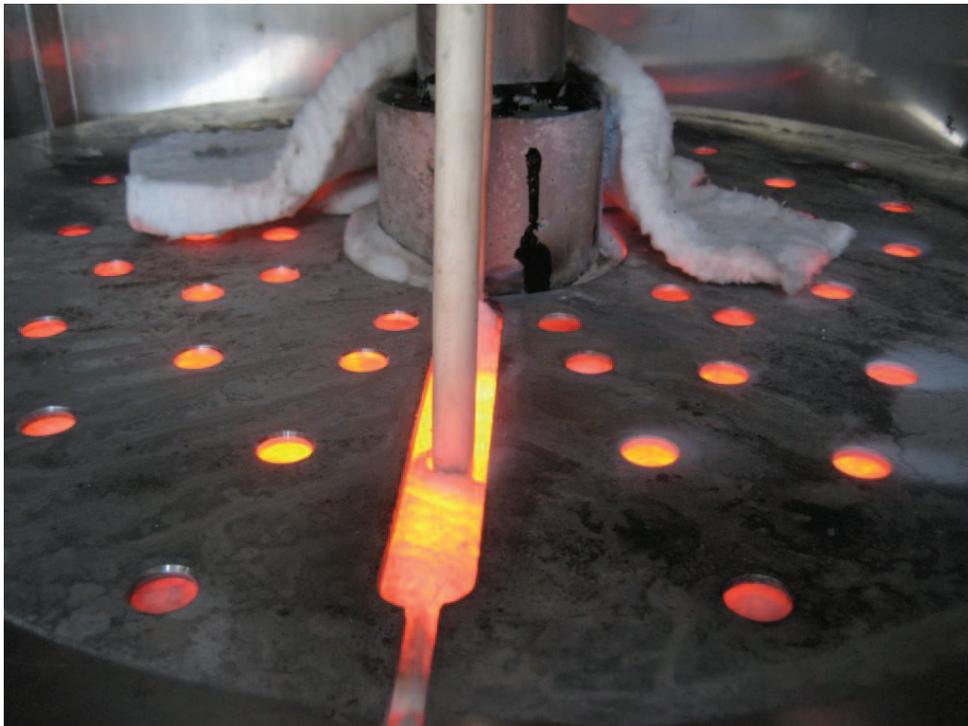


INTERMEDIATE PROGRESS REPORT INDU CARB (INOX)-DEVELOPMENT FOR RECOVAL (BELGIUM)

Contents

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Preface – Management Abstract

Reference is made on the agreement between Recoval SPRL of Belgium, Tribovent Process Development Ltd. in Austria and Patco Engineering GmbH of Switzerland signed in June 2007.

It was agreed by all parties to concentrate development efforts on high Ni-containing steelmill dusts, with the aim to reuse Ni in Form of FeNi as a premelt.

For this target Tribovent selected the InduCarb (RecoDust) Process-System to be adapted for direct introduction of Ni-containing steelmill dusts, together with some acidic additive to maintain flow ability of resulting melts.

„InduCarb“ in this sense means melting-reduction of high Ni-containing steelmill dusts via inductively heated coke bed to FeNi as a premelt for stainless steel making (austenitic steel) by direct feeding and dosing the educts.

The original RecoDust-Concepts feeds the educt („rawmaterial“) in form of a melt ex Flashreactor to the InduCarb-Reactor to get a purified Zn. In this case the recovery of Ni and not Zn was the target, so direct feeding of dust was agreed by Recoval. This meant the development of a dust feeding and dosing system, as well as changing the entire InduCarb from counter current to a parallel flow system.

During development phase quality of steelmill dusts has been changed by Recoval demand to high chromium containing steelmill dusts from the ferritic steel production. Processing of high chromium containing dusts needs significant additional enforcement of the InduCarb-Principle due to different properties and thermochemical behaviour of chromium and its compounds, compared to the nobler Ni. This was to take under consideration by Tribovent and leads to a respectively adapted InduCarb-Process concept, potentially fulfilling demands for recovering Ni and additional Chromium in form of a FeNiCr premelt. The new adapted InduCarb-Process concept additional will potentially avoid expensive metallurgical coke (meantime coke prices rise significantly!), minimize specific CO₂-emission and reduce production costs:

The new InduCarb(Inox) – Process approach avoids expensive metallurgical coke by dividing both functions, namely upheating the reactive partners by an inductive heated non-consumable graphite bed and melt reducing by application of cheaper coaldust, but still in one InduCarb-Reactor. Steelmill dusts together with some additives and coaldusts will be fed by Tribovents own dust dosing – system technology via some nitrogen gas to the InduCarb reactor. A more detailed description will be given with the separate report „Next Steps“ after approval of this report by Recoval.

Acidic additives have also been evaluated. Suitable for the process are e.g. waste foundry sand, waste glass, shale, used acidic refractory from steel industry and foundries.

In parallel to the testing phase and developing phase for the new InduCarb (Inox)-process approach, heavy supply delay of the different steel mill dusts by the steel producer causes serious organizational troubles within the Tribovent-organization.

This development phase was supported by the Montanuniversität Leoben (scientific support), Leoben, Austria; Fa. Schenck – Prozess, Braunau, Austria (dust dosing tests); Fa. Dynamic Systems, ZH, Switzerland (mathematical modeling); and Eldec, D (Induction plant).



Friendly support regarding coke/graphite evaluation by SGL-Carbon, Meitingen (D) and Linde Gas (A) regarding consultancy of gas supply and waste gas treatment. Important consultancy by a not named highly qualified and experienced design engineer, in the field of high temperature furnace development, design and construction.

A qualified dust feeding and melting trial on the testing plant in Bludenz, similar to the developed concept for the InduCarb-pilot plant, was not possible due to the high technical and also safety demand on the waste gas system (e.g. post combustion, temperature quench and maintaining low pressure inside the system for safety reasons). Such dust feeding and –dosing tests would exceed the available budget of this phase by far.

However, a simplified, but risky (safety conditions, CO emission, etc.) “handfed show test“, but irrelevant to the foreseen pilot feeding/dosing system, would be possible for tilting demonstration of liquid melt out of the testing InduCarb plant.

A revised budget plan and time schedule will therefore be supplied within the separate report regarding „Next Steps“.

By the way, it has to be mentioned, that „InduCarb“ has been awarded with the Austrian State Innovation Price „Econovius“ 2007!

Summary – Management Abstract

The development work originally was dedicated and focused on pyroprocessing of high Ni-containing steelmill dusts by the InduCarb-Process from the austenitic steel plate production of Genk and Charleroi to recover Ni for its reuse in the processing of stainless steel, e.g. as a premelt (FeNi) in the EAF, AOD-Converter or Secondary Metallurgy.

During the ongoing development phase the project target was extended by Recoval on demand to develop the InduCarb (Inox)-Process also for the melting-reduction of high chromium containing dusts from the ferritic steel production, accordingly given chemical analysis of several dust qualities. This means consequently the development of a new highly flexible InduCarb-Process concept to fulfill also demands regarding melt reduction of the extremely oxygen-affine Chromium content of the dusts as well as the significantly increased melting point of the yielded melt. Special attention has also to be paid on the process-feasibility due to the low Cr-Price in comparison to the high Ni Price (see also LME). The „conventional“ InduCarb process operates with metallurgical coke (meantime high increased coke price), which makes the economic feasibility questionable, based solely on the recovery of FeCr as premelt. Also attention regarding specific CO₂-emissions should be considered. Very problematic to Tribovens capacity planning and organization was also the heavy delay of the supply of the steel mill dust samples by the steel plant from Belgium to Austria.

However, this report concentrates on the valuable and important results of the testing phase, supported by the Montanuniversität Leoben, Austria. A separate report (Proposal Next Steps) will show the new InduCarb-Process-Approach, which is based on the testing results and considerations mentioned above. A revised budget plan and time schedule shall be implemented.

The following intensive development and preparation work for the „dust- and gasless“ InduCarb-Testplant (200 kW electric power) was done up to the plant commissioning:

1. Electrical and mechanical development, design and optimization for the induction generator as well as inductor successfully completed by Eldec specialists
2. Development, design and optimization of the „dustless“ InduCarb-testing plant, coke extraction system, instrumentation, safety considerations and evaluation
3. Development of a thermo-couple system that allows to measure temperature in the high frequency inductive field of the dustless InduCarb coke bed
4. Refractory evaluation, design and construction of refractory parts and elements performed
5. Design, construction and testing of an auxiliary cooling water system
6. Design and construction of PLC, mechanical testing equipment, water-/cooling infrastructure, devices for heat – and mass balances
7. Development of test-evaluation program and graphic software, implementation in PLC
8. Pre evaluation of several coke qualities (Eldec) and purchasing of sufficient coke amounts (TVT)
9. Theoretical evaluation of potential dust additives to decrease melting point (MU-Leoben)

10. Establishing several heat- and mass balances for high Ni- and Cr- containing dust treatment on theoretical base (MU-Leoben)
11. Very first approximation on electrical energy demand (without introduction of oxygen for carbon-degasification!) done by Tribovent shows numbers in the order between 1.5 to 2.5 MWh/t of dust, depending on the chemistry and humidity of the dust
12. Preparation work of dynamic process model, but without dust feeding and gas development done by Dr. Hans Musch, Dynamic Systems, Zurich
13. Cold and hot commissioning of the entire testing plant successfully done
14. Separate pretests performed for dust dosing system, based on Tribovens own technology, done with hardware supplier (Schenck-Process) with success. The dust-feeding and –dosing was one of the most critical items of the basic development work, a lot of tests have been performed with several systems and suppliers, more or less insufficient. E.g. pneumatic dosing leads to instable process conditions and huge waste gas amounts. Accordingly to these test results it is not possible to use the counter current flow principle for InduCarb (Inox), so the parallel flow system with decreased process pressure was chosen for the pilot-concept, avoiding also heavy dust extraction within the waste gas system and potential of dangerous process-gas Emission from the InduCarb-Reactor. Dosing system, similar to Tribovens system for the Tribovent Flash-Reactor was chosen for the Pilot Concept
15. Very first considerations regarding waste gas treatment concept done for the Pilot Installation

After successful hot commissioning of the „dust- and gasless“ InduCarb-Testing plant accordingly a testing period has been performed with the following main results

1. Fast coke upheating velocity (typical 25 - 45 K/min, max. 55 K/min), limited by power supply of induction generator, high energy density on coke bed periphery
2. Good conversion efficiency of electric energy to coke heat is observed with about 60 %, even with not optimized thermal insulating of the testing plant. Testing electro-inductive frequency between 50 and 200 kHz
3. High temperature levels are achievable (more than 1800°C possible, which is more than sufficient for metallurgical reasons), limited only by thermal properties/ destruction of thermocouples and refractory material. Typical testing temperature between 1550°C and 1685 °C
4. Finalization of modeling work, done by Dr. H. Musch, Dynamic Systems, Zurich
5. According to coke grain size and induction frequency a radial temperature gradient from cokebed - periphery to the cokebed - center (300 mm) between min. 167 K and max. 602 K are observed, due to the thermal conductivity properties of the porous coke and radiation between coke particles, under dynamic conditions with implemented central cooling finger
6. Sharp skin effect observed and evaluated. This leads to a „Dead Man“ in the center of the InduCarb, more or less similar as known in the blast furnace. Temperature and heat equalization (steady state conditions) from cokebed-periphery to center therefore needs some time (heat conductivity of coke)
7. As previous stated, there was no gas movement/-dynamic due to CO-development of melting-reduction of the dust (convective heat transport) nor heat transport via hot and radiant dust particles available and considered due to testing set up and testing conditions



8. Also a vertical temperature deviation of minor importance was observed
9. Potential process modifications/adaptations and optimization opportunities shall be described within the separate report „Proposal Next Steps“
10. According to investigation results and considerations regarding above described high chromium containing dusts and its consequences a new budget plan and time schedule will be worked out and presented within „Proposal Next Steps“
11. A basic concept for the pilot-plant in Charleroi has been developed and worked out

Conclusion and Recommendation – Management Abstract

Results of the described trial phases show the principle technical feasibility of the InduCarb-concept: good energy transformation efficiency from electric power to thermal heat, fast upheating velocity, feasible inductive frequency.

Problematic items are the radial temperature gradient from coke bed periphery to its center due to skin effect and low thermal conductivity of coke. This also means superheating the cokebed-periphery due to skin effect by relatively high electro-inductive frequency needed due to relatively high electric resistivity of coke. Meantime high market price and problematic availability of metallurgical coke also in Europe occurs.

The next development step consequently will be proposed based on the adapted InduCarb (Inox)-process concept, separating the two functions

- „inductive upheating“, using no consumable graphite-briquettes also on needed high temperature level instead of coke and
- „melt reduction“ of the up-heated dust to Ferro-alloy and slag melt, using coaldust instead of expensive metallurgical coke as the reduction agent.

The use of graphite instead of coke includes the following significant advantages, e.g.:

- Very high thermal conductivity of graphite leads to decreased temperature- and heat-gradient to the bed center. This means higher specific productivity per given bed diameter and therefore less investment in the InduCarb-Reactor. Also less heat losses and therefore less energy costs due to decreased outer surface of the Reactor.
- Very low electric resistivity of graphite means lower electro-inductive frequency demand and therefore a lower skin effect (peripheral superheating of graphite bed) and cheaper investment of electrical plant-equipment
- High thermochemical resistivity of graphite under reductive process-conditions leads to minimized graphite consumption, especially if highly reactive coaldust as reduction agent is applied.

Verification tests of the graphite advantages should be considered and could be arranged upon request.

The next development steps to be proposed are to be focused in preparation work of an „Integrated InduCarb-Plant“, combining e.g. storage, preparation and dust dosing – equipment with InduCarb and waste gas-treatment. The next phase therefore will be performed in cooperation with our specialized engineering company/plant supplier INTECO GmbH, demonstrating also the economical feasibility and preparing the documents for a basic plant and process approval by the Belgium authorities.

A pilot plant (including infrastructure like storage silos, dust pre-blending with additives, coaldust storage, feeding and dosing systems, waste gas system, etc.) has to be constructed in Charleroi.



Further and sustainable scientific support, scientific cooperation and independent consultancy e.g. with the Montanuniversität Leoben (A), Prof. Dr. H. Raupenstrauch (Thermoprozesstechnik) and Prof. Dr. Antrekowitsch (Nonferrous-Metallurgy) are also strongly suggested due to the fact of developing a worldwide new process. Of course there are also other specialists from other Universities possible to be consulted, according to Recovals preferences.



Montanuniversität Leoben - University of Leoben

Department Metallurgie - Department of Metallurgy

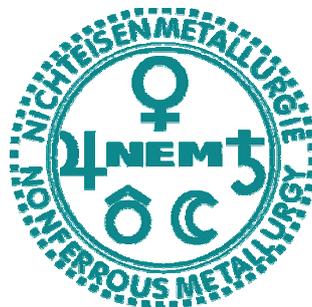
Nichteisenmetallurgie - Nonferrous Metallurgy

Head: Ao.Univ.-Prof. Dipl.-Ing. Dr.mont. Helmut Antrekowitsch



Experimental report:

***Investigations on the treatment of nickel containing
residues in an inductively heated coke bed***



Dipl.-Ing. Dieter Offenthaler

Dipl.-Ing. Dr.mont. Jürgen Antrekowitsch

Leoben, May 2008

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1. Introduction

The recycling of steel mill wastes nowadays heads for a multi-purpose facility that shows the opportunity to treat several residues from different dusts to slag. The InduCarb technology - invented by the company Tribovent G.m.b.H - should meet this specification and furthermore implements various advantages. Compared to other reduction-processes the inductively heated coke bed (InduCarb) should offer

- an economical operation even at low yearly tonnage,
- high yields of valuable metals,
- a simultaneous recovery of different metals,
- high quality products,
- low off gas volumes and
- a fast reduction process.

Especially for the recovery of high price metals like nickel or chromium the recycling process should focus on fast and complete extraction beside low amounts of emissions to fulfill economical and ecological requirements of the future. Competitive processes based on plasma technology or submerged arc technique, are characterized by a very high energy input resulting in a low degree of efficiency. Compared to this, in the InduCarb-reactor the energy is concentrated in a relatively small area in which melting and reduction takes place rapidly and therefore heat losses can be minimized, leading to a high efficiency of the provided energy. In case of using inductive heating as single energy-support (no addition of oxygen) an essential advantage can be seen in the very low off gas volume generated during the process.

2. Characterization of provided materials

Nine different samples of residues from steel mill processes were provided by Recoval.

The characterization was done by Scanning Electron Microscopy (SEM), Laser Diffractometry (Cilas) and by means of chemical analysis especially the Inductively Coupled Plasma (ICP). To allow a more simple identification the nine samples were designated with P1 to P9. The correlation to the original labelling is given in table 1.

P1	déchet nettoyage slabs "B"
P2	Ex Filtre Filter
P3	déchet nettoyage de slabs 0,40m x 5m "A"
P4	Filter Stega
P5	Otk Droog stof silo
P6	Filler BB6 "B" 19.04.07
P7	Filler BB6 "A" 19.04.07
P8	UGINE & ALZ Belgium Dry Dust
P9	Filter Beton 2 DW

table 1: identification of samples

In table 2 the results of the chemical analysis are summarized.

	P1	P5	P8	P2	P4	P9	P6	P7	P3
Zn	1.80	1.02	1.59	-	-	-	-	-	1.81
Pb	0.13	0.31	0.68	0.12	0.03	-	-	-	0.39
Cd	0.02	-	-	-	-	-	-	-	-
Cu	0.18	0.19	0.22	0.19	-	0.18	-	-	0.58
Fe	16.60	9.15	3.26	11.70	1.63	0.89	0.53	0.43	12.80
Fe ²⁺	2.56	3.49	6.03	2.51	5.39	4.76	1.24	1.85	9.58
Fe ³⁺	4.25	14.23	18.54	39.30	49.98	50.67	1.99	1.29	22.52
CaO	28.90	24.00	19.30	-	-	-	40.20	40.20	3.12
MgO	3.96	5.64	6.40	-	-	-	8.60	8.72	1.36
Al ₂ O ₃	2.11	0.18	0.30	-	-	-	4.32	4.23	0.21
SiO ₂	13.70	4.90	3.80	-	-	-	29.80	29.30	0.10
MnO	1.66	4.53	4.96	1.48	1.40	1.41	1.70	1.70	3.98
Ni	2.55	2.80	3.49	3.50	2.90	3.11	0.23	0.27	4.00
Cr	8.72	9.40	9.67	8.05	6.90	7.37	7.38	7.86	9.36
Cl	0.14	0.11	0.12	-	-	-	-	-	-
F	0.42	0.80	0.59	-	-	-	-	-	0.50
C	-	-	-	0.18	0.10	0.10	-	-	0.21

table 2: chemical analysis

Sample P1 is characterized by a relatively big grain size and a high amount of metalized iron accompanied by a high amount of slag components. Samples P5 and P8 are more typical for steel mill dusts with grains mainly sized below 20 μm . The grain size distribution as well as the analysis of P2, P4 and P9 indicates that these materials are typical scales. Samples P6 and P7 are slags with low contents of valuable metal oxides. Sample 3 is more or less an exception because the grain size distribution and the included elements are typical for a dust on the one side, while the amount of metalized iron is very high and the slag components (SiO₂ and CaO) are very low, what is on the other side untypical for steel mill dusts.

The Nickel content can be found typically around 3 % for the dusts and the scales, which is an amount that should enable an efficient and economical recovery of the metal.

The content of chromium in the form of chromium oxide is rather high for most of the samples. With this, a high melting point of the resulting slag in a melting process can be predicted if the reduction of chromium oxide does not take place in an adequate time. The meltability of the remaining slag containing only SiO₂, CaO and MgO and its suggestibility by adding SiO₂ is discussed in one of the following chapters.

3. Mass and energy balances – thermochemical calculations

Based on thermochemical databases from the software “FactSage 5.1” and from “HSC Outokumpu research” a theoretical modelling was done, that delivers important process data like mass and energy balances as well as the composition and the amount of the resulting slag and the off gas.

For this, the necessary energy for heating up the dust components, the fusion heat, the reduction energy, the energy losses and the coke preheating were carefully calculated for different temperatures. The process temperature, the coke preheating, the reduction grade of various compounds and the option to add oxygen were kept variable in the model.

In the following such a mass and energy balance for the dust P8 is demonstrated, as P8 was indicated by Recoval as the most readily available dust. Coke preheating was assumed with 1000 °C. Further assumptions are:

The grade of iron and nickel oxide reduction should be 100 %, the one of chromium oxide 60 %. One half of the energy should be provided by induction heating while the other half should be generated by combustion of coke.

The result of the calculation is displayed in the following diagrams and tables:

In figure 1 the energy output is shown for the described procedures for 1 kg of dust.

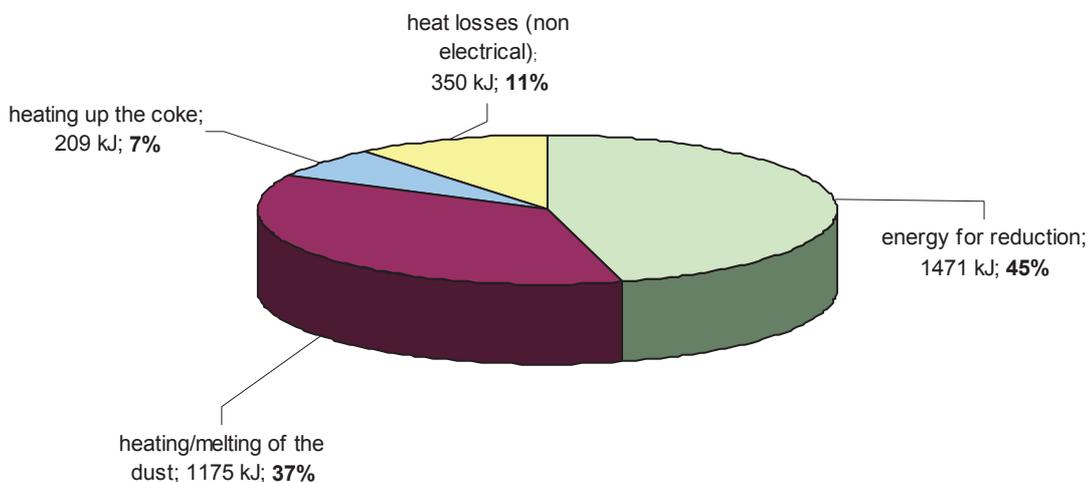


figure 1: energy output, split into different categories

In figure 2 the energy input is displayed for the treatment of 1 kg of dust for the above mentioned conditions:

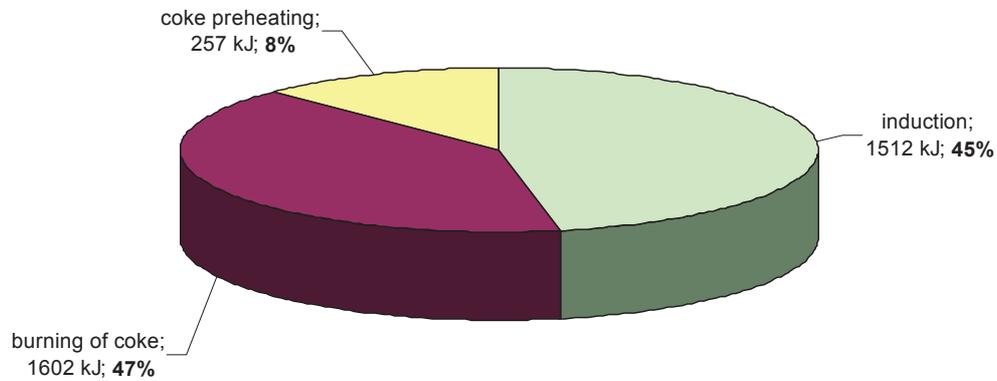


figure 2: energy input, split into different categories

With coke preheating at an assumed 1000 °C it becomes obvious, that related to the whole energy demand the coke preheating plays a secondary role. The expected compositions of the produced metal alloy and slag are shown in table 3 and table 4. About 370 kg of alloy and 440 kg of slag are produced per ton of charged dust. The difference (190 kg) is caused by the oxygen removed during reduction and by some volatile components (e.g. Zn, F, Cl)

	Fe	Ni	Cr
mass-%	74.97	9.40	15.63

table 3: calculated composition of the produced alloy

	Cr ₂ O ₃	SiO ₂	CaO	MnO	MgO	Al ₂ O ₃
mass-%	8.84	8.78	55.44	11.46	14.78	0.69

table 4: calculated composition of the produced slag

4. Meltability of the produced slag

As mentioned above, the meltability of the dust plays an important role for the process. A fast smelting leads to a more intensive contact of the material with the hot coke and therefore yields in a high reaction surface that offers a fast reduction process. Most responsible for the meltability are the various slag components (SiO_2 , CaO , MnO , MgO and Cr_2O_3). If Chromium(III) oxide is not reduced to Chromium(II) oxide or to chromium metal very high melting points are the result because of the high content in the samples. For such systems only little information can be found from literature and also thermochemical databases are rare. An attempt to calculate such a system with "FactSage 5.5" for 2000 °C is shown in figure 3.

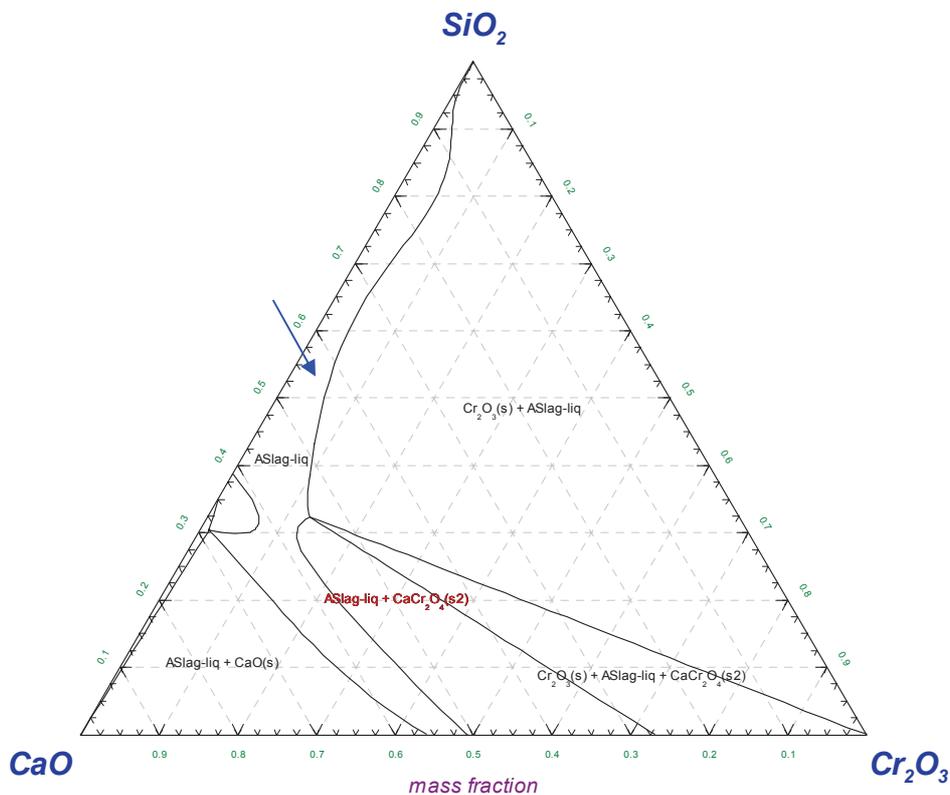


figure 3: system SiO_2 - CaO - Cr_2O_3 at 2000 °C

When the calculation was done for 2000 °C the area where a liquid slag is possible (highlighted by the blue arrow) is rather small.

Assuming that chromium oxide reduction takes place, the meltability of the slag components can be described by the system $\text{SiO}_2\text{-CaO-MgO}$. This system was calculated for different temperatures from 1500 °C to 2000 °C. The area where a liquid slag is possible for the various temperatures is shown in the ternary system in figure 4. The triangles show the position of the samples P1, P5 and P8. Only P1 gets close to the areas where molten slags are possible. For the other two a significant addition of SiO_2 is essential to make a melting at lower temperatures possible.

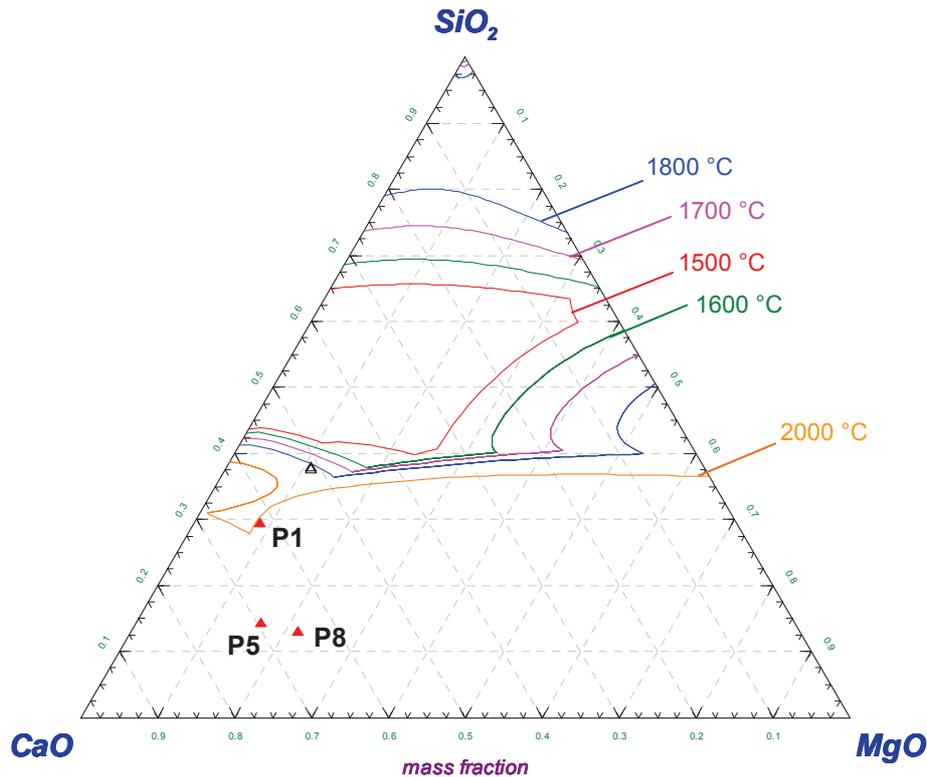


figure 4: system $\text{SiO}_2\text{-CaO-MnO}$ at various temperatures

These diagrams were received from theoretical data and not all interactions with other compounds can be taken into account with this method, but the results deliver enough information concerning the necessary preparation of the dust as well as the temperature that should be chosen to run a successful process. In view of this high temperature needed for the reduction process it became necessary to practically test the energy distribution induced by induction into the coke bed in order to verify if the available energy is distributed well enough in order to allow the total reduction of the material, assuming that the melting energy is coming from coke burning.

5. Experimental investigations

The aim of these basic investigations (without feeding of material onto the coke bed) was to characterize the influence of induction heating on a coke bed. For this a mathematical model was developed (see chapter 5.3), that allows to describe the inductive heating process of the coke bed in radial and vertical direction. To fit the parameters of the model and to verify the model itself, it was reverted on experimentally measured time-temperature graphs (see chapter 5.2). The main advantage of the model must not be seen in the ability to simulate the time-temperature graphs in dependence of frequency, coke particle size, power supply and heat losses but in the fact that it allows to analyze the measured graphs and quantify the different influencing variables in a very complex system with lots of interactions. It is a tool to determine the penetration depth of induction, to quantify the amount of heat conduction and radiation in the coke bed and to estimate with sufficient accuracy heat losses as well as the efficiency of the whole system. All this information and knowledge is not only of scientific interest but it is essential for a future design of the InduCarb-reactor and the scale up of the test plant. Finally an evaluation is possible whether, to which extent or under which conditions the principle of induction is appropriate to heat up and keep on temperature an energy absorbing coke bed of a certain diameter.

Another main aspect of the tests was to determine the optimum induction frequency with respect to coke particle size. Additionally the preliminary trials should help to reveal possible and unpredictable problems linked to the applied high frequency induction-technology as well as to the InduCarb process and design itself. By identifying such problem areas in an early stage of process development, there is a chance to consider these cognitions in the following pilot-plant design, avoiding the linked problems.

5.1 *Experimental setup*

As mentioned above, this first testing-facility was designed to characterize and study the influence of induction heating on a coke bed. It has to be underlined that further smelting and reduction trials with a continuous melt flow and representative dust amounts require extensive modifications and a redesign of the facility respectively. However key-components of the present plant (induction coil, capacitors, high-frequency generator, cooling circuit, instrumentation . . .) can directly or with small modifications be used for a further testing-facility. Although the testing-facility seems to be quite simple in the present form, the design, construction and assembling took quite a lot of time, lasting until January 2008. Especially the access to high-frequency induction technology turned out to be difficult and the delivery times associated with this equipment (special copper profiles for the induction coil,

capacitors, generator, . . .) were crucial for the start of the planned test series. A detailed description to the structural design and the instrumentation of the testing plant is given in the following text.

5.1.1 Structural design of the testing facility

Figure 5 and figure 6 show a schematic illustration and a photo of the testing plant. Its main components are:

- the high frequency generator
- the induction coil with the associated capacitors and cooling devices
- a heat sink, placed in the coke bed and called “cooling finger”
- the mechanical construction holding the refractory lining and
- the instrumentation.

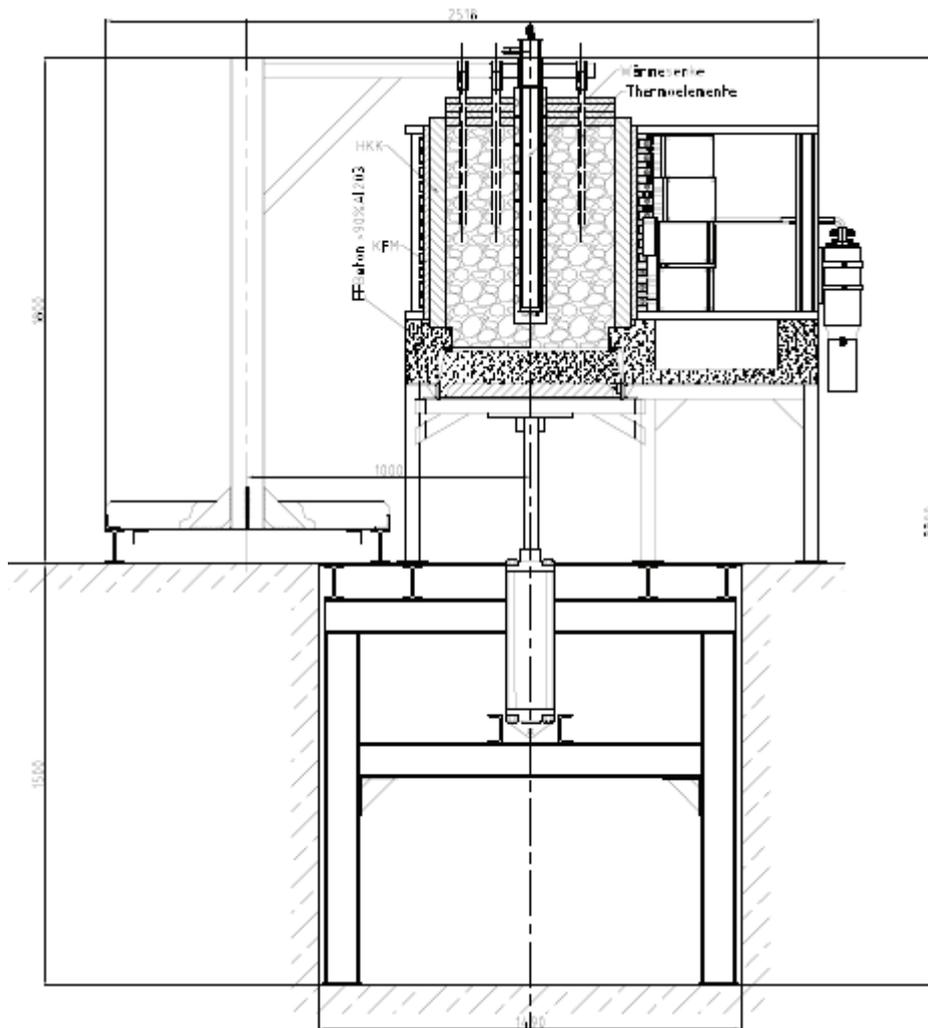


figure 5: structural design of the testing facility



figure 6: testing facility

Figure 6 shows the testing plant in operation during a coke-heating-trial – for a better understanding the main plant components are labelled. Without going too much into detail, the following text should give an overview of the single plant components and describe its main characteristics.

5.1.1.1 High frequency generator

The HF-generator was delivered from “eldec Schwenk Induction GmbH”, for reasons of economy and especially to save time an existing generator at Eldec was modified for the purposes of the trials and rented by Tribovent for the time of the tests.

The high frequency generator (type: Eldec HFG200) is electronically power regulated and offers a maximum power output of 200 kW (at a mains capacity of 240 kVA), which is infinitely variable between 2 and 100 percent. The connection between the generator and the directly compensated induction coil is realized via water cooled, flexible power cables. Operating frequency of the generator can be changed within a range of 50 to 200 kHz by adjusting the capacities at the induction coil.

5.1.1.2 Mechanical construction and refractory lining

The framework bearing the InduCarb-reactor was manufactured from austenitic stainless steel which was partially plated with copper. This was necessary in order to avoid an inductive heating of the metal parts by stray fields originating from the coil. To protect the metal construction against the heat, generated in the coke bed, the InduCarb-reactor itself was placed upon a refractory block, lying on the framework (see figure 7).

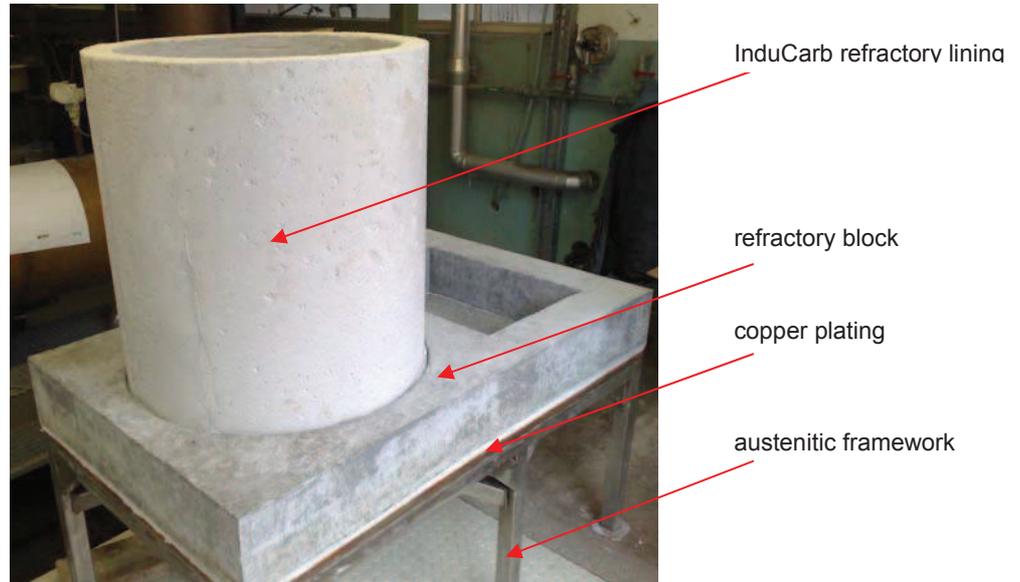


figure 7: mechanical framework with refractory block and InduCarb refractory-lining

In order to be able to quickly remove (hot) coke from the InduCarb reactor, a pneumatically operating bottom discharge unit, as displayed in figure 8, was integrated into the construction. This was especially important for accelerating the experimental operation, because otherwise it would have been necessary to manually remove the coke through the top opening of the InduCarb after each trial.



figure 8: InduCarb bottom discharge unit

The InduCarb-reactor itself had an inner diameter of 600 mm and a height of 800 mm, which corresponds to a volume of about 225 litres. Depending on charging level, type, humidity and grain size of coke this volume is equal to 85 and 150 kg of coke respectively.

To minimize heat losses during the tests it was tried to insulate the reactor as good as possible. This was realized by the use of bubble alumina for the InduCarb cylinder and additional fibre blankets for the insulation between the InduCarb cylinder and the induction coil. Those blankets have also been used for top (up to 5 blankets) and bottom (up to 3 blankets between coke bed and bottom discharging unit) insulation of the hot coke bed. All other refractory components were made of alumina rich, castable, refractory concrete.

5.1.1.3 Induction coil

The induction coil (see figure 9) was manufactured from hollow, water cooled, rectangular (40 x 15 mm) copper profiles. From the 12 turns of the whole coil always 4 turns were connected in parallel and these 3 parallel linked groups of turns were then connected in series (the coil had 3 x 4 turns). The coil itself was supported by a fibreglass construction.

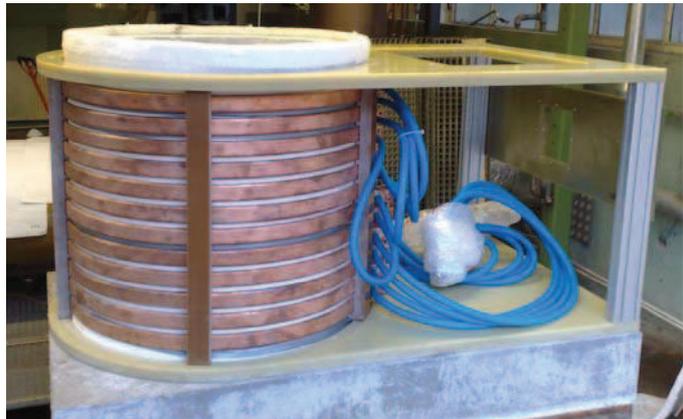
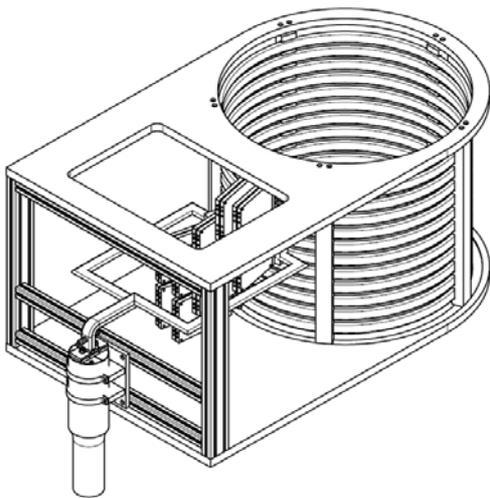


figure 9: construction of the induction coil

5.1.1.4 Heat sink – “cooling finger”

A cross section of the heat sink can be found in figure 5 – it is essentially a water cooled austenitic stainless steel cylinder that is reveted with a protective refractory layer (outer diameter: 116 mm) and placed axially in the centre of the coke bed. The idea behind the cooling finger is to simulate an energy consuming process (e.g. smelting of dust, energy for reduction of Fe, Ni, Cr, . . .) within the coke bed. This in turn gives an indication which temperature gradients can be expected during a continuous dust-processing and whether or

to which extent energy is transferred from the outside margin of the coke bed (where it is expected to mainly be induced) to the centre.

Depending on coke bed temperature the heat sink is able to remove between 10 and 15 kWh of energy, which corresponds to a throughput between 11 and 17 kg of dust per hour (energy for smelting and reduction of the dust).

5.1.2 Instrumentation

The instrumentation associated with the testing plant was a rather troublesome issue, since sensor signals were subjected to severe interferences originating from the strong, high frequency electromagnetic fields of the induction coil. A measurement of reliable signals was just possible if the voltage signals of the sensors were transformed to less sensitive 0/4 – 20 mA signals by special transducers. Sometimes even this did not help and there was a significant and visible influence of induction (and the associated high frequency, electromagnetic fields) on the signals. To solve this problem and ensure signals, that are not sensitive to any electromagnetic disturbances, it is recommended (for a possible, future pilot plant) to only use optical signal transmissions in fibre optical cables.

The instrumentation of the pilot plant comprises the following sensors:

- 1 flow rate measuring device for the cooling water of the generator
- 1 flow rate measuring device for the cooling water of the induction coil
- 1 flow rate measuring device for the cooling water of the cooling finger
- 1 Pt100 temperature sensor for the cooling water of the generator
- 1 Pt100 temperature sensor for the cooling water of the induction coil
- 1 Pt100 temperature sensor for the cooling water of the cooling finger
- 1 Pt100 temperature sensor for the returning cooling water from generator, induction coil and cooling finger
- 1 pressure sensor in the cooling water pipe of the cooling finger (in order to detect a possible overheating or leakage in the cooling finger)
- 4 Typ B thermocouples for measuring coke bed temperatures
- 1 CO-sensor (for safety reasons, to detect a possible CO development)
- a signal informing about the actual power output of the generator

All these sensor signals are collected by a SPS and displayed as well as stored on a computer close to the testing plant.

Special emphasis should be placed on the temperature measurement in the coke bed, which was realized with 3 to 4 (depending on the trials) type B (Pt/PtRh) thermocouples. In order to avoid inductive coupling of the thermocouple wires and especially of the thermo-pearl itself, the maximum wire-diameter was limited to 0.35 mm. In this context it is important to know, that with increasing frequency the penetration depth of induction drops and as a consequence even very small and thin metallic particles (e.g. thermocouple wires) start to couple and become inductively heated. This of course has to be avoided, since it would cause completely wrong temperature measurements and immediate smelting of the thermocouples.

It has to be outlined, that a coke bed temperature measurement with thermocouples is just limited to these preliminary tests. In trials where dusts are smelted and slags are formed in the reactor, thermocouples would suffer from immediate chemical corrosion and become destroyed. The only viable temperature measurement method in this case is a pyrometer, which has the big disadvantage of just being able to measure surface temperatures - a look "into" the coke bed is not possible anymore. Therefore temperature measurement and the linked process and power control will be a challenging task for further tests as well as for an industrial prototype of the InduCarb.

5.1.3 Coke types

As already mentioned, one main focus of this test series is to find out if and to which extent a different coke grain size requires changes in induction frequency, so that an optimal heating behaviour of the coke bed is guaranteed. To check this, blast furnace coke in 3 different grain size classes:

type 1: 10 – 20 mm

type 2: 20 – 40 mm

type 3: 40 – 60 mm

was ordered for the trials. The choice for the use of blast furnace coke fell in investigations in the run-up to this test series, where among several investigated coke types blast furnace coke turned out to have the best properties for a use in the InduCarb. The listed grain size classes were selected with respect to procedural requirements.

5.2 Testing procedure & results

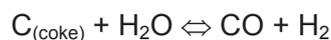
The aims of these trials were already discussed in the introduction to chapter 5 and can be summarized as follows:

- characterization and modelling of induction heating on a coke bed (determination of penetration depth, quantification of heat transfer by thermal conductivity and radiation, estimation of electrical efficiency factor)
- determination of optimal induction frequency in dependence of coke grain size

In order to achieve these aims and to be able to draw correct and reliable conclusions the following trials, listed in table 5, were carried out. The listed trials are those, which were taken for interpretation purposes. Preliminary test to check the instrumentation and to become acquainted with the testing plant are not included in the listing, also trials that had to be cancelled due to technical or other problems (and were not useful for an interpretation) are not listed. As table 5 shows, the 15 trials can be divided into 3 groups:

5.2.1 Trials of group 1

The tests of group 1 had the aim to optimize the experimental procedures as well as to find a reasonable testing procedure for the following trials. Apart from this, the coke needed for all further experiments was dried during these tests. This became necessary as a consequence of the high water content of the coke samples (up to 20 weight-%). Due to the potential risk of a CO formation – according to the following reaction equation – the drying process had to be done with great care.



Especially since the equilibrium of the reaction noticeably shifts to the right with increasing temperature a “slow” heating process was required. To avoid this time-consuming process in other tests, one proportion from each coke type was dried once and then used for all further trials.

5.2.1.1 Experimental procedure to trials of group 1

The 3 tests of group 1 all were done with heat sink and at a frequency of 105 kHz, but with different coke grain size classes. To better understand the testing procedure, it should be explained with the graph of trial number 3, shown in figure 10.

The 3 temperature graphs of the diagram show coke-bed temperatures at three different, horizontal positions. As figure 11 displays, T_1 is placed close to the heat sink with a distance of 21 cm from the outside margin of the coke bed. T_2 and T_3 are placed in a distance of 15 and 7.5 cm respectively from the margin. All thermocouples normally have the same depth of immersion into the coke bed and are positioned at the transition of the upper to the middle third of the induction coil – more exactly between the 4th and 5th turn of the coil (which has 12 turns). In addition to the temperature graphs, the diagram also shows the generator power on a second y-axis.

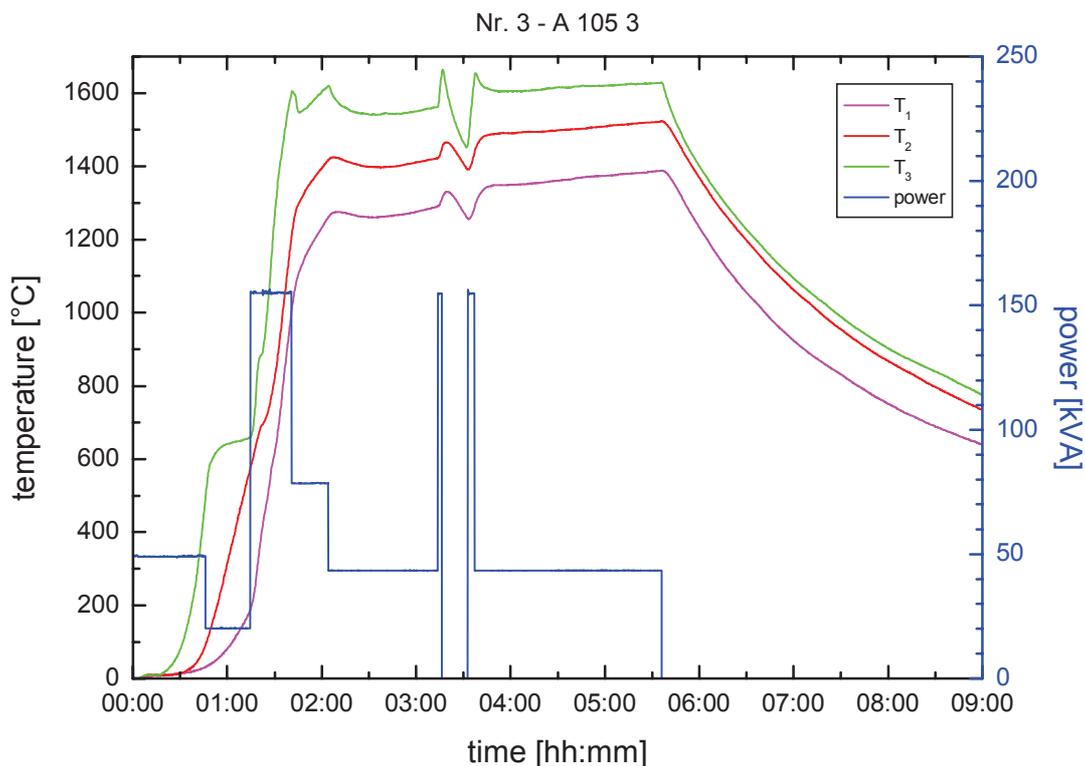


figure 10: time-temperature graph of trial nr. 3

According to the diagram in figure 10 and the testing conditions listed in table 5, trial nr. 3 started with preheating and drying of coke at powers of 25 and 10 % (50 and 20 kVA) respectively. The power reduction from 25 to 10 % was necessary in order to maintain temperature of the outer thermocouple (T_3) at about 600 °C and to improve temperature equalisation in the coke bed. When the drying process was completed, the power was increased to 80 % (160 kVA) and the coke bed heated up to process temperature. Within a time of about 23 minutes T_3 increased starting from 660 °C to 1600 °C (T_2 from 575 to 1205 °C, T_1 from 190 to 1000 °C). To avoid damage of both thermocouples and refractory lining the power was then reduced again to 40 % (80 kVA) for the next 23 minutes.

During this time T_3 first began to fall a little bit down to 1547 °C and then started to increase again up to 1620 °C. Unlike T_3 , T_2 and T_1 continuously increased, reaching temperatures of 1415 °C and 1260 °C respectively.

Although the temperature differences between the thermocouples decreased during this “equalization period” they are still as follows:

$$\begin{aligned} T_3 - T_2: & \quad 205 \text{ °C} \\ T_2 - T_1: & \quad 155 \text{ °C} \\ T_3 - T_1: & \quad 360 \text{ °C} \end{aligned}$$

After another lowering of power to 22 % for 01:09 hours, no significant changes in temperature distribution or temperature level could be identified. What then followed was a short heating period for 2 and a half minutes with 80 % of power. During this period T_3 rose from 1560 to 1650 °C (T_2 from 1422 to 1447 °C, T_1 from 1291 to 1310 °C).

A power cut off to cool down to 1450 °C at T_3 , followed by a short heating period (80 % power, 4 min) and a temperature holding period (22 %, 2 hours) completed this trial.

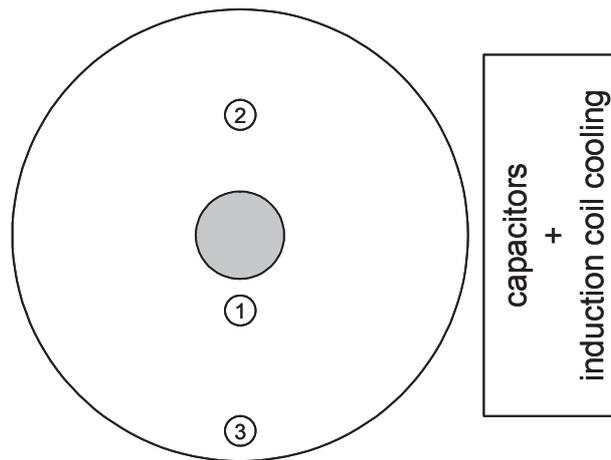


figure 11: schematic diagram to the positioning of the thermocouples in trials 1 – 11

5.2.1.2 Analysis and conclusions of trials from group 1

Characteristic for the measured time-temperature graphs are very fast heating periods, but the resulting temperature profiles show considerable radial temperature gradients with higher temperatures at the margin and lower temperatures towards the coke bed centre. A possible explanation for these gradients is a rather small penetration depth of induction, combined with low values for heat conduction and radiation within the coke bed. This would explain why considerably higher temperatures are found at the margin of the coke bed (where the energy is induced) and why this heat quantity (associated with the high temperatures) is not transferred into the centre of the bed (due to low heat transfer rates).

If the heat transport (by conduction or radiation) would be better, a more equal temperature distribution could be expected.

An important observation during trial number 2 was that the maximum generator power was limited to 60 % – above this value the generator showed an overvoltage alarm. Trial number 2 was the one, where the smallest coke grain size class (10 – 20 mm) was tested. This indicates that grain size has a strong effect on coupling behaviour of coke. It becomes obvious that with decreasing grain size, induction frequency has to increase in order to guarantee optimal heating conditions. Thinking on an InduCarb process operation it can be expected, that the coke grain size is continuously reduced during the process as a consequence of different reduction reactions. This fact has to be considered when selecting an optimal induction frequency.

5.2.2 Trials of group 2 & 3

The trials of group 2 are characterized by a systematic testing procedure that is very similar in all 8 trials. As an example for the testing procedure figure 12 shows the data of trial nr. 5 that ran as follows:

1. preheating of coke and thermocouples with 20 % of power
2. first heating period to target temperature 1600 °C with 70 % of power
3. temperature equalisation period with 25 % of power
4. power cut off – cooling phase (with T_3 target temperature 1400 °C)
5. heating period with 90 % of power (with T_3 target temperature 1600 °C)

Step 4 and 5 were then repeated with heating powers of 80, 70, 50 and finally again 90 % of power. Temperature T_3 was always oscillating between 1600 and 1400 °C during heating and cooling periods respectively.

6. power cut off and cooling down to room temperature

During the 8 listed trials (nr. 4 – nr. 11) 3 coke grain size classes (10 – 20 mm, 20 – 40 mm, 40 – 60 mm) were systematically tested with three different frequencies (75, 105, 150 kHz). It was expected from these experiments to identify differences regarding heating behaviour in dependence of coke grain size class and frequency as well as to find an optimal combination of frequency and coke grain size.

The horizontal and vertical thermocouple positioning was the same as during the group 1 trials. The only exception is test number 11, where an additional thermocouple was placed at the outer coke bed margin at the same position as T_3 , but displaced for 180 °.

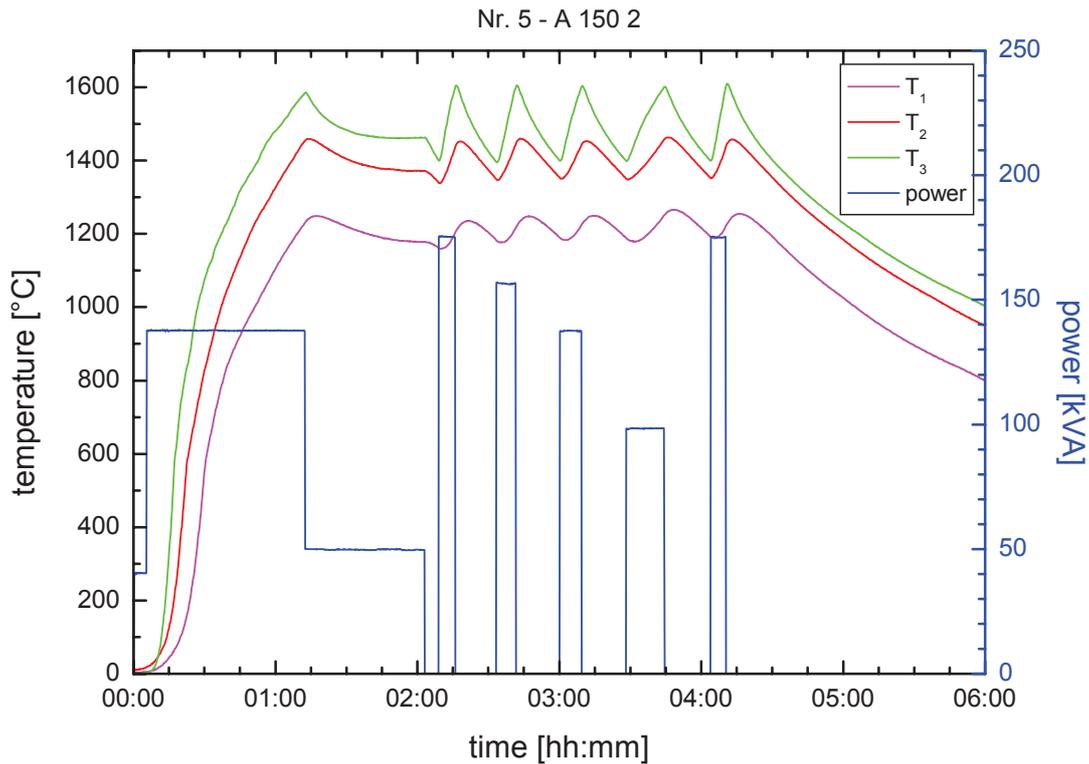


figure 12: time-temperature graph of trial nr. 5

The trials of group 3 had two aims:

- assessment of possible vertical temperature distribution in the coke bed
- characterization of horizontal and vertical temperature distribution in case that no energy is removed from the coke bed (no “cooling finger” in operation)

Unfortunately the removal of the heat sink was linked to problems with the thermocouple signals that suddenly showed major interferences, when induction was turned on. Despite several attempts to suppress these interferences, it was not possible to get reliable and unaffected signals. Since a solution to these problems would have meant a complete (cost and time intensive) redesign of the thermocouple measurement setup it was decided to cancel these trials. This decision was also supported by the results of the preceding trials that delivered sufficient information to draw the necessary conclusions for the future project progress.

In trials 13 to 15 the heat sink was removed and instead of it a 4th thermocouple was placed in the centre of the coke bed (see figure 13). The vertical position of the thermocouples varied – in trial 13 they were placed in the normal position between 4th and 5th turn, in trial 14 all thermocouples were placed 20 cm deeper in the coke bed between turn 8 and 9 and in trial 15 $T_1 + T_3$ were again between turn 8 and 9, while $T_2 + T_4$ were placed between turn 6 and 7.

As already mentioned, these trials did not deliver the expected results regarding a possible vertical temperature distribution in the coke bed, since the thermocouple signals were subjected to major interferences.

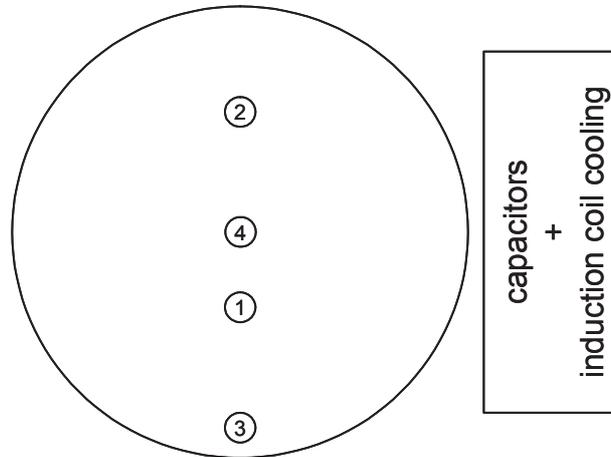


figure 13: schematic diagram to the positioning of the thermocouples in trials 13-15

Interesting results were found in trial nr. 12, where the heat sink was still in operation but 3 (T_{1-3}) of 4 thermocouples were positioned 10 cm higher in the coke bed than in trial nr. 11, which except from this had identical testing conditions to trial nr. 12. Especially the combination of these trials with temperature measurements at the refractory lining of the InduCarb reactor during trials 7, 8, 11 and 12 brought some interesting findings.

5.2.2.1 Analysis and conclusions of trials from group 2 & 3

One of the most important issues concerning an industrial InduCarb reactor is a homogenous temperature distribution in the coke bed. Unfortunately the trials clearly showed that even with moderate heat removal from the coke bed centre through the heat sink (10 – 15 kW) horizontal temperature gradients appear. Table 6 shows the measured temperature gradients from trial 4 to 11 (for the time-temperature graphs see chapter 6.2, “Graphs to trials of group 2”) at the beginning (min.) and at the end (max.) of the last heating period of each trial. The corresponding diagrams to the data in table 6 are displayed in figure 14 and figure 15. The graphs show the following tendencies:

- Independent of frequency the smallest coke grain size class 1 shows considerably larger temperature differences than coke grain size class 2 and 3, which just marginally differ.
- During heating periods temperature differences between the outer (T_3) and inner thermocouple (T_1) of about 350 °C are typical. Considering that the distance between T_1 and T_3 is just 15 cm, these gradients indicate an inhomogeneous temperature distribution.

- The temperature difference between T_2 and T_1 is higher than between T_2 and T_3 .
- Different frequencies have only small influence on the measured temperature gradients. Nevertheless there is a slight tendency that with lower frequency also the temperature gradients become smaller. A possible and logical explanation for this is an increased penetration depth of induction with lower frequency.
- During cooling periods all temperature differences clearly decrease, especially the difference between T_3 and T_2 is minimized to around 30 – 40 °C.

	coke 1		coke 2		coke 3	
	power	max.	min.	max.	min.	max.
150 kHz						
$T_2 - T_1$	245	283	160	215	144	153
$T_3 - T_2$	145	306	47	164	33	178
$T_3 - T_1$	390	589	207	379	177	331
105 kHz						
$T_2 - T_1$	-	-	163	226	139	212
$T_3 - T_2$	-	-	35	141	39	137
$T_3 - T_1$	-	-	198	367	178	349
75 kHz						
$T_2 - T_1$	245	292	152	214	123	188
$T_3 - T_2$	161	310	29	114	44	122
$T_3 - T_1$	406	602	181	328	167	310

table 6: temperature differences before and after the last heating period of each trial

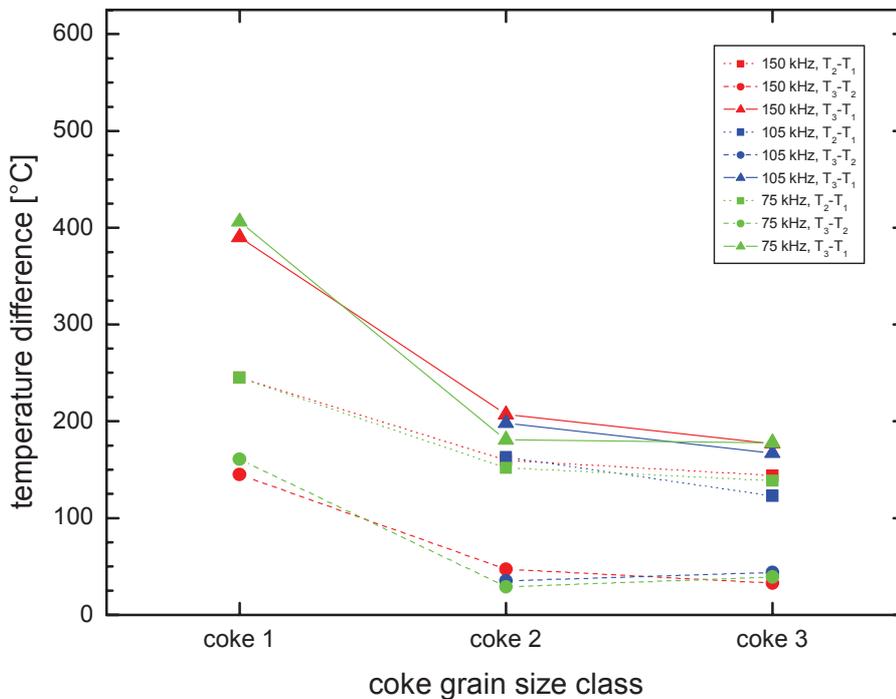


figure 14: temperature differences after cooling period

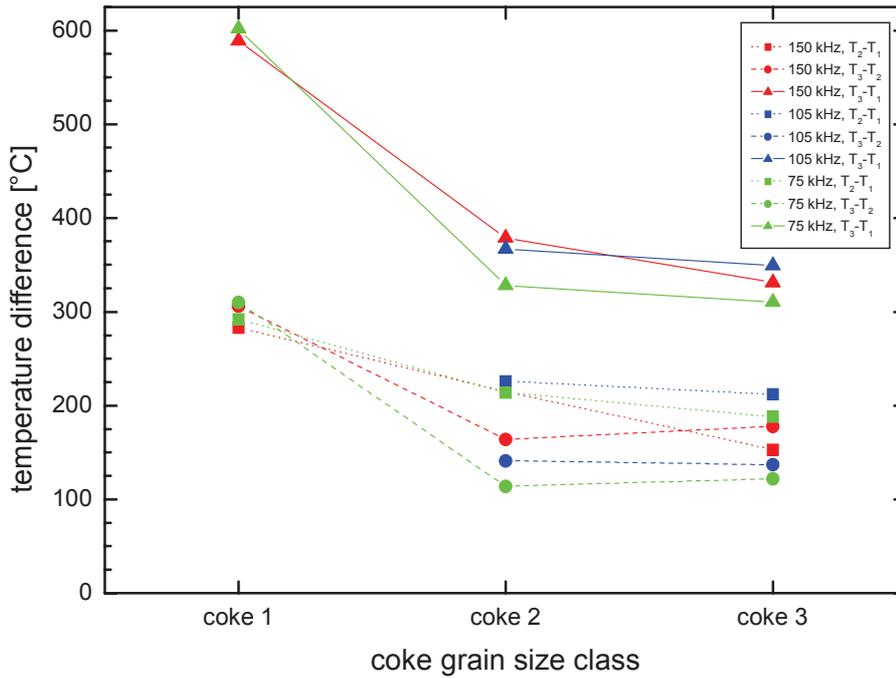


figure 15: temperature differences after heating period

Although frequency seems to have only a small influence on the measured temperature differences, there is strong effect regarding the coupling behaviour. In all trials with a frequency of 75 kHz the maximum power output of the generator was limited to 70 % since above this value an overvoltage alarm occurred. The same problem occurred during the group 1 trials when the small coke grain size class (coke type 1) was tested with a frequency of 105 kHz. This is also the reason why coke grain size class 1 was not tested with a frequency of 105 kHz during the group 2 trials. The other coke grain size classes (2 + 3) didn't show any problems at 105 kHz – the same is for a frequency of 150 kHz where even coke grain size class 1 worked well.

Except from the horizontal temperature distribution in the coke bed also the vertical temperature distribution is of certain interest. Unfortunately the trials of group 3, where the characterization of the vertical temperature distribution was one aim, were cancelled due to problems with the thermocouple signals; therefore the corresponding data is very limited, especially regarding temperatures deeper in the coke bed. Nevertheless during the trials 7, 8, 11 and 12 temperature measurements at the outside of the InduCarb refractory lining (between the insulating fibre blanket and the bubble alumina refractory cylinder) were done. In order not to risk any interferences of the measured temperatures with the high frequent fields of the induction coil the temperature measurements were always done during the cooling phases (1600 → 1400 °C). For the measurement a type K thermocouple was pushed through the fibre blanket, so that the outside temperature of the bubble alumina cylinder

could be measured. Measuring points were above turn 1 (0. turn) between the space of turn 2 and 3 (2. turn), turn 4 and 5 (4. turn), turn 6 and 7 (6. turn), turn 8 and 9 (8. turn), turn 10 and 11 (10. turn) and below the last turn 12 (12. turn).

In figure 16 and figure 17 the results of the measured refractory temperatures during trial 11 and 12 are displayed. The trend lines in the diagrams show the area in which the refractory temperatures fluctuated. The points that lie outside the area (some red and black points) originate from the beginning phase of the test, where the refractory lining and the other plant components still have not reached their thermal equilibrium. Both graphs clearly show that there is a vertical temperature distribution in the coke bed. In trial 12 the gradients are more distinctive than in trial 11, but the tendency is always the same: At the beginning of the test, the temperature differences between the “centre turns” (turn. 6) and the top (0. turn) and bottom (12. turn) turns are higher than at the end of the test. In addition to this the general temperature level increases with proceeding testing time.

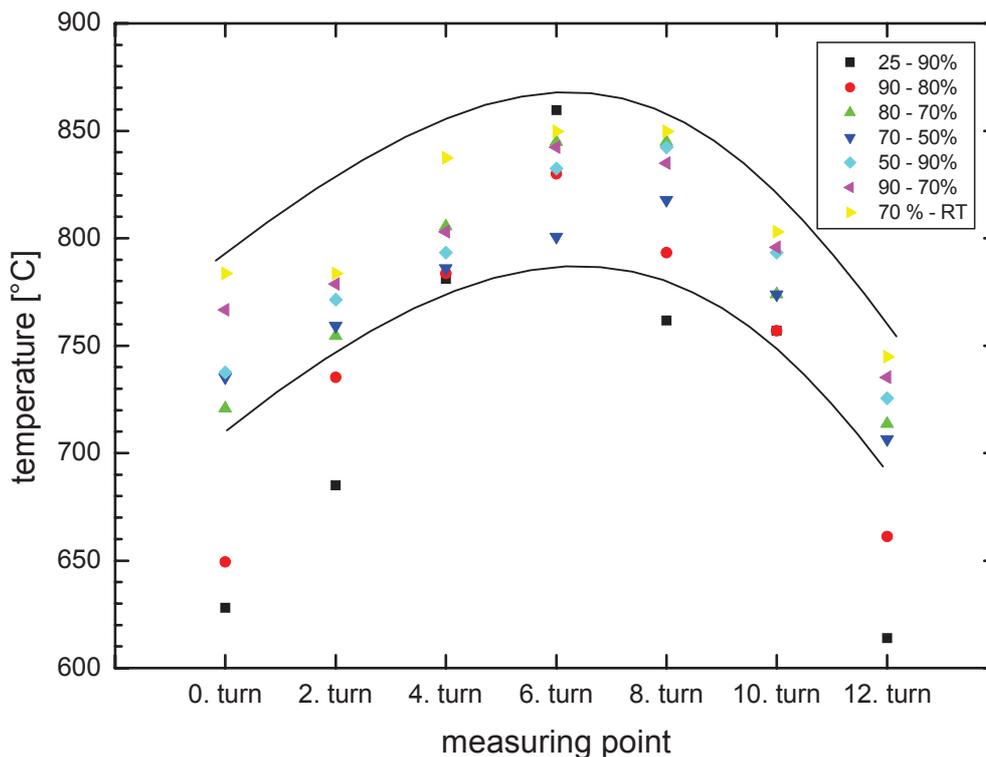


figure 16: refractory temperatures measured during cooling phases of trial 11

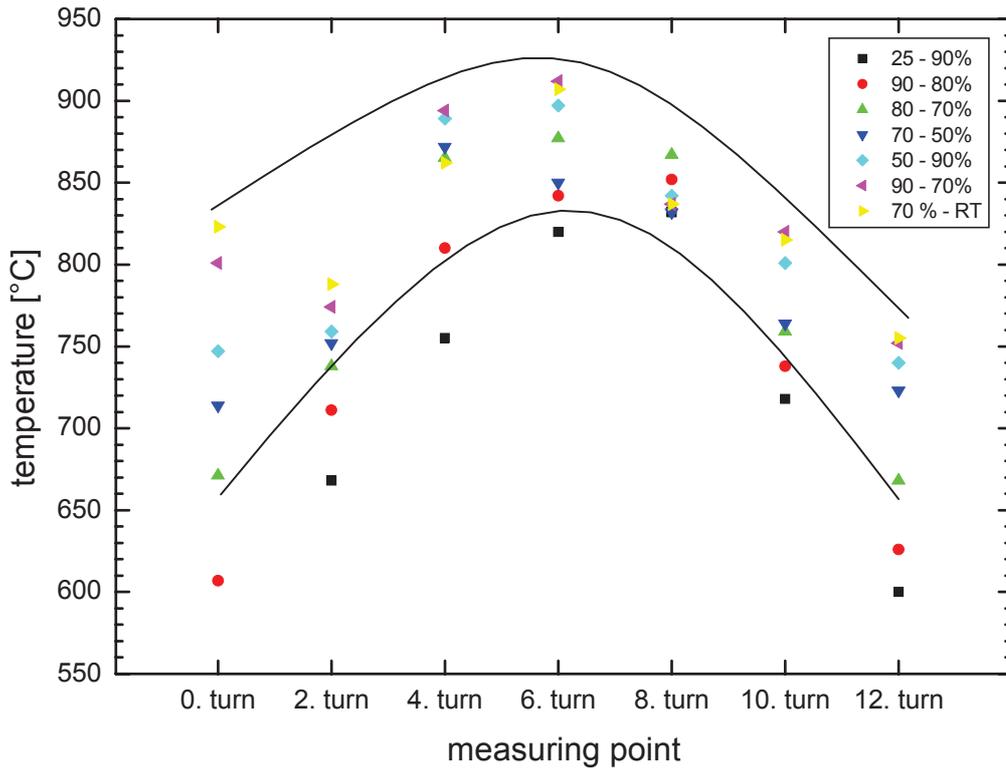


figure 17: refractory temperatures measured during cooling phases of trial 12

A comparison of the time-temperature graphs of trial 11 and 12 (see figure 18) draws a similar picture. As already mentioned, both trials had completely identical testing conditions – the only difference was that in trial 12 thermocouple 1, 2 and 3 were positioned 10 cm higher in the coke bed than in trial 11. To be able to compare both tests, in trial 12 T_4 remained in the same position as in trial 11 (between turn 4 and 5) and the test was repeated according to the temperatures measured by T_4 .

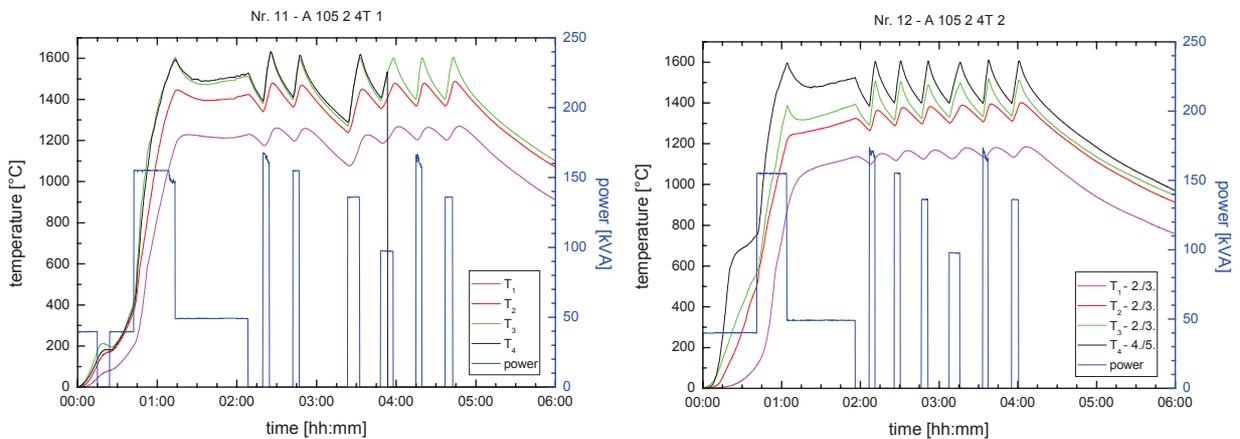


figure 18: comparison of time-temperature graphs from trail 11 + 12

From the left graph of figure 18 it can be seen that the 10 cm vertical displacement of T_1 , T_2 and T_3 caused a constant temperature difference of about 100 °C at all three thermocouples. These measurements combined with the refractory temperature measurements confirm the assumption that the energy input by induction is stronger in the centre region of the induction coil than at the top and bottom of it. Of course also heat losses through the top and bottom of the reactor contribute to these temperature gradients, but especially the higher gradients at the beginning phase of the tests (the black and red points in figure 16 and figure 17), where the reactor isn't in thermal equilibrium already, indicate that an inhomogeneous power input by induction mainly causes the resulting, vertical temperature profile. However, in view of an industrial InduCarb operation it can be expected that a melt and gas flow in the reactor will balance these differences in large part. In this context it should be outlined again, that the performed preliminary trials were done without any melt or gas flow in the coke bed. Therefore it is possible that in presence of a slag as well as a (electroconductive) metal flow in the reactor the heating behaviour of the reactor changes. However it can be concluded, that the energy input will always be limited to the marginal zone of the reactor (as in any other induction heating process), but the heat transfer from this marginal zone to the centre can possibly be improved by gas and melt flow in the coke bed. To gain certainty about that, further trials are absolutely necessary.

5.3 Modelling of experimental data

In this part mainly the results of the radial and vertical modelling of the coke bed heating process are presented, since a full documentation of the mathematical would go beyond the scope of this report. The mathematical model was developed by Dr. Hans Musch from "dynamic systems AG", Zurich. He also did the simulations and provided the simulation results to trials 6, 7, and 9 that are presented in the following:

The graphs in figure 19, figure 21 and figure 23 show a comparison between the measured and modelled time-temperature graphs of trial nr. 6, 7 and 9 respectively. The red lines represent the modelled data while the blue lines show the measured graphs. Except from the lower temperature region during the heating up period, the measured and modelled data show a good correlation. A main conclusion from the modelling is the penetration depth of induction into the coke bed. In figure 20, figure 22 and figure 24 the horizontal and vertical distribution of the power input by induction into the coke bed is graphically displayed (one segment corresponds to about 27 mm). These pictures show the following tendencies that have also been found in similar form in chapter 5.2.2.1:

- the power input is limited to a marginal zone
- power input also slightly varies vertically – in the (vertical) centre region of the coil the power input is higher than at the top and bottom of it

- with increasing coke grain size penetration depth increases (trial nr. 7 vs. 9)
- with increasing frequency penetration depth decreases (trial nr. 6 vs. 7)

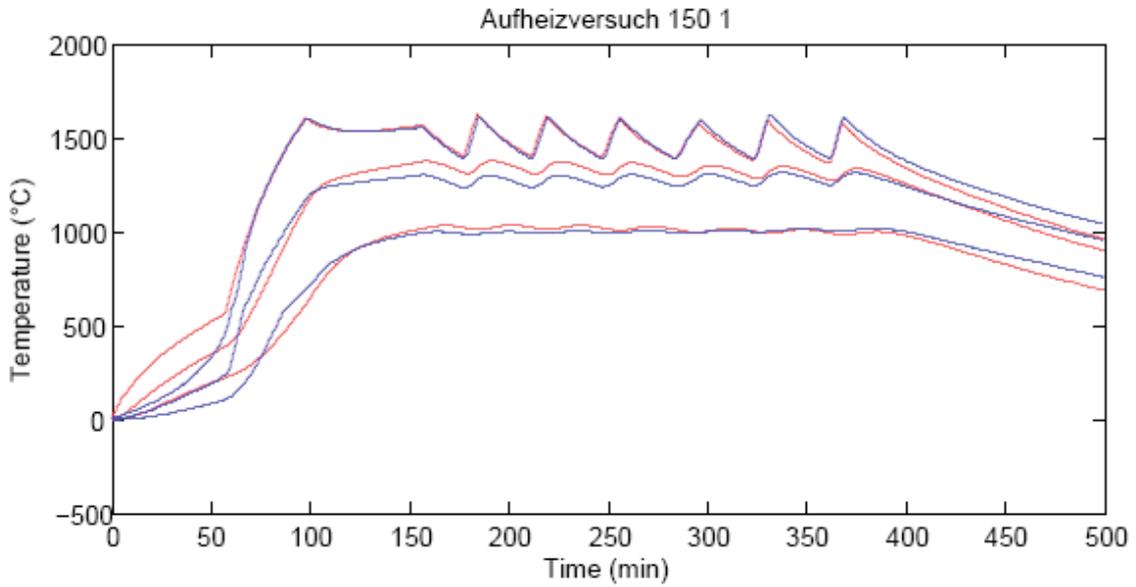


figure 19: comparison of measured (blue) and modelled (red) time-temperature graphs to trial nr. 6

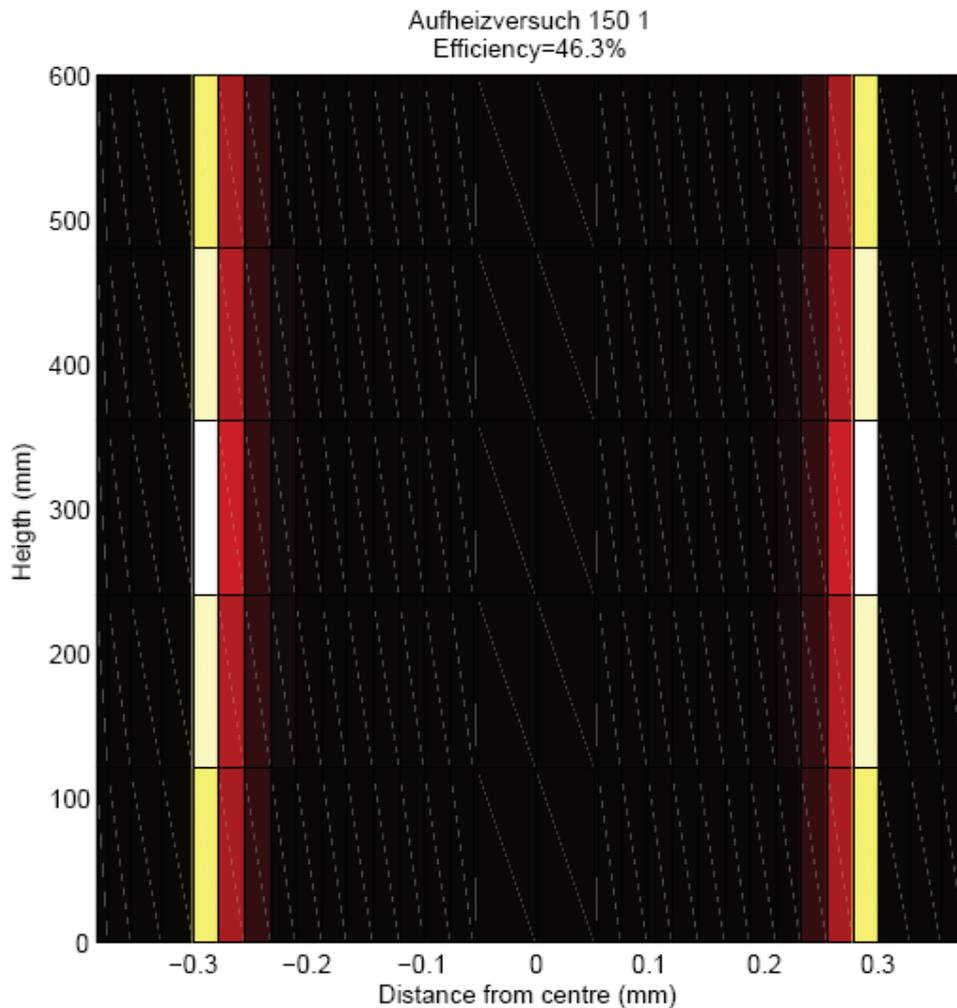


figure 20: graphic display of horizontal and vertical power input into the coke bed for trial nr.

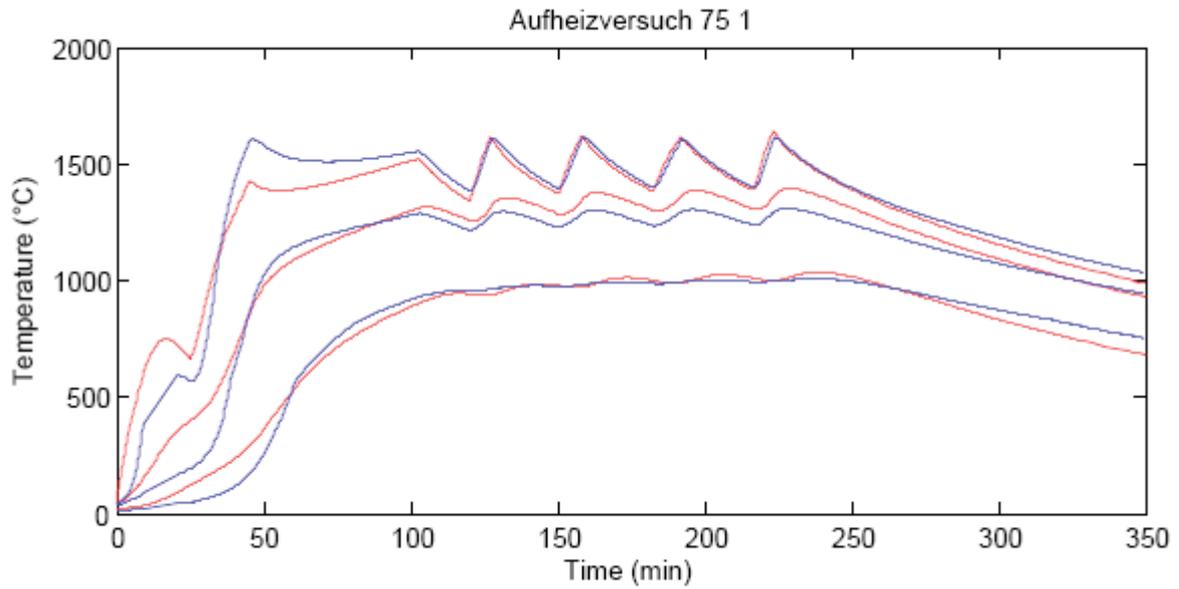


figure 21: comparison of measured (blue) and modelled (red) time-temperature graphs to trial nr. 7

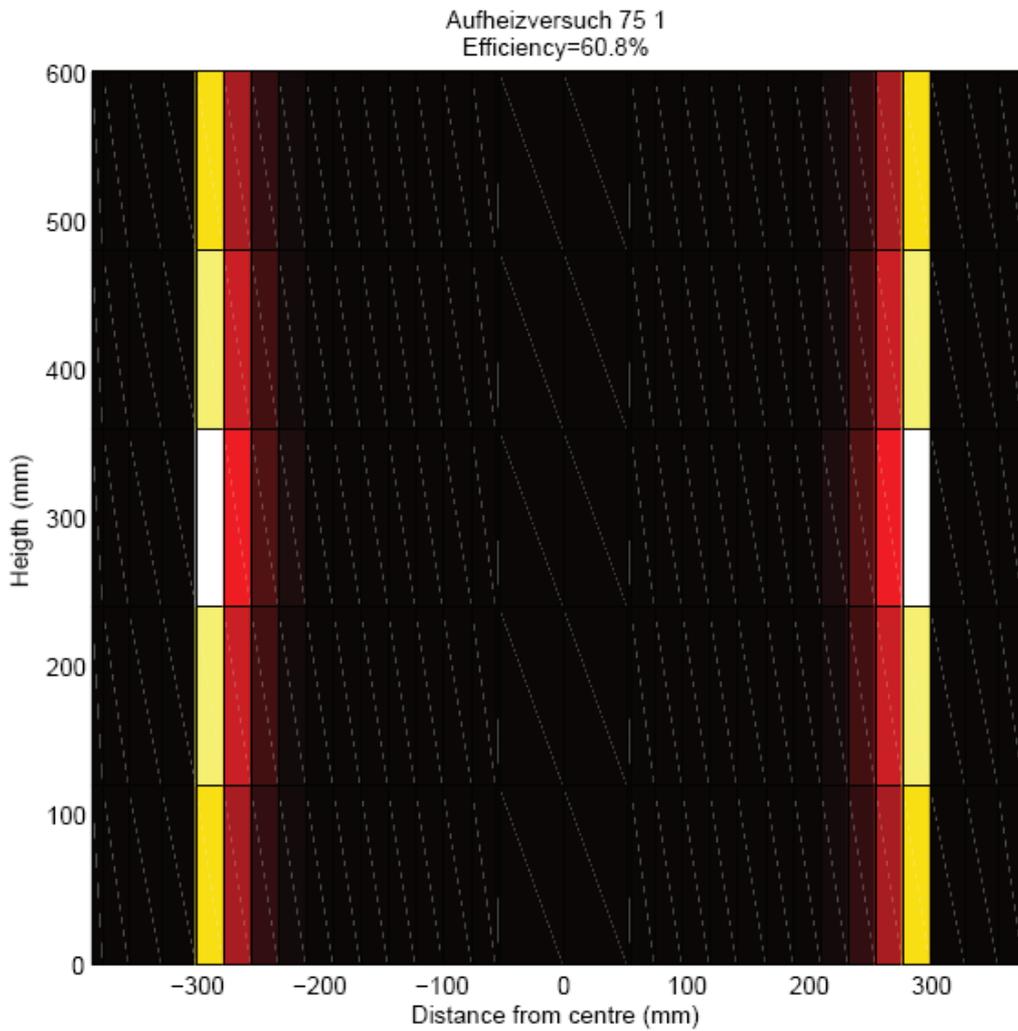


figure 22: graphic display of horizontal and vertical power input into the coke bed for trial nr. 7

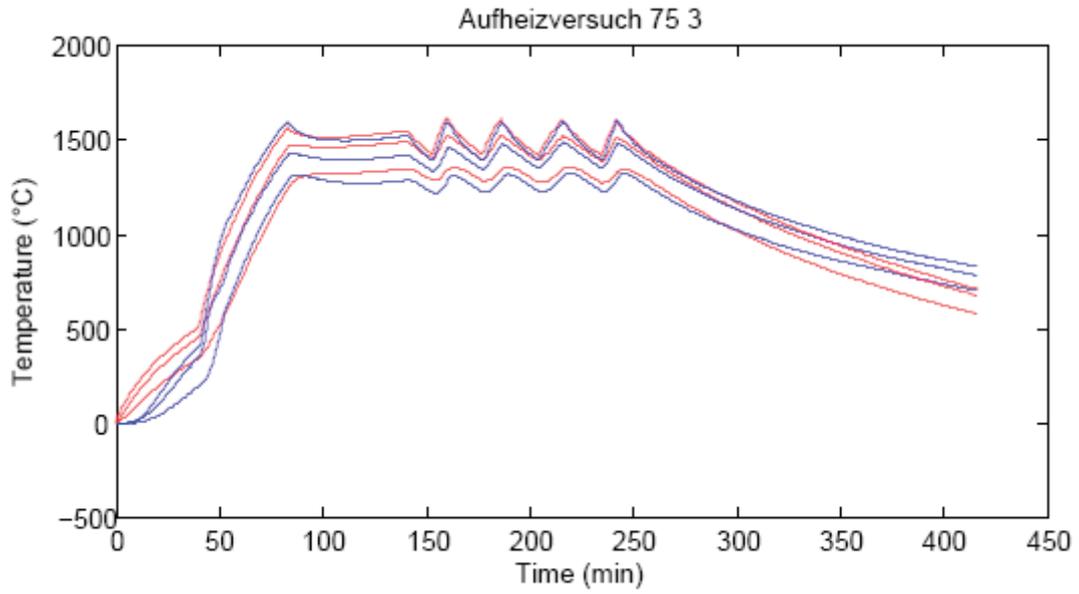


figure 23: comparison of measured (blue) and modelled (red) time-temperature graphs to trial nr. 9

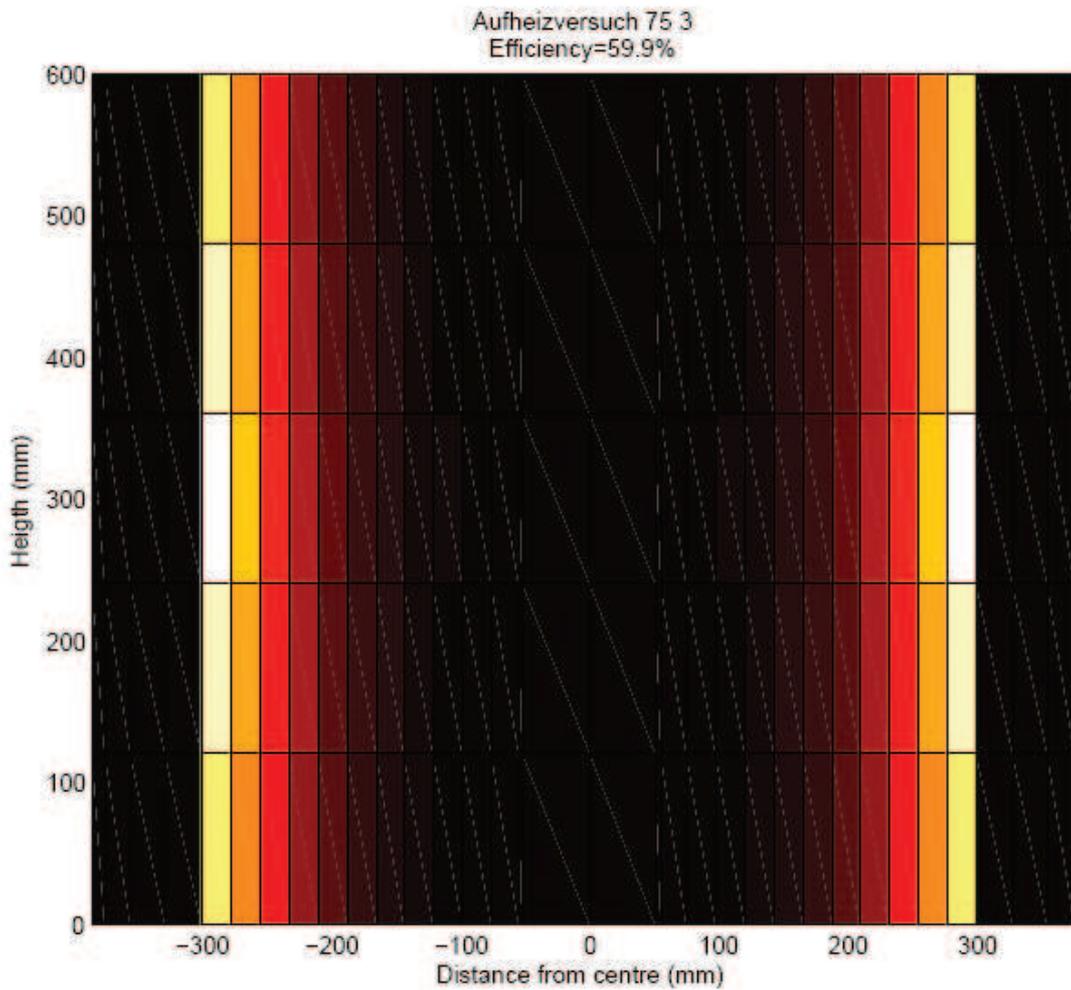


figure 24: graphic display of horizontal and vertical power input into the coke bed for trial nr. 9

The pictures also show the calculated efficiency factor for the electrical energy of the InduCarb. This factor indicates how much of the consumed electrical energy is converted into thermal energy in the coke bed. In the modelled trials the calculated electrical efficiency ranges from 46 to 61 %.

For the modelled trials the calculated penetration depths (graphically displayed in figure 20, figure 22 and figure 24) are also listed in table 7. To get an idea which energy input distribution is linked to a certain penetration depth, figure 25 shows this distribution for 4 different penetration depths (0.01 m, 0.02 m, 0.04 m and 0.06 m). To be able to compare these curves, the area under each curve is of course always the same.

trial nr.	06	07	08	09	10
	150 kHz coke 1	75 kHz coke 1	75 kHz coke 2	75 kHz coke 3	105 kHz coke 3
penetration depth [m]	0.028	0.033	0.046	0.068	0.071

table 7: calculated penetration depths of modelled trials

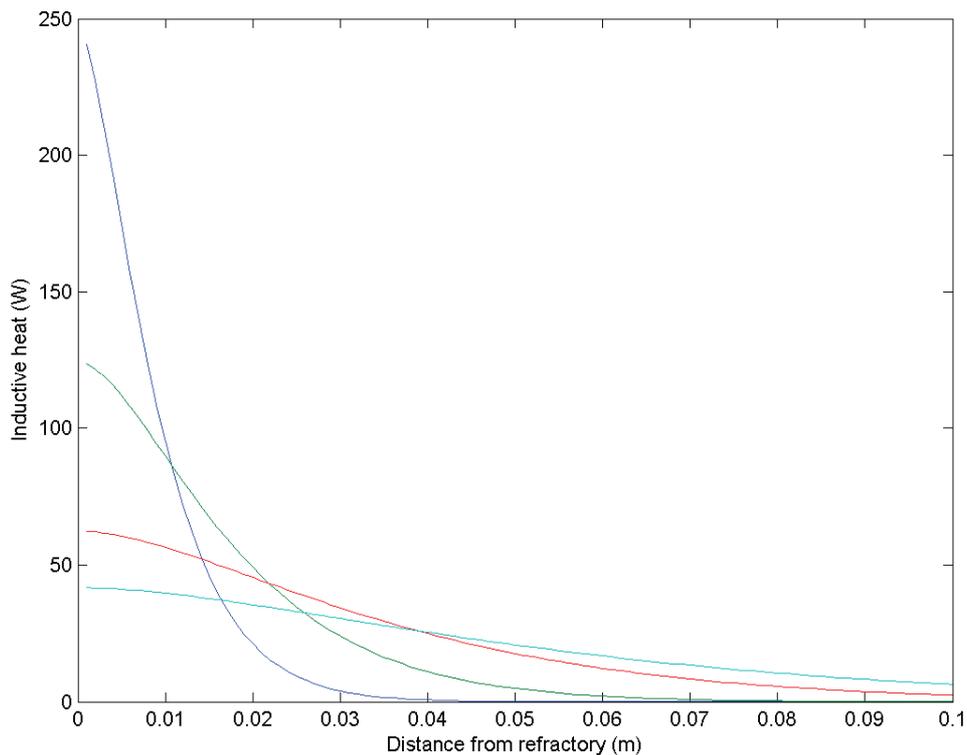


figure 25: graphic display of radial energy input for penetration depths of 0.01 m (blue), 0.02 m (green), 0.04 m (red) and 0.06 m (turquoise)

Another main question the model had to answer was how the considerable horizontal temperature gradients in the coke bed – from the margin to the heat sink – can be explained. Of course it is obvious that the heat removal by the “cooling finger” causes these gradients,

but with an improved heat transport within the coke bed (or a higher penetration depth of induction) the gradients should not be that high. The main conclusion from the modelling work to this question is that heat transport in the coke bed is characterized by a relatively low heat conductivity of the coke itself and that also radiation seems to be of minor importance in the bulk. Especially the low heat conductivity of coke is well documented in literature and also the explanation why in the coal coking process it takes about 20 hours to heat up a coke bed of a thickness of 400 mm to a temperature of about 1200 °C, although the coking chamber is heated from both sides. That at these high temperatures of around 1600 °C even radiation doesn't contribute to a more homogenous temperature distribution was surprising. The only chance to get a more homogenous temperature distribution within the coke bed seems to be the convection of a gas in the bulk, but this was neither modelled nor could it be tested in the present form of the pilot plant. Since also in classical shaft furnaces a major part of the heat transfer is attributed to this mechanism, this is certainly an important issue that would be tested in one of the first steps of a possible further testing series with a modified pilot plant setup.

6. Appendix

On the following pages all time-temperature graphs of the 15 trials listed in table 5 are displayed:

6.1 Graphs to trials of group 1

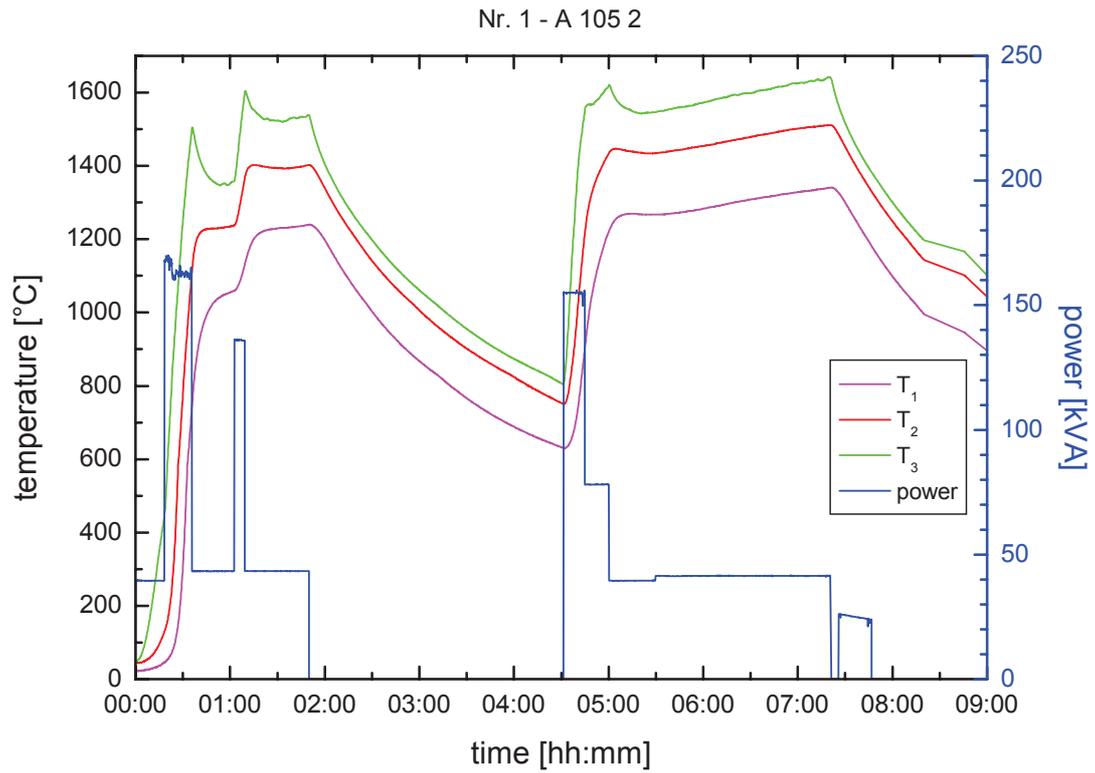


figure 26: time-temperature graph of trial nr. 1

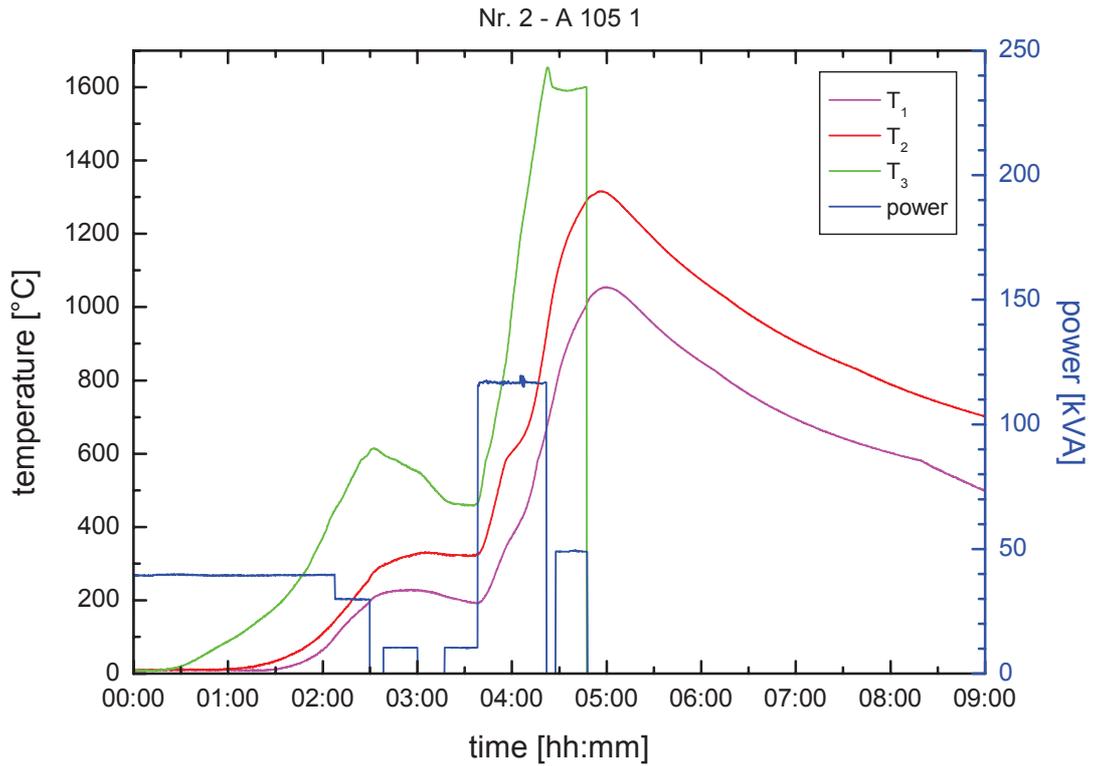


figure 27: time-temperature graph of trial nr. 2

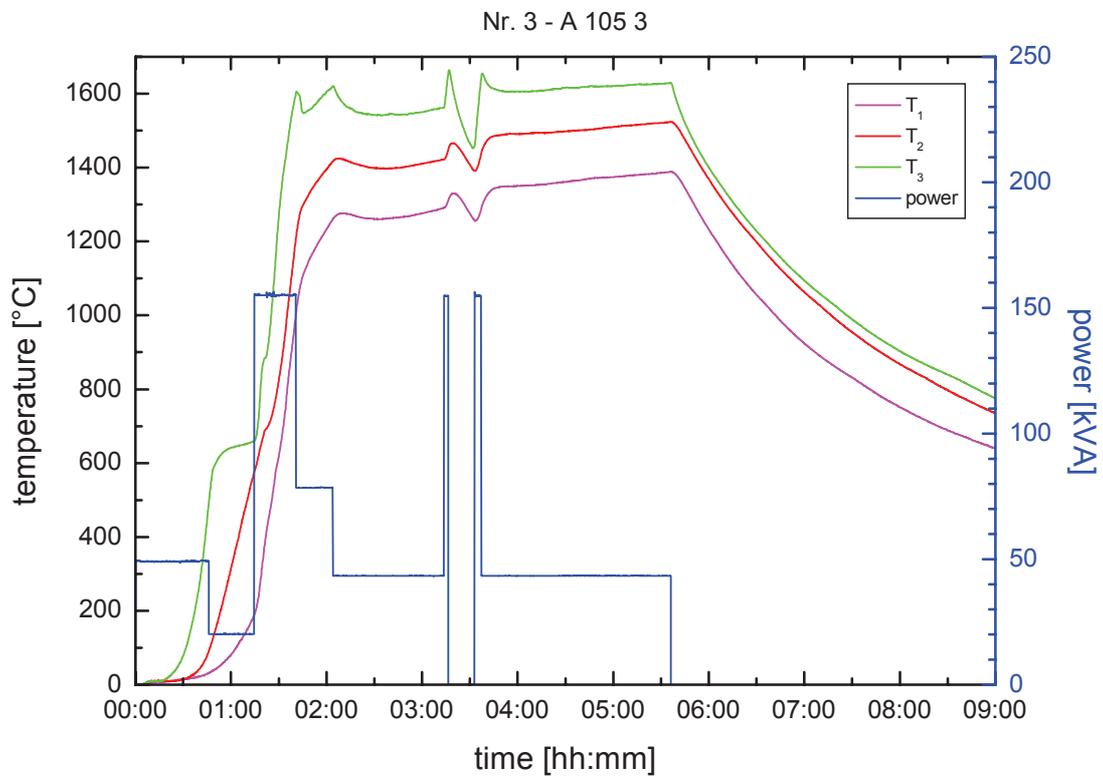


figure 28: time-temperature graph of trial nr. 3

6.2 Graphs to trials of group 2

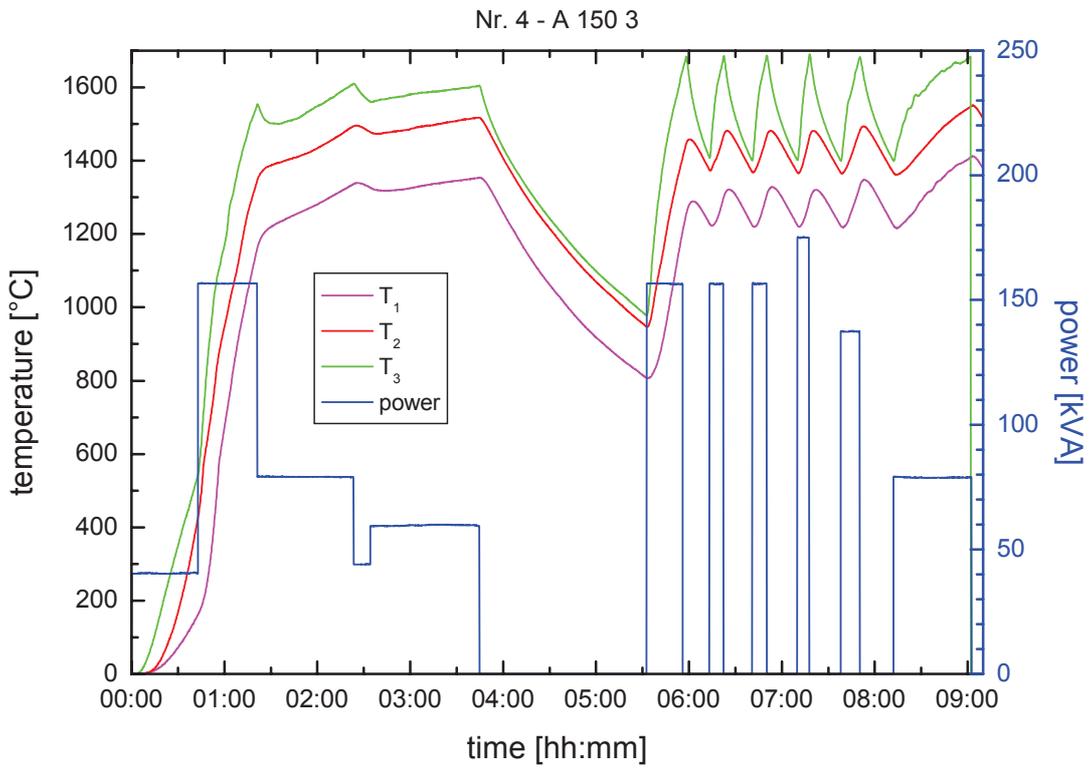


figure 29: time-temperature graph of trial nr. 4

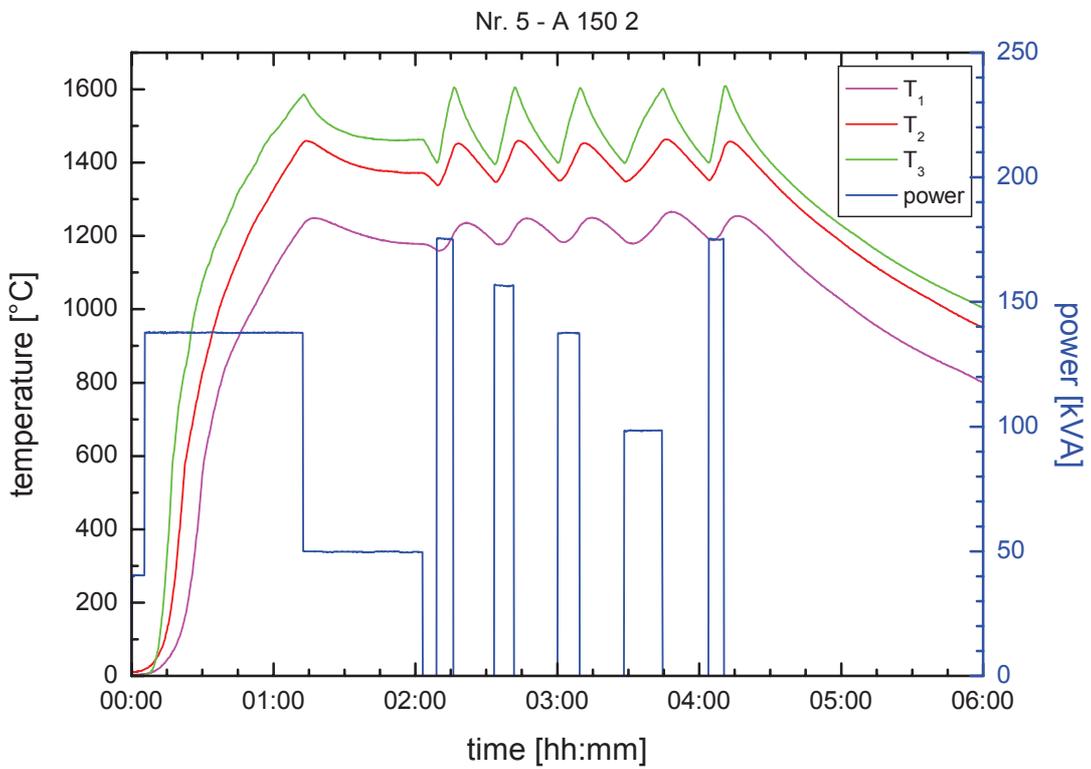


figure 30: time-temperature graph of trial nr. 5

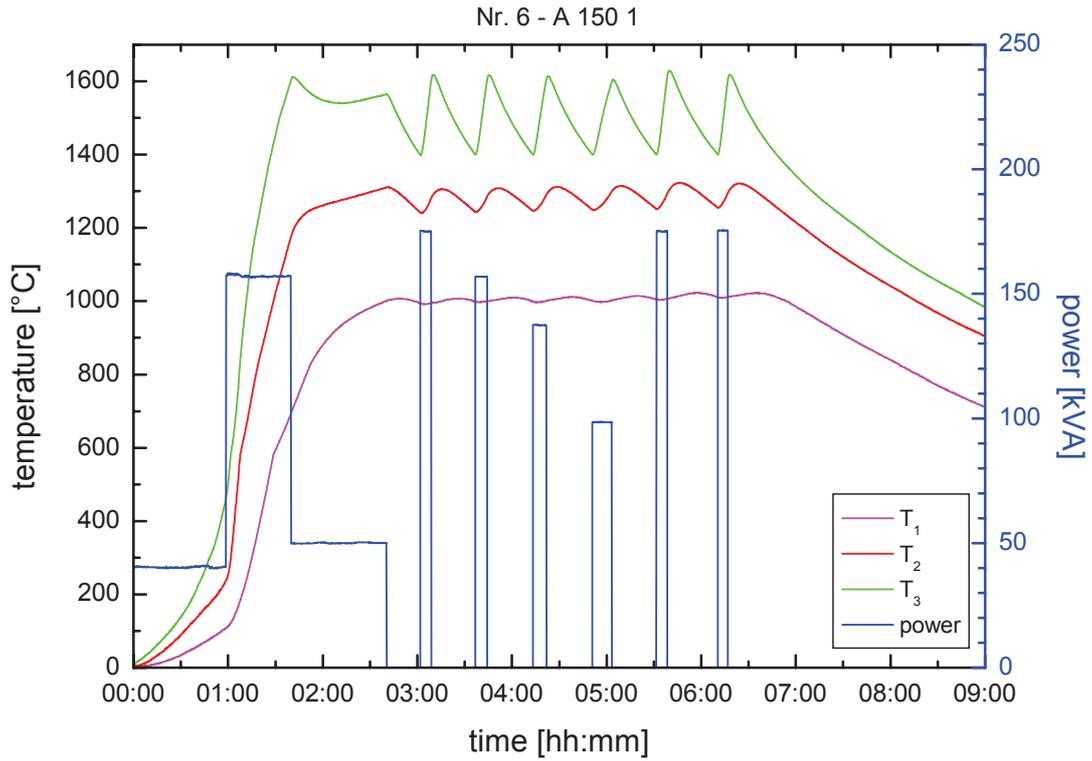


figure 31: time-temperature graph of trial nr. 6

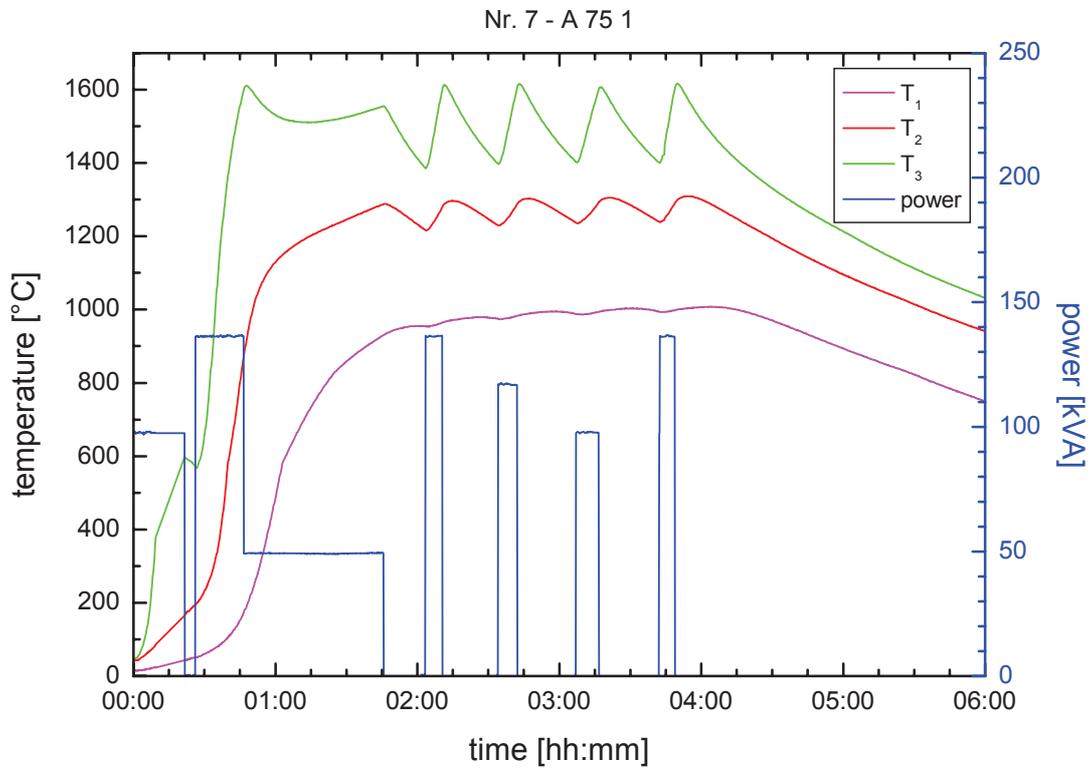


figure 32: time-temperature graph of trial nr. 7

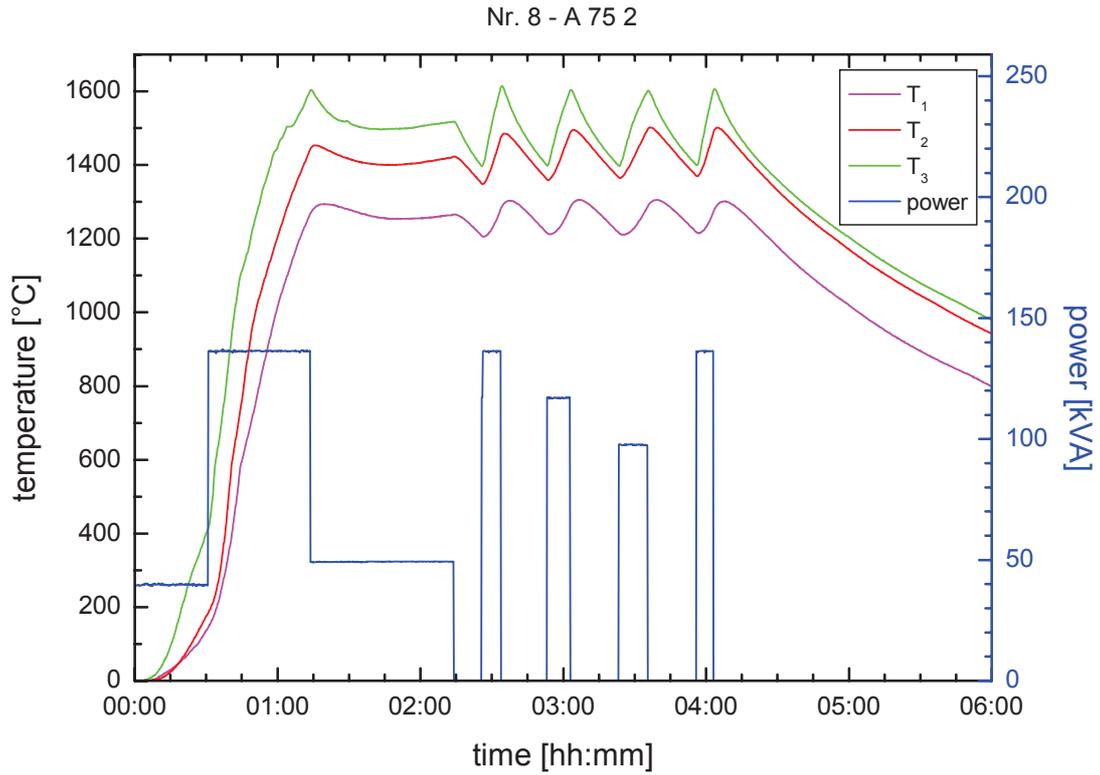


figure 33: time-temperature graph of trial nr. 8

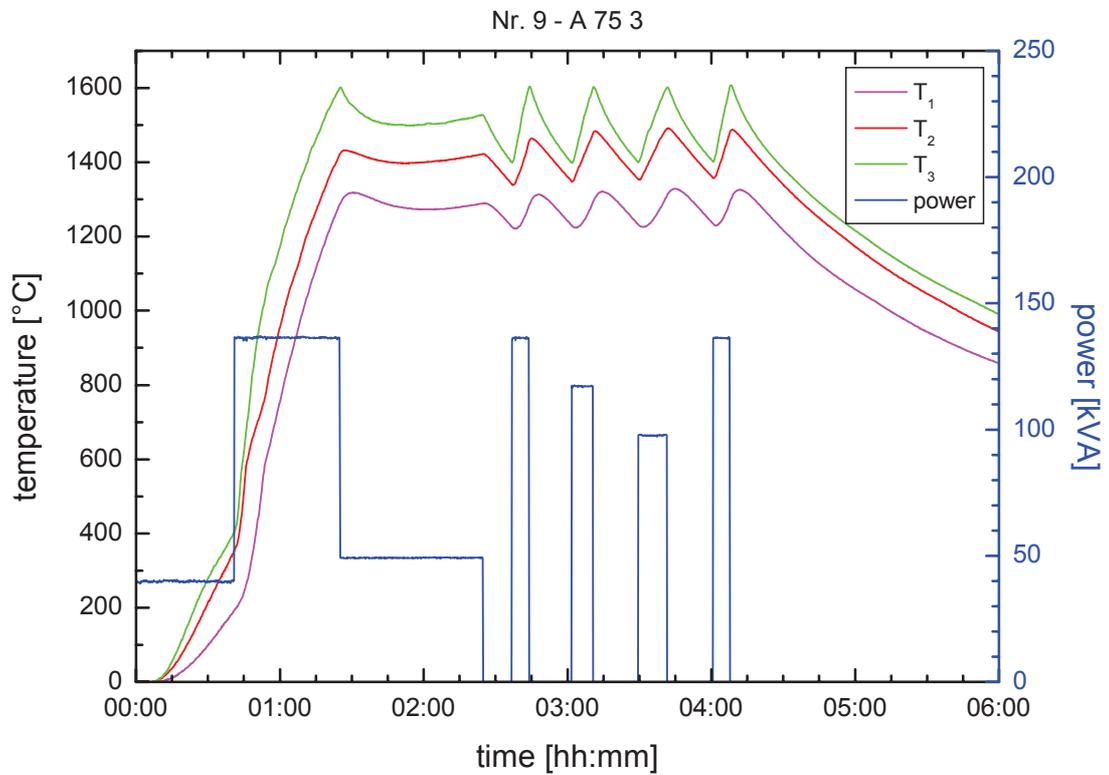


figure 34: time-temperature graph of trial nr. 9

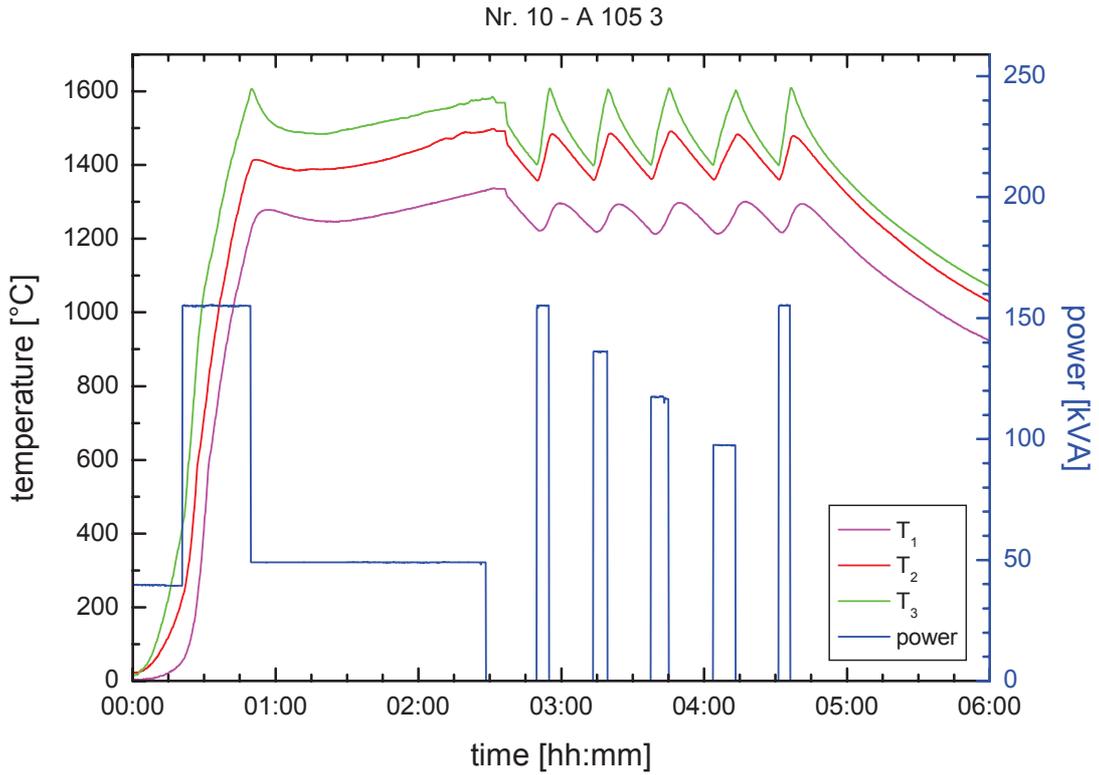


figure 35: time-temperature graph of trial nr. 10

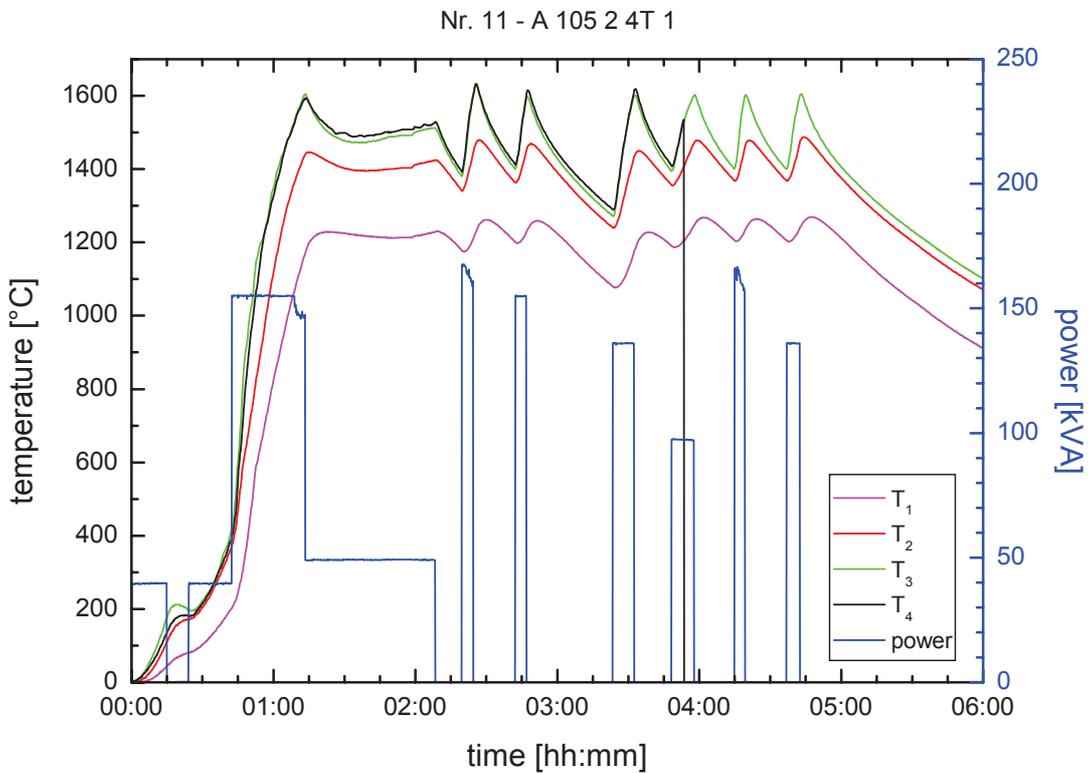


figure 36: time-temperature graph of trial nr. 11

6.3 Graphs to trials of group 3

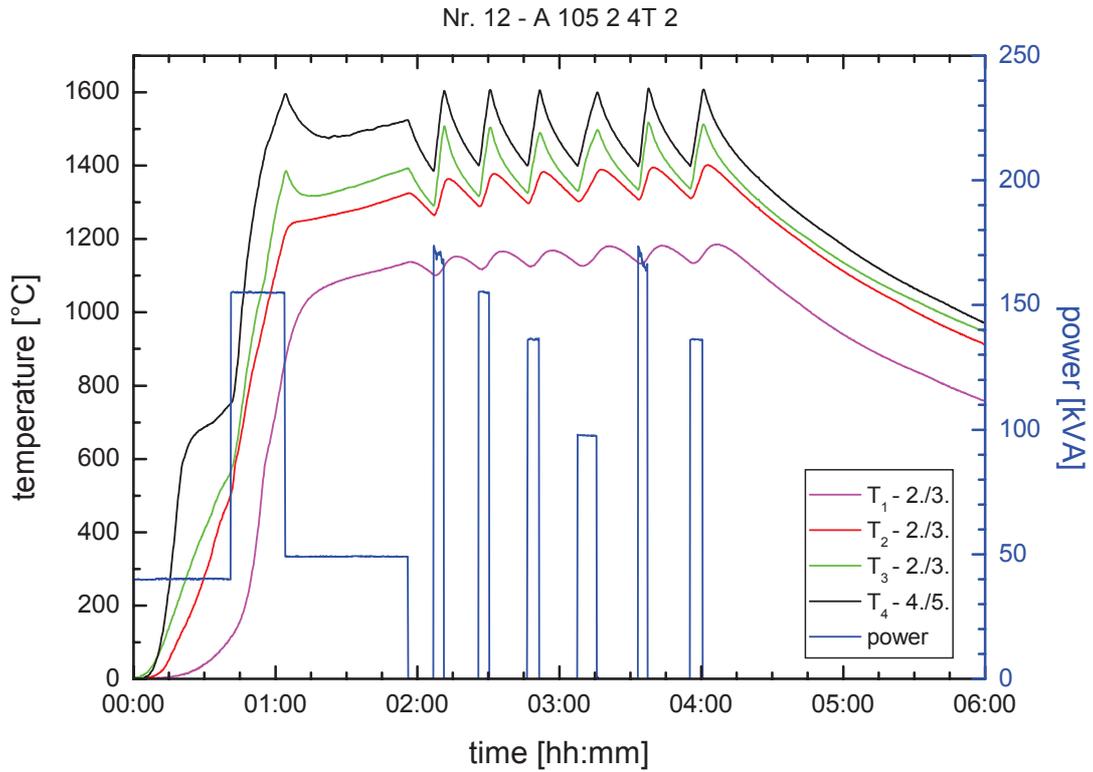


figure 37: time-temperature graph of trial nr. 12

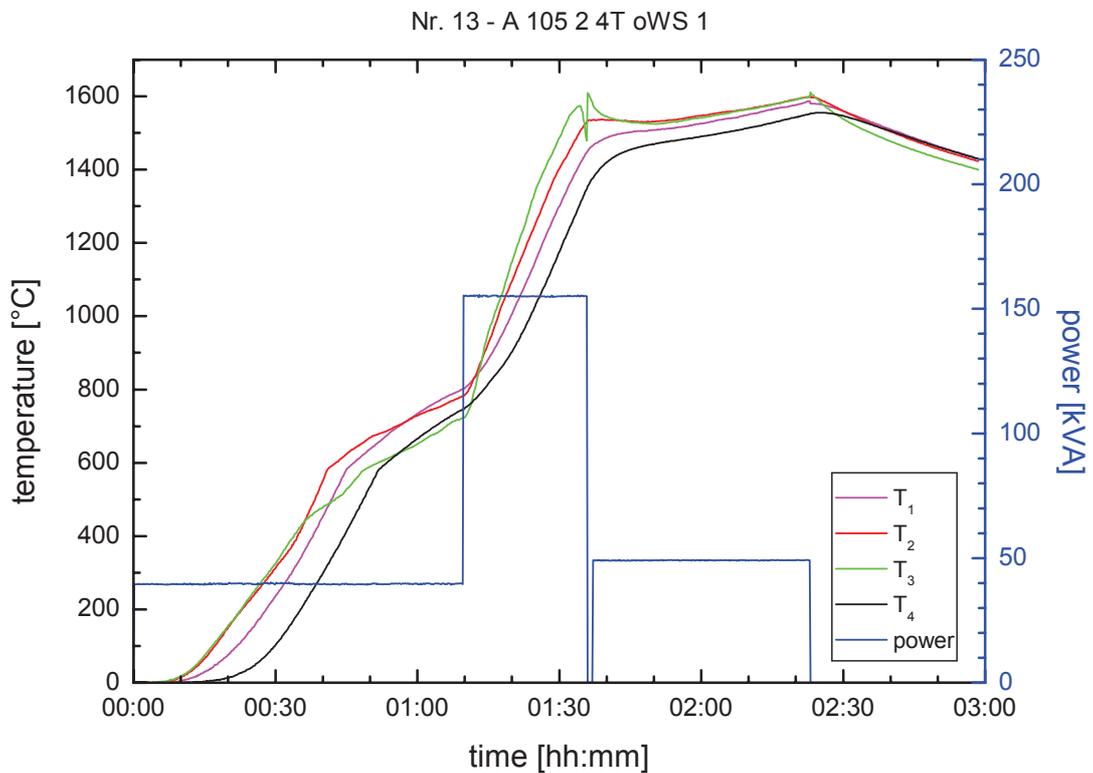


figure 38: time-temperature graph of trial nr. 13

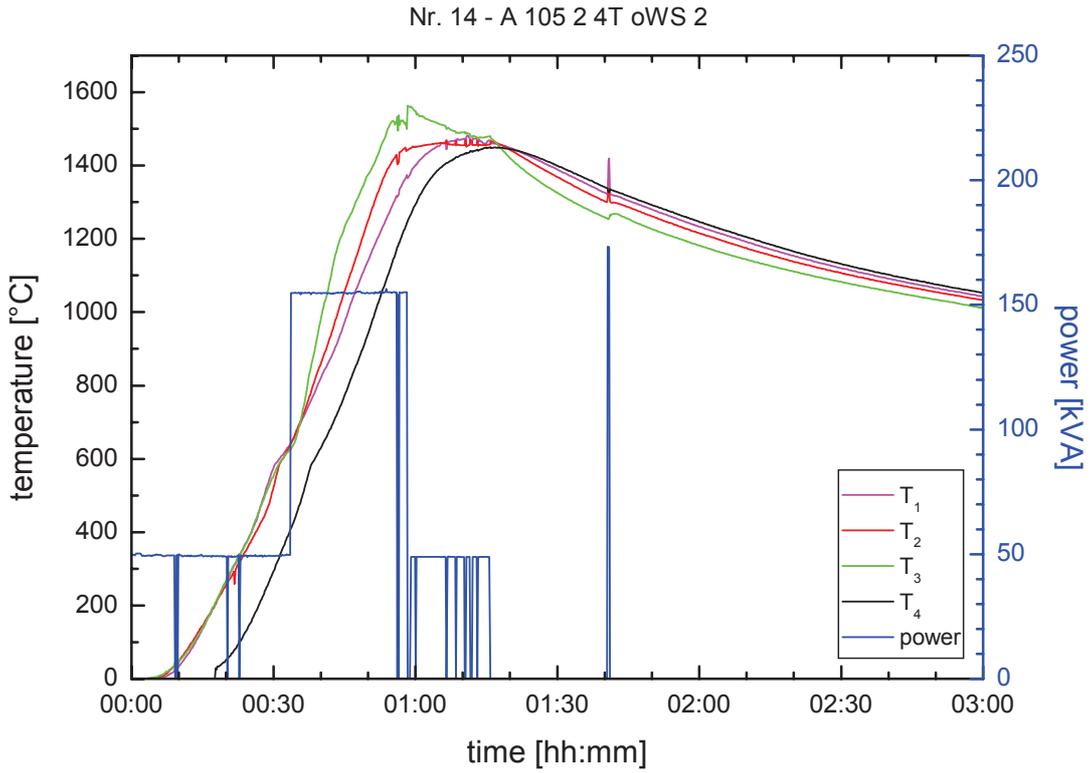


figure 39: time-temperature graph of trial nr. 14

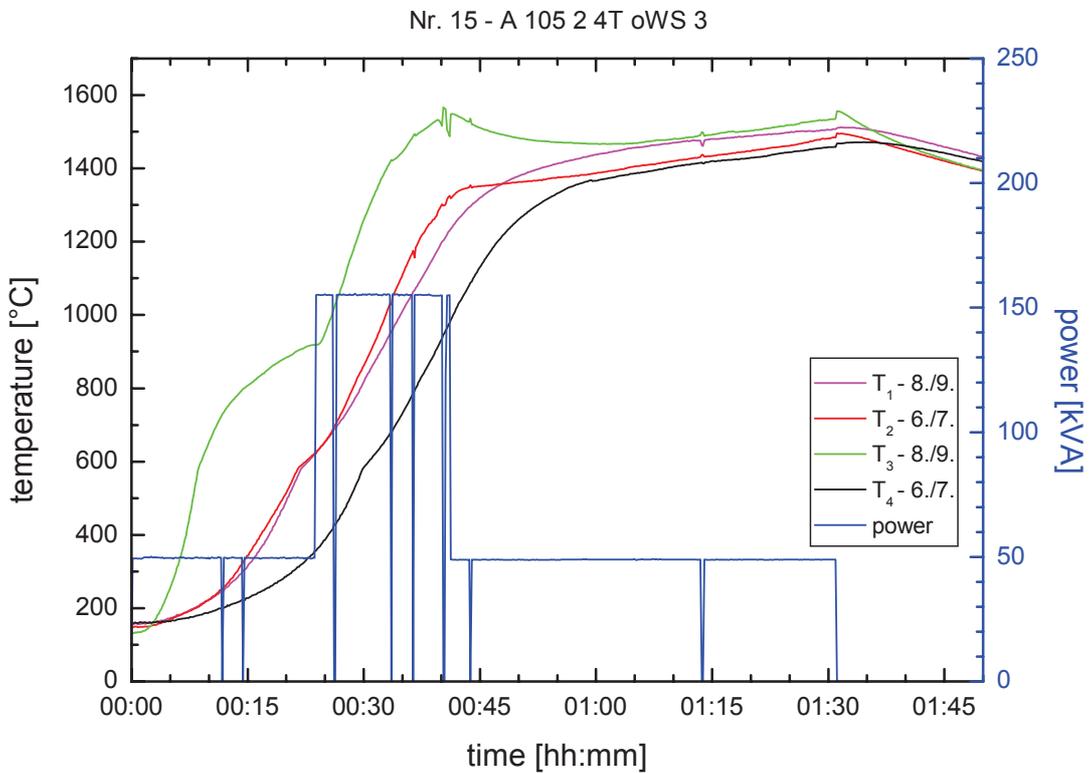


figure 40: time-temperature graph of trial nr. 15

6.4 Refractory temperatures of trials 7, 8, 11 and 12

	60 min	120 min	185 min
1. turn	343	468	569
3. turn	661	740	825
5. turn	735	840	912
7. turn	586	774	825
9. turn	395	616	723
11. turn	239	438	572

table 8: refractory temperatures of trial 7

	60 min	120 min	185 min
1. turn	666	793	757
3. turn	842	892	869
5. turn	887	929	897
7. turn	899	877	892
9. turn	837	847	874
11. turn	774	757	850
ref. block	64	102	137

table 9: refractory temperatures of trial 8

	25 – 90	90 – 80	80 – 70	70 – 50	50 – 90	90 – 70	after 70
0. turn	628	649	721	735	738	76	784
2. turn	685	735	754	759	771	779	784
4. turn	781	784	805	786	793	803	837
6. turn	860	830	845	801	832	842	850
8. turn	762	793	845	818	842	835	850
10. turn	757	757	774	774	793	796	803
12. turn	614	661	714	706	726	735	745
ref. block		57	74	117	134	149	162

table 10: refractory temperatures of trial 11

	25 – 90	90 – 80	80 – 70	70 – 50	50 – 90	90 – 70	after 70
0. turn	529	607	671	714	747	801	823
2. turn	668	711	738	752	759	774	788
4. turn	755	810	865	872	889	894	862
6. turn	820	842	877	850	897	912	907
8. turn	832	852	867	832	842	837	837
10. turn	718	738	759	764	801	820	815
12. turn	600	626	668	723	740	752	755
ref. block	47	59	83	100	122	129	149

table 11: refractory temperatures of trial 12