermal stress, it is stated that some internal stresses are established during the process of electrode paste shrinkage below the contact shoes area, where the paste is baked at temperatures in excess of the baking isotherm temperature (Larsen *et al.*, 2013). However, as previously stated, Søderberg electrode paste consists of a binder and a solid filler. In this case, the solid filler was calcined anthracite of various size fractions, mixed to obtain the optimum composition. Although more environmentally friendly binders have been proposed than coal tar pitch (e.g. (Ciesińska *et al.*, 2009)), it remains the most commonly used binder for Søderberg electrode pastes and was also used in the paste consumed by the case study electrode. In previous studies, it was proven that anthracite calcined at 1 400 °C expands less than 1% and coal tar pitches prebaked at the maximum baking isotherm temperature (i.e. 475 °C, (Shoko *et al.*, 2013)) shrink approximately 12%, if heated in a non-oxidising environment up to 1 300 °C (Beukes *et al.*, 2013). These opposing thermal expansion characteristics likely cause the thermal stresses below the contact shoes area referred to by Larsen *et al.* (2013). In contrast to calcined anthracite, uncalcined anthracite shrink 6 to 8% over the same temperature range. The significant difference between calcined and uncalcined anthracite clearly indicates how important the calcination process is to avoid additional thermal stresses forming just below the contact shoes area. Industrial calcination of anthracite to be used in electrode paste typically occurs at temperatures above 1 400 °C (as tested by Beukes *et al.* 2013); therefore, DOG similar to the blue area in Figure 6.3b (corresponding to  $DOG \leq 0.5$ ) is imparted on the electrode paste solid filler even before it is mixed in the electrode paste. The candidate was not privileged to know the exact mixing ratio of the binder and filler used in the case study electrode. However, typically, the mixture is dominated by filler, with the binder fractional contribution limited to just enable solid filler interparticle contact and proper paste flow characteristics above the softening point (to fill the casing in the area above the baking isotherm temperature).

## 6-3 Bulk density

### 6-3-1 ASTM-C20-00 method

Figure 6.4 presents the average measured bulk density determined with ASTM C20-00 (2015) (Section 4-3-1), as a function of length below the contact shoes and distance from the delta side of the electrode. Since ASTM C20-00 involves aqueous saturation of the samples via boiling, repeat analyses were not attempted. The density differences were relatively small (Figure 6.4); therefore, even slight modification of the original characteristics that might occur during analysis or re-drying of

the samples in preparation for reanalyses, could make such reanalyses' results misleading. Additionally, larger cylindrical sample pellets were used in this specific method (Section 4-3-1), which implied that significant additional sample material would have been required for reanalysis. This was not feasible, since a finite amount of material was available after sampling (Section 3-2-2). As is evident from the ASTM C20-00 results (Figure 6.4), bulk densities did not indicate significant differences as a function of length below the contact shoes. Therefore, a single linear fitted line was drawn through all the data, which indicated a slight increase in densities toward the delta side of the electrode.

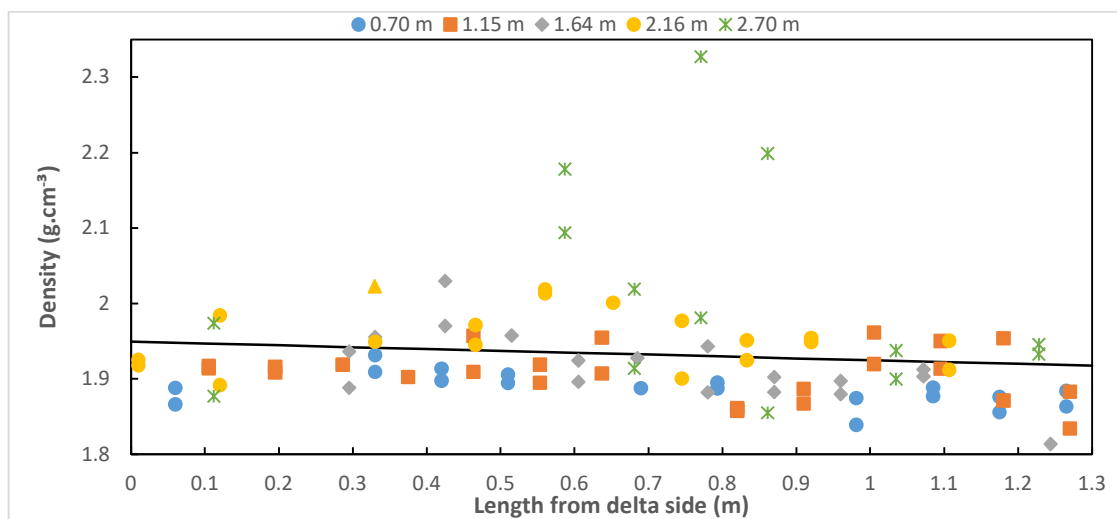


**Figure 6.4** ASTM C20-00 average measured bulk densities (markers) as a function of length below the contact shoes and distance from the delta side of the electrode. A single linear fitting of all this data (line) is also indicated.

### 6-3-2 Gas pycnometer

To verify that densities did not differ as a function of length below the contact shoes, and to determine standard deviations, bulk densities were also determined with gas pycnometry (Section 4-3-2). The gas pycnometry densities (Figure 6.5), in contrast to the ASTM C20-00 results (Figure 6.4), did indicate that bulk densities differed as a function of length below the contact shoes. This indicates that gas pycnometry is possibly a more precise method than ASTM C20-00, or alternatively that the difference in atom/molecule size (i.e. He used in gas pycnometry vs. H<sub>2</sub>O used ASTM C20-00) plays a significant role in the measured results.

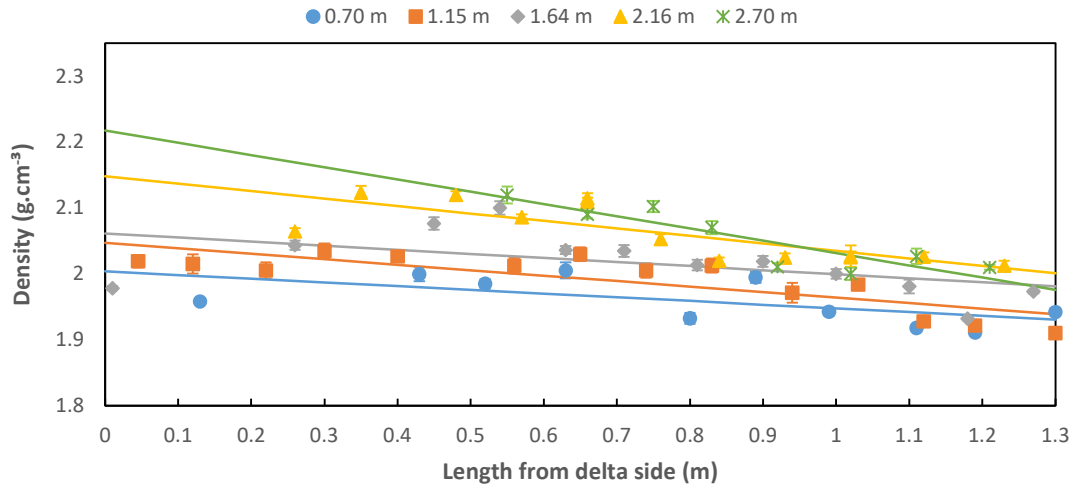


**Figure 6.5** Gas pycnometry average measured bulk densities (markers) and linear fitting of this data (lines), as a function of length below the contact shoes and distance from the delta side of the electrode.

To visualise these gas pycnometry results, Figure 6.6 presents the associated spatially interpolated 3D image, based on the fitted data presented in Figure 6.5. Considering the very similar perspectives indicated by the bulk densities presented here (Figure 6.5) and the DOG presented earlier (Figure 6.3b), it is inferred that the increased bulk densities observed, particularly on the delta side of the electrode as a function of length below the contact shoes, were due to increased DOG. Increased DOG will result in further shrinkage of the carbonaceous Söderberg electrode paste material.

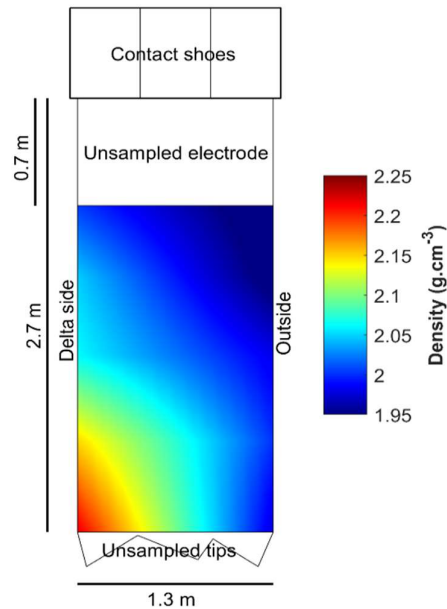
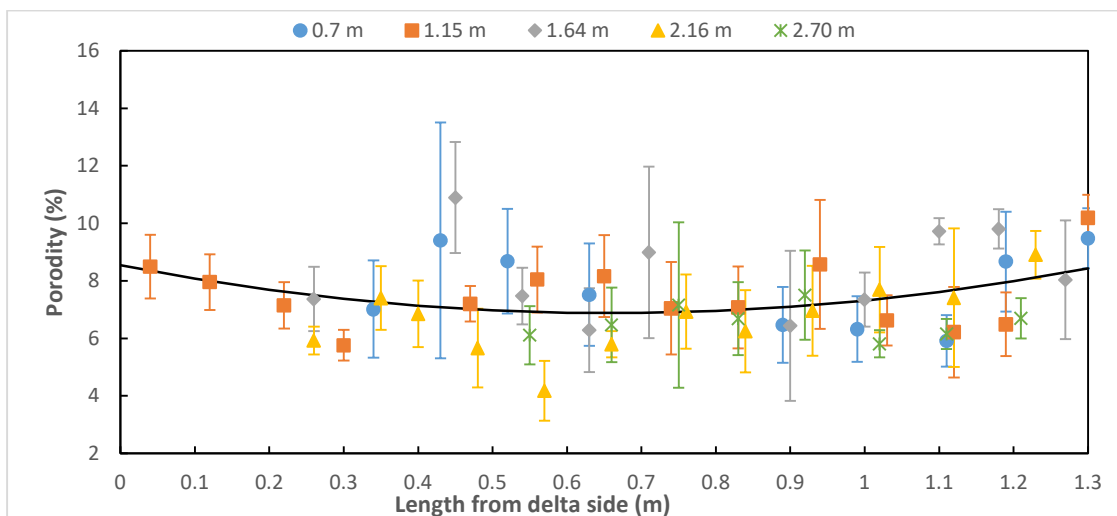


**Figure 6.6** The fitted lines data associated with gas pycnometry average bulk density at 10 mm intervals across the electrode at each of the lengths below the contacts shoes indicated in Figure 6.5 were spatially interpolated across the entire investigated area (70–270 cm below the contacts shoes) by using the ‘grid data’ function in Matlab, with triangulation-based linear interpolation.

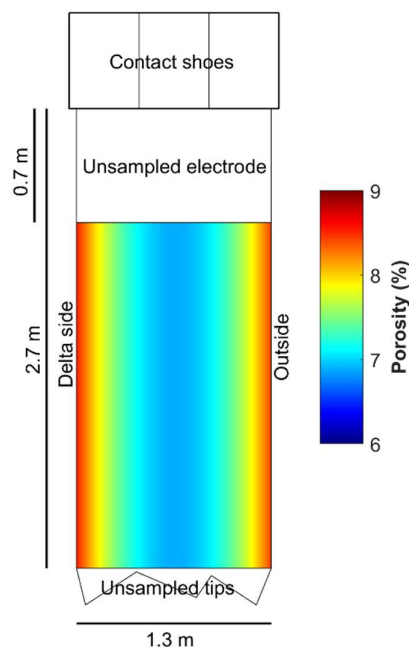
## 6-4 Porosity determination with computerized tomography (CT)

Figure 6.7a presents the average measured porosity. As is evident, no significant difference in porosity as a function of length below the contact shoes was observed. However, a slight increase in porosity was observed, near the electrode perimeter on both the delta and non-delta side. Therefore, a second-order polynomial (solid line in Figure 6.7a) fitted the data best. The corresponding spatially interpolated 3D image is presented in Figure 6.7b.

Since the industrial application of the Söderberg electrode started, it has been proven that volatiles released from the liquid paste just above the baking isotherm (indicated by black dotted line in Figure 6.8a) cannot rise if proper (high enough) liquid and solid paste levels are maintained, e.g. Sem (1954). Conversely, if 'smoke' is observed at the top of the electrode column casing, it serves as a serious warning signal to operational personnel that the liquid paste level of that electrode is critically low and/or that the liquid paste is too hot. Since the volatiles just above the baking isotherm cannot rise, they escape downward through the pores of the already baked electrode (Sem, 1954). Cracking of these descending volatiles (due to the high temperature of the baked electrode) leaves a considerable amount of carbon in the pores of the baked electrode paste just below the contact shoes area, which improves its quality (Sem, 1954). This process is illustrated by the purple arrows in the enlarged area of Figure 6.8a. The blue temperature isotherm in the enlarged area of Figure 6.8b represents a slightly hotter isotherm temperature than the baking isotherm. Considering this diagram, one would assume that the route of least resistance for volatiles to escape would be towards the outside of the electrode (i.e. shortest route of baked electrode paste), not the electrode core. Theoretically, this process would therefore slightly reduce the porosity of the electrode towards its perimeter, which is contrary to the experimental results presented (Figures 6.7a and 6.7b).



(a)



(b)

**Figure 6.7** (a) Average measured porosity (markers), as a function of length below the contact shoes and distance from the delta side of the electrode, as well as a single second-order polynomial fit of this data (line); (b) The fitted line data associated with porosities at 10 mm intervals across the electrode at each of the lengths below the contact shoes spatially interpolated across the entire investigated area (70-270 cm below the contact shoes) by using the 'grid data' function in Matlab, with triangulation-based linear interpolation.

There could be two other factors that counter the possible decrease in porosity towards the electrode perimeter, caused by descending volatile cracking. Firstly, liquid electrode paste segregation (i.e. partial separation of the pitch binder and solid filler), as illustrated by Nelson and Prins (2004), could result in significant differences in the baked electrode properties considered diagonally across an

electrode. However, calcined anthracite has a much higher DOG than coal tar pitch baked above the maximum baking isotherm temperature (i.e. 475 °C, (Shoko *et al.*, 2013)). Therefore, if paste segregation caused the differences in observed porosities (Figure 6.7b), corresponding differences in DOG diagonally across the case study electrode would likely also have been evident at the first level below the contact shoes investigated (i.e. 0.7 m). This was not the case (Figure 6.3b); therefore, paste segregation is unlikely to be the cause. Furthermore, visual inspection of the sampled electrode did not indicate any segregation. The second possible contributing factor that could increase porosity of the case study electrode, is chemical corrosion, i.e. oxidation of the electrode carbon above the bed material. As previously stated, this can especially occur in an open SAF (Beukes *et al.*, 2017), as considered here. Figure 6.8b shows the diagonal cross-section of another electrode from the same SAF that was cut off approximately 1.0 m below the contacts shoes. As is evident from this electrode, corrosion on the outer perimeter was more significant in the areas where the casing fins occurred. This indicated that the gaps remaining after the casing burned off and/or melted away, formed paths for oxygen penetration and associated oxidation of the carbon material. This describes the process on a macroscale, but it is postulated that the same process occurs on a pore-size scale, which will increase pore volumes and therefore porosity preferentially on the electrode perimeter.

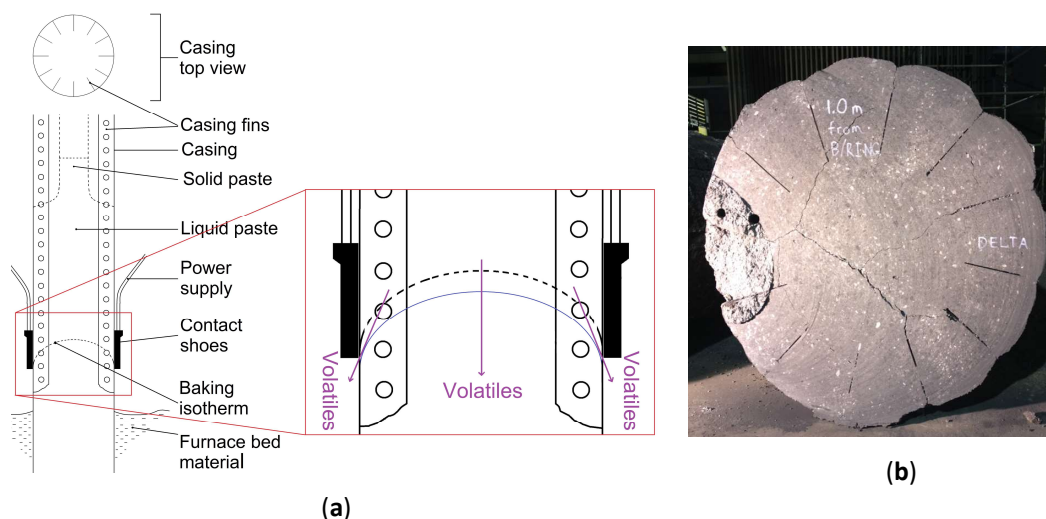


**Figure 6.8** (a) Representation of a partial Søderberg electrode column, adapted from Beukes *et al.* (Beukes *et al.*, 2013), with the contact shoes area enlarged (indicated by the square red block); (b) Cross-section of another electrode from the same SAF that was cut off with a diamond wire saw approximately 1.0 m below the contact shoes.

To test the hypothesis that oxidation above the SAF bed increased the pore sizes and volumes (and therefore porosity) on the case study electrode perimeter, scanning electron microscopy (SEM) micrographs of polished samples from the electrode perimeter and interior were compared. Figures 6.9a to d show various SEM micrograph images of polished samples taken from the delta side edge of

the core sample, collected at 1.64 m below the contact shoes. These micrograph images indicate that the pore sizes can differ from extremely small, to sizes greater than 2 mm in diameter (Figure 6.9c) in the electrode perimeter area. As expected, the micrographs also prove that the electrode is not very homogeneous, due to the mixture of solid filler (calcined anthracite) and coal tar pitch binder.

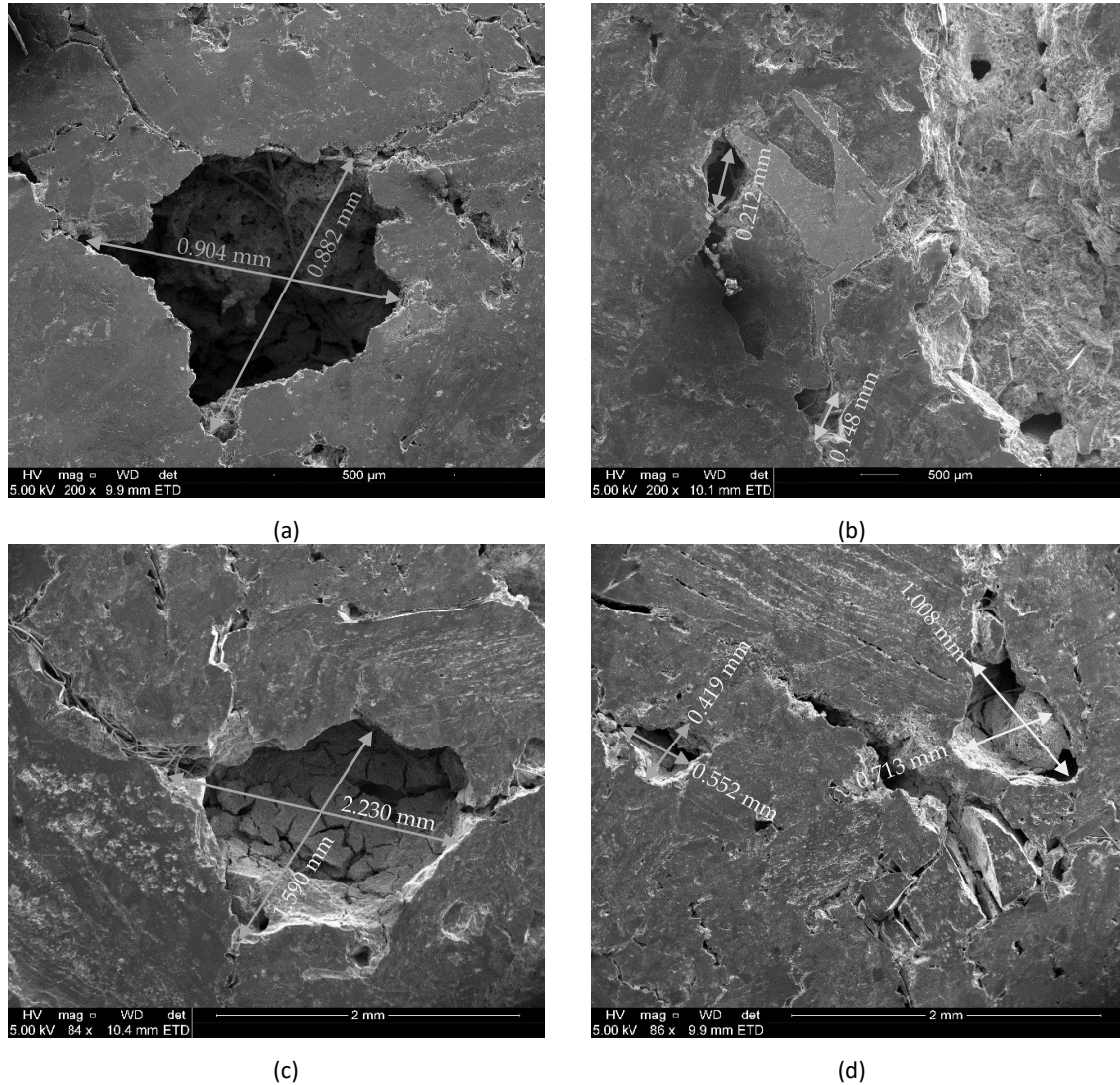


**Figure 6.9** SEM micrograph images of the polished sample taken from the delta edge, 1.64 m below the contact shoes. **(a)** The image was taken at a 200x magnification and shows a single pore with a dimension of 0.904 x 0.882 mm. **(b)** The image was also taken at a 200x magnification and shows a number of smaller pores with the largest having a length of 0.212 mm. **(c)** The image was taken at a 84x magnification and shows a single pore with dimensions of 1.590 x 2.2301 mm. **(d)** The image was taken at a 86x magnification and shows a number of smaller pores with the largest having dimensions of 1.008 x 0.713 mm in size.

Figures 6.10a to d show various SEM micrograph images of polished samples taken 90 mm from the delta edge of the core sample that was collected 1.64 m below the contact shoes. As is evident, numerous pores were also present in these samples, with the largest being 0.808 x 0.296 mm. However, by comparing these micrograph images and the pores indicated thereon, with those

presented in Figure 6.9, it is evident that the pores on the electrode perimeter were in general, much larger than the pores just 90 mm into the electrode. Therefore, although these results are relatively non-quantitative, they support the hypothesis that oxidation above the SAF bed increased the pore sizes and volumes (and hence porosity) on the case study electrode perimeter, which was used to explain the porosity results presented in Figure 6.7.

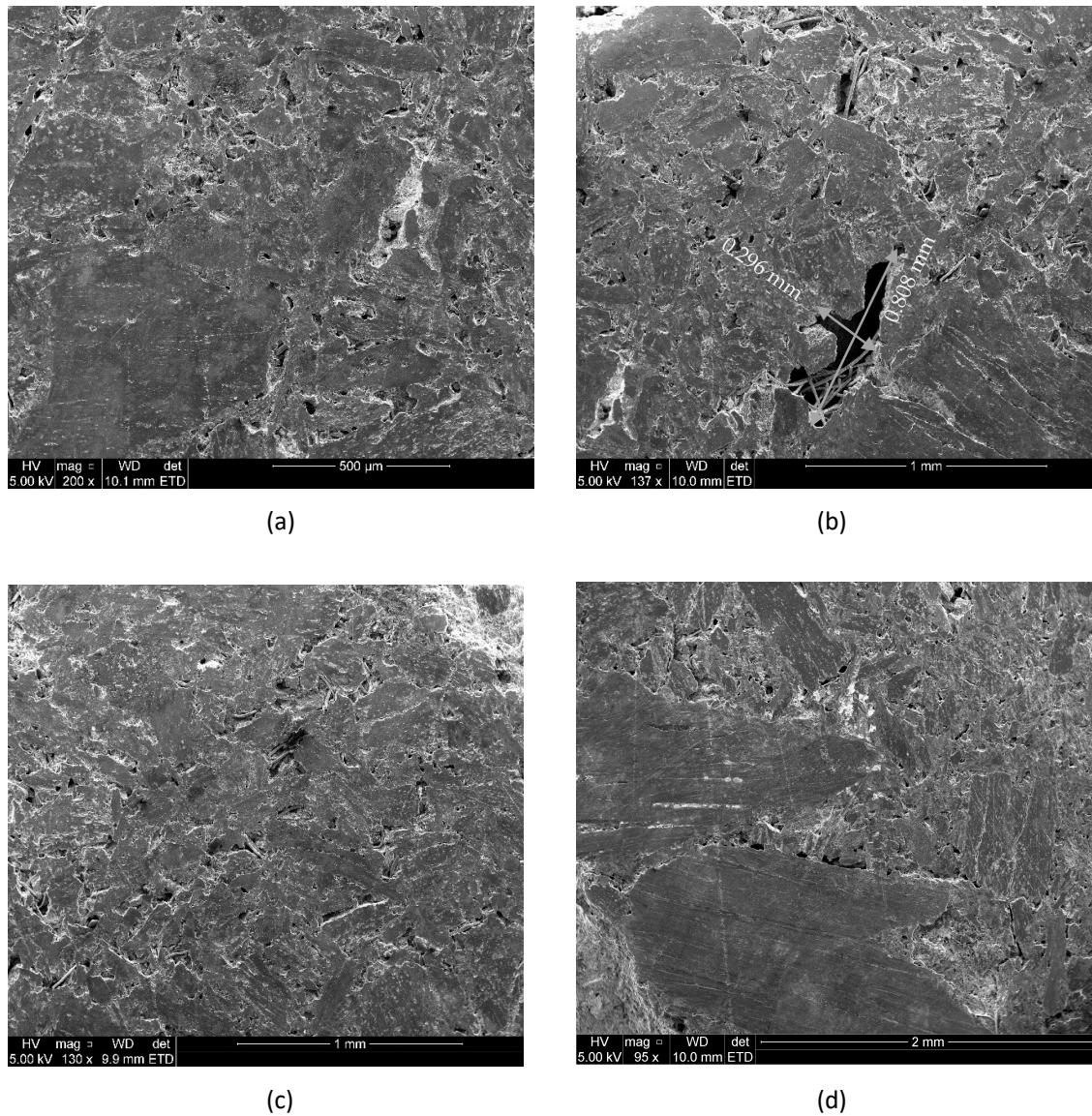


**Figure 6.10** SEM micrograph images of the polished sample taken 90 mm from the delta edge, 1.64 m below the contact shoes. **(a)** The image was taken at a 200x magnification and has numerous small pores. **(b)** The image was taken at a 137x magnification and shows a single big pore with dimensions of 0.296 x 0.808 mm along with numerous smaller pores. **(c)** The image was taken at a 130x magnification and shows a number of small pores. **(d)** The image was taken at a 95x magnification and shows a number of small pores.

## 6-5 Compressive strength test

Figure 6.11 presents the average measured compressive breaking strength of the 8 x 10 mm cylindrical sample pellets as a function of length below the contact shoes and distance from the electrode delta side. The trend line indicated a slight increase in breaking strength from the delta to the non-delta side of the electrode, but the overall increase across the electrode diameter (approximately 0.063 kN) is far smaller than the standard deviations of the measurements. This indicates that compressive breaking strength was not related to DOG, as was the case for electrical resistivity and bulk density. Nor was it influenced by physical and/or chemical processes, as postulated for the porosities. No published comprehensive strength analysis or models were found to compare to these results.

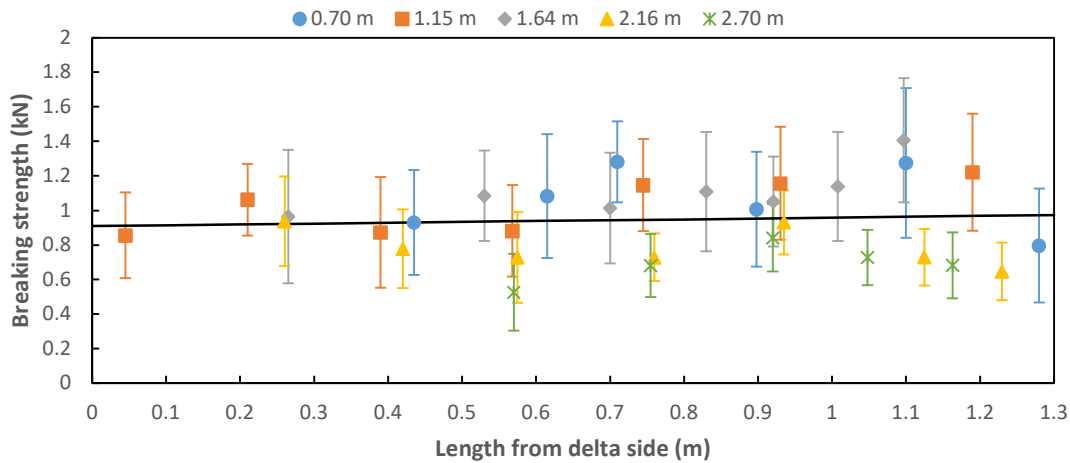


**Figure 6.11** Average measured compressive breaking strength (markers) of the 8 x 10 mm cylindrical sample pellets, as a function of length below the contact shoes and distance from the delta side of the electrode, as well as a single linear fit of this data (line).

## 6-6 Thermal shock

Figure 6.12 presents the average measured compressive breaking strength of the 8 x 10 mm cylindrical sample pellets, which were thermally shocked (see section 4-6), as a function of length below the contact shoes and distance from the electrode delta side. Considering the trend line, there was a slight decrease in breaking strength from the delta to the non-delta side of the electrode (approximately 0.078 kN). However, this difference is far smaller than the standard deviations of the measurements, and therefore it is uncertain whether this trend is meaningful.

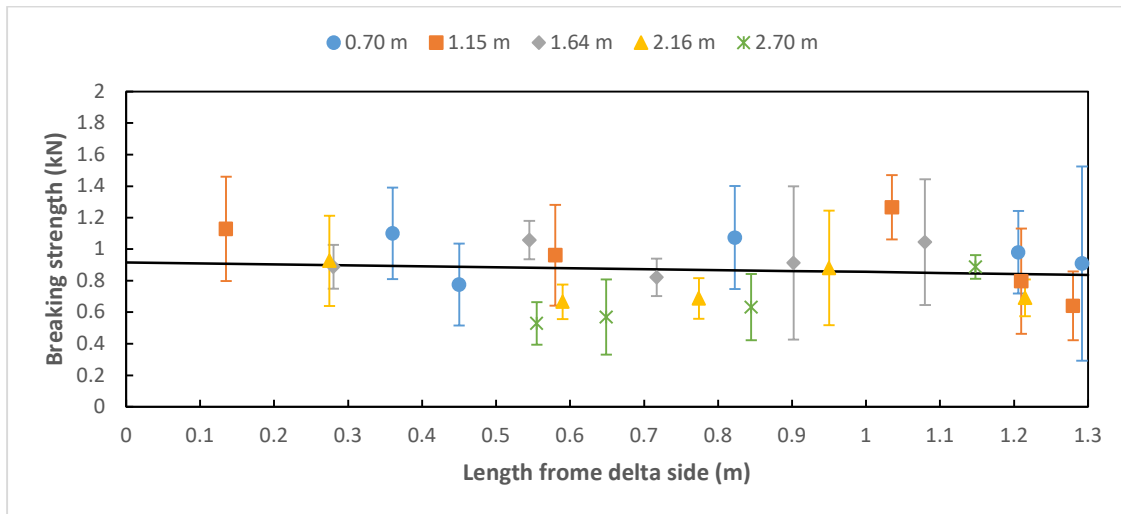


**Figure 6.12** Average measured compressive breaking strength (markers) of the 8 x 10 mm cylindrical sample pellets that were thermally shocked (see Section 4-6), as a function of length below the contact shoes and distance from the delta side of the electrode, as well as a single linear fit of this data (line).

In order to further investigate the meaningfulness of the trend line presented in Figure 6.12, the thermal shock data were presented with the normal compressive breaking strength data (Figure 6.11) in Figure 6.13. This comparison shows that there is not much difference between these two datasets, except for a difference in trend line slope. The normal compressive breaking strength data had a small positive slope from the delta to the non-delta side, while the thermal shock breaking strength data had a small negative slope. This comparison indicated that the delta side of the electrode might be slightly less susceptible to thermal shock breakage than the non-delta side, since its compressive breaking strength was not influenced by the thermal shock treatment. This might be due to the DOG (Figure 6.3) and bulk density (Figure 6.6) of the delta side of the electrode being a bit higher and increasing with length below the contact shoes, if compared to the non-delta side. However, the small differences in thermal shocked and non-thermal shocked breaking strength results might also indicate that the sizes of the samples considered here (i.e. 8 x 10 mm) were simply too small to test the effect of thermal shock thoroughly and/or that the test procedure (Section 4-6) needs to be modified to assess this aspect better. It does, however, show that there is a trend in both the breaking strength and the shock resistance that can be investigated in the future. As far as the candidate could determine, no test procedure has been published in the peer-reviewed public domain to investigate the thermal shock characteristics of S derberg electrode material.

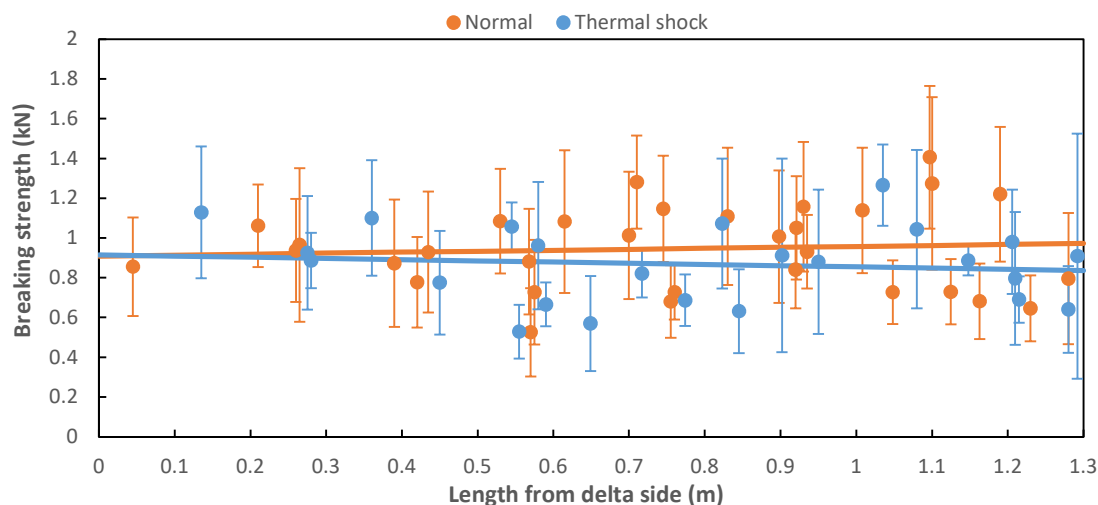


**Figure 6.13** Comparing average normal compressive breaking strength measurements and the average thermally shocked compressive breaking strength measurements, of the 8 x 10 mm cylindrical sample pellets, as a function of the distance from the delta side of the electrode, as well as the linear fits of their data.

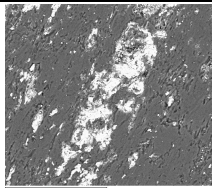
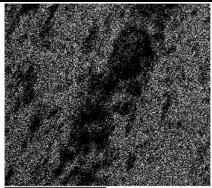
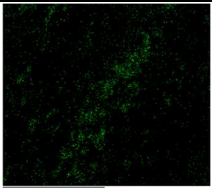
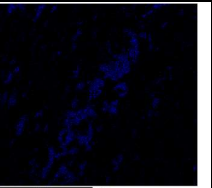
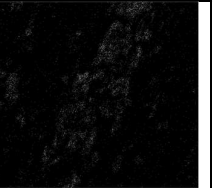
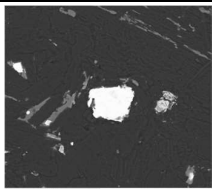
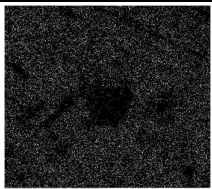
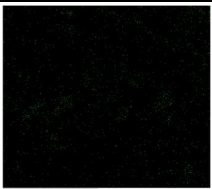
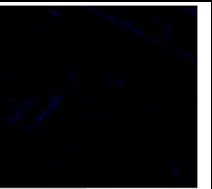
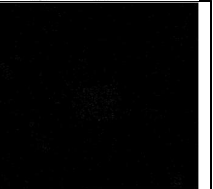
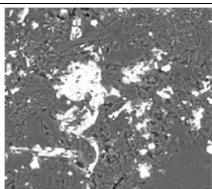
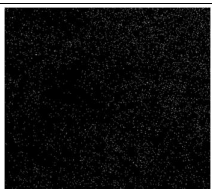
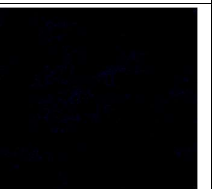
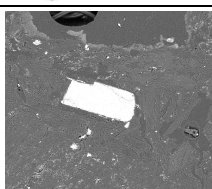
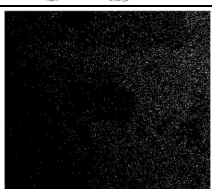
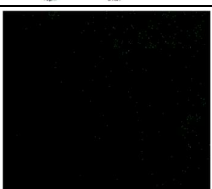
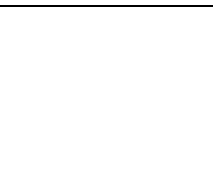
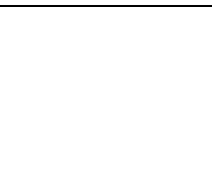
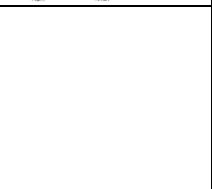
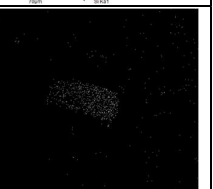
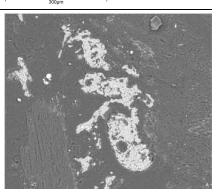
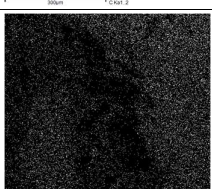
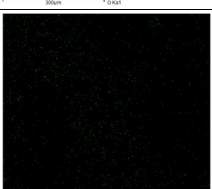
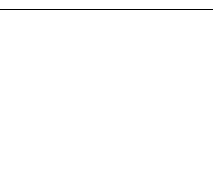
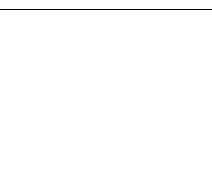
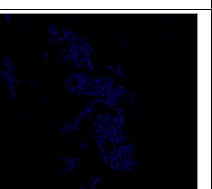
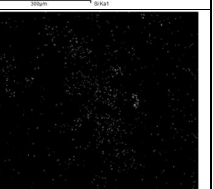
## 6-7 SEM EDS

SEM backscatter images and EDS analysis of samples obtained from the delta end, non-delta end and the middle of the 1.64 core sample are summarised in Table 6.1. Brighter (lighter coloured) sections in these micrographs can be attributed to some impurities that were present in the electrode, which include small amounts of iron, magnesium, aluminium, and silicon as indicated by the EDS analysis. It seems that aluminium and silicon are present in the electrode paste itself and did not originate from the furnace, since these species are present throughout the electrode. Iron and magnesium, however, are only present on the delta side of the electrode, which indicate that these elements in the electrode originate predominantly from the furnace during the smelting process. The carbon EDS analysis shows that the electrode mainly comprise carbon (as expected), while oxygen EDS analysis reveals that the two ends of the electrode had higher amounts of oxygen than the middle section of the electrode. This is indicative of oxidation of the electrode during cool down. This also supports the hypothesis in Section 6-4 that the outside of the electrode has increased pore sizes and volumes.

Table 6.2 lists the SEM backscatter images and EDS analysis of samples obtained from the rusted leftover parts of the casing fins (see Figure 3.12) of the 1.64 m core sample. EDS analysis reveals higher levels of magnesium, aluminium and silicon in these samples, which can be attributed to the electrode fin being an alloy of these metals. Aluminium is a better conductor of electricity than iron or steel. The oxygen EDS analysis show that there is significantly more oxygen present in these cavities than on the delta and non-delta sides of the electrode, which is due to metal species being easier oxidised than carbon. This is also indicative of the electrode being exposed to oxygen as the outer casing of the

electrode burns away, which causes oxidation on the outside of the electrode resulting in a smaller electrode diameter than the diameter of the casing (see sections 6-1 and 6-4). This also contributes to indents where the fins are located as shown in Figure 3.6.

**Table 6.1.** The SEM backscatter images, as well as the EDS analysis for carbon, oxygen, iron, magnesium, aluminium, and silicon for samples taken from the delta- and non-delta end, as well as in the middle of the 1.64 m core sample.

| Name   | SEM                                                                                 | C                                                                                   | O                                                                                   | Fe                                                                                    | Mg                                                                                    | Al                                                                                    | Si                                                                                    |
|--------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|---------------------------------------------------------------------------------------|
| Delta  |    |    |    |    |    |    |    |
|        |    |    |    |    |    |    |    |
| Middle |    |    |    |    |    |    |    |
|        |   |   |   |   |   |   |   |
|        |  |  |  |  |  |  |  |

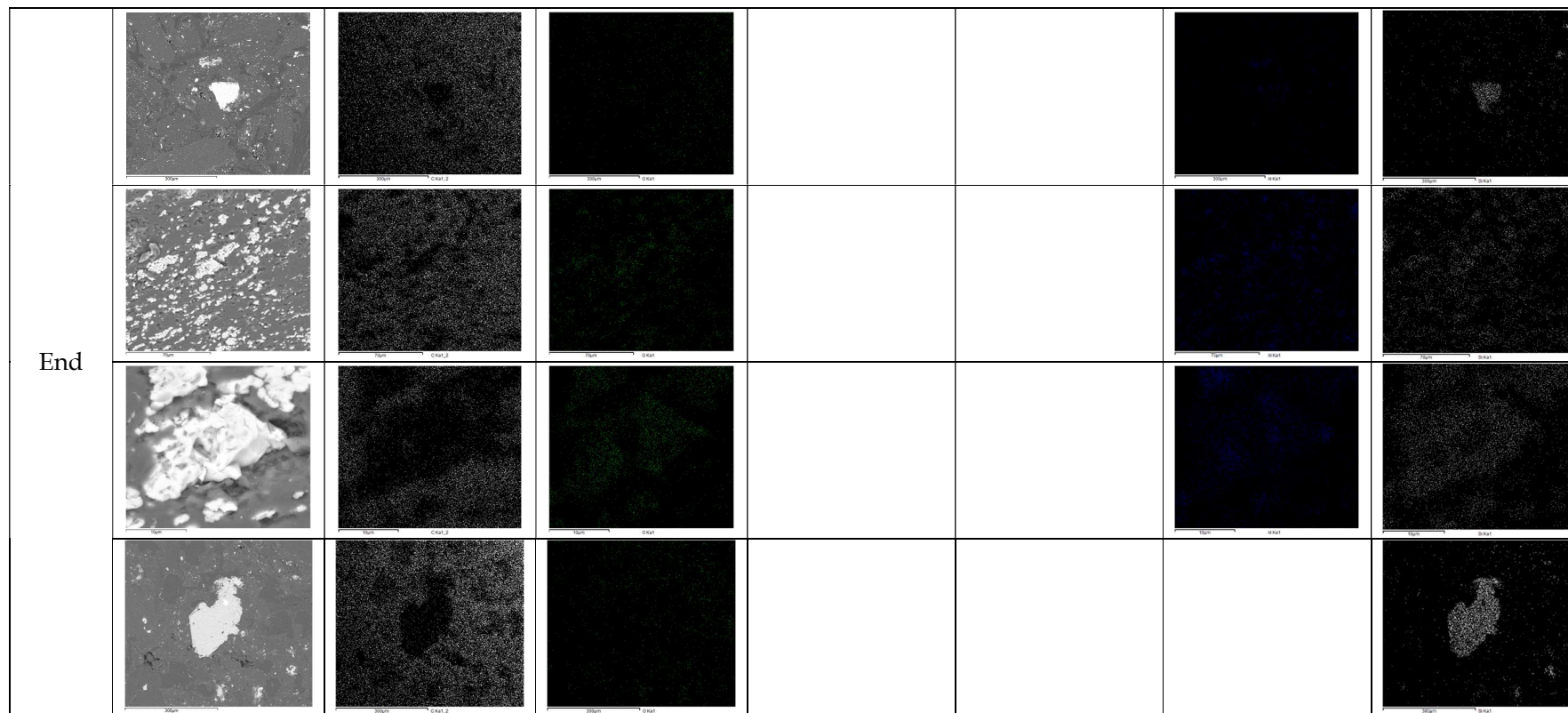
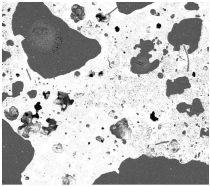
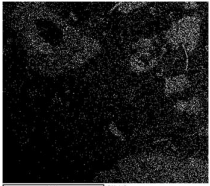
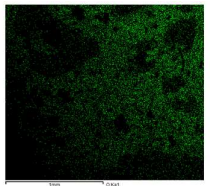
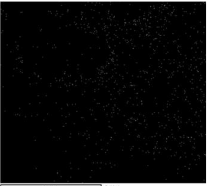
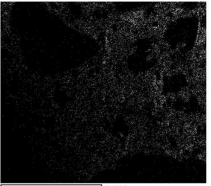
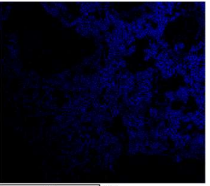
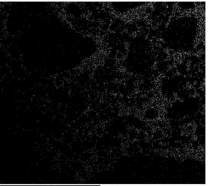
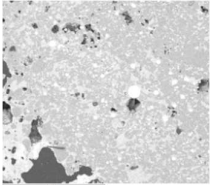
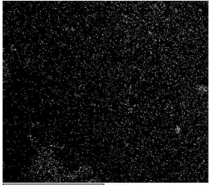
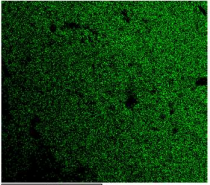
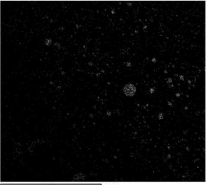
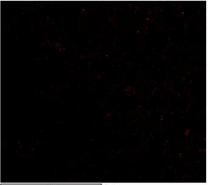
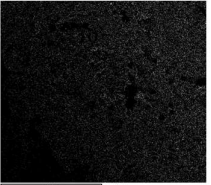
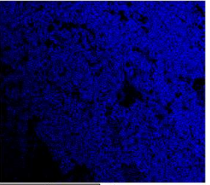
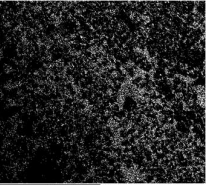
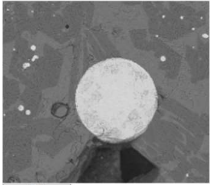
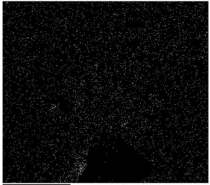
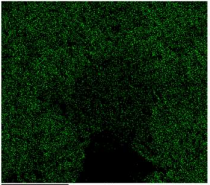
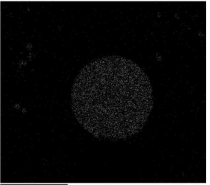
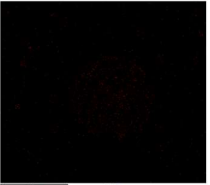
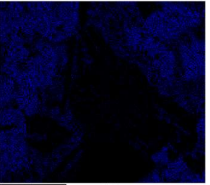


**Table 6.2.** The SEM backscatter images, as well as the EDS analysis for carbon, oxygen, chromium, iron, magnesium, aluminium, and silicon for samples taken from the rusted leftover parts of the casing fins (see Figure 3.12) of the 1.64 m core sample.

| Name                                     | SEM                                                                               | C                                                                                 | O                                                                                 | Cr                                                                                 | Fe                                                                                  | Mg                                                                                  | Al                                                                                  | Si                                                                                  |
|------------------------------------------|-----------------------------------------------------------------------------------|-----------------------------------------------------------------------------------|-----------------------------------------------------------------------------------|------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|
| Rusted leftover parts of the casing fins |  |  |  |  |  |  |  |  |
|                                          |  |  |  |  |  |  |  |  |
|                                          |  |  |  |  |  |  |  |  |

## 6-8 Future use data

The temperature to which S derberg electrode paste is exposed determines to a large degree the DOG. As shown in this study, the DOG, in turn, determines, to a large extent, the resistivities and bulk densities. Therefore, it would be advantageous if S derberg electrode models could accurately replicate the DOG presented here. To aid such comparison and use of the data presented in this study, the resistivities, DOG, and bulk densities associated with the fitted lines at 10 cm intervals across the diameter of the electrode at each of the investigated lengths below the contacts shoes prior to being spatial interpolated to generate the 3D images are presented in Table 6.3.

In Table 6.4 the fit parameters and correlation coefficients for the trendlines presented in Figures 6.1, 6.3a, 6.4, 6.5, 6.7, 6.11 and 6.12 in this chapter are listed. The correlation coefficients show that electrode parameters varies significantly at the top of the electrode. Better correlations are observed lower in the electrode, which is indicative of the electrode being more stable at the bottom. These fit parameters and correlation coefficients could also be used to improve current S derberg electrode models.

**Table 6.3.** The electrical resistivity, DOG and bulk density data associated with the fitted lines in Figures 6.1, 6.3a and 6.5, at 10 cm intervals across the diameter of the electrode at each of the investigated lengths below the contacts shoes, respectively. The data were spatially interpolated to generate the 3D images presented in Figures 6.2, 6.3b and 6.6, respectively.

| Distance from<br>delta side (m)  | Distance below the contact shoes (m) |                       |                       |                       |                       |
|----------------------------------|--------------------------------------|-----------------------|-----------------------|-----------------------|-----------------------|
|                                  | 0.70                                 | 1.15                  | 1.64                  | 2.16                  | 2.70                  |
| Resistivity ( $\Omega \cdot m$ ) |                                      |                       |                       |                       |                       |
| 0                                | $7.42 \times 10^{-5}$                | $7.34 \times 10^{-5}$ | $4.66 \times 10^{-5}$ | $2.92 \times 10^{-5}$ | $2.41 \times 10^{-6}$ |
| 0.1                              | $7.51 \times 10^{-5}$                | $7.37 \times 10^{-5}$ | $4.98 \times 10^{-5}$ | $3.37 \times 10^{-5}$ | $9.25 \times 10^{-6}$ |
| 0.2                              | $7.59 \times 10^{-5}$                | $7.41 \times 10^{-5}$ | $5.30 \times 10^{-5}$ | $3.82 \times 10^{-5}$ | $1.61 \times 10^{-5}$ |
| 0.3                              | $7.67 \times 10^{-5}$                | $7.45 \times 10^{-5}$ | $5.62 \times 10^{-5}$ | $4.27 \times 10^{-5}$ | $2.29 \times 10^{-5}$ |
| 0.4                              | $7.75 \times 10^{-5}$                | $7.49 \times 10^{-5}$ | $5.94 \times 10^{-5}$ | $4.72 \times 10^{-5}$ | $2.98 \times 10^{-5}$ |
| 0.5                              | $7.83 \times 10^{-5}$                | $7.52 \times 10^{-5}$ | $6.26 \times 10^{-5}$ | $5.17 \times 10^{-5}$ | $3.66 \times 10^{-5}$ |
| 0.6                              | $7.91 \times 10^{-5}$                | $7.56 \times 10^{-5}$ | $6.58 \times 10^{-5}$ | $5.62 \times 10^{-5}$ | $4.35 \times 10^{-5}$ |
| 0.7                              | $7.99 \times 10^{-5}$                | $7.60 \times 10^{-5}$ | $6.91 \times 10^{-5}$ | $6.07 \times 10^{-5}$ | $5.03 \times 10^{-5}$ |
| 0.8                              | $8.07 \times 10^{-5}$                | $7.64 \times 10^{-5}$ | $7.23 \times 10^{-5}$ | $6.52 \times 10^{-5}$ | $5.71 \times 10^{-5}$ |
| 0.9                              | $8.15 \times 10^{-5}$                | $7.68 \times 10^{-5}$ | $7.55 \times 10^{-5}$ | $6.97 \times 10^{-5}$ | $6.40 \times 10^{-5}$ |
| 1.0                              | $8.24 \times 10^{-5}$                | $7.71 \times 10^{-5}$ | $7.87 \times 10^{-5}$ | $7.42 \times 10^{-5}$ | $7.08 \times 10^{-5}$ |
| 1.1                              | $8.32 \times 10^{-5}$                | $7.75 \times 10^{-5}$ | $8.19 \times 10^{-5}$ | $7.88 \times 10^{-5}$ | $7.77 \times 10^{-5}$ |
| 1.2                              | $8.40 \times 10^{-5}$                | $7.79 \times 10^{-5}$ | $8.51 \times 10^{-5}$ | $8.33 \times 10^{-5}$ | $8.45 \times 10^{-5}$ |
| 1.3                              | $8.48 \times 10^{-5}$                | $7.83 \times 10^{-5}$ | $8.83 \times 10^{-5}$ | $8.78 \times 10^{-5}$ | $9.13 \times 10^{-5}$ |

| DOG |      |      |      |      |      |
|-----|------|------|------|------|------|
| 0   | 0.32 | 0.39 | 0.48 | 0.59 | 0.86 |
| 0.1 | 0.33 | 0.39 | 0.47 | 0.58 | 0.81 |
| 0.2 | 0.34 | 0.39 | 0.46 | 0.56 | 0.77 |
| 0.3 | 0.34 | 0.39 | 0.45 | 0.55 | 0.73 |
| 0.4 | 0.35 | 0.39 | 0.45 | 0.54 | 0.69 |
| 0.5 | 0.36 | 0.38 | 0.44 | 0.53 | 0.65 |
| 0.6 | 0.37 | 0.38 | 0.43 | 0.52 | 0.61 |
| 0.7 | 0.37 | 0.38 | 0.42 | 0.50 | 0.57 |
| 0.8 | 0.38 | 0.38 | 0.41 | 0.49 | 0.53 |
| 0.9 | 0.39 | 0.38 | 0.41 | 0.48 | 0.49 |
| 1.0 | 0.40 | 0.37 | 0.40 | 0.47 | 0.45 |
| 1.1 | 0.40 | 0.37 | 0.39 | 0.46 | 0.41 |
| 1.2 | 0.41 | 0.37 | 0.38 | 0.44 | 0.38 |
| 1.3 | 0.42 | 0.37 | 0.37 | 0.43 | 0.34 |

| Bulk density (g.cm <sup>-3</sup> ) |      |      |      |      |      |
|------------------------------------|------|------|------|------|------|
| 0                                  | 2.00 | 2.05 | 2.06 | 2.15 | 2.22 |
| 0.1                                | 1.99 | 2.04 | 2.06 | 2.14 | 2.20 |
| 0.2                                | 1.99 | 2.03 | 2.05 | 2.13 | 2.18 |
| 0.3                                | 1.99 | 2.02 | 2.04 | 2.11 | 2.16 |
| 0.4                                | 1.98 | 2.01 | 2.04 | 2.10 | 2.14 |
| 0.5                                | 1.98 | 2.01 | 2.03 | 2.10 | 2.12 |
| 0.6                                | 1.97 | 2.00 | 2.02 | 2.08 | 2.11 |
| 0.7                                | 1.96 | 1.99 | 2.01 | 2.07 | 2.09 |
| 0.8                                | 1.96 | 1.98 | 2.01 | 2.06 | 2.07 |
| 0.9                                | 1.95 | 1.97 | 2.00 | 2.05 | 2.05 |
| 1.0                                | 1.95 | 1.96 | 2.00 | 2.04 | 2.03 |
| 1.1                                | 1.94 | 1.96 | 1.99 | 2.02 | 2.01 |
| 1.2                                | 1.94 | 1.95 | 1.99 | 2.01 | 2.00 |
| 1.3                                | 1.93 | 1.94 | 1.98 | 2.00 | 1.98 |

**Table 6.4.** Fit parameters and correlation coefficients for trendlines presented in Figures 6.1, 6.3a, 6.4, 6.5, 6.7, 6.11 and 6.12

| Graph          | Trendline | Fit Parameters                                                | Correlation Coefficients (R <sup>2</sup> ) |
|----------------|-----------|---------------------------------------------------------------|--------------------------------------------|
| Figure 6.1     | 0.70 m    | $y = 8.11\text{E-}08x + 7.42\text{E-}05$                      | 0.1133                                     |
|                | 1.15 m    | $y = 3.79\text{E-}08x + 7.34\text{E-}05$                      | 0.0408                                     |
|                | 1.64 m    | $y = 3.20\text{E-}07x + 4.66\text{E-}05$                      | 0.5783                                     |
|                | 2.16 m    | $y = 4.51\text{E-}07x + 2.92\text{E-}05$                      | 0.7956                                     |
|                | 2.7 m     | $y = 6.84\text{E-}07x + 2.41\text{E-}06$                      | 0.894                                      |
| Figure 6.3 (a) | 0.70 m    | $y = 5.79\text{E-}02x + 3.31\text{E-}01$                      | 0.0611                                     |
|                | 1.15 m    | $y = -2.19\text{E-}02x + 3.95\text{E-}01$                     | 0.0277                                     |
|                | 1.64 m    | $y = -7.90\text{E-}02x + 4.77\text{E-}01$                     | 0.1951                                     |
|                | 2.16 m    | $y = -1.21\text{E-}01x + 5.88\text{E-}01$                     | 0.2626                                     |
|                | 2.7 m     | $y = -3.91\text{E-}01x + 8.45\text{E-}01$                     | 0.8516                                     |
| Figure 6.4     | Average   | $y = -2.46\text{E-}04x + 1.95\text{E+}00$                     | 0.0132                                     |
| Figure 6.5     | 0.70 m    | $y = -5.62\text{E-}02x + 2.00\text{E+}00$                     | 0.3555                                     |
|                | 1.15 m    | $y = -8.33\text{E-}02x + 2.05\text{E+}00$                     | 0.666                                      |
|                | 1.64 m    | $y = -6.18\text{E-}02x + 2.06\text{E+}00$                     | 0.2549                                     |
|                | 2.16 m    | $y = -1.13\text{E-}01x + 2.15\text{E+}00$                     | 0.6185                                     |
|                | 2.7 m     | $y = -1.86\text{E-}01x + 2.22\text{E+}00$                     | 0.792                                      |
| Figure 6.7     | Average   | $y = 3.81\text{E+}00x^2 - 5.05\text{E+}00x + 8.54\text{E+}00$ | 0.1213                                     |
| Figure 6.11    | Average   | $y = 4.88\text{E-}02x + 9.09\text{E-}01$                      | 0.0057                                     |
| Figure 6.12    | Average   | $y = -6.01\text{E-}02x + 9.15\text{E-}01$                     | 0.012                                      |

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# Chapter 7

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## Project evaluation and future perspectives

In Section 7-1 of this chapter, the project is evaluated within the context of the objectives stated in Chapter 1 (Section 1.2). In Section 7-2, the recommendation for possible future work will be discussed.

### 7-1 Project evaluation

To evaluate the project, the specific objectives stated in Section 1.2 are repeated, followed by a brief overview of achievements related to each objective.

- (i) Objective 1: *Obtain a full-size Söderberg electrode from a ferroalloy producer to serve as a case study electrode, together with a reasonable period of operational history of this electrode, prior to it being removed from the submerged arc furnaces (SAFs). Obtaining sample material would usually not be considered an objective for a PhD study, since such materials are usually readily available. However, finding and convincing a ferroalloy producer to donate a full-size Söderberg electrode and supplying the operational history thereof for research is not easy.*

In 2013, a large South African ferrochrome producer decided to rebuild two (named Furnace 1 and Furnace 3) of their operational submerged arc furnaces (SAFs). Electrode 2 from Furnace 3 was obtained as a case study electrode. This electrode was 1.4 m in diameter, including the steel casing, before it burned/melted off. The electrode was approximately 3 m long from just below the contact shoes to the tip.

The ferrochrome producer also provided the operational data for the SAF from which the case study electrode was removed for a month prior to the furnace rebuild. This data indicated that the amount of electrode paste blocks (Figure 5.1) loaded into, as well as the number of casings (Figure 5.2) added to each electrode during its last month of operation were normal. The provided electrode slipping data also showed normal and relatively consistent slipping, with only one longer than a normal slip (i.e. 40 cm) taken on Electrode 1 (Figure 5.3a) on 19 July 2013. This is not considered a long slip (i.e. usually defined as  $\geq 100$  cm). Except for a lower current on Electrode 1 during the period of 16 to 19 July 2013 (Figure 5.5), the currents were relatively stable and within the targeted range on all three electrodes for the entire month. This lower current (on Electrode 1 during the period 16-19 July 2013) was probably due to a suspected shorter than normal electrode. However, the current on this

electrode recovered to normal after the aforementioned 40 cm slip was taken on 19 July 2013. Lastly, both the solid (Figures 5.6 and 5.7) and liquid (Figures 5.8 and 5.9) paste levels were stable, with no abnormal levels during the month.

Considering all the above-mentioned, in conjunction with a visual inspection of the case study electrode (that occurred during sampling) and its length before being cut off, it can be concluded with a very high level of confidence that it was representative of stable SAF operation.

- (ii) Objective 2: *Sampling of the case study Söderberg electrode, to yield representative samples that can be handled conveniently in the laboratory. Due to the size of typical industrial Söderberg electrodes, sampling will be challenging.*

As the full-size case study Söderberg electrode would have been impossible to analyse, it was processed into smaller workable samples. This was done firstly by cutting off the electrode 0.5 m below the contact shoes (Figure 3.3). The operational personnel did not want to cut the electrode off closer to the contact shoes, since it would have increased the operational risk during start-up. The case study electrode was removed from the SAF with appropriate rigging equipment (Figure 3.5).

Next, 100 mm (outer diameter) core samples (an example of which is presented in Figure 3.7) were drilled out at lengths of 0.70, 1.15, 1.64, 2.16, and 2.70 m below the contact shoes. The core drilling was done from the outside towards the delta side, straight through the electrode.

These core samples were then further processed by cutting them into discs (Figure 3.13) that were 10 mm thick, with a fit-for-purpose modified cut-off grinder (Figure 3.8). Cylindrical sample pellets (Figure 3.15b) were then drilled out from these disks. The cylindrical sample pellets and the leftover pieces from the drilling (Figure 3.15a) were used in most of the experiments.

- (iii) Objective 3: *To characterise the case study Söderberg electrode in terms of electrical resistivity, degree of graphitisation, bulk density, porosity, compressive breaking strength and thermal shock resistance. Changes in these parameters will be investigated as a function of electrode length below the contact shoes, as well as from the delta to the non-delta side.*

The characterisation results indicated no significant electrical resistivity (Figure 6.2), degree of graphitisation (DOG) (Figure 6.3b), and bulk density (Figure 6.6) changes from 0.7 to 2.7 m on the non-delta side of the electrode, while these characteristics changed relatively significantly on the delta side of the electrode. The area where the submerged arc would mostly like jump off the electrode clearly had the lowest resistivity, as well as highest DOG and bulk density.

The determination of porosity with computerised tomography (CT) scanning combined with image processing (Figure 4.6) was found to likely be more accurate than a conventional method. From the presented porosity data (Figure 6.7a), no significant differences in porosities, as a function of length below the contact shoes, were evident. However, slight increases in porosities were observed near the electrode perimeter on both the delta and non-delta sides, which were fitted best with a second-order polynomial function (Figure 6.7b). It was postulated that chemical corrosion, i.e. oxidation of the carbonaceous material, increases the pore volumes in the baked electrode paste near the perimeter. Scanning electron microscopy images seemed to confirm this postulation (Figures 6.9 and 6.10), as the pores directly on the delta side were significantly larger than those further into the electrode. The SEM-EDS images in Table 6.1 show an increase in oxygen on the out side of the electrode. This is an indication that oxidation might have occurred which helps prove the above-mentioned hypothesis.

The compressive breaking strength data revealed no significant trends (Figure 6.11). Therefore, operational personnel should not be concerned about breaking strength in the region well below the baking isotherm being during stable operation. Thermal stresses that form in and just below the baking isotherm, as well as thermal stress differences between the perimeter and core of the electrode formed due to prolong furnace downtime, remain the most likely reasons for breaks of electrodes with no other apparent dysfunctionalities.

A very small reduction in compressive strength of thermally shocked cylindrical sample pellets, as measured from the delta side to the non-delta side of the electrode (only 0.1356 kN), was observed (Figure 6.12). This might indicate that the non-delta side of Söderberg electrodes is slightly more susceptible to thermal shock breakages than the delta side. However, as the change was very small, it could be statistically insignificant.

As far as the candidate could assess, this work presents the first characterisation of a real-world Söderberg electrode, published in the peer-reviewed journal public domain. Notwithstanding this novelty, this study had several limitations. For instance, measurements of the characteristics were conducted at room temperature, instead of at the operational temperatures. Furthermore, the study was conducted on an electrode removed from an open-SAF producing ferrochrome, for which some characteristics might differ from Söderberg electrodes operating in other furnace types and smelting different materials. However, all these limitations are similar to furnace dig-out studies (e.g. (Nell & Joubert, 2013; Steenkamp *et al.*, 2018; Wedepohl & Barcza, 1983), which have, to a large degree, formed our current understanding of zones within furnaces. Therefore, the results presented are valuable, notwithstanding the aforementioned limitations. Another data limitation of the current

work was that the case study electrode did not include the area directly above, in, and just below the contact shoes. Understanding this area is critical. Unfortunately, this section of the electrode could not be obtained, since that would have entailed starting up the furnace with a green (unbaked) electrode tip, which would have increased the operational risk significantly.

- (iv) Objective 4: *Contextualise the above-mentioned characterisation results within the context of previous findings and/or modelling studies.*

To contextualise the resistivity results (Figure 6.2), they were firstly compared to high temperature bulk bed electrical resistivity values reported relatively recently for carbonaceous reductants (Surup *et al.*, 2020). Such high temperature bulk bed resistivities simulate the so-called coke bed (area around and below electrode tips in a SAF), where reductants serve as one of the primary electrical conductors. At 1 600 °C, it was found that the reductant bulk bed resistivities ranged from approximately  $2 \times 10^{-2}$  to  $5 \times 10^{-3} \Omega \cdot m$  (Surup *et al.*, 2020). These values were approximately 60 to 250 times higher (therefore less conductive) than the approximate  $8.40 \times 10^{-5} \Omega \cdot m$  resistivity observed from 0.7 to 2.7 m below the contact shoes on the non-delta side of the case study electrode. Correspondingly, the high temperature reductant bulk bed resistivities were approximately 2 000 to 8 000 times higher than the  $2.4 \times 10^{-6} \Omega \cdot m$  obtained at 2.7 m below the contact shoes on the electrode delta side.

The resistivity data were further compared to S  derberg electrode paste resistivity values derived from finite element thermo-electrical modelling (McDougall *et al.*, 2004; Tangstad *et al.*, 2019). Although the modelled values from McDougall *et al.* (2004) and Tangstad *et al.* (2019) and the values presented in the current work are in the same order, the differences in the corresponding values ( $5.88 \times 10^{-6} - 2.63 \times 10^{-6} = 3.25 \times 10^{-6} \Omega \cdot m$  versus  $8.48 \times 10^{-5} - 2.41 \times 10^{-6} = 8.24 \times 10^{-5} \Omega \cdot m$ ) suggest that the models do not quickly enough account for lower resistivities as a function of electrode length below the contact shoes. Furthermore, no evidence could be found that the models (Larsen *et al.*, 2013; McDougall *et al.*, 2004; Tangstad *et al.*, 2019) accounted for the very apparent asymmetrical variation of resistivities observed in the case study electrode (Figure 6.2).

As far as the candidate could assess, no real-world, or modelled S  derberg electrode has yet been presented in the peer-reviewed public domain for any of the DOG, bulk density, porosity, compressive breaking strength or thermal shock resistance; therefore, it was not possible to contextualise the measured results with such data.

- (v) Objective 5: *Indicate topics that should receive research attention to advance knowledge in this under-investigated study field.*

Future perspectives are discussed in the next section.

Notwithstanding the limitations mentioned, all the objectives of this study were successfully achieved. Therefore, the study can be considered successful. Furthermore, the results presented in Chapter 6 were published (Glastonbury *et al.*, 2021, which was included as Appendix A) in the journal *Metals* (an MDPI journal). *Metals* is a high-impact (impact factor 2.117 for 2019), internationally-accredited, peer-reviewed journal. This proves that the candidate made a contribution to science, which is one of the main criteria for a PhD study. The results of this study will be of help to optimise in-house unpublished models and operational models of smelters.

## 7-2 Future prospective

Although this study was successfully completed, it was not without limitations, and additionally, the study also revealed new and/or additional research questions/topics that need to be considered in future research. Therefore, to help stimulate this much-understudied research field, several questions/topics were identified:

- (i) It is currently not evident from the literature whether the very asymmetrical characteristics of the electrical resistivity (Figure 6.2), DOG (Figure 6.3b), and bulk density (Figure 6.6) can be accurately predicted by Söderberg electrode models, e.g. (Bermúdez *et al.*, 1998; Larsen *et al.*, 2013; McDougall *et al.*, 2004). All such models predict temperature isotherms/curves. The temperature to which Söderberg electrode paste is exposed determines the DOG, to a large degree. As shown in this study, the DOG determines the resistivities and bulk densities to a large extent. Therefore, it would be advantageous if Söderberg electrode models could accurately replicate the DOG presented in this study.
- (ii) The DOG of calcined anthracite and coal tar pitch should be investigated separately (not as a mixture in electrode paste), in relation to electrical resistivity and bulk density, as well as other characteristics such as thermal expansion. This will enable a better understanding of the individual contribution of these two components, to the combined results observed for electrode paste.
- (iii) The manual marking of the areas on the CT images (e.g. Figure 4.6b), to be included in the calculations for porosity, makes the method time consuming and less attractive. Research should be done to automate the determination of the particle rims within the CT image slices and calculation of porosities and pore volumes.



- (iv) Thermal shock remains the most likely cause of Söderberg electrode breaks. However, the thermal shock results presented in this study (Figure 6.12) were not conclusive. Therefore, it is proposed that an entire postgraduate study be dedicated to formulating and testing various thermal shock procedures, so that this very important characteristic can be quantified better.
- (v) Considering the value of studies such as this, the candidate strongly recommends that smelters should donate similar case study electrodes to researchers, and if possible, include the area directly above and below the contact shoes in such case study electrodes. As an example, the candidate belatedly became aware of two separate 63 MVA closed-SAFs, for which the electrode outer casing diameters were increased from 1.5 to 1.6 m. In order to do so, six entire electrode columns, including the casings with un-melted solid paste at the top, down to the baked electrode tips at the bottom, were removed and discarded. However, no one in the research community was notified, nor did the company/operational personnel reach out to researchers. Such large-scale, real-world samples are scarce; therefore, opportunities such as this should not be missed.

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# Characterisation of a Real-World Søderberg Electrode

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**Abstract:** Very little research on Søderberg electrodes has been published in the journal peer reviewed public domain. The main aim of this work is to characterise a Søderberg electrode that was cut off approximately 0.5 m below the contacts shoes of a submerged arc furnace. Additionally, the characterisation data can be used to verify if Søderberg electrode models accurately predict important electrode characteristics. The operational history (slipping, current, and paste levels) proved that the case study electrode was a representative specimen. The characterisation results indicated no significant electrical resistivity, degree of graphitisation (DOG), and bulk density changes from 0.7 to 2.7 m on the non-delta side (outward facing), while these characteristics changed relatively significantly on the delta side (inward facing) of the electrode. The area where the submerged arc would mostly like jump off the electrode had the lowest resistivity, as well as highest DOG and bulk density. No significant difference in porosity as a function of length below the contact shoes were observed; however, slight increases occurred near the perimeters. It was postulated that oxidation of carbon resulted in increased pore volumes near the electrode perimeter. No significant difference in



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compressive breaking strength was observed over the electrode area investigated.

**Keywords:** Søderberg electrodes; submerged arc furnace (SAF); ferro-alloy production; ferrochrome; electrical resistivity; degree of graphitisation; bulk density; porosity; compressive breaking strength

## 1. Introduction

Since its development in the early 1900s [1], Søderberg electrodes have been used extensively in submerged arc furnaces (SAFs) to conduct electrical current from a transformer(s) to the smelting zone [2]. Various authors have presented representations of a Søderberg electrode column and/or described the processes associated with it, e.g., [2–10]. Such a column typically consists of a cylindrical steel casing that extends from a building level well above the furnace, down into the smelting zone. The casing serves as the mould for the electrode paste, which contains a binder (e.g., coal tar pitch) mixed with a solid aggregate (e.g., calcined anthracite or coke), to form the electrode during operation. The casing also has longitudinal fins, which add dimensional stability to the casing and the unbaked electrode. Resistive heating of current passing through the casing, fins, and paste, as well as heat conducted upward from the SAF in the electrode column melt the electrode paste at temperatures above its softening point to fill the casing. Electrode paste softening temperatures have been reported to be in the range of 65 to 134 °C [11]. Solid electrode paste

is added at regular intervals via the top of the electrode casing to maintain a steady level of solid (un-melted) and liquid paste (that have filled the casing). The casing is slipped (lowered in relation to clamping devices that hold the electrode) relatively small distances on a shift basis (usually 8 h long), and longer slips are taken if an electrode break occurs, or if the electrode is shorter than ideal. All the afore-mentioned result in the melted paste being exposed to increasing temperatures as it approaches the so-called contact shoes. Contact shoes are large copper (or copper alloy) components that are pressed against the casing to conduct electrical current into the electrode. Before the liquid electrode paste reaches the bottom of the contact shoes, it should ideally be transformed into solid carbonaceous material. The temperature at which the paste transforms from a liquid to a solid is termed the baking isotherm. This transformation temperature has been reported to be between 450 and 475 °C [11]. As the carbonaceous electrode descends further into the SAF temperatures increase, which results in further baking.

Good Söderberg electrode management, together with other aspects such as maintaining a suitable metallurgical balance (to obtain product within specifications and the correct slag chemistry), high electrical power input (assuming fixed specific energy consumption, production volume is directly related to power input), and feeding the furnace appropriately (without feed it cannot produce and asymmetrical feed could lead to damage) form the basis for safe (occupational health and safety of workers), environmentally responsible (limiting emissions), and profitable smelting operations. However, good Söderberg electrode management implies that a multitude of actions and tracking of parameters need to take place on a shift and daily basis.

Very harsh conditions in the SAFs lead to severe mechanical and thermal stresses, which result in unavoidable wear of the electrode tips. Furthermore, thermal stresses associated with shutdowns and operational instability may cause anything from inconsequential hard electrode breaks to disastrous green breaks [7,8]. Broken electrodes can negatively impact process productivity (e.g., lower electrical power input, damage to equipment, or even prolonged shutdowns to repair damage), personnel occupational health and safety, as well as environmental emissions (e.g., process instability often result in higher than normal atmospheric emissions).

Notwithstanding the importance of Söderberg electrode design and management to ensure responsible, safe, and profitable smelting operations, very little research focussing specifically on Söderberg electrode related aspects have been published in the journal peer reviewed public domain. For instance, as far as the authors could assess (using the “Scopus” scientific search engines with the key words “Soderberg electrode”), only 5 such papers have been published in the last decade (i.e., 2011 to 2020) [4,11–14]. Several conference proceeding papers have also been published during this time, e.g., [2,3,5,15]. These conference proceeding papers also gave valuable topic specific insights. However, many such contributions were made principally to demonstrate the capability of commercial companies to serve their clients instead of divulging all relevant information as is common in academic journal papers.

The main aim of this work was to characterise a largely intact Söderberg electrode, removed from a ferrochrome SAF during a shutdown to rebuild many of the furnace components. The operational history for a month prior to the shutdown, procedures followed to sample the electrode and experimentally determined characteristics (e.g., electrical resistivity, degree of graphitisation, bulk density, porosity, and compressive breaking strength) are presented. As far as the authors could assess, such a characterisation of a real-world Söderberg electrode have not yet been presented in the peer reviewed public domain. Such data will contribute towards a better understanding of electrode management by operational personnel and assist researchers in identifying unresolved questions. Additionally, the characterisation data can be used to verify such characteristics in Söderberg electrode models, which have up to now mainly been restricted to temperature, current densities, and thermal stress profiles, e.g., [2,3,5,6].

## 2. Materials and Methods

### 2.1. Söderberg Electrode Sampling

On 31 July 2013, an operational ferrochrome SAF with 1.4 m casing outer diameter Söderberg electrodes was switched off, after following the appropriate preparations for a furnace rebuild. The furnace was allowed to cool down for 5 days. Thereafter, the number 2 electrode (Figure 1a indicating the numbering from a top view), was cut off with a diamond wire saw approximately 0.5 m below the contact shoes, as indicated in Figure 1b. The electrode stump was removed from the furnace with appropriate rigging equipment. Then, 100 mm outer diameter core drill samples were collected, with the 100 mm sample centred at 0.70, 1.15, 1.64, 2.16, and 2.70 m below the contact shoes. This core drilling was done through the entire diameter of the electrode, from the outside towards the delta side of the electrode. Here the term “delta” refers to the side of the electrode facing the centre of the SAF (see Figure 1a). An example of such a core drilled sample is presented in Figure 1c. The 100 mm diameter core samples were then cut into 10 mm disks (examples indicated in Figure 1d) with a commercial cut off grinder that was modified for the purpose. Precise lathe turned; tight fitting bushings were installed in the pivot point of this cut-off grinder to ensure accurate cutting. Additionally, it was permanently fitted on a table with specially designed cradles to hold the 100 mm tube-like core drilled electrode sample perpendicular to the blade of the cut-off grinder. Cylindrical sample pellets with outer diameters of 8 mm were then drilled out from these 10 mm thick disks with an appropriate diamond coated core drill fitted in a bench drill, as indicated in Figure 1e,f. The condition of a specific 100 mm thick disk determined how many such 8 mm 10 mm cylindrical sample pellets could be obtained from it, with 10 being the norm. These 8 mm 10 mm cylindrical sample pellets were used in all electrode characterisation experiments, unless stated otherwise. Since many of the characterisation techniques involved destructive analysis, the investigated characteristics were not performed on material sampled from the same positions within the case study electrode. Therefore, it was not possible to directly correlate (via correlation graphs) characteristics with one another.

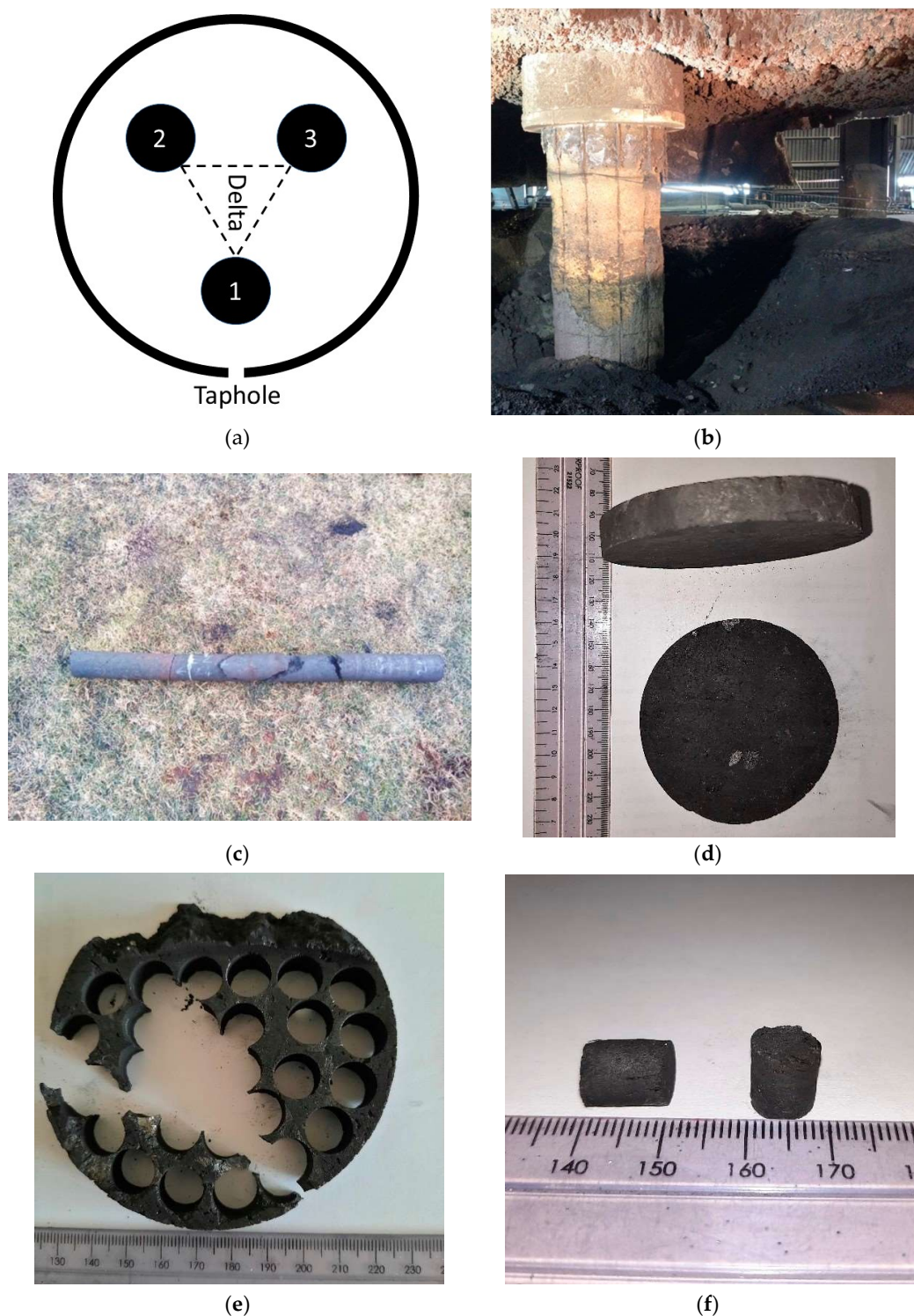


**Figure 1.** (a) Diagram from a top view indicating the electrode numbering of the SAF from which the electrode was removed; (b) photo indicating how the electrode was cut off, approximately 0.5 m below the contact shoes with a diamond wire saw; (c) an example 100 mm outer diameter core drill sample, collected by core drilling through the entire diameter of the electrode, from the outside towards the delta side of the electrode; (d) examples of 10 mm thick disks that were cut from the 100 mm diameter core samples; (e) example 10 mm thick disk, after 8 mm outer diameter cylindrical sample pellets were drilled out; (f) example 8 mm × 10 mm cylindrical sample pellets that were used in most experiments.

## 2.2. Electrical Resistivity Measurements

The electrical resistivities of cylindrical sample pellets were measured at room temperature using a GW Instek resistance precision meter (Model LCR-6020 Series). The meter can be used to measure inductance (L), capacitance (C), and resistance (R) of samples over a wide frequency (10 Hz–20 kHz), resistance (0.99–99.9 MΩ), and voltage range (10.00 mV–2.00 V). As indicated in Figure 2a, a two-probe device was manufactured (by the North-West University Instrument Makers) similar to the design presented by Heikkilä et al. [16] and connected to the LCR meter. This two-probe device had a bottom and a top probe, which were both turned from solid copper. The stainless steel tightening wheel mechanism (Figure 2a) was used to gently tighten a cylindrical sample pellet in-between these copper probes prior to a measurement. A torque wrench was then used to fasten the hex/Allen key located on top of the stainless steel tightening wheel mechanism further. This ensured that each sample was set under the same torque pressure (i.e., 0.5 Nm). The crocodile clamps of the LCR meter were clamped into fit-for-purpose holes drilled in the copper probes. These holes ensured consistent placement of the clamps on the probes, as well as consistent contact area between the clamps and the probes. The frame and connectors that attached the probes to the frame were made of polyvinyl chloride (PVC) plastic, to isolate the frame from the sample. As illustrated in Figure 2b, the face of the bottom copper probe on which the cylindrical sample pellet sat had a small rim on the outside, while the corresponding face of the top copper probe was flat. These shapes were used to ensure that the cylindrical sample pellets were held in-between the two copper probes in a repeatable manner. Both copper probes were also set on a 360° articulating spring mechanism. This kept the ends of the cylindrical sample pellet perpendicular to the probe faces, even if the cylindrical sample pellet ends were not perfectly perpendicular to the cylindrical sample pellet sides.

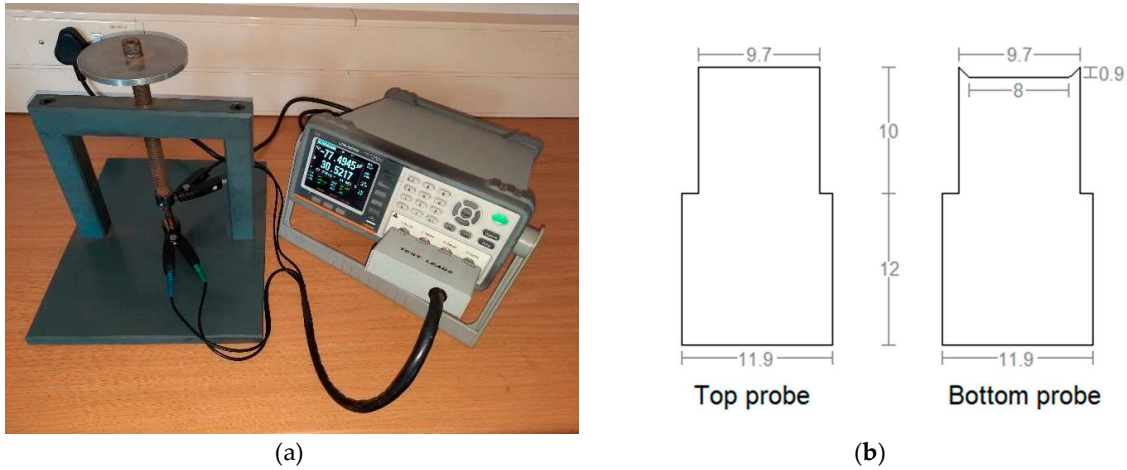


**Figure 2.** (a) Photo indicating the GW Instek resistance precision meter (Model LCR-6020 Series) and two-probe device, similar to the design presented by Heikkilä et al. [16], used to measure electrical resistivity of the cylindrical sample pellets; (b) illustration indicating the shapes of the copper probes, in-between which the cylindrical sample pellets were held.

To ensure precision and accuracy, the LCR meter was switched on for 30 min and a daily calibration was conducted before measurements commenced. Cylindrical copper, aluminium, and stainless steel samples, which had dimensions similar to the cylindrical sample pellets, were measured during calibration. The calibration values were stored by the LCR meter to correct for errors caused by impedance of the experimental setup. The resistance values of the measured cylindrical sample pellets were calculated from Ohm's law:

$$R = V/I, \quad (1)$$

where  $R$  is the resistance ( $\Omega$ ),  $V$  is the voltage across the sample (V), and  $I$  is the measured current (A). Assuming uniform current distribution, the resistivity was calculated by Equation (2):

$$\rho = (V \cdot A) / (I \cdot H), \quad (2)$$

where  $\rho$  is the material resistivity ( $\Omega \cdot \text{m}$ ),  $V$  is the voltage across the sample,  $I$  is the measured current (A),  $A$  is the cross-sectional area of the cylindrical sample ( $\text{m}^2$ ), and  $H$  is the height of the sample (m).

### 2.3. Degree of Graphitisation Determinations

The degree of graphitisation (DOG) was determined with the so-called interlayer spacing or  $d_{002}$  method, based on X-Ray Diffraction (XRD) analysis [17]. This method is one of at least three recognised methods to determine DOG [18]. The powder generated during drilling of the 8 mm  $\times$  10 mm cylindrical sample pellets (as explained in Section 2.1) were used for this purpose. A small amount of silicon metal powder, used as a reference material, was added to the powdered Söderberg electrode material and mixed thoroughly, prior to analysis. The prepared samples were analysed on a Bruker D8 A25 DaVinci X-ray Diffractometer and LynxEye™ SuperSpeed Detector with xyz-stage mounted and using Cu-K radiation. The DOG was calculated with Equation (3) [17]:

$$\text{DOG} = (3.440 - d_{002}) / (3.440 - 3.354) = (3.440 - d_{002}) / 0.086, \quad (3)$$

where DOG is the degree of graphitisation (unitless, with 1 indicating fully graphitised and 0 fully un-graphitised), 3.440 is the interlayer spacing of fully un-graphitised carbon (nm), 3.345 is the interlayer distance for pure graphite (nm), and  $d_{002}$  is the interlayer spacing derived from XRD analysis (nm).

### 2.4. Bulk Density Measurements

Bulk densities were measured using the ASTM C20-00 method [19]. Cylindrical sample pellets with 18 mm  $\times$  10 mm dimensions, obtained in a similar manner as the 8 mm  $\times$  10 mm cylindrical sample pellets (described in Section 2.1), were used in these determinations. The samples were first air dried at 105 °C for 3 h in an oven. After cooling in a desiccator, the samples were weighted ( $D$ ). They were then saturated by boiling them in distilled water for 2 h. Thereafter, the samples were weighted in water to get the saturated weight ( $S$ ). The bulk density was calculated with the following formula:

$$\rho = [D / (D - S)(\rho_o - \rho_L)] + \rho_L, \quad (4)$$

where  $\rho$  is the bulk density of the sample ( $\text{g} \cdot \text{cm}^{-3}$ ),  $D$  the sample weight after drying,  $S$  the saturated sample weight,  $\rho_o$  the density of water (taken as  $0.9982 \text{ g} \cdot \text{cm}^{-3}$ ), and  $\rho_L$  the density of air (taken as  $0.0012 \text{ g} \cdot \text{cm}^{-3}$ ).

Bulk densities were also measured with an AccyPyc II 1340 gas pycnometer. Söderberg electrode pieces remaining after the 8 mm  $\times$  10 mm cylindrical sample pellets were drilled out of the 10 mm thick disk (Figure 1e) were used for this purpose. Each sample was first weighed and then placed in a 35  $\text{cm}^3$  sample holder in the instrument chamber. The chamber was purged 5 times with 99.99% helium. A known quantity of gas at a known pressure was then expanded into the chamber and the new pressure measured. The difference between the initial and final pressures and the known volume of the chamber is then used (with the gas law) to calculate the volume of the sample. And with the sample mass, the sample bulk density could be calculated.

### 2.5. Porosity Determinations

Computerized tomography (CT) scanning was used to generate sample image slices, which could be processed to determine porosity. A Nikon XT H 225 ST with a 225 kV reflection target source and a Perkin Elmer 1620 detector CT instrument was used. Similar to the bulk density determinations (described in Section 2.4), Söderberg electrode pieces remain-

ing after the 8 mm  $\times$  10 mm cylindrical sample pellets were drilled out of the 10 mm thick disk (Figure 1e) were used. The samples were placed in glass poly tops stacked 6 high and wrapped in cellophane to keep them aligned and easy to handle. Each poly top was scanned individually, with 2000 sample image slices being generated per poly top. From these 2000 image slices per sample, 10 images which were evenly spaced (every 160 images between images 200 and 1800) were used for the actual porosity determinations. An example of a CT image slice is presented in Figure 3a. ImageJ (<https://imagej.nih.gov/ij/>, accessed 20/10/2020) was used to analyse the CT image slices. This was done by adjusting the threshold value so that the pores and background was red in colour (Figure 3b) in all images. In each image, the boundaries of the solid particle(s) visible in the CT image slice were then manually marked (indicated by the yellow line and white markers in Figure 3b). Thereafter, the measurement tool of ImageJ was used to determine the surface area of red pixels within the selected area(s), as a percentage of all the pixels within the selected area(s). This corresponded to the percentage pores in the particular CT image slice.

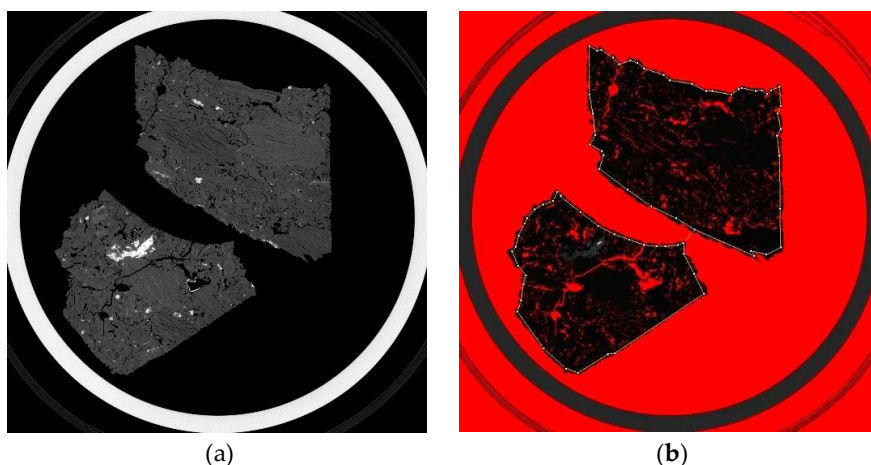


**Figure 3.** (a) An example of a CT image slice; (b) ImageJ (<https://imagej.nih.gov/ij/>, accessed 20/10/2020) was used to analyse the CT image slices. The threshold value was adjusted so that the pores and background was red in colour. The boundaries of the solid particle(s) visible in the CT image slice were manually marked (indicated by the yellow line and white markers).

## 2.6. Compressive Breaking Strength Measurements

The compressive breaking strengths of 8 mm  $\times$  10 mm cylindrical sample pellets were determined using an Ametek Lloyd Instruments LRXplus 5 kN strength tester. NEXYGEN-Plus material test and data analysis software was used to control the instrument and capture the generated data. The speed of the compression plates moving towards one another was kept constant at 10 mm min<sup>-1</sup> during all compressive strength tests. The 8 mm  $\times$  10 mm cylindrical sample pellets were placed upright in the breaking strength machine (flat sample faces on compression plates), and the maximum load to induce breakage of each sample pellet was recorded.

## 2.7. Statistical Handling of Data

For electrical resistivity, between 10 and 30 of the 8 mm  $\times$  10 mm cylindrical sample pellets from a single 10 mm thick sample disk was measured, and each such pellet was measured 15 times. DOG determinations were only done once on each sample considered. In the ASTM C20-00 density experiments, 2 of the 18 mm  $\times$  10 mm sample pellets from a single 10 mm thick sample disk were considered, and each such pellet was used only once. In the gas pycnometry analyses, cuts off of sample pellets were used, and each analysis was repeated 5 times. Porosities based on CT scanning and image processing was conducted

as indicated in Section 2.6. For compressing breaking strength, between 10 and 30 of the 8 mm 10×mm cylindrical sample pellets were tested from each considered 10 mm thick sample disk.

Where multiple repetitions were done, outliers were identified with a commonly applied method (e.g., <https://www.mathworks.com/help/stats/box-plots.html>, accessed 30/10/2020). This was done by multiplying the interquartile range (IQR) with 1.5, and datapoints that were above the 75th quartile plus the IQR and below the 25th percentile minus the IQR were disregarded in further calculations. Where applicable, standard deviations were presented with average values to indicate data repeatability.

### 3. Results and Discussion

#### 3.1. Operational History

The results presented in this paper will only be relevant to Søderberg electrode use in SAF operations, if the case study electrode represented normal/stable operation. To explore this, total daily slipping taken on the electrode, daily average current passing through it, as well as daily average solid and liquid paste levels in the month prior to the furnace shutdown, are presented in Figure 4. From this data, it is evident that the slipping was relatively consistent (varied between 14 to 32 mm per day). No long slips (usually defined as >100 mm) were taken on this electrode, which indicates that the operational personnel considered its length as normal and that no breaks occurred during this 1 month period. The daily average current passing through the electrode varied between approximately 63 and 76 kA, and had an average of 72.2 kA in the period between 1 and 28 July 2013. This again indicates that no major operational issues occurred. In the last three days prior to the shutdown (29 to 31 July 2013) the energy input was purposefully reduced, which resulted in the average current passing through the electrode on the last operational day being 58.0 kA. Such a gradual reduction in power input contributes significantly to prevent thermal shock on the electrode [5]. Average daily solid (varying between 6.7 and 9.3 m) and liquid (varying 2.9 and 4.1 m) paste levels were normal, again with no indication that paste consumption increased or decreased significantly during the last month of operation. Considering all the afore-mentioned, in conjunction with visual inspection of the electrode and its length before being cut off (it was approximately 3 m long), it can be concluded that this case study electrode was representative of stable SAF operation.

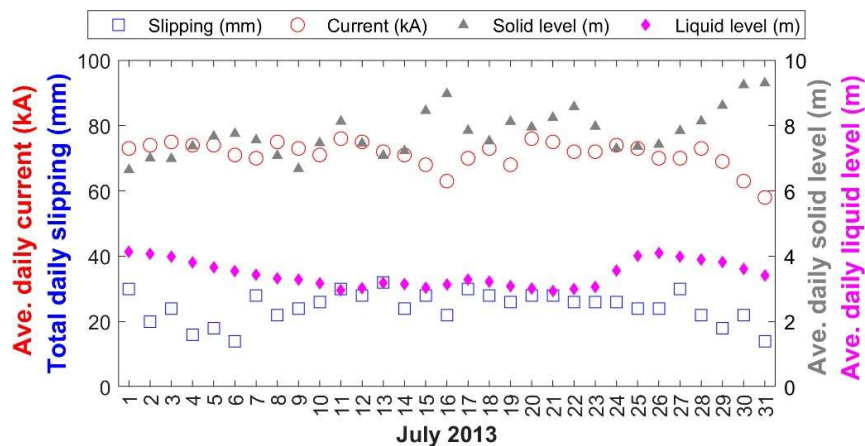


**Figure 4.** Total daily slipping taken on the case study electrode, daily average current passing through it, as well as daily average solid and liquid paste levels in the month prior to the furnace shutdown.

#### 3.2. Electrical Resistivity

The main function of Søderberg electrodes is to conduct electrical current into the smelting zone of the furnace. It is therefore fitting that electrical resistivity is the first

characteristic that is considered. Figure 5a presents the average measured resistivity and linear fitting of this data, as a function of length below the contact shoes and distance from the delta side of the electrode. It should be noted that no experimental data could be collected on, or very close to 0 and 1.4 m from the electrode delta side, since the 100 mm core drilled samples (Figure 1c) were typically 1.3 m long. This smaller electrode diameter than the original outer casing diameter of 1.4 m (introduced in Section 2.1), is due to several factors. Firstly, the mild steel casing usually burns off and/or melts within the first approximately 0.5 m below the contact shoes, which is also shown in Figure 1b for the specific electrode considered here. The casings used on the case study electrode was 3 mm thick. Therefore, 6 mm (3 mm on each side) can be attributed to the casing loss. Secondly, the electrode paste will shrink at temperatures above the baking isotherm [5]. This occurs since the carbonaceous material structure becomes more organised and closer to that of graphite, due to the increased temperature the electrode experiences as it descends. Thirdly, chemical corrosion, i.e., oxidation of the electrode carbon above the bed material, can also contribute, especially since the furnace considered here was an open-SAF. In open-SAFs, atmospheric air containing oxygen can relatively freely enter (determined by off-gas flow rate) and results in a partially oxidising environment above the bed material [20]. Lastly, physical erosion, due to frictional and mechanical forces exerted on the electrode, can also erode its outer perimeter. Due to all the afore-mentioned reasons, all result figures wherein the case study electrode diameter is specified will indicate it as 1.3, not 1.4 m.

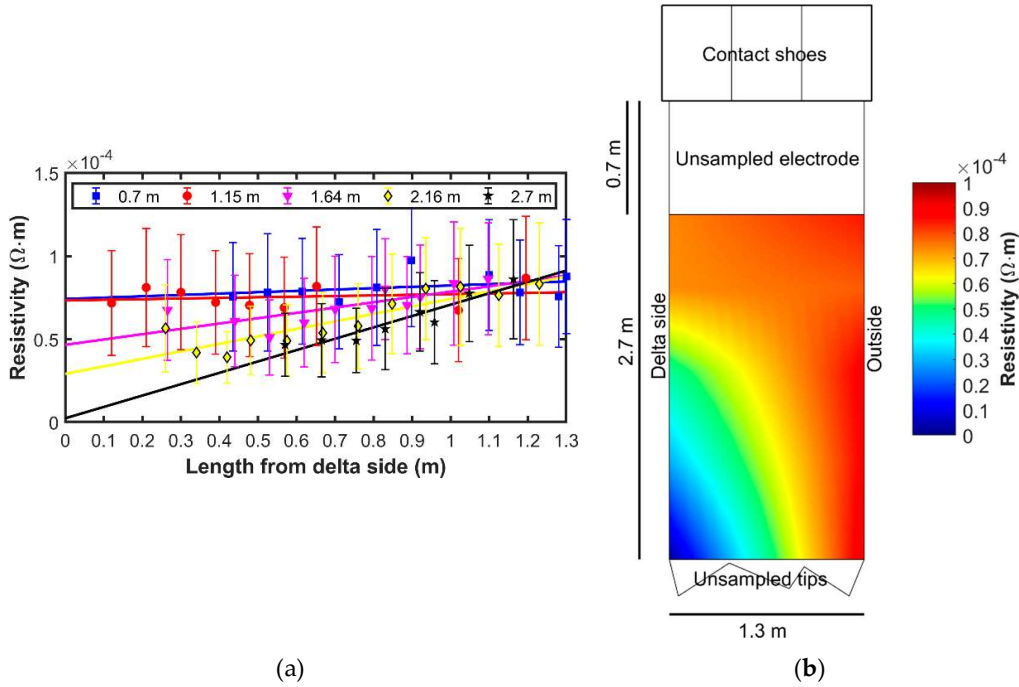


**Figure 5.** (a) Average measured electrical resistivity (markers) and linear fitting of this data (lines), as a function of length below the contact shoes and distance from the delta side of the electrode; (b) 3D rendering of the fitted line data associated with resistivities at 10 mm intervals across the electrode at each of the lengths below the contacts shoes indicated in Figure 5a.

The standard deviations of the resistivity measurements presented in Figure 5a were relatively large. This was mostly due to the actual resistivities being so low (i.e.,  $10^{-5}$  to  $10^{-6} \Omega \cdot m$ ). Therefore, to ensure that the average values could be used to identify trends, very large numbers of measurements (i.e.,  $N = 150$  to  $450$ ) were conducted. Considering the average values, Figure 5a clearly indicates that the electrical resistivity on the outside (non-delta side) of the electrode between 0.7 and 2.7 m below the contact shoes, were essentially consistent. In contrast, the resistivity decreased (implying increased conductivity)

significantly from 0.7 to 2.7 m below the contact shoes on the delta side of the electrode. To visualise the electrical resistivity results better, the average data associated with the fitted lines presented in Figure 5a at 10 mm intervals across the diameter of the electrode at each of the investigated lengths below the contact shoes were spatially interpolated across the entire investigated area (0.7 to 2.7 m below the contact shoes), as indicated in Figure 5b. This was done using the “grid data” function in Matlab, with triangulation-based linear interpolation. In effect, this representation gives a 3-dimensional (3D) cross section perspective of the investigated electrode, excluding the unsampled electrode area directly below the contact shoes and the uneven electrode tips, with colour indicating the 3rd dimension. This 3D perspective indicates that resistivity was at its lowest at the bottom end on the delta side of the electrode, where the submerged arc would most likely jump off the electrode.

To contextualise the resistivity results presented in Figure 5a,b, they were firstly compared to high temperature bulk bed electrical resistivity values reported relatively recently for carbonaceous reductants (i.e., metallurgical coke, semi-coke, coal, and charcoal used in SAF smelting) [21]. Such high temperature bulk bed resistivities simulate the so-called coke bed (area around and below electrode tips in a SAF), where reductants serve as one of the primary electrical conductors [22]. At 1600 °C, it was found that the reductant bulk bed resistivities ranged from approximately  $2 \times 10^{-2}$  to  $5 \times 10^{-3} \Omega \cdot m$  [21]. These values were approximately 60 to 250 times higher (thus less conductive) than the approximate  $8.40 \times 10^{-5} \Omega \cdot m$  resistivity observed from 0.7 to 2.7 m below the contact shoes on the non-delta side of the electrode (reddish area on the right-hand side of Figure 5b). Correspondingly, the high temperature reductant bulk bed resistivities were approximately 2000 to 8000 times higher than the  $2.4 \times 10^{-6} \Omega \cdot m$  obtained at 2.7 m below the contact shoes on the electrode delta side (dark blue area in bottom left-hand corner of Figure 5b). Obviously, interparticle contact in a carbonaceous reductant bed is far inferior to the relatively solid carbonaceous mass of a Söderberg electrode, but the afore-mentioned comparison does indicate on a relative scale the conductivity ability of a Söderberg electrode.

To further contextualize the resistivity data presented in Figure 5a,b, it was compared to Söderberg electrode paste resistivity values derived from finite element thermoelectrical modelling [9,23]. These studies postulated resistivities to be  $5.88 \times 10^{-6} \Omega \cdot m$  and  $2.63 \times 10^{-6} \Omega \cdot m$  in the electrode where the temperatures were 1000 and 2600 °C, respectively. Such temperatures correspond roughly to the 0.7 m (both delta and non-delta side) and 2.7 m (delta side only) positions below the contact shoes in Figure 5b, where resistivities were approximately  $8.48 \times 10^{-5}$  and  $2.41 \times 10^{-6} \Omega \cdot m$ , respectively. Although the modelled values [9,23] and values presented in the current work are in the same order, the differences in the corresponding values ( $5.88 \times 10^{-6} / 2.63 \times 10^{-6} = 3.25 \times 10^{-6} \Omega \cdot m$  versus  $8.48 \times 10^{-5} / 2.41 \times 10^{-6} = 8.24 \times 10^{-5} \Omega \cdot m$ ) suggest that the models do not quickly enough account for lower resistivities as a function of electrode length below the contact shoes. Compared to the afore-mentioned models [9,23], a more comprehensive Söderberg electrode model has also previously been introduced [5]. This model might contain more detailed Söderberg electrode paste properties and might better account for the variation of such properties as a function of electrode length below the contact shoes. However, this model was essentially introduced as a black box to demonstrate the capabilities of a commercial company [5]; therefore, it was impossible to include the material properties specified therein in the current comparison. Furthermore, no evidence could be found that either of the three models [5,9,23] accounted for the very apparent asymmetrical variation of resistivities observed in the case study electrode (Figure 5b).

### 3.3. Degree of Graphitisation (DOG)

Figure 6a presents the average measured DOG and linear fitting of this data, as a function of length below the contact shoes and distance from the delta side of the electrode. Figure 6b presents the associated spatially interpolated 3D image. From both these figures (but particularly from Figure 6b) that visually represent the entire electrode cross section, it is evident that DOG is inversely related to the electrode resistivity (Figure 5b). This makes

sense, since increased DOG of the electrode paste will result in lower resistivity (i.e., higher conductivity). It is particularly insightful to also note that only the electrode tip on the delta side of the electrode (red area in bottom left-hand corner of Figure 6b, which corresponded to a maximum DOG of approximately 0.84), approached full graphitisation. As previously stated (Section 2.3), DOG is unitless, with 1 indicating fully graphitised and 0 fully ungraphitised. The majority of the investigated cross-sectional electrode area had  $DOG \leq 0.5$ . As far as the authors could assess, DOG for a real world or a modelled Söderberg electrode has not yet been presented in the peer reviewed public domain; therefore, it was not possible to contextualise the measured DOG with such data.

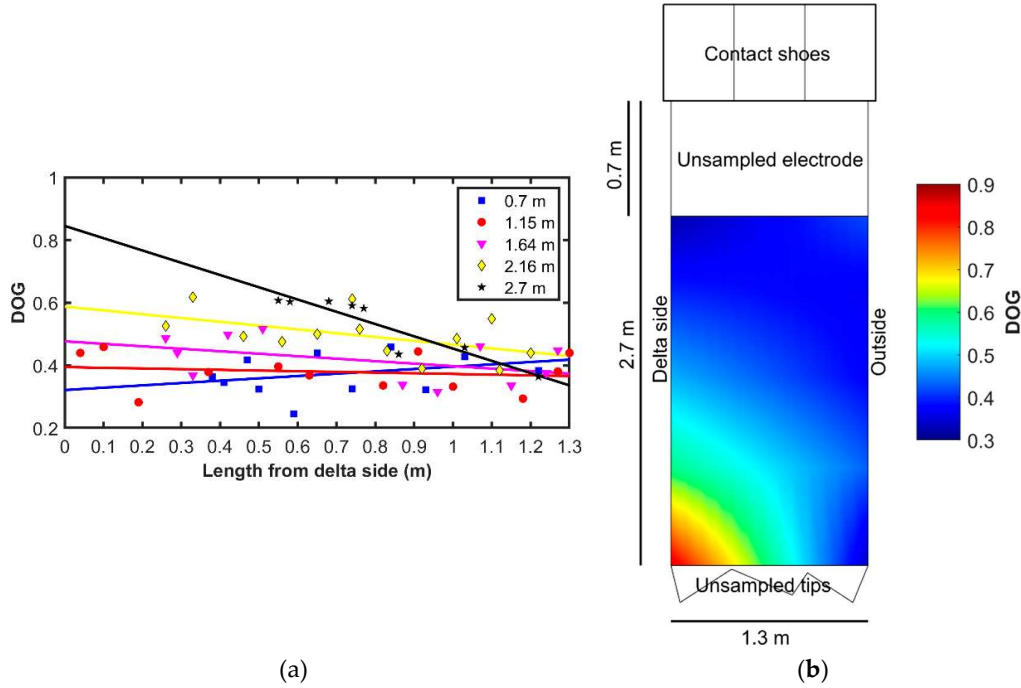


**Figure 6.** (a) Average measured degree of graphitisation (DOG) (markers) and linear fitting of this data (lines), as a function of length below the contact shoes and distance from the delta side of the electrode; (b) 3D rendering of the fitted line data associated with DOG at 10 mm intervals across the electrode at each of the lengths below the contacts shoes indicated in Figure 6a.

As indicated in the previous paragraph, to a large extent DOG determines the electrical resistivity of the Söderberg electrode. However, the DOG of the electrode paste is also related to other important characteristics. For instance, DOG is associated to possibly the most important aspect that cause electrode breaks, i.e., thermal stress. Particularly, green breaks (implying exposure of liquid paste in the break zone) in the contact shoes area are feared by operational personnel, due to the devastating consequences such breaks could have. In the most authoritative work on Söderberg electrode thermal stress, it is stated that some internal stresses are established during the process of electrode paste shrinkage below the contact shoes area, where the paste is baked at temperatures in excess of the baking isotherm temperature [5]. However, as previously stated, Söderberg electrode paste consists of a binder and a solid aggregate. In this case, the solid aggregate was calcined anthracite of various size fractions, mixed to obtain the optimum composition. Although more environmentally friendly binders have been proposed than coal tar pitch, e.g., [24], it remains the most commonly used binder for Söderberg electrode pastes and was also used in the paste consumed by the case study electrode. In previous studies, it was proven that anthracite calcined at 1400 °C expands less than 1% and coal tar pitches prebaked at the maximum baking isotherm temperature (i.e., 475 °C, [11]) shrink approximately

12% if heated in a non-oxidising environment up to 1300 °C [4]. These opposing thermal expansion characteristics likely cause the thermal stresses below the contact shoes area referred to by Larsen et al. [5]. In contrast to calcined anthracite, uncalcined anthracite shrink 6 to 8% over the same temperature range. The significant difference between calcined and uncalcined anthracite clearly indicates how important the calcination process is to avoid additional thermal stresses forming just below the contact shoes area. Industrial calcination of anthracite to be used in electrode paste typically occur at temperatures above 1400 °C (as tested by Beukes et al. [4]); therefore, DOG similar to the blue area in Figure 6b (corresponding to DOG 0.5) is imparted on the electrode paste solid aggregate even before it is mixed into the electrode paste. The authors were not privileged to know the exact mixing ratio of the binder and aggregate used in the case study electrode. But, typically the mixture is dominated by aggregate, with the binder fractional contribution limited to just enable solid aggregate interparticle contact and proper paste flow characteristics above the softening point (to fill the casing in the area above the baking isotherm temperature).

### 3.4. Bulk Density

Figure 7a presents the average measured bulk density determined with ASTM C20-00 [19] (Section 2.4, 1st paragraph) as a function of length below the contact shoes and distance from the delta side of the electrode. Since ASTM C20-00 involves aqueous saturation of the samples via boiling, repeat analyses were not attempted. The density differences were relatively small (Figure 7a), therefore, even slight modification of the original characteristics that might occur during analysis or re-drying of the samples in preparation for reanalyses could make such reanalyses results misleading. Additionally, larger cylindrical sample pellets were used in this specific method (Section 2.4, 1st paragraph), which implied that significant additional sample material would have been required for reanalyses. This was not feasible, since a finite amount of material was available after sampling (Section 2.1). As is evident from the ASTM C20-00 results (Figure 7a), bulk densities did not indicate significant differences as a function of length below the contact shoes. Therefore, a single linear fitted line was drawn through all the data, which indicated a slight increase in densities toward the delta side of the electrode.

To verify that densities did not differ as a function of length below the contact shoes and to determine standard deviations, bulk densities were also determined with gas pycnometry (Section 2.4, 2nd paragraph). The gas pycnometry densities (Figure 7b), in contrast to the ASTM C20-00 results (Figure 7a), did indicate that bulk densities differed as a function of length below the contact shoes. This indicates that gas pycnometry is possibly a more precise method than ASTM C20-00, or alternatively that the difference in atom/molecule size (i.e., He used in gas pycnometry vs. H<sub>2</sub>O used ASTM C20-00) play a significant role in the measured results. To visualise these gas pycnometry results, Figure 7c presents the associated spatially interpolated 3D image, based on the fitted data presented in Figure 7b. Considering the very similar perspectives indicated by the bulk densities presented here (Figure 7c) and the DOG presented earlier (Figure 6b), it is inferred that the increased bulk densities observed, particularly on the delta side of the electrode as a function of length below the contact shoes, were due to increased DOG. Increased DOG will result in further shrinkage of the carbonaceous Söderberg electrode paste material.

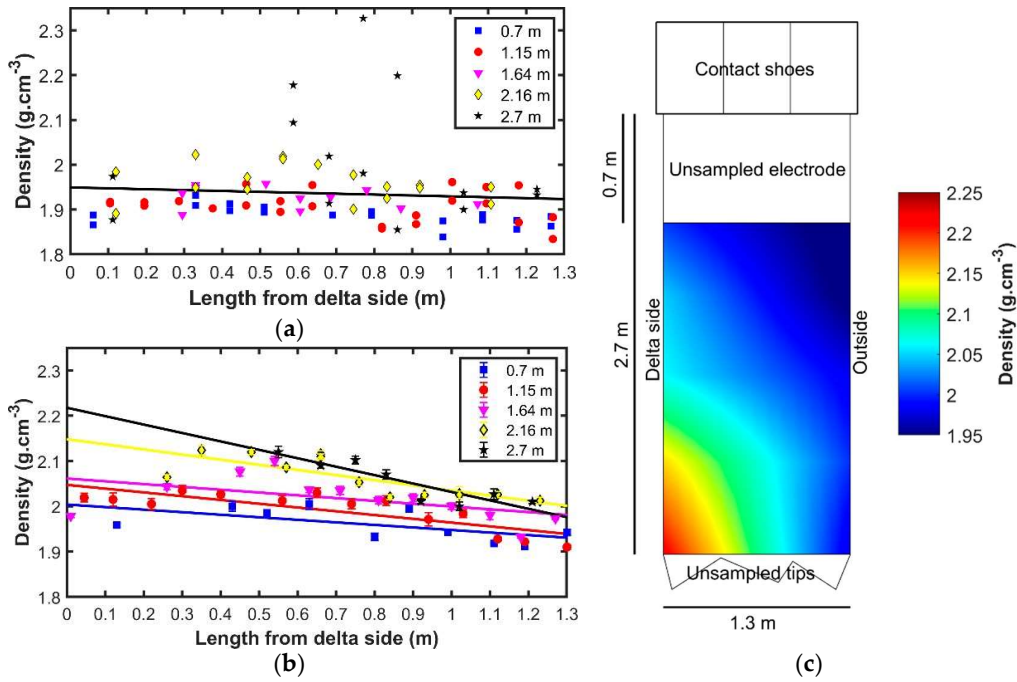


**Figure 7.** (a) ASTM C20-00 average measured bulk densities (markers) as a function of length below the contact shoes and distance from the delta side of the electrode. A single linear fitting of all this data (line) is also indicated; (b) gas pycnometry average measured bulk densities (markers) and linear fitting of this data (lines), as a function of length below the contact shoes and distance from the delta side of the electrode; (c) 3D rendering of the fitted line data associated with bulk densities at 10 mm intervals across the electrode at each of the lengths below the contacts shoes indicated in Figure 7b.

### 3.5. Porosity

Figure 8a presents the average measured porosity. As is evident, no significant difference in porosity as a function of length below the contact shoes were observed. However, a slight increase in porosity was observed, near the electrode perimeter on both the delta and non-delta side. Therefore, a 2nd order polynomial (solid line in Figure 8a) fitted the data best. The corresponding spatially interpolated 3D image is presented in Figure 8b.

Since the industrial application of the Söderberg electrode started, it has been proven that volatiles released from the liquid paste just above the baking isotherm (indicated by black dotted line in Figure 9a) cannot rise if proper (high enough) liquid and solid paste levels are maintained e.g., [25]. Conversely, if “smoke” is observed at the top of the electrode column casing, it serves as a serious warning signal to operational personal that the liquid paste level of that electrode is critically low and/or that the liquid paste is too hot. Since the volatiles just above the baking isotherm cannot rise, they escape downward through the pores of the already baked electrode [25]. Cracking of these descending volatiles (due to the high temperature of the baked electrode) leaves a considerable amount of carbon in the pores of the baked electrode paste just below the contact shoes area, which improves its quality [25]. This process is illustrated by the purple arrows in the enlarged area of Figure 9a. The blue temperature isotherm in the enlarged area of Figure 9b represents a slightly hotter isotherm temperature than the baking isotherm. Considering this diagram, one would assume that the route of least resistance for volatiles to escape would be towards the outside of the electrode (i.e., shortest route of baked electrode paste), not the electrode core. Theoretically, this process would therefore slightly reduce the porosity of the electrode towards its perimeter, which is contrary to the experimental results presented (Figure 8a,b).

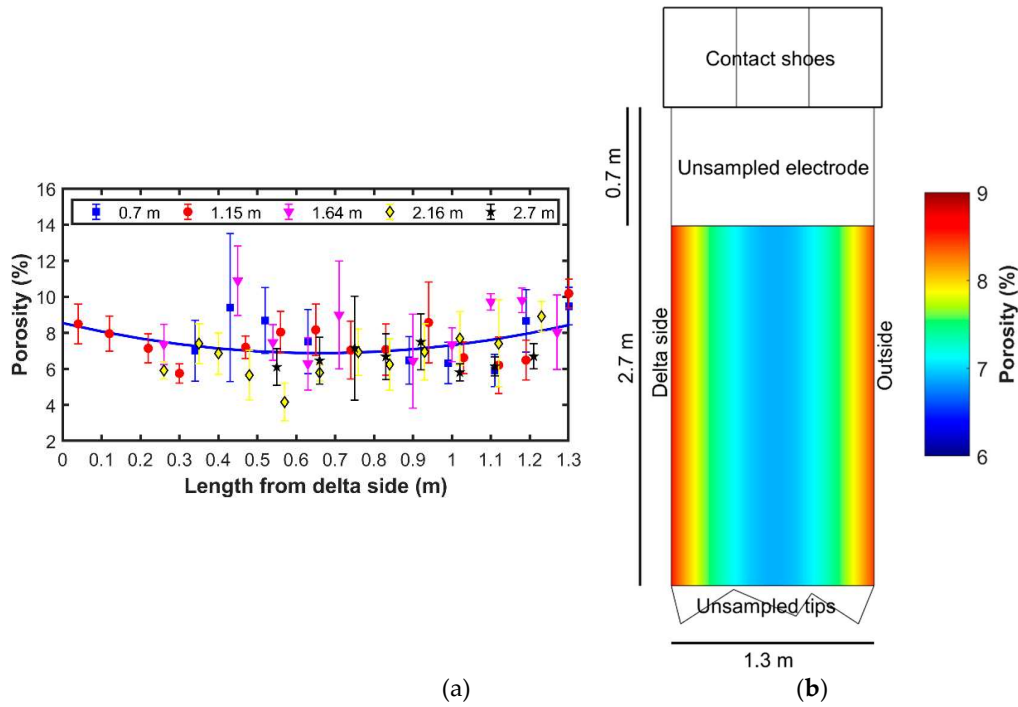


**Figure 8.** (a) Average measured porosity (markers), as a function of length below the contact shoes and distance from the delta side of the electrode, as well as a single 2nd order polynomial fit of this data (line); (b) 3D rendering of the fitted line data associated with porosities at 10 mm intervals across the electrode at each of the lengths below the contacts shoes indicated in Figure 8a.

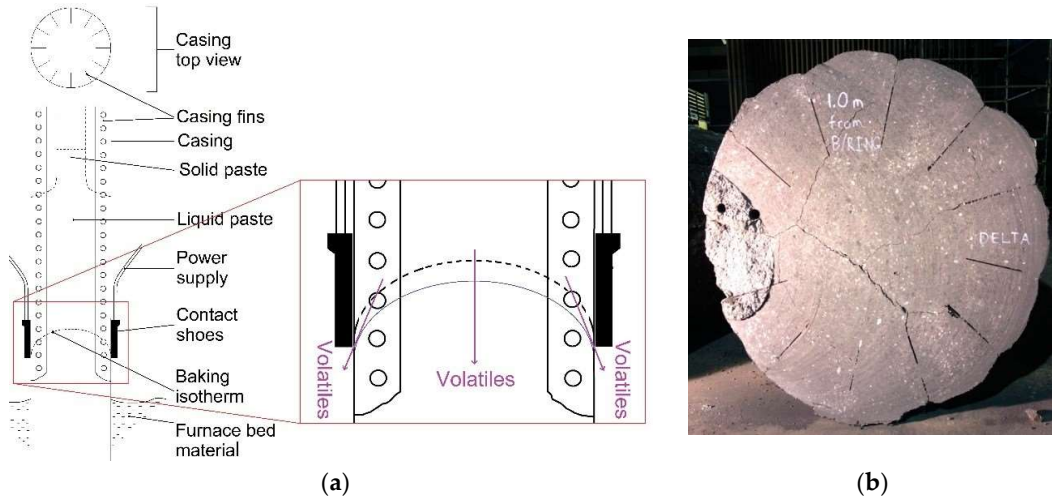


**Figure 9.** (a) Representation of a partial Söderberg electrode column, adapted from Beukes et al. [4], with the contact shoes area enlarged (indicated by the square red block); (b) cross section of another electrode from the same SAF that was cut off with a diamond wire saw approximately 1.0 m below the contact shoes.

There could be two other factors that counter the possible decrease in porosity towards the electrode perimeter, caused by descending volatile cracking. Firstly, liquid electrode paste segregation (i.e., partial separation of the pitch binder and solid aggregate), as illustrated by Nelson and Prins [8], could result in significant differences in the baked electrode properties considered diagonally across an electrode. However, calcined anthracite has

a much higher DOG than coal tar pitch baked above the maximum baking isotherm temperature (i.e., 475 °C, [11]). Therefore, if paste segregation caused the differences in observed porosities (Figure 8b), corresponding differences in DOG diagonally across the case study electrode would likely also have been evident at the first level below the contact shoes investigated (i.e., 0.7 m). This was not the case (Figure 6b); therefore, paste segregation is unlikely to be the cause. Also, visual inspection of the sampled electrode did not indicate any segregation. The second possible contributing factor that could increase porosity of the case study electrode is chemical corrosion, i.e., oxidation of the electrode carbon above the bed material. As previously stated, this can especially occur in an open-SAF, as considered here [20]. Figure 9b shows the diagonal cross section of another electrode from the same SAF that was cut off approximately 1.0 m below the contact shoes. As is evident from this electrode, corrosion on the outer perimeter was more significant in the areas where the casing fins occurred. This indicates that the gaps remaining after the casing burned off and/or melted away, formed paths for oxygen penetration, and associated oxidation of the carbon material. This describes the process on a macroscale, but it is postulated that the same process occurs on a pore size scale, which will increase pore volumes and hence porosity preferentially on the electrode perimeter.

### 3.6. Compressive Breaking Strength

Figure 10 presents the average measured compressive breaking strength as a function of length below the contact shoes and distance from the electrode delta side. As is evident, no significant difference was observed over the entire electrode area investigated. This indicates that compressive breaking strength was not related to DOG, as was the case for electrical resistivity and bulk density, nor was it influenced by physical and/or chemical processes, as postulated for the porosities.

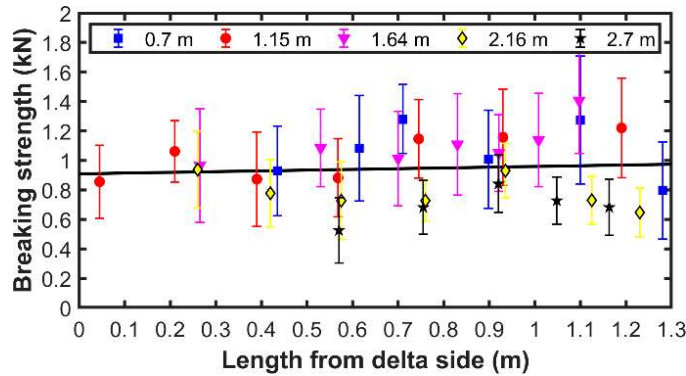


**Figure 10.** Average measured compressive breaking strength (markers), as a function of length below the contact shoes and distance from the delta side of the electrode, as well as a single linear fit of this data (line).

## 4. Conclusions

As far as the authors could assess, this work presents the first characterisation of a real-world Söderberg electrode, presented in a peer reviewed journal in the public domain. Obviously, a study such as this has limitations. For instance, measurements of the characteristics were conducted at room temperature, instead of at the operational temperatures. Also, the study was conducted on an electrode removed for an open-SAF producing ferrochrome, for which some characteristics might differ from Söderberg electrodes operating in other furnace types and smelting different materials. However, all these limitations are similar to furnace dig-out studies, e.g., [26–28], which have to a large degree formed our current understanding of zones within furnaces. Another data limitation of the current work was that the case study electrode did not include the area directly above, in, and just below the contact shoes. Understanding this area is critical.

Unfortunately, this section of the electrode could not be obtained, since that would have entailed starting up the furnace with a green electrode tip.

The results presented in this paper provided valuable insights, but also indicated questions/topics that need to be considered in future research. The operational data for the month prior to the furnace rebuild, during which the case study electrode was removed, proved that the electrode was a representative specimen. The characterisation results indicated no significant electrical resistivity, DOG, and bulk density changes from 0.7 to 2.7 m on the non-delta side of the electrode, while these characteristics changed relatively significantly on the delta side of the electrode. The area where the submerged arc would mostly like jump off the electrode clearly had the lowest resistivity, as well as highest DOG and bulk density. It is currently not evident from literature if these very asymmetrical characteristics can be accurately predicted by Söderberg electrode models, e.g., [5,9,23]. However, all such models predict temperature isotherms/curves. The temperature to which Söderberg electrode paste is exposed determines the DOG, to a large degree. As shown here, the DOG in turn determine to a large extent, the resistivities and bulk densities. Therefore, it would be advantageous if Söderberg electrode models could accurately replicate the DOG presented here. To aid such comparison, the resistivities, DOG, and bulk densities associated with the fitted lines at 10 mm intervals across the diameter of the electrode at each of the investigated lengths below the contacts shoes prior to being spatially interpolated to generate the 3D images are presented in Appendix A. For future experimental studies, it is recommended that the DOG of calcined anthracite and coal tar pitch be investigated separately (not as a mixture in electrode paste), in relation to electrical resistivity and bulk density, as well as other characteristics such as thermal expansion. This will enable better understanding of the individual contribution of these two components, to the combined results observed for electrode paste.

The determination of porosity with CT scanning combined with image processing is likely more accurate than conventional methods. However, the manual marking of the areas to be included in the calculations for porosity made the method time consuming and less attractive. Research has already been initiated to automate the determination of the particle rims within CT image slices and calculation of porosities and pore volumes. From the presented porosity data, no significant difference in porosities, as a function of length below the contact shoes, were evident. However, slight increases in porosities were observed near the electrode perimeter on both the delta and non-delta sides, which was fitted best with a 2nd order polynomial function. It was postulated that chemical corrosion, i.e., oxidation of the carbonaceous material, increases the pore volumes in the baked electrode paste near the perimeter.

The compressive breaking strength data revealed no significant trends. Therefore, operational personnel should not be concerned about breaking strength in the region well below the baking isotherm being during stable operation. As previously stated [5], thermal stresses that form in and just below the baking isotherm, as well as thermal stress differences between the perimeter and core of the electrode formed due to prolonged furnace downtime, remain the most likely reasons for breaks of electrodes with no other apparent dysfunctionalities.

Considering the value of studies such as this, hopefully the current work will also encourage the donation of similar case study electrodes to researchers. As an example, the authors belatedly became aware of 2 separate 63 MVA closed-SAFs, for which the electrode outer casing diameters were increased from 1.5 to 1.6 m. In order to do so, 6 entire electrode columns, including the casings with un-melted solid paste at the top, down to the baked electrode tips at the bottom, were removed and discarded. However, no one in the research community was notified, nor did the company/operational personnel reach out to researchers. Such large scale, real-world samples are scarce; therefore, opportunities such as this should not be missed.

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draft preparation, J.P.B.; writing—review and editing, all co-authors; study supervision, J.P.B. and P.G.v.Z.; funding acquisition, J.P.B., P.G.v.Z., M.T., and E.R. All authors have read and agreed to the published version of the manuscript.

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## Appendix A

**Table A1.** The electrical resistivity, DOG, and bulk density data associated with the fitted lines in Figure 5a, Figure 6a, and Figure 7b, at 10 mm intervals across the diameter of the electrode at each of the investigated lengths below the contacts shoes, respectively. This data was spatially interpolated to generate the 3D images presented in Figure 5b, Figure 6b, and Figure 7c, respectively.

| Distance from<br>Delta Side (m)  | Distance below the Contact Shoes (m) |                       |                       |                       |                       |
|----------------------------------|--------------------------------------|-----------------------|-----------------------|-----------------------|-----------------------|
|                                  | 0.70                                 | 1.15                  | 1.64                  | 2.16                  | 2.70                  |
| Resistivity ( $\Omega \cdot m$ ) |                                      |                       |                       |                       |                       |
| 0                                | $7.42 \times 10^{-5}$                | $7.34 \times 10^{-5}$ | $4.66 \times 10^{-5}$ | $2.92 \times 10^{-5}$ | $2.41 \times 10^{-6}$ |
| 0.1                              | $7.51 \times 10^{-5}$                | $7.37 \times 10^{-5}$ | $4.98 \times 10^{-5}$ | $3.37 \times 10^{-5}$ | $9.25 \times 10^{-6}$ |
| 0.2                              | $7.59 \times 10^{-5}$                | $7.41 \times 10^{-5}$ | $5.30 \times 10^{-5}$ | $3.82 \times 10^{-5}$ | $1.61 \times 10^{-5}$ |
| 0.3                              | $7.67 \times 10^{-5}$                | $7.45 \times 10^{-5}$ | $5.62 \times 10^{-5}$ | $4.27 \times 10^{-5}$ | $2.29 \times 10^{-5}$ |
| 0.4                              | $7.75 \times 10^{-5}$                | $7.49 \times 10^{-5}$ | $5.94 \times 10^{-5}$ | $4.72 \times 10^{-5}$ | $2.98 \times 10^{-5}$ |
| 0.5                              | $7.83 \times 10^{-5}$                | $7.52 \times 10^{-5}$ | $6.26 \times 10^{-5}$ | $5.17 \times 10^{-5}$ | $3.66 \times 10^{-5}$ |
| 0.6                              | $7.91 \times 10^{-5}$                | $7.56 \times 10^{-5}$ | $6.58 \times 10^{-5}$ | $5.62 \times 10^{-5}$ | $4.35 \times 10^{-5}$ |
| 0.7                              | $7.99 \times 10^{-5}$                | $7.60 \times 10^{-5}$ | $6.91 \times 10^{-5}$ | $6.07 \times 10^{-5}$ | $5.03 \times 10^{-5}$ |
| 0.8                              | $8.07 \times 10^{-5}$                | $7.64 \times 10^{-5}$ | $7.23 \times 10^{-5}$ | $6.52 \times 10^{-5}$ | $5.71 \times 10^{-5}$ |
| 0.9                              | $8.15 \times 10^{-5}$                | $7.68 \times 10^{-5}$ | $7.55 \times 10^{-5}$ | $6.97 \times 10^{-5}$ | $6.40 \times 10^{-5}$ |
| 1.0                              | $8.24 \times 10^{-5}$                | $7.71 \times 10^{-5}$ | $7.87 \times 10^{-5}$ | $7.42 \times 10^{-5}$ | $7.08 \times 10^{-5}$ |
| 1.1                              | $8.32 \times 10^{-5}$                | $7.75 \times 10^{-5}$ | $8.19 \times 10^{-5}$ | $7.88 \times 10^{-5}$ | $7.77 \times 10^{-5}$ |
| 1.2                              | $8.40 \times 10^{-5}$                | $7.79 \times 10^{-5}$ | $8.51 \times 10^{-5}$ | $8.33 \times 10^{-5}$ | $8.45 \times 10^{-5}$ |
| 1.3                              | $8.48 \times 10^{-5}$                | $7.83 \times 10^{-5}$ | $8.83 \times 10^{-5}$ | $8.78 \times 10^{-5}$ | $9.13 \times 10^{-5}$ |
| DOG                              |                                      |                       |                       |                       |                       |
| 0                                | 0.32                                 | 0.39                  | 0.48                  | 0.59                  | 0.86                  |
| 0.1                              | 0.33                                 | 0.39                  | 0.47                  | 0.58                  | 0.81                  |
| 0.2                              | 0.34                                 | 0.39                  | 0.46                  | 0.56                  | 0.77                  |
| 0.3                              | 0.34                                 | 0.39                  | 0.45                  | 0.55                  | 0.73                  |
| 0.4                              | 0.35                                 | 0.39                  | 0.45                  | 0.54                  | 0.69                  |
| 0.5                              | 0.36                                 | 0.38                  | 0.44                  | 0.53                  | 0.65                  |
| 0.6                              | 0.37                                 | 0.38                  | 0.43                  | 0.52                  | 0.61                  |
| 0.7                              | 0.37                                 | 0.38                  | 0.42                  | 0.50                  | 0.57                  |
| 0.8                              | 0.38                                 | 0.38                  | 0.41                  | 0.49                  | 0.53                  |
| 0.9                              | 0.39                                 | 0.38                  | 0.41                  | 0.48                  | 0.49                  |
| 1.0                              | 0.40                                 | 0.37                  | 0.40                  | 0.47                  | 0.45                  |
| 1.1                              | 0.40                                 | 0.37                  | 0.39                  | 0.46                  | 0.41                  |
| 1.2                              | 0.41                                 | 0.37                  | 0.38                  | 0.44                  | 0.38                  |
| 1.3                              | 0.42                                 | 0.37                  | 0.37                  | 0.43                  | 0.34                  |

Table A1. Cont.

| Distance from<br>Delta Side (m)    | Distance below the Contact Shoes (m) |      |      |      |      |
|------------------------------------|--------------------------------------|------|------|------|------|
|                                    | 0.70                                 | 1.15 | 1.64 | 2.16 | 2.70 |
| Bulk density (g·cm <sup>-3</sup> ) |                                      |      |      |      |      |
| 0                                  | 2.00                                 | 2.05 | 2.06 | 2.15 | 2.22 |
| 0.1                                | 1.99                                 | 2.04 | 2.06 | 2.14 | 2.20 |
| 0.2                                | 1.99                                 | 2.03 | 2.05 | 2.13 | 2.18 |
| 0.3                                | 1.99                                 | 2.02 | 2.04 | 2.11 | 2.16 |
| 0.4                                | 1.98                                 | 2.01 | 2.04 | 2.10 | 2.14 |
| 0.5                                | 1.98                                 | 2.01 | 2.03 | 2.10 | 2.12 |
| 0.6                                | 1.97                                 | 2.00 | 2.02 | 2.08 | 2.11 |
| 0.7                                | 1.96                                 | 1.99 | 2.01 | 2.07 | 2.09 |
| 0.8                                | 1.96                                 | 1.98 | 2.01 | 2.06 | 2.07 |
| 0.9                                | 1.95                                 | 1.97 | 2.00 | 2.05 | 2.05 |
| 1.0                                | 1.95                                 | 1.96 | 2.00 | 2.04 | 2.03 |
| 1.1                                | 1.94                                 | 1.96 | 1.99 | 2.02 | 2.01 |
| 1.2                                | 1.94                                 | 1.95 | 1.99 | 2.01 | 2.00 |
| 1.3                                | 1.93                                 | 1.94 | 1.98 | 2.00 | 1.98 |

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