MEASUREMENT OF MATERIAL RESISTIVITY AND CONTACT RESISTANCE OF METALLURGICAL COKE

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ABSTRACT

In the submerged arc production of ferromanganese, sillicomanganese and ferrochromium, the coke bed characteristics play an important role. The basic mechanisms of electric current conduction in the coke bed are not yet fully understood, but important parameters are the material resistivity of coke and the contact resistance between the coke particles. Preliminary tests indicate that the contact resistance is of great importance to the bulk resistance of the coke bed. An apparatus measuring the material resistivity and contact resistance of carbon materials has been developed. Both graphite and industrial coke have been tested. The material resistivity tests were performed on single particles to ensure that the effects of pores and cracks were included and the contact resistances were excluded. Two geometries were tested to obtain the contribution of the contact resistance to the total resistance of two connecting particles. The tests were performed in an inert atmosphere at temperatures and current levels found in industrial submerged arc furnaces. The results show that the method has a satisfactory reproducibility. It was found that the contact resistance is about 10 times higher than the resistance of the material when two particles are in perfect contact and that the resistivity of SSAB coke is more than 10 times higher than that of the graphite at room temperature, but with increasing temperature the difference decreases. For SSAB coke the general trend is that both the resistivity and the contact resistance decrease with increasing temperature.

INTRODUCTION

In several submerged arc furnace processes, e.g. the FeMn, SiMn and FeCr processes, the coke bed is the resistance element where the heat is developed. Although the definition of the coke bed varies, a coke enriched area has been reported in several dig-outs of industrial furnaces [1-4]. There have been numerous investigations on the electrical resistivity of coke beds with various particle sizes and compositions [5-16]. These investigations are very useful for understanding how the bulk resistivity of a coke bed is influenced by particle size, charge composition and slag on the coke bed. Based on these earlier investigations it was proposed to investigate two of the basic characteristics of a dry coke bed, the material resistivity, $\rho_{material}$ and the particle-to-particle contact resistance, r_c.

In Figure 1, the equivalent circuit diagram for the total resistance of the coke bed, R_{tot}, is shown. It consists of series and parallel connected resistances. The main factors affecting the total resistance are: 1) The geometrical shape of the coke bed and the arrangement of the particles, which determines the number of particles in series and parallel. 2) The material resistivity combined with the shape of the particles according to equation (1) to get the particle material
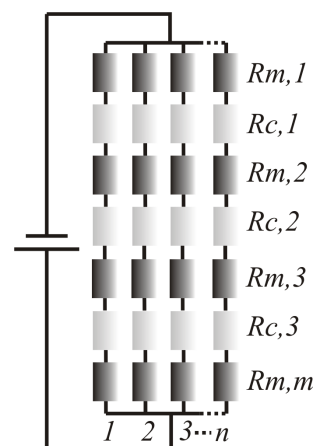

Figure 1: Equivalent electric circuit diagram of a cubically packed coke bed with material resistances R_m and contact resistances r_c

resistance, R_m, and 3) the contact resistance, r_c, between the particles. It is thus necessary to estimate these factors to get a deeper understanding of what affects the coke bed resistivity.

For an arbitrary shaped, inhomogeneous particle, the general relation between the particle resistance, R[Ω] and the material resistivity, ρ [Ω_m] is:

$$R = \int_0^h \frac{\rho(x)}{A(x)} dx \qquad (1)$$

where *h* is the sample height [m] and *A* is the cross-section area [m^2] normal to the current. For a homogeneous sample with uniform temperature, the resistivity, ρ, is calculated from the measured resistance, *R*, by means of equation (1).

In these calculations a uniform current distribution is assumed over the cross-section area of the sample, and thus equipotential lines that are parallel to the end surface. This assumption is not strictly correct since the equipotential lines at all times will be normal to the sides of the test particle, and a par-

Figure 2: Illustration of contribution of reduced cross-section area to total resistance of a particle. Dotted line indicates the particle shape

ticle shaped as a double cone, as tested in this paper, will have equipotential lines that are not parallel to the end surfaces of the sample. As a result the real current conducting area will be smaller than the apparent area *A*. This effect will increase as the ratio between the minimum and maximum diameter decreases. If this is not compensated for, the resistivity calculated from the measured resistance will be higher than the true value. With the radius ratios used in the present work the corrections to the resistivity is thought to be insignificant.

In a standard material resistivity test, one piece of test material of a given geometrical shape is used. The resistivity measured includes the effect of pores and cracks that may be naturally present in the material. For materials that are delivered in large blocks of relatively homogenous material, like metals and graphites, this is fairly trivial to do at room temperature using *the four-electrode-principle*. For graphite this is standardized through e.g. DIN 51911 [17].

The contact area is of great importance to the bulk resistivity and total resistance of the coke bed since the current is constricted in these areas, resulting in local current densities far exceeding the average current density. The resistance of the contact is dependent upon the contact area. This is illustrated in Figure 2, which shows a particle with $\rho_{material} = 0.0003$ Ω_m. The calculations show that the middle 5 mm of the sample contributes more than 36% of the total resistance. If the contact is less than perfect the contribution will increase drastically. This leads to a higher temperature in the material volume in the immediate surroundings of the contact spot and thus an increased reaction rate in this zone, which may again lead to consumption of the contact point. If a gas filled gap is formed, or non-conducting material is deposited the contact resistance will increase, but there might be an increase of the contact area and thus a decrease in contact resistance. A study of this area is, therefore, important. The theory for contact between metals is well established [18]. To the authors' knowledge, the matter of contact between coke particles has not been investigated. For metals the general rules are that the current carrying contact spot area is only a fraction of the apparent contact area. The contact resistance decreases with increasing pressure on the contact area and surface films of non-conducting materials, e.g. oxides, substantially reduce the contact conductance. This is, however, counteracted by a rough surface which increases the pressure in the contact points and cracks the surface film. For a contact between two coke particles the surface is very rough. This will initially lead to only very small contact points. When the pressure is increased, the protruding contacts will eventually crack, leading to a larger contact area. If a
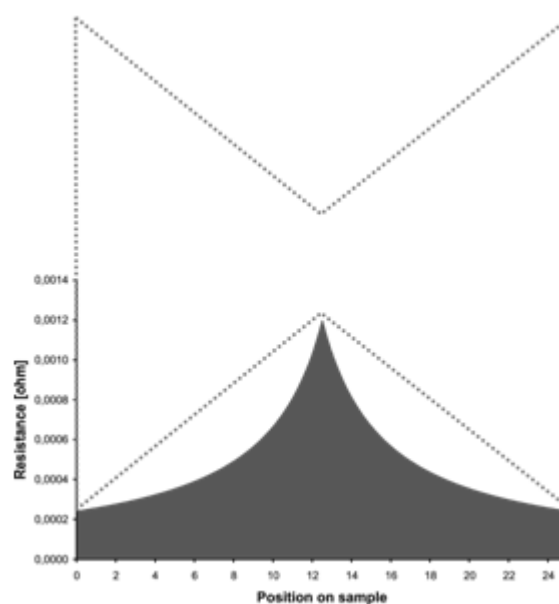
surface film, e.g. deposits from reacted coke, is present in the contact area this may break, and a new or better contact will be established.

The contact resistance can be estimated by measuring the potential drop over two sample bodies in contact and made of materials of known resistivities. By subtracting the calculated contribution of the material resistance from the total measured resistance, the contact resistance is estimated. This method was used by Sørlie and Gran [19] to determine the contact resistance between the collector bar and the cathode used in aluminium cells up to 1000°C. If both bodies on either side of the contact joint are made up of the same material it is also possible to calculate the contact resistance by measuring the resistance of two equal geometrical shapes, e.g. as the shape shown in Figure 2, where one is and one is not connected at the neck. The difference in resistance will thus be the contact resistance.

2. EXPERIMENTAL

2.1 Samples

Experiments were done on one type of graphite and one coke manufactured at SSAB in Luleå, Sweden. Proximate analyses of the materials are given in Table 1. From the graphite bars and coke particles the shapes shown in Figure 3 were made. Both the 30mm diameter cylinder and the double cone, with notation ø30/15mm to indicate the maximum and minimum diameters, were used to measure the material resistivity. To measure the contact resistance identical but *divided* shapes were made of the cylinder for graphite and of the double cone shape for both materials.

Table 1: Proximate analysis of the materials tested

Material	Ash (wt.%)	Fixed C (wt.%)	Volatiles (wt.%)
Graphite [20]	0.1	99.9	0
SSAB coke	13.10	85.71	1.22

The machining of the coke was a challenge, since the coke had numerous internal cracks and pores. It was, however, possible to obtain the desired shapes, but the surfaces were not as smooth as the graphite surfaces. Small grooves were made at the measuring points to ensure that the molybdenum wire used to measure the voltage drop stayed in the same position during the experiment. The grooves were made 3mm from the end surfaces, making the distance between the potential measuring points 24mm. In the centre of each sample there was a 3mm diameter hole.

2.2 Measurement of Electrical Resistivity

Since no standard method for this kind of measurement has been published, the goal was to develop an apparatus that would give reliable results that may be used for obtaining the material resistivity and contact resistance of carbon materials. This apparatus is shown in Figure 4. In this graphite tube furnace 1600ºC can be reached, which is within the temperature range assumed to exist in the industrial coke bed. The size of the apparatus, i.e. the sample including the measurement circuit, is limited by the inner diameter of the graphite tube, 56 mm, as shown in Figure 4.

The support, shown in Figure 5, is made of graphite and is coated with boron nitride (BN). BN spacers are mounted on the support to keep the measuring circuit from get-
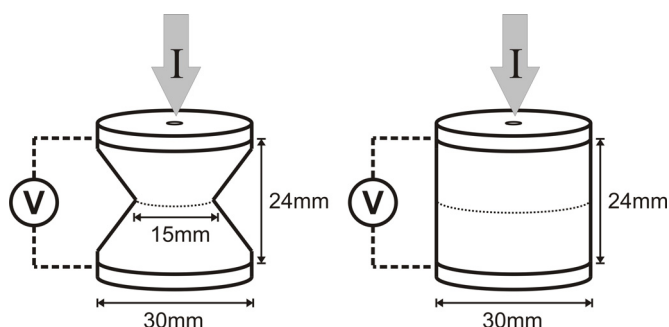
Figure 3: Illustration of the sample shapes. To determine the contact resistance, the sample is divided into two equal parts at the dotted line.
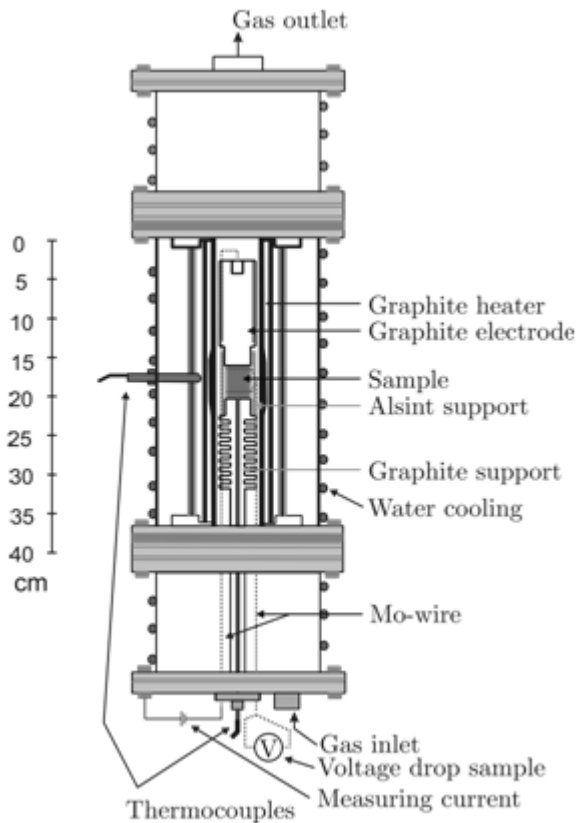
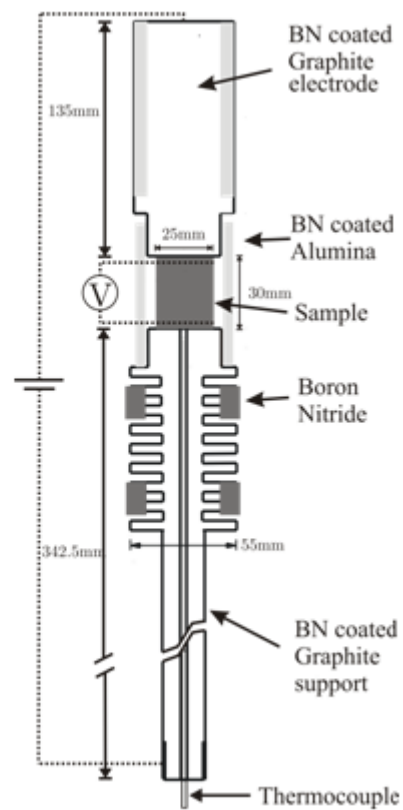*Figure 4: Sketch of apparatus and furnace with main wiring*

Figure 5: Measuring circuit placed inside the graphite tube furnace

ting into electrical contact with the graphite furnace tube. The measuring current going through the sample is supplied by a current controlled DC power source and led to the top electrode by a BN coated molybdenum wire, as indicated in Figure 4. The measuring current was kept at about 3.9 A during the experiments, which for the double cone sample corresponds to a current density of 22 kA/m^2 at the neck. In a 45 MVA furnace the current running through one of the electrodes, diameter 1.9m, would typically be 100 kA, which corresponds to a current density of 35 kA/m^2 in the electrode. If the coke bed is bell shaped, as reported by Barcza et al [1], the current density will decrease as the radius of the coke bed increases. To measure the voltage drop over the sample, a thin molybdenum wire is wrapped around the sample. The wires are placed 3 mm from the top and bottom of the cylinder or double cone to get a uniform current distribution over the sample cross-section area. The Mo-wires are insulated from the graphite support by running them in grooves in the graphite heat shields, separated from the graphite by the BN spacers. The temperature is measured below the sample, inside the graphite support. The furnace is controlled by another thermocouple, indicated in Figure 4. Tests show that the control thermocouple measures a temperature approximately 150°C below the measuring thermocouple, when the latter is indicating 1600°C. Based on these tests and other practical considerations beyond the authors' control, it was decided to use the temperatures registered by the measurement thermocouple for the results. Axial temperature profile measurements show a variation of about 10°C within the area of interest at 1400°C.

The graphite tube furnace is supplied with AC current (typically 1 kA) from a thyristor controlled transformer. This may potentially create electrical noise on the DC low voltage measurements (below 0.1V). This can be averaged away, but may still affect the final result. A possible effect is thus best eliminated by turning off the furnace for a few seconds while the measurements are made. Afterwards the measurements are aver-

aged over a period of 3 seconds. The parameters of the sample and furnace were continuously monitored throughout the experiments by using a data logger. The graphite furnace was flushed with argon gas throughout the experiments.

The weight of the electrode was 461g. The pressure exerted by this will have little effect on the material resistivity but, as mentioned previously, it is well known from metal contact theory that an increasing contact pressure decreases the contact resistance. How this affects the contact resistance between coke particles has not yet been researched, but it is likely that an increasing contact pressure will crush small contact points and thus create larger ones. Since the force from the graphite electrode onto the sample was fixed, the contact pressure would only vary with a changing contact area. For contact diameters of 30 and 15 mm the contact pressures are 659 and 2717 kg/m^2, respectively.

When the sample had been mounted in the furnace a measurement was taken at room temperature. Then the sample was heated to 700°C at a rate of 55°C per minute. After a holding time of 5 minutes to stabilize the temperature, the heating continued to 1600°C at a rate of 10°C per minute. At 700, 1000, 1200, 1400 and 1600°C the furnace was turned off and measurements were made. No measurements were done at decreasing temperature.

The programming of the furnace was based on the fact that the furnace controller was unstable below 900°C and did not give a stable power input, and thus the temperature did not increase evenly. Since the main interest is the electrical properties at temperatures above this temperature, a heating rate of 10°C/min was judged to be a sufficiently slow heating rate to equalize temperature gradients in the system.

3. RESULTS AND DISCUSSION

3.1 Reproducibility and variation in resistivity due to geometrical shape

Table 2 shows the mean resistances at the tested temperature levels with the standard deviation. The duplicate runs give an estimate of the variance over the temperature range. From the data in Table 2 it is not possible to see any trend in the standard deviations suggesting that the variance, i.e. the squared standard deviation, is not constant, at least within each temperature level. A pooled estimate of variance was therefore calculated for each temperature level as well as for the total set of measurements. This was done by calculating the mean squares of the pure error, which is an estimate of the error variance, according to Box and Draper [21].

Table 2: Mean resistance (mΩ) ± std. dev. (%) of parallel runs of the graphite samples tested with pooled estimate of the standard deviation for each temperature

Temperature	19±3°C	707±6°C	1001±2°C	1201±2°C	1399±2°C	1598±1°C
Graphite – divided cylinder	4.14 mΩ ± 11 %	1.03 mΩ ± 4 %	0.915 mΩ ± 2 %	0.851 mΩ ± 7 %	0.802 mΩ ± 9 %	6.52 mΩ ± 6 %
Graphite - d.cone ø30/15mm	0.635 mΩ ± 10 %	0.402 mΩ ± 2 %	0.530 mΩ ± 7 %	0.574 mΩ ± 6 %	0.635 mΩ ± 9 %	0.657 mΩ ± 5 %
Graphite – divided d.cone ø30/15mm	7.10 mΩ ± 4 %	1.61 mΩ ± 2 %	1.68 mΩ ± 5 %	1.73 mΩ ± 4 %	1.69 mΩ ± 3 %	1.47 mΩ ± 4 %
Pooled standard deviation	8.9 %	2.9 %	5.2 %	5.7 %	7.6 %	5.3 %

Pooled estimate of the standard deviation of all the duplicate runs: s = 6.2 %.

The pooled standard deviation shows that the standard deviation at the 700°C level is slightly lower than the standard deviation of the other levels. This suggests that the soaking time is sufficient to equalize temperature gradients that may be present in the system. Preliminary tests did, however, show that this soaking time was too long for the higher temperatures, as the temperature started to drop more rapidly at the higher tem-

perature levels. The results are also satisfying for the other temperature levels. The pooled estimate of the standard deviation, *s*, of the test is 6.2%.

The results show that the test has a high degree of reproducibility.

Table 3: Measured resistivity of graphite double cones and variation from cylinder data

Temperature (°C)	Resistivity ($\mu\,\Omega\,$m)	Std. deviation	Deviation from graphite cylinder data
21	10.2	10 %	12 %
704	6.46	2 %	42 %
1001	8.50	7 %	11 %
1200	9.22	6 %	16 %
1401	10.2	9 %	7 %
1600	10.5	5 %	-1 %

Table 3 displays the measured resistivities of the graphite double cone with the standard deviation of the mean resistivities and the deviation from the resistivity of the graphite cylinder. The resistivity was calculated according to Equation (1). Figure 6 shows the measured resistivity of the graphite, both cylinders and double cone ø30/15mm, and the resistivity reported on the data sheet for the graphite quality used. As mentioned in the introduction, if a uniform current distribution is assumed, the calculated resistivity of the double cone should be higher than that of the cylinder. The results show that the graphite cylinder resistivity is indeed lower than that of the double cone, but the difference decreases with increasing temperature and at 1400 and 1600°C the differences between the resistivities are smaller than the standard deviations of the two parallels. From the available data it is not possible to isolate the previously described effect caused by the shape of the particle. Due to this, the resistivity of the material should not be based on other shapes than a cylinder.

3.2 Comparison to producer data

The producer reports typical averaged data for the graphite, and not specifically for the graphite specimens used in these samples [20]. The general trend is the same in the measured values as in the data from the producer. This may be seen in Figure 6. The resistivities at room temperature and 1600°C are about the same, with a minimum between 400 and 800°C. The measured values do, however, deviate significantly from the producer values. At room temperature the measured resistivity is 14% higher than the reported typical value. At the 700°C level the measured value is 33% lower than the reported value, but at 1000°C the resistivity is yet again higher than the reported value. From 1000 to 1600°C the difference increases from 22 to 48%. Unfortunately no data is given by the producer on the accuracy of the test used, but it is reasonable to believe that the variation within the data the producer values is lower than the differences reported. A publication on

Table 4: Measured resistivity of graphite cylinder and deviation from producer values.

Temperature (°C)	Resistivity ($\mu\,\Omega\,$m)	Deviation from producer values
17	9.08	14 %
695	4.54	-33 %
1000	7.64	22 %
1200	7.97	24 %
1399	9.53	39 %
1600	10.7	48 %

the method used by the producer would have been of great help to understand the significant difference between the measured and reported resistivities.

3.3 Material Resistivity

In Figure 7 the calculated resistivity of the SSAB coke is shown. The general trend is that of a decreasing resistivity with increasing temperature. The cylinder and one of the double cones tested have the same slope, with a marked decrease of resistivity from 1400 to 1600°C. The upper double cone line shows a much higher resistivity at room temperature, but also a steeper decline in the resistivity to the same level as the other coke samples at 1400°C. The marked decrease in resistivity above 1400°C that is evident for all the tested samples is probably due to the fact that the coke was heated to a temperature exceeding the production temperature. This would give further graphitization and a further devolatilization. Above 1400°C SiO_2 in the ash, typically 50wt% of the ash components, reacts at a considerable rate to SiC [22]. It is, however, hard to determine the dominating effect, as the type and amount of SiC formed is not known.

The large variation in the resistivity results between the coke samples is most likely due to the variation in the sample material. This is to be expected, as these are industrial materials, and just a few samples are taken from several tons produced. In the light of this, the spread of the results at 1200 to 1600°C is acceptable.

Figure 6: Measured resistivity of graphite compared to the typical values reported by the manufacturer

Figure 7: Measured resistivity of SSAB coke

As expected, the resistivity of the SSAB coke is significantly higher than the resistivity of the graphite tested. This is due to the higher degree of graphitization of the graphite. From Figure 8 it is evident that the resistivity of the SSAB coke is more than an order of magnitude higher than that of the graphite. The difference decreases as the temperature rises above 1400°C. The reason for this may be increased graphitization of the SSAB coke.

3.4 Contact Resistance

Figures 9 and 10 show the calculated contact resistance and the measured resistances of the whole and divided samples for graphite and SSAB coke, respectively. The results clearly show that the contact resistance decreases from room temperature to 1600°C. It also confirms that the contact resistance is much larger than the contribution of the material to the total resistance between two particles.

For the graphite the difference in resistance between the connected and the divided samples is about one order of magnitude at room temperature. The contact resistance decreases from room temperature to about 700°C. From 700°C the resistance measured for the whole samples increase, and the decrease in contact resistance from 700 to 1600°C is markedly smaller than the change from room temperature to 700°C. For SSAB coke the divided double cone has a resistance that is about 7.5 times higher than that of the whole double cone.
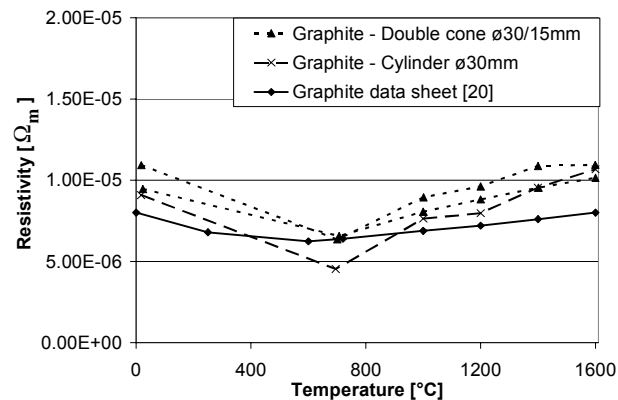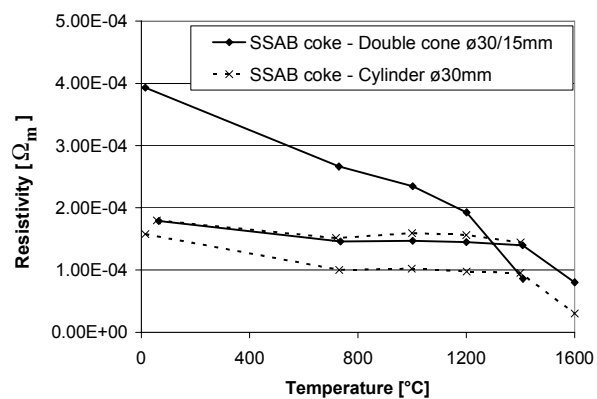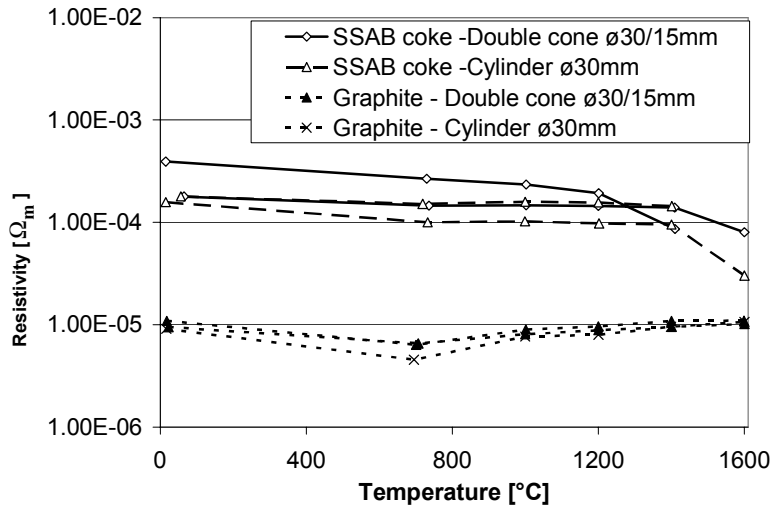
Figure 8: The resistivity of the SSAB coke is about ten times higher than the resistivity of the graphite tested
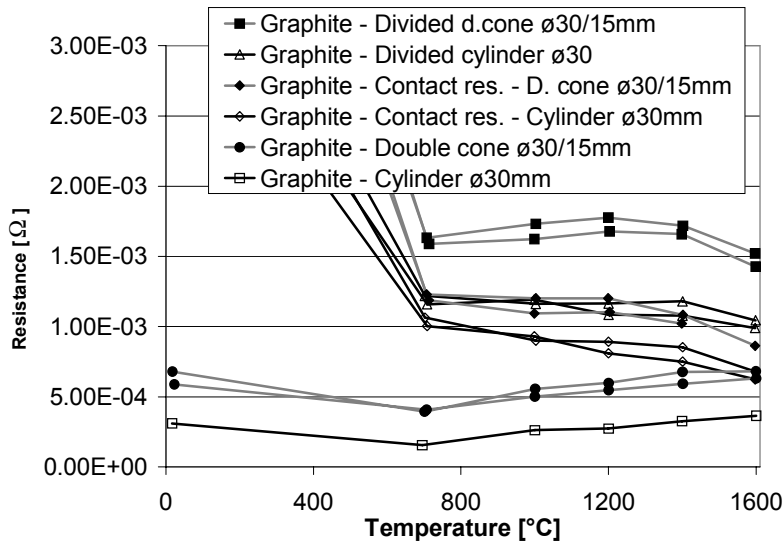

Figure 9: Measured sample resistances and calculated contact resistances of the graphite samples

The contact resistance declines more evenly for the SSAB coke than for the graphite. The different slopes of the contact resistances of graphite and SSAB coke may be due to the way the resistivities change with temperature.

From the theory of metal contacts it is well known that the temperature in the actual contact point can be significantly higher than the bulk temperature of the contact [23]. This is due to the fact that only a fraction of the apparent contact area is actually current carrying. The small volume of material that the current runs through will get heated to a temperature higher than the bulk temperature, and thus the resistivity of the material at temperatures higher than the bulk temperature is important. For graphite the resistivity decreases up to about 700°C and increases from there on to at least 2000°C [20], and thus the increased resistance in the contact point will counteract other effects that may lower the contact resistance as the temperature is increas-
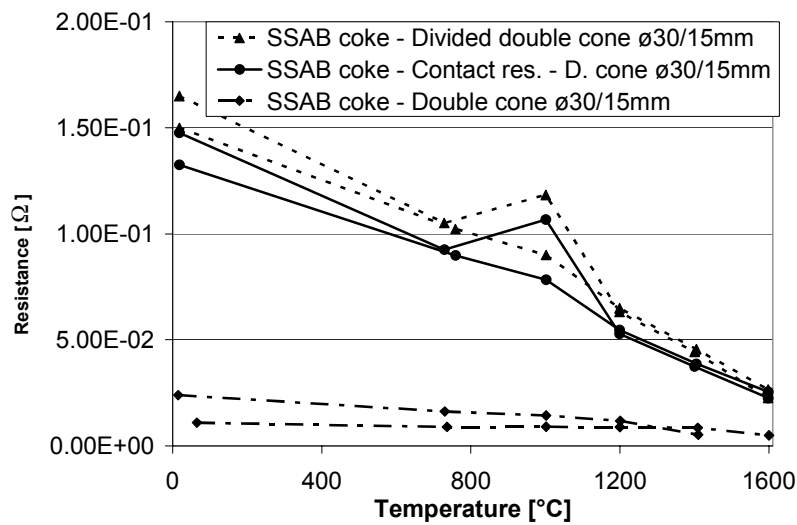
Figure 10: Measured sample resistances and calculated contact resistances of the SSAB coke samples

ing. For the SSAB coke the measurements have shown that the resistivity decreases with increasing temperature, and from Figure 10 it is evident that the contact resistance decreases quite steadily.

It is quite clear that the increased surface of the cylinder gave better electrical contact as shown in Figure 9, even though this decreased the contact pressure. This is most likely due to the low and relatively similar contact pressures and the hardness of the graphite. The contact pressures would need to be considerably higher and the surface rougher to get the anticipated enlargement of the contact area with increasing pressure.

Figure 11 shows the contact resistance of both the graphite and the SSAB coke. The difference between the two is quite significant. The difference may be explained by the fact that the structure of the coke makes the surface considerably less smooth, and thus decreasing the areas of potential electrical contacts.
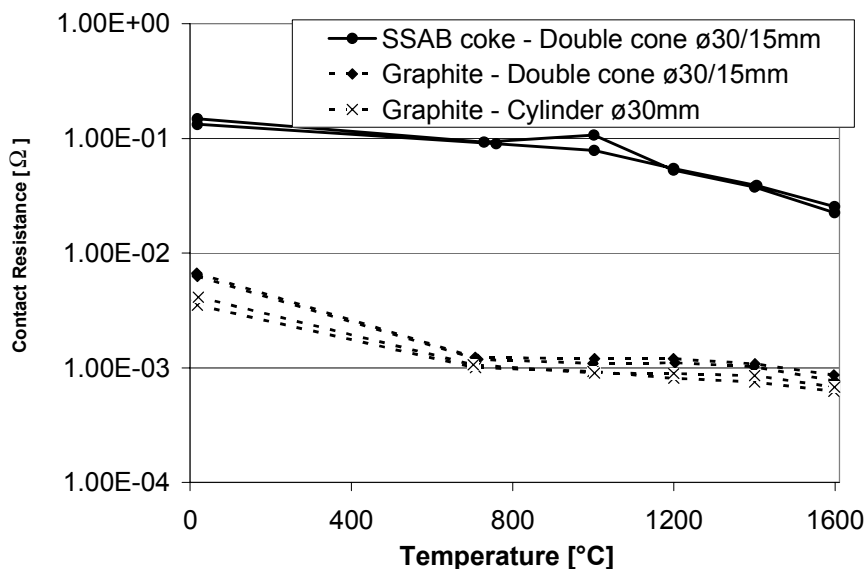

Figure 11: The contact resistance of the SSAB coke is more than 10 times higher than the contact resistance of graphite

4. CONCLUSIONS AND FURTHER WORK

The method developed for measuring the electrical resistivity and the contact resistance of carbon materials has a pooled standard deviation of 6.2% for the temperature levels tested, which is acceptable. The resistivity trend with respect to temperature is the same as that reported by the manufacturer for the graphite, but the measured resistivity is higher.

The resistivity of the SSAB coke is, as expected, considerably higher than that of the graphite. At room temperature the graphite resistivity was measured as $9.08\mu\Omega m$, and of the SSAB coke as $168\mu\Omega m$. As the temperature rises the graphite resistivity decreases towards 700°C and then increases to $10.7\mu\Omega m$ at 1600°C, whereas the resistivity of the coke decreases continuously from room temperature to $30.2\mu\Omega m$ at 1600°C.

The contact resistance generally decreases with increasing temperature, and the difference between graphite and SSAB coke is pronounced. For the graphite double cone ø30/15mm it decreases from $6.46m\Omega$ at room temperature to $0.817m\Omega$ at 1600°C. By comparison, the corresponding figures for the SSAB coke are $140m\Omega$ and $24m\Omega$.

It was shown that the shape of the sample influences both the contact resistance and the sample resistance. In both instances the resistance increased when the minimum diameter was decreased. This effect was stronger than the increased contact pressure caused by the decreased apparent contact area.

The next step should be to do more replicates of the SSAB coke and other cokes to check the ability of the method to distinguish these. The results show a variation between the coke replicates that is larger than the variation between the graphite replicates. More replicates and other material would create a picture of the variance of the cokes, and thus the ability to differentiate between these. Work is also done on making samples from coke particles with a diameter below 50mm.

The final goal is to use the information on the material resistivity and the contact resistance to build a mechanistic model that describes the resistance of the coke bed. In this model variables as packing, electrical resistivity of slag, pressure and the geometrical shape of the coke bed must be included.

By obtaining a more fundamental knowledge of what influences the electrical resistivity of the coke bed, it will be easier to make a qualified guess on what choice of raw materials to include in the charge mix.

5. ACKNOWLEDGMENTS

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