R&D Carbon

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EVALUATION OF SÖDERBERG PASTE FOR ARC FURNACES

SAMPLE PREPARATION AND TEST METHODS

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1 Introduction

The quality of Söderberg paste for Si and Si/Fe arc furnaces is determined by measuring different properties on the green paste and then by preparing baked electrode specimen which are eventually tested.

For the green paste the following properties are determined:

- Water content
- Green apparent density
- Flowability

After baking and core drilling the following properties on the baked specimen:

- Baked apparent density
- Specific electrical resistance
- Dynamic elasticity modulus
- Thermal conductivity
- Flexural strength
- Fracture Energy
- Compressive strength with static elasticity modulus
- Coefficient of thermal expansion
- Air Permeability
- Real Density
- Impurities
- Ash content

The interpretation of these different test results allows a good prediction of the behavior of the paste in the arc furnaces.

In this document the testing conditions and methodology are first described followed by the preparation of the baked electrode and cores for property testing .The test schedule and the corresponding equipment and test arrangement are reviewed. Typical values for wet and dry paste are eventually listed and the relevance of each property for the anode behavior in the pots are commented.

Green Paste Testing

1.1 Sample Preparation

Approximately 500 grams of Söderberg paste are placed in two porcelain dishes and heated in the RDC 185 air forced cabinet (Figure 1) to 170°C. The temperature of the paste is measured with a thermometer.

Figure 1: RDC185 air forced cabinet for paste preparation and testing isometric cylindrical samples of 50 mm

Two molds are preheated in the same cabinet. To prevent sticking, the molds are slightly treated with oil. When the paste temperature reaches 170°C (~ 1h to 1h30 heating) the hot paste dish is weighed to determine the water content. The hot paste is then added in three or four steps to each mold (see Figure 2).Tamping with the stopper using the nylon hammer after each paste addition is performed to guarantee a uniform densification. Excess paste on the mold top is removed with a steel spatula.

After the molds are cooled with water, the test specimens are removed, dried with a cloth and then weighed. The difference between the weights of the two test specimens should not exceed 2 grams.

The operations are repeated twice to obtain a total of 4 green paste samples.

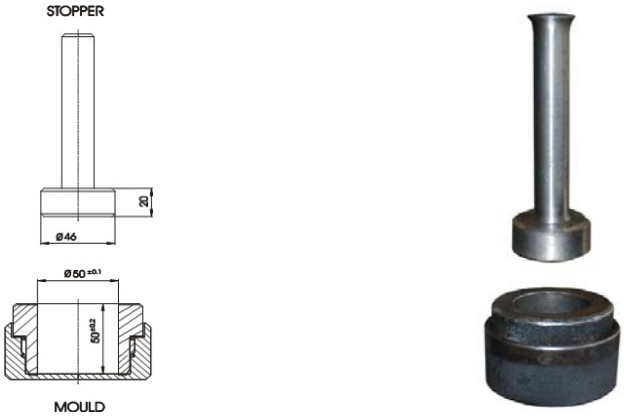

Figure 2: RDC 190 mould and stopper for the green paste cylinder preparation

1.2 Water Content (WC)

The water content is determined prior the preparation of the test specimen by measuring the weight loss (0.01g scale precision) between the ambient temperature and the heated (170°C) Söderberg paste sample.

The calculation is made according the following equation:

$$WC = \frac{m_b - m_a}{m_b} \cdot 100$$

where

WC: Water content [%]

m_b: Mass of the cold sample [g]

m_a: Mass of the heated sample[g]

The mean value of the two determinations is rounded to the first decimal.

1.3 Green Apparent Density (GAD)

The green apparent density is determined on each cylinder from their weight (0.01 g precision balance) at the end of the sample preparation. Their volume is constant as isometric sample of 50 mm are automatically prepared.

The calculation is made on each cylinder according to the following equation:

$$GAD = \frac{m_{green}}{98.17}$$

where

GAD: Green Apparent Density [kg/dm^3]

m_{green}: Mass of the sample [g]

The mean value of the four determinations is rounded to the second decimal.

1.4 Flowability (FL)

For dry ferro-alloys pastes (low flowability), the method with load is applied in order to increase the sensitivity of the analysis.

1.4.1 Flowability with load (FL)

The forced air oven is preheated to 170±3°C. The PTFE coated plate is placed on the middle shelf of the oven. Two cold test specimens are placed on the hot plate and the steel cylinders mounted on the furnace (2 kg each) are placed on the top of the samples. After 30 minutes, the plate is taken out of the oven and immediately cooled with water. The diameter of the deformed sample is measured four times (4 times with a 45 degree angle progression from an arbitrary diameter position) and the average calculated.

The flowability is calculated from the ratio of diameters (after and before the test) according the following equation:

$$F_L = \frac{d_{after}}{50}$$

where

F_L: Flowability with load [-]

d_{after}: Mean diameter after test [mm]

The mean value of the two determinations is rounded with two decimals.

2 Preparation of Test Electrodes

Two Söderberg paste samples (10 kg each) are weighed in two 20 l metallic buckets that will be placed in a forced air oven heated to about 180°C with a cover to minimize the risk of pitch fumes loss.

After 6 hours, the hot paste is filled in two 5 liters buckets (28.5 /30 cm diameter and 34 cm height) used as molds. To prevent sticking, they are lined with a heavy weight brown wrapping paper, so that there is a 100 mm overlap, and the paper extends 150 mm beyond the top of the bucket. The buckets are filled in 5 portions. The paste is tamped manually after each addition with a piston of Ø80 mm. The buckets are filled until the tamped paste is 20 mm underneath the top of the basket. When the paste has cooled down, the excess paper on the top of the bucket is folded down over the paste.The bucket containing the paste is placed in the furnace RDC 165 Söderberg baking furnace BF12S and positioned under the pressure rod. A pressure of 50 kPa (4.2 bar) is applied to the top of the electrode sample by means of a pneumatic pressure device and lifted automatically when the temperature reaches 600°C. The automatically controlled heating rates for the baking procedures are:

- 100°C/h from 20°C to 200°C
- 15°C/h from 200°C to 600°C
- 50°C/h from 600°C to 1000°C

The heat soaking time by 1000°C is 5 hours.

Figure 3: RDC 165 Söderberg baking furnace BF12S

Under these conditions, the baked test electrode samples have physical and chemical properties close to those of industrial anodes.

3 Preparation of the testing cores

After cooling, five test cylinders of 50 mm diameter are drilled from each baked electrode using the RDC 179 core drilling machine The top 20 mm of the cores is discarded by using the RDC 148 diamond wheel saw machine and eventually a 130 mm long cylinder is cut, as shown on the testing schedule shown on the next page. A grinding machine (RDC 149) is also used to prepare 20 mm plan parallel discs for the measurement of the thermal conductivity and air permeability. The samples are dried over a period of 12 hours at 180°C.

Figure 4: RDC 179 Pilot core drilling machine

Figure 5: RDC 148 Diamond wheel saw

Figure 6: RDC 149 Grinding machine

4 Baked Cores Testing

The properties of the baked cores are determined according to the test schedule below. Most of testing methods are ISO standards. A description of each determination is given in the following pages together with the corresponding RDC equipment.

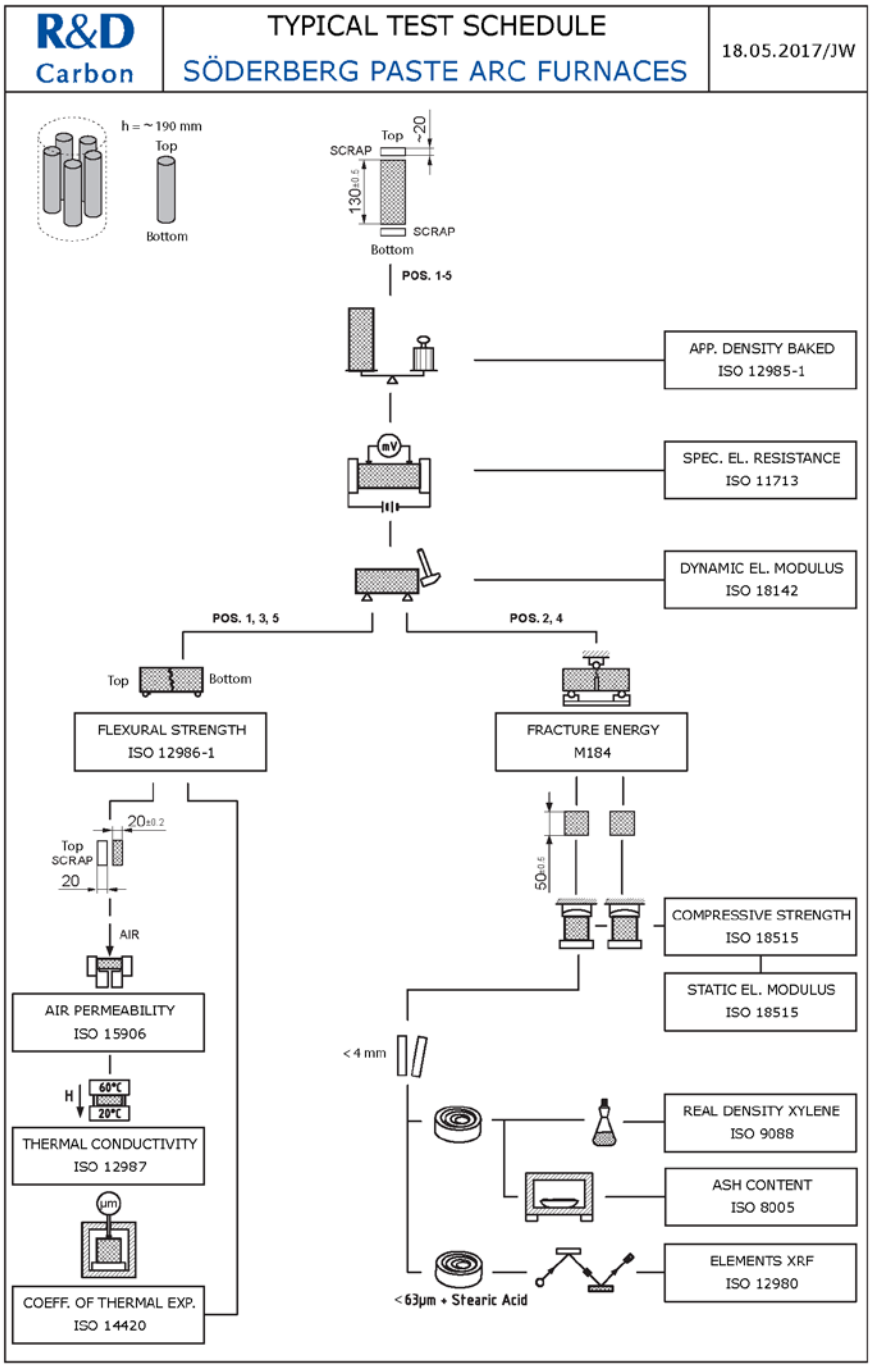

Figure 7: Test Schedule

4.1 Baked Apparent Density (BAD): ISO 12985-1

The BAD is determined by weighing and measuring the dimensions of a dried 130 mm long test specimen. The results are given in kg/dm^3. The apparent density of baked electrodes is an important quality figure to characterize the electrode performance. Most of the physical properties are strongly influenced by the apparent density of the material.

4.2 Specific Electrical Resistance (SER): ISO 11713

The specific electrical resistance is determined by measuring the voltage drop on a 50 mm diameter by 130 mm long sample at a constant current of 1.00 Ampere. The test arrangement is shown below. The results are expressed in [µΩm].

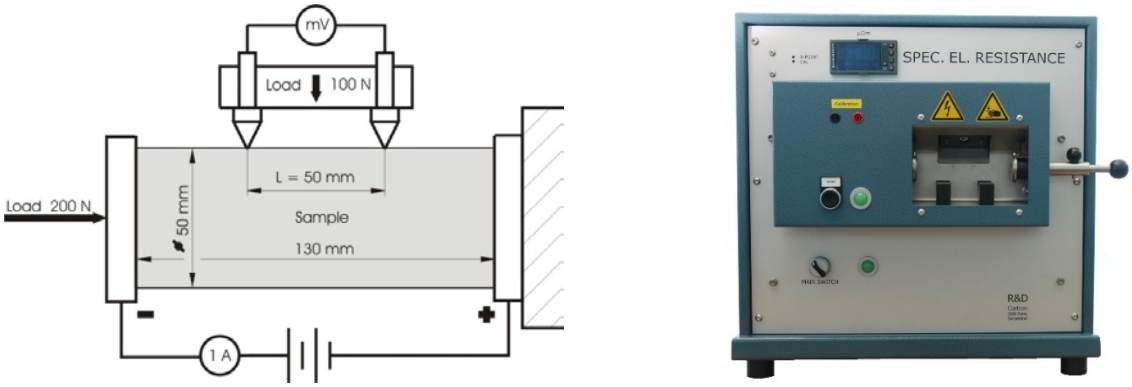

Figure 8: Test arrangement for the determination of the SER (RDC 150)

Generally, a low resistivity is desired, with the restriction that the other properties will thereby not be influenced negatively. The specific electrical resistance is strongly influenced by the density of the electrode. A high density results in a low specific electrical resistance. The calcinations degree of the raw materials, especially anthracite, as well as the addition of graphite scraps also influence the SER. The measurement of the specific electrical resistance is also used to detect cracks in the material.

4.3 Dynamic Elasticity Modulus (DEM): DIN 51915

It is determined by causing the specimen to vibrate by impulse exciting and measuring the frequency of the oscillation as shown below (taken from Grindosonic, Lemmens-Elektronika).

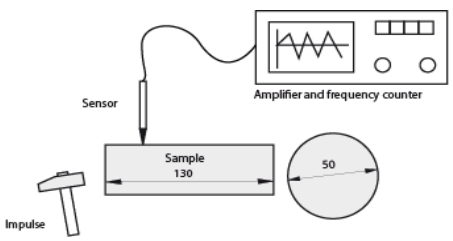

Figure 9: Test arrangement for the determination of the DEM (EXT 110)

The Elasticity dynamic is than calculated from the frequency, taking into account the sample length and apparent density, as follows:

$$DEM = 4 \cdot ADB \cdot l^2 \cdot f^2 \cdot 10^{-12}$$

with

ADB: Apparent Density Baked in kg/dm^3

l: Length of the Ø 50mm core in mm

f: Resonant frequency in Hz

The elasticity modulus is expressed in GPa. A high dynamic elasticity modulus might be deleterious for the thermal shock resistance.

Usually the dynamic elasticity modulus is about twice higher than the static elasticity modulus.

4.4 Comp. Strength and Stat. Elasticity Modulus (CS+SEM): ISO 18515

Compressive strength and static elasticity modulus are determined from the breaking load and from the deformation (strain) under load of a sample with a length of 50 mm. The values are reported in MPa for the compressive strength and in GPa for the static elasticity modulus.

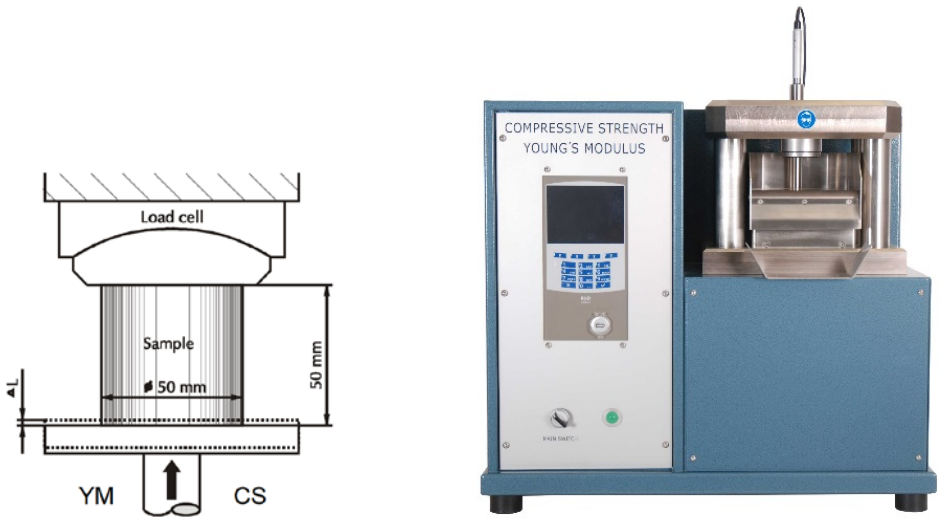

Figure 10: Test arrangement for the determination of the compressive strength and of the static elasticity modulus (RDC 144)

4.5 Flexural Strength (FS): ISO 12986-1

The flexural strength is determined using three-point loading on a sample with a diameter of 50 mm and a length of 130 mm. The test arrangement is shown below.

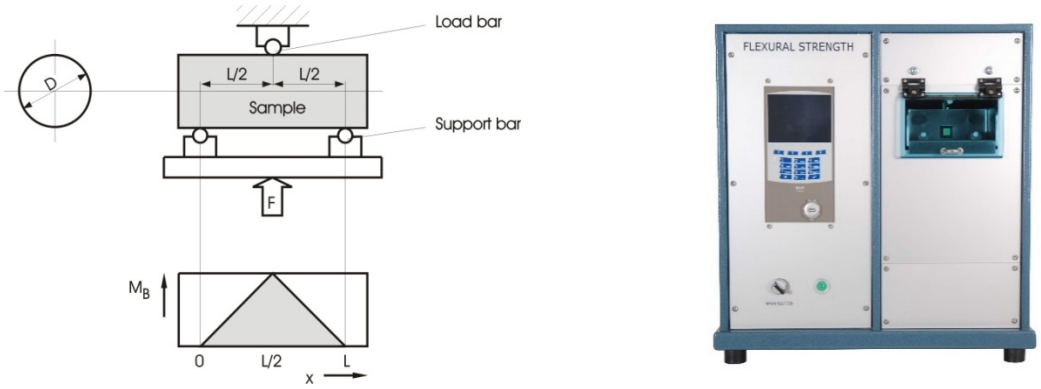

Figure 11: Test arrangement for the determination of the flexural strength (RDC 187)

The values are reported in MPa (10^6 N/m^2). The flexural strength is mainly influenced by the raw materials quality (QI of the binder, grain stability of the coke) as well as by the intensity of the paste mixing. Presence of cracks massively decreases the level of the flexural strength.

4.6 Fracture Energy (FE): ISO 11706

The fracture energy is determined by applying a force in the centre of a notched sample with a diameter of 50 mm and a length of 130 mm until it fractures. The test arrangement is shown below.

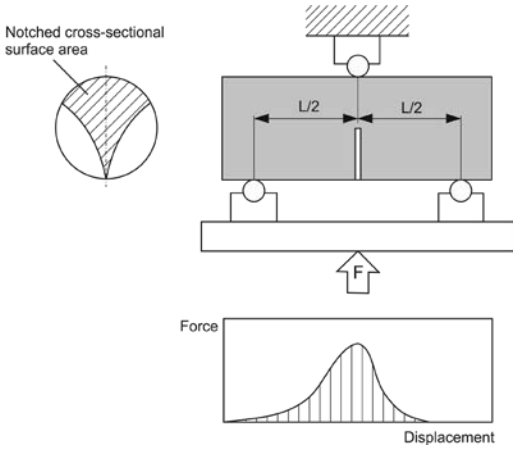

Figure 12: Test arrangement for the determination of the fracture energy (RDC 184)

The values are reported in J/m^2. A high fracture energy is desirable, since the respective material exhibits a ductile behavior with a high resistance to crack propagation. This is an important mechanical property in order to withstand the thermal shock.

4.7 Air Permeability (AP): ISO 15906

The air permeability is determined by measuring the time taken for a certain volume of air to pass through a sample disc of 50mm diameter and a height of 20 mm. The results are expressed in nanoperms (nPm).

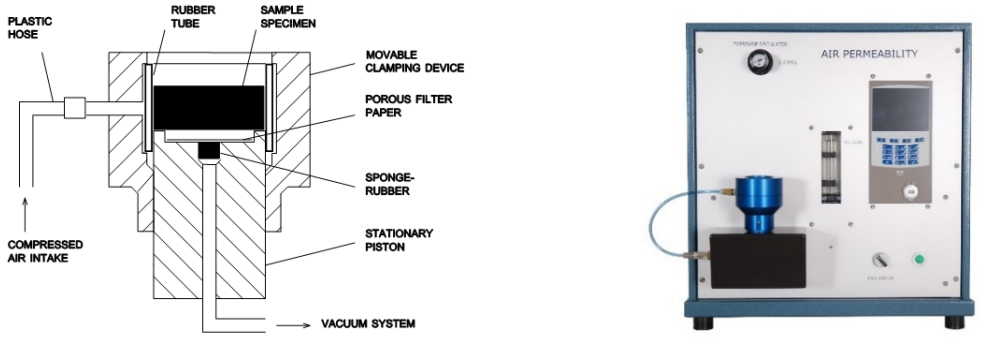

Figure 13: Test arrangement for the determination of air permeability (RDC 145)

The air permeability of the material has a great influence on the electrode air burning. High gas permeability leads to an internal attack of the baked electrode and to an increased consumption.

4.8 Thermal Conductivity (TC): ISO 12987

The thermal conductivity is determined by a comparative method using sample discs with a diameter of 50 mm and a length of 20 mm. The surfaces of the sample have to be plan parallel and very smooth to guarantee a defined thermal transfer to the metallic cells. The use of a special grinding machine with two blades is mandatory (see figure 6 on page 9).

The heat flow is measured between electrically heated (60°C) and water-cooled (20°C) metallic measuring heads as shown below. The results are expressed in W/mK.

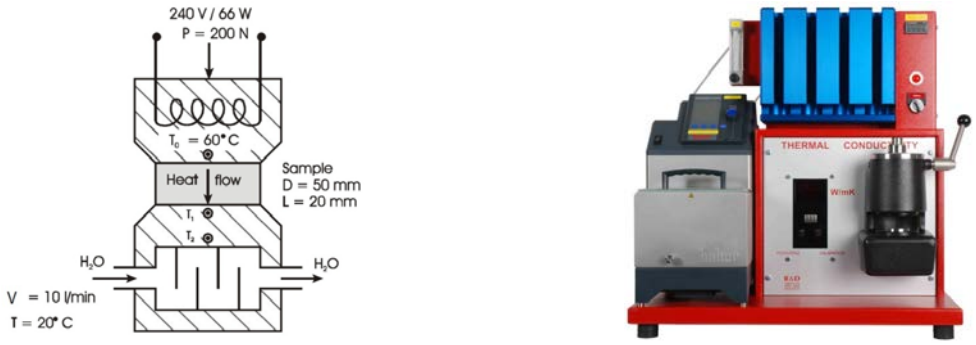

Figure 14: Test arrangement for the determination of the thermal conductivity (RDC 143)

The thermal conductivity is mainly a function of the density of the electrode and of the calcination degree of the dry aggregate. A high density generally leads to a high thermal conductivity as well. High thermal conductivity is favorable for the thermal shock resistance.

4.9 Coefficient of Thermal Expansion (CTE): ISO 14420

The coefficient of thermal expansion is determined by measuring the change in length of a sample with 50 mm diameter and 50 mm length placed in a furnace pre-heated at 300°C during 3 hours. The test arrangement is shown in the figure below.

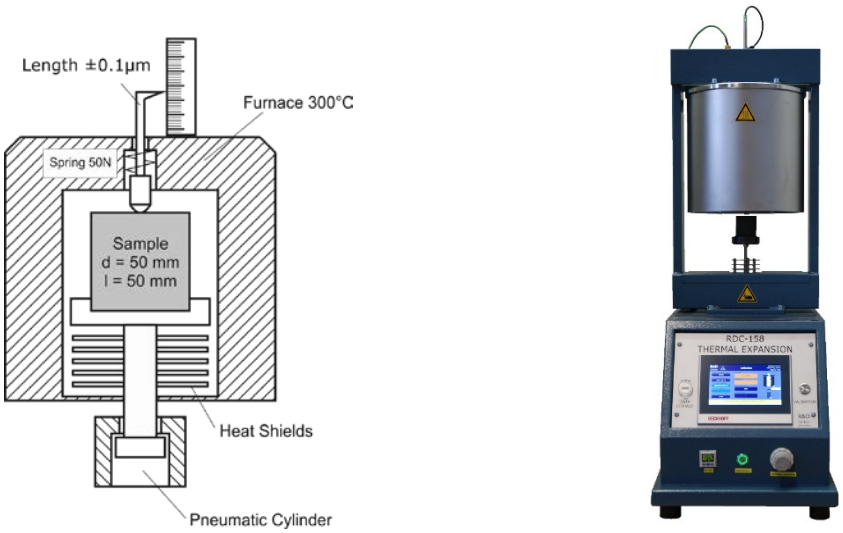

Figure 15: Test arrangement for the determination of the thermal expansion (RDC 158)

The thermal expansion is mainly given by the dry aggregate used for the production of the Söderberg paste. A low CTE is desirable as the thermal stresses in the electrode are directly proportional to the CTE and to the temperature gradient.

4.10 Real Density (RD): ISO 9088

The real density in xylene is measured in a pycnometer with xylene on a sample ground to <63µm particle size.

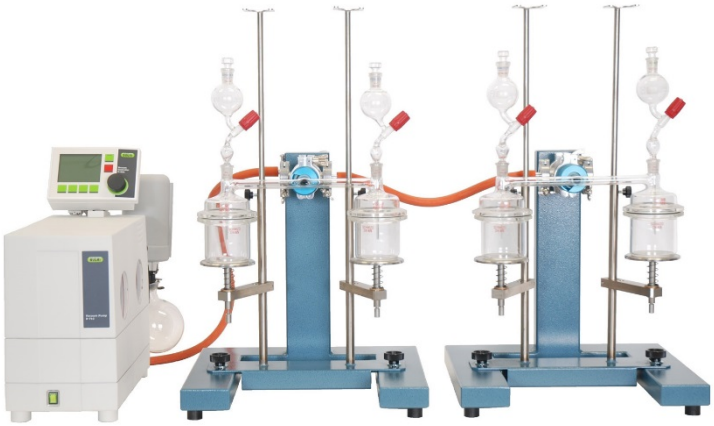

Figure 16: Real density determination apparatus (RDC 152 / 186 / 198)

The real density depends on the real density of the dry aggregate.

4.11 Elements XRF (XRF): ISO 12980

The contaminants are determined by use of an X-Ray fluorescence apparatus (see figure below). The changes in the impurity levels give an indication of inconsistent raw materials quality. The results are expressed in % for the sulfur and in ppm for the other impurities.

Figure 17: XRF spectrometer EXT 103

For Si metal purity, the Fe and Ti contents in baked paste are important.

4.12 Ash Content (AC): ISO 8005

The ash content is determined in powder milled to a size down to 63 µm. The 2 grams sample is burned at 800°C during 12 hours. The ash content is obtained by dividing the residual weight of sample after burning by the initial weight. The results are expressed in %.

The total amount of impurities measured by X-Ray spectrometry can be correlated to the ash content in order to see if other impurities are present or simply to confirm the XRF measurements.

Figure 18: Furnace for Ash Content RDC 169

5 Typical Values

Property		Method	Unit	Typical Range	
				Mean	2STD
Water Content		ISO 11412	%	0.00-0.20	-
Green Apparent Density		ISO 12985-1	kg/dm³	1.55-1.68	-
Flowability (without load)		M185	-	1.20-1.80	-
Baked Apparent Density		ISO 12985-1	kg/dm³	1.44-1.54	0.01
Specific Electrical Resistance		ISO 11713	µΩm	58-72	2
Dynamic Elasticity Modulus		DIN 51915	GPa	4.0-7.0	0.5
Flexural Strength		ISO 12986-1	MPa	4.0-8.0	1.0
Compressive Strength		ISO 18515	MPa	18-35	4
Elasticity Modulus Static		ISO 18515	GPa	2.0-4.0	0.5
Fracture Energy		M184	J/m²	120-220	25
Coefficient of Thermal Expansion		ISO 14420	10^{-6}/K	3.00-4.00	0.30
Thermal Conductivity		ISO 12987	W/mK	3.00-6.00	0.30
Real Density in Xylene		ISO 9088	kg/dm³	1.84-2.04	0.01
Air Permeability		ISO 15906	nPm	5.0-15.0	-
XRF Analysis	S	ISO 12980	%	0.20-0.80	-
	V		ppm	0-100	-
	Ni		ppm	0-100	-
	Si		ppm	100-10000	-
	Ti		ppm	100-200	
	Fe		ppm	200-5000	-
	P		ppm	0-100	-
	Al		ppm	100-5000	-
	Na		ppm	50-500	-
	Ca		ppm	100-1000	-
	K		ppm	100-500	
	Mg		ppm	50-300	
	Pb		ppm	10-100	-
	Zn		ppm	10-100	-
	F		ppm	100-300	-
Ash Content		ISO 8005	%	0.3-5.0	-