



# Bulk density and INternal Gas pressure in coke Ovens **(BINGO)**

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**Bulk density and INternal Gas pressure in coke Ovens (BINGO)**

European Commission

Directorate-General for Research and Innovation

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Unit D.3 — Low Emission Future Industries

Contact Hervé Martin

E-mail [rtd-steel-coal@ec.europa.eu](mailto:rtd-steel-coal@ec.europa.eu)

[RTD-PUBLICATIONS@ec.europa.eu](mailto:RTD-PUBLICATIONS@ec.europa.eu)

European Commission

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# Research Fund for Coal and Steel

## ***Bulk density and INternal Gas pressure in coke Ovens***

**(BINGO)**

Matthieu LANDREAU (Coordinator)

**ArcelorMittal Maizières Research SA**

BP 30320 – Voie Romaine, F-57283 MAIZIÈRES-LÈS-METZ Cedex

P. BARAN, M. LANDREAU

**ArcelorMittal Maizières Research SA**

BP 30320 – Voie Romaine, F-57283 MAIZIÈRES-LÈS-METZ Cedex

S. ALVARO, C. PHENG

**ArcelorMittal Atlantique Et Lorraine SAS**

Rue Luigi Cherubini 1-5, F- 93200 SAINT-DENIS

M.A. SCHULTEN, V. STISKALA

**Thyssenkrupp Steel Europe Ag**

Kaiser Wilhelm Strasse 100 - 47166 DUISBURG

R. LIN, M. NIEBERGALL, F. RULLANG

**Aktien-Gesellschaft der Dillinger Hüttenwerke**

Werkstraße 1 - 66763 DILLINGEN

M. HÖLZL, K. PILZ

**voestalpine Stahl GmbH**

voestalpine Straße 3 - 4020 LINZ

D. GAJIC

**DMT GMBH & CO. KG**

Am Technologiepark 1 - 45307 ESSEN

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### **Final Report**



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## Final summary

### **WP 1 Comparison and Analysis of different measuring methods of IGP**

#### **Task 1.1: Measuring methods used in gravity charging**

This first task was devoted to the method description used by the industrial partners and research centers in gravity charging. This description was based on:

- The design of the probe (single or multiple probes, length, slits, holes, etc.)
- The method used to insert the probes in the charge
- The location of the measurements (Coke Side, Pusher Side, doors or charging holes...)
- The method to measure the Internal Gas Pressure (IGP) itself (sensors, data transmission, counter current nitrogen flow, etc.)
- The equipment used to remove the probes before coke pushing
- The additional parameters measured at the same time (coal blend characteristics, temperature inside the charge, heating wall temperature, etc.)
- The interpretation of the measurements

Each partner described their measuring method, and this task allows to exchange and share the best methodology. The following table presents the main results.

Table 1: Methodology used in gravity charging in July 2015

	<b>CPM/AMAL</b>	<b>CPM (pilot oven)</b>	<b>AMO-CCL (pilot oven)</b>	<b>AMO</b>	<b>DMT (pilot oven)</b>	<b>tkSE (pilot oven)</b>	<b>tkSE</b>	<b>voestalpine</b>
<b>Design of the probe</b>	Triple probes 2 slits	Triple probes 2 slits	Single probe Squeezed tube ending	Single probe Squeezed tube ending	2 x Single probes 3 holes ( $\varnothing$ 3mm)	Single probe 1 hole ( $\varnothing$ 3mm)	Single probe 1 slit	Single probe 4 holes ( $\varnothing$ 5mm)
<b>Length / Diameter</b>	2.6m $\varnothing$ 10mm	Center of the pilot oven $\varnothing$ 10mm	Length 1.5 m Center of the pilot oven $\varnothing$ 12mm	2m $\varnothing$ 12mm	Center of the pilot oven $\varnothing$ 18mm	Center of the pilot oven $\varnothing$ 10mm	2.8m $\varnothing$ 16mm	2m Inner $\varnothing$ :28.5mm Outer $\varnothing$ :33.7mm
<b>Position</b>	Coke Side 0.5m above the oven sole	Coke Side 0.5m above the oven sole	Coke Side 0.435m above the oven sole	Pusher Side 1.2m above the oven sole	Charging hole	Pusher Side 0.25 m above the oven sole	Coke Side 0.65m above the oven sole	Pusher Side 0.25m above the oven sole
<b>Insertion / Removal</b>	Manually	Manually	Manually	Manually	Manually	Manually	Manually	Manually
<b>Frequency of measurements</b>	According plant request	Each charge	Each charge	Not applied regularly	Each blend	Each blend	1 measurement per blend	6 measurements per week

#### **Task 1.2: Measuring methods used in stamp charging**

This task was devoted to the description of the methodology used in case of stamp charging batteries with the same items than in the Task 1.1. Similar results were obtained, but due to difficulty to insert probe in stamp charging conditions, this specific point was discussed by the

AGDH and AMO. The following table summarizes the main characteristics of measurements of Internal Gas Pressure in stamp charging.

Table 2: Methodology used in stamp charging in July 2015

	<b>AGDH</b>	<b>CPM (pilot oven)</b>	<b>AMO-CCL (pilot oven)</b>	<b>AMO</b>
<b>Design of the probe</b>	Single probe Squeezed tube ending 2 slits	2 x Single probes 2 slits	Single probe Squeezed tube ending	Single probe Squeezed tube ending
<b>Length / Diameter</b>	2m	1m Ø 10mm	1.5m Ø 12mm	2m Ø 12mm
<b>Position</b>	Pusher Side Probe is inclined (~0.8m above the oven sole)	Coke Side + Pusher Side 0.5m above the oven sole	Coke Side 0.435m above the oven sole	Pusher Side 1.1m above the oven sole
<b>Insertion</b>	Drilling equipment	Manually	Steel tube in stamping box during stamping	Drilling
<b>Removal</b>	Manually	Manually	Manually	Manually
<b>Frequency of measurements</b>	1 measurement per week	Each charge	Each charge	Not applied regularly

#### Task 1.3: Organization of a dedicated workshop

AMMR has invited all the European cokemakers involved in measurements of Internal Gas Pressure to participate to a technical meeting in order to exchange procedures, to define possible ways of improvements and to discuss the use of the results for preventing the degradation of coke oven batteries. Despite the weak number of participants, this workshop allowed an exchange between the different partners and to present common results in different topics.

#### Task 1.4: Ways of improvements

AMMR/CPM worked to improve the IGP measurements in terms of safety, installation of the probes and accuracy of the measurements. A new system was designed and submitted to patent.

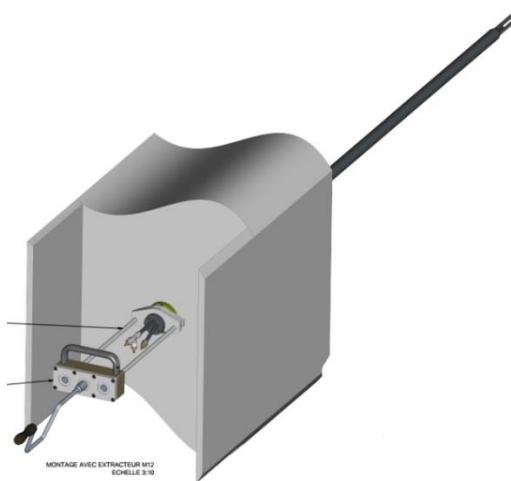


Figure 1: New system and new design of triple probe

The good point for this new system is the handling. Indeed, insertion/removal of this probe could be manually done without using hammer or a hand winch and the most important, without disturbing the coke production. This new solution will be implemented in all ArcelorMittal coke plant

with the assistance of AMMR/CPM team for the next series of tests. This new design of probe was proposed to be patented.

#### Task 1.5: Analysis of best practices

During the 3.5 years of BINGO project, improvement and test of different types of probe endings were investigated at industrial scale, such as single probe with slits, squeezed tip in horizontal, as well as vertical arrangement and with an angle of 45°. Main problem was to be certain to correctly hit the junction of the plastic layer. The correlation of the IGP-Peak with the position of the Soprecco valve was investigated and well established at Dillinger. The results of the long-term observation were positive and improve the centering inside the oven.

### **WP 2 Investigation of parameters influencing IGP by industrial measurements**

#### Task 2.1: Configuration of the measurements

The measurement of Internal Gas Pressure in industrial coke ovens is one of a very few possibilities for obtaining empirical data indicating the expansion behavior of the coal charge, thus the threat of potential oven wall damage or operation failures to occur due to a pressure generated by coal in coke production cycles. As there is no standard method available, the IGP probes have either series of holes or slots located at the tip of the lance, some plants use double-lance or even triple-lance probes, probes are inserted into the charge from either Coke Side or Pusher Side of the oven, the height of the lance above the oven sole is not standardized, etc. These factors have direct influence on the obtained IGP results. The task was to get information on IGP measurement configurations in order to be able to study and conclude whether any change from the existing to "recommended" configuration improves the accuracy of IGP measurement or could be standardized further.

AMMR/CPM and AMMR/AMO studied the influence of these parameters in both stamping and wet gravity charging techniques. Measurements were carried out through pusher side and coke side doors, at different heights above the oven sole and at different depths of insertion. Table hereafter presents the Comparison of investigated probes design.

Table 3: Comparison of investigated probes design

		squeeze probe		triple probe (kPa)	classical slot design (kPa)	holes endings (kPa)	delta (%)	standard deviation
		vertical (kPa)	horizontal (kPa)					
<b>probes design</b>								
2.1 a	MV coal	20.9		22.9			8.7	1.4
	MV coal	42.5		41.0			3.7	1.1
	LV coal	112.2		112.3			0.1	0.1
2.1 b	MV coal	11.2			11.5		2.6	0.2
	LV coal	27.5			27.7		0.7	0.1
2.1 c	MV coal	35.4				38.9	9.0	2.5
	MV coal	34.4				34.3	0.3	0.1
	LV coal	122.7				115.5	6.2	5.1
2.1 d	LV coal	95.6	91.9				4.0	2.6
	MV coal	25.2	25.4				0.8	0.1
	LV/MV coal	85.0	86.6				1.8	1.1
<b>average</b>							<b>3.5</b>	<b>1.3</b>

Main results show that probe endings do not have any influence on measurement of IGP, and so the simple and cheaper ending (squeeze ending) allows to accurately measure the IGP. Dealing with the position of probes, results showed that values are more scattered on Coke Side and IGP value are lower when distance from oven sole is increased.

#### Task 2.2: Properties of coal charge

Inherent quality of coal determines the amplitude of Internal Gas Pressure. Typical values of IGP measured for a given coal under conventional top charge conditions get even higher after the coal charge has been densified and its bulk density has increased, i.e. when oil addition technique or stamp charge process is employed, or when a change in moisture and/or grain size of the charge

leads to a change in coal bulk density. Also, the carbonization rate has an influence on the measured values of IGP.

This task was devoted to a long-term investigation about evolution of IGP according to the coal rank in both charging methods. Indeed, three industrial partners (tkSE, AGDH and voestalpine) carried out more than 50 measurements of Internal Gas Pressure, during the whole project period. In the frame of these measurements, AGDH and tkSE followed IGP evolution according to the inert components. The studies focusing on the charging methods have confirmed that the charge bulk density has a significant influence on IGP whereas later investigations demonstrated that the bulk density influencing factors have no important impact on IGP. The research conducted on top charge blends has determined the volatile matter content and various petrographic data as the most decisive factors. Study about influence of blend preparation method on homogeneity of coal charge has showed that inhomogeneity of the blend can be considered an influencing factor contributing to other conditions producing remarkable IGP fluctuation (cf. Figure 2). For both blending methods (Chevron method, system of bunkers) regular homogeneity checks should be applied to avoid large number of ovens being charged with potentially critical blend compositions.

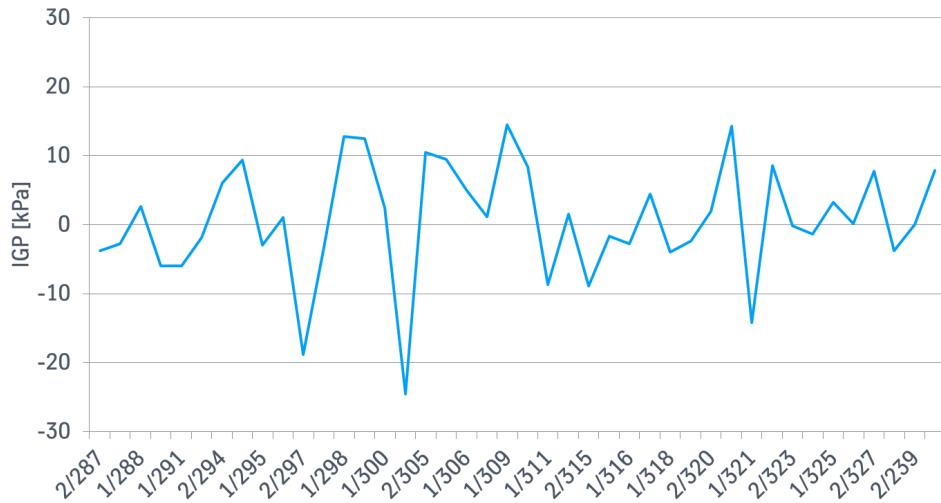


Figure 2: Difference between first and second measurement per blend

#### Task 2.3: Coke oven operations & design

While Internal Gas Pressure was investigated according to coal blend properties in Task 2.2, the Task 2.3 was devoted to study influence of coking rate, charging methods as well as moisture or grain size on Internal Gas Pressure results.

Effects of grain size, properties of the coal charge or influence of the heating rate were highlighted during industrial measurements. It was observed when grain size of the blend got coarser, the measured gas pressure significantly rose.

An original test was carried out in the frame of this work: ArcelorMittal Ostrava has prepared a coal blend for stamp charging battery and this blend was charged in gravity charging battery with the aim to check influence of charge density of the same blend on the value of IGP. Results showed that there is an important impact of charging method on Internal Gas Pressure values. Indeed, the same blend gives 2 kPa in gravity charging versus 19 kPa in stamp charging.

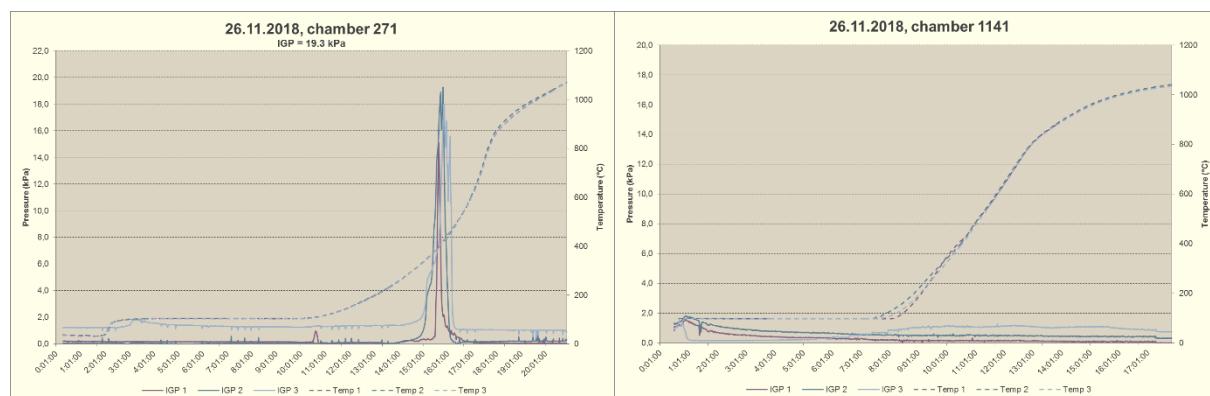


Figure 3: Same blend tested in stamp charging conditions and gravity charging at AMO



## **WP 3 Relationship between IGP and OWP in pilot oven and reflection on industrial IGP measurements with pilot oven results**

### **Task 3.1 Comparison between pilot and industrial measurements**

In task 3.1 each project partner compared the results of the pilot plant with their industrial coke ovens. Couples of pilot and industrial partners were established in order to compare and correlate Internal Gas Pressure.

Comparisons between CPM/AMAL, CPM/ZKS or AMO, highlighted that without any modification of Pilot Oven setup (heating rate, bulk density, etc..), a direct comparison between Pilot and Industrial measurement was not successful. Similar conclusions were observed by voestalpine, DMT and tkSE: values obtained at pilot scale, underestimate the industrial measurement due to bulk density parameter.

tkSE carried out IGP measurements in its new pilot scale coking facility with a coal charge capacity of 60 kg (wet) from coal blend sampled at coke plant Schwegern. Modifications of operating conditions of their MWO were performed to be relevant to IGP measurement. Thus, relationship between industrial and pilot oven IGP results displays a noticeable trend confirming that the measures have been efficient (Figure 4). This setup can be applied for carbonization tests the results of which deliver an indication of a corresponding IGP to be obtained in industrial ovens.

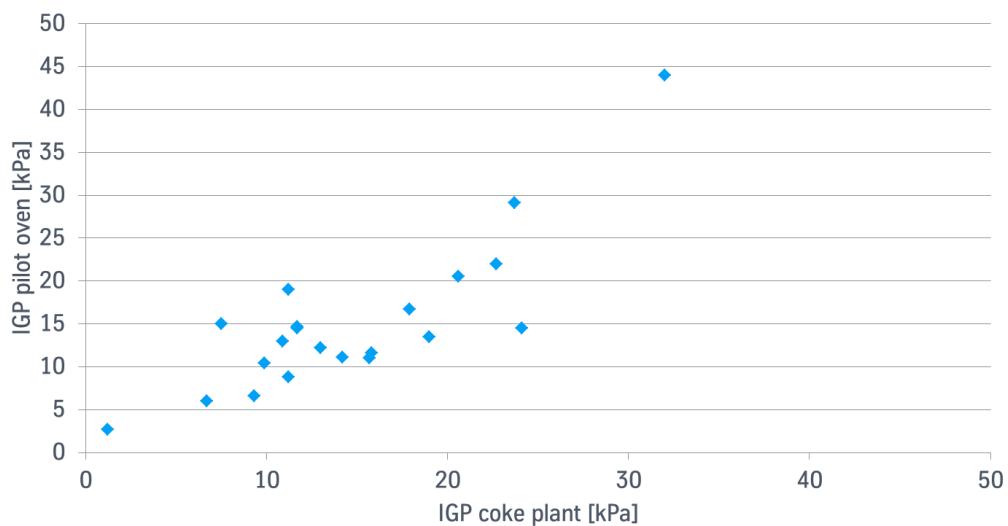


Figure 4: Relationship between industrial and pilot scale IGP results at tkSE

### **Task 3.2 Round robin on pilot ovens on two blends already prepared**

In order to correctly analyze values of internal gas pressures (IGP) from different pilot facilities, round robin tests were organized within the task 3.2, when two different blends representing gravity and stamp charging conditions were prepared and distributed among all partners. Each project partner gets one coal blend representing gravity charging and one blend representing stamp charging. The goal was to carry out carbonization tests at each project partner's facility and compare firstly the results of the internal gas pressure measurement. Within the round robins, also oven wall pressure (OWP) values and coke quality was compared.

During the round robin in gravity charging, no significant oven wall pressure or internal gas pressure was detected on any participant pilot facility. Detected internal gas pressures were in the range 0.4 – 1.6 kPa which can be considered as negligible compared to values in stamp charging conditions. In stamp charging, values of detected internal gas pressure were recorded within the range 7.4 kPa (DMT) and 15.9 kPa (tkSE) (cf. Table 4). This difference could be due to the difference in term of methodology between DMT and other labs. DMT used to measure the IGP through the oven roof whereas other labs performed the measurement through the door.

Table 4: Pilot oven tests output - stamp charging

Pilot oven parameters for STAMP CHARGING		CPM		DMT		Thyssen		Ostrava	
Coal charge preparation before the test		Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
< 3.15 mm	%	90.2	90.2	92.0	91.0	92.7	92.7	91.0	91.2
< 0.5 mm	%	42.3	41.7	48.0	48.0	42.6	42.6	37.7	38.2
Type of sieves	Square/round	square		square		square		square	
Charge moisture	%	10.9	11.1	11.0	11.0	10.8	10.8	11.0	11.0
Charge density	kg/m <sup>3</sup> d.b.	1008	1008	1000	1000	990	990	1001	1000
<b>carbonization parameters</b>									
Coking time	h:mm	22:46	22:56	22:38	22:29	14:30	14:30	21:50	21:50
Final coke temperature	°C	1102	1076	1102	1112	1000	993	1032	1033
real coking rate	mm/h	9.9	9.8	10.4	10.4	10.3	10.3	9.8	9.8
Max oven wall pressure As recorded	kPa	12.0	10.6	7.4	7.1	6.7	7.5	9.1	11.1
Time at OWP max	h:mm	3:42	3:54	14:42	13:36	11:26	11:40	3:16	2:45
Max oven wall pressure during the junction of the plastic layer	kPa	6.6	6.8	6.5	5.6	6.7	7.5	5.1	5.1
Max Internal gas pressure	kPa	8.2	10.1	7.4	11.0	13.4	15.9	9.7	10.6
Time at IGP max	h:mm	14:30	15:24	18:00	18:21	11:25	11:32	14:30	14:37
% of rank iron time during the junction of the plastic layer	%	66	67	80	81	78	79	66	67

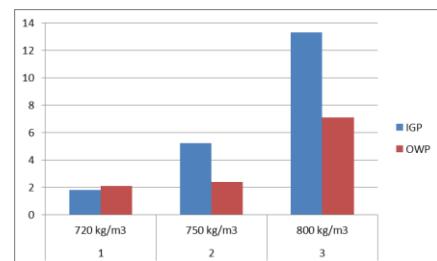
### Task 3.3 Investigations of influence of parameters under defined operation conditions in pilot oven

In Task 2.2 and 2.3, influences of properties of coal charge and coke oven operation & design on the internal gas pressure were investigated in the industrial scale. However, wall pressures which are generated through the IGP and transmitted to the coking oven walls cannot be directly measured industrially. Therefore, in this task carbonization tests were performed, at which there will be a variation of the following parameters moisture, grain size distribution, bulk density, coking rate and rank of the blend. In 2017, DMT studied the influence of the previous parameters in wet gravity charging whereas AMMR/CPM carried out the same investigation in stamp charging technique in 2018.

Results show that coal grain size and moisture have significant impact on the charge bulk density. These results demonstrate that the charge bulk density is the determinant factor dominating over other factors that influence the charge bulk density as presented on the following table.

Table 5: Influence of bulk density on IGP and WP

Bulk density	kg/m <sup>3</sup>	700	750	800
IGP	kPa	1.8	5.2	13.3
WP	kPa	2.1	2.4	7.1



### WP 4 Wall deformation and force transmitted to the chamber walls.

#### Task 4.1: Measurements of wall deformation in an empty oven during the carbonization of the adjacent ovens

In this task, the objective was to measure the heating wall deformation generated by IGP. In order to estimate the maximum wall deformation, an optical device was used. A new device was developed by CPM, and finally used in the frame of BINGO project. The 3D-Laser tool was easy to use in a coke plant, resistant to high temperature and sufficiently accurate to measure the small displacement of the wall without disturbing the production for a long time.

The aim of this measurement was to obtain maximum displacement of a heating wall during the junction of plastic layers. To obtain this value, measurements were carried out in an empty oven during the maximum peak of Internal Gas Pressure of the neighbor oven. Tests were carried in ZKS coke plant, and maximal measured deformations are below 5mm.

Task 4.2: Use of heating wall model with different sets of boundary conditions. Comparison with industrial observations

This task was devoted to run calculation from the 3D model of a complete heating wall. In collaboration with University of Orleans, modelling of Gijon and AGDH coke plant was done during the BINGO project.

Simulations were performed in 2 steps: thermal simulation, then mechanical simulation using the mechanical loads and the stress due to the thermal calculations. The results obtained are promising since thanks to this model, it is possible to obtain numerically the location of weak zones on the masonry. Indeed, the red and yellow parts of the following figures indicate the tension area.

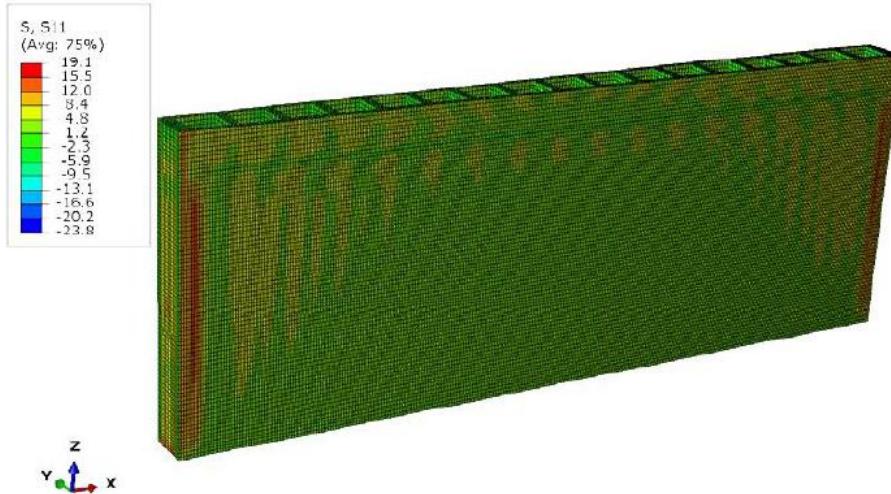


Figure 5: Stress field in direction 1 of AGDH heating wall

This task allows to confirm that methodology based on homogenization and Finite Element Method is correct to estimate wall deformation from a numerical point of view. Drawback of this approach is the maturity of this kind of models to be used industrially on a routine basis.

## **Scientific and technical description of the results**

### **1.1    Objectives of the project**

The objectives of BINGO project are to investigate if measurements of Internal Gas Pressure at industrial scale are suitable as a monitoring technique to detect possible deviation in the coal blend preparation and thus, avoid deformation of the coke ovens. This project will contribute to increase the service life of European coke plants. The work packages and tasks have been carefully defined to achieve this final objective.

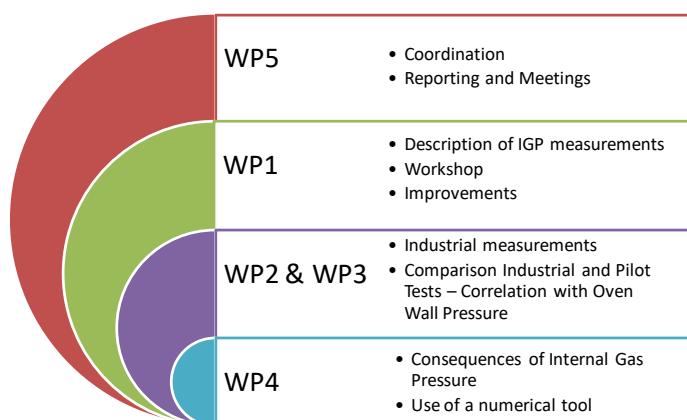
The first work package is fundamental for the project. The tasks will consist in gathering all information necessary for the project progress. This work package will allow different partners to share their know-how in the Internal Gas Pressure measurements. Indeed, different methods are used depending on the charging method or their industrial experience. The organization of a workshop with European coke makers will allow to extend the discussion about performing accurate measurements and to have new ideas in order to improve these Internal Gas Pressure measurements.

The second work package deals with investigations of industrial parameters which could influence Internal Gas Pressure measurements (design of the probes, location of the measurements, coal blend properties, etc.). Several measurements will regularly be carried out during the whole project period and the results will be analyzed in order to optimize coal blend for minimizing Internal Gas Pressure, to protect coke ovens from damage and to detect possible deviation in coal blending.

The third work package will correlate the Internal Gas Pressure measured at industrial scale with different pilot facilities. Indeed, different couples of partners (1 industrial + 1 R&D center) were defined in order to test the industrial blend in pilot facilities during the project period. Thanks to the diversity of partnership, movable wall pilot ovens of different capacity (60 to 600 kg), but also a small retort (11 kg) will be used. A round robin will be carried out to compare the results of Internal Gas Pressure measured on the same blend in different pilot facilities. This round robin will allow to find a relationship between Internal Gas Pressure and Oven Wall Pressure measured at pilot scale.

The fourth work package will be devoted to quantifying the effects of Internal Gas Pressure on heating wall conditions. The aim of this work package is to measure the heating wall deformation generated by Internal Gas Pressure and to define threshold values. A mechanical or optical device will be implemented to measure the wall displacement during coal carbonization. The industrial results will be compared to the results of the heating wall model developed in the frame of previous RFCS projects [1,2]. Then the mathematical model will allow to better understand Oven Wall Pressure consequences on wall degradation. The results of calculations will be used to estimate the deformation of heating wall and the maximum acceptable Oven Wall Pressure

The last work package will be dedicated to coordination work and reporting. Indeed, the project cannot succeed without good exchanges between partners. Coordination meetings will be held every 6 months for discussing the progress, results and to agree project reports and presentations to the Commission. All the work packages have a strong interrelation as illustrated on the next figure.



## **1.2 Description of activities and discussion**

This section covers the description of the experimental work performed on a task per task basis, highlighting the main results achieved.

### **WP 1 Comparison and Analysis of different methods of measuring IGP**

#### ***Task 1.1 - Measuring methods used in gravity charging [AMMR, AMAL, tkSE, voestalpine, DMT, CPM, AMO]***

This Task was devoted to draw a state of art in terms of measurement of Internal Gas Pressure. Each partner (CPM/AMAL, AM Ostrava, voestalpine and tkSE) has described the methodology used for the internal gas pressure measurement in industrial scale under gravity charging conditions with focus on the following aspects:

- The design of the probe (single or multiple probes, length, slits, holes, etc.)
- The method used to insert the probes into the charge
- The location of the measurements (Coke or Pusher Side, doors or charging holes, etc.)
- The method to measure the IGP itself (sensors, data transmission, etc.)
- The equipment used to remove the probes before coke pushing
- The additional parameters measured at the same time (coal blend characteristics, temperature inside the charge, heating wall temperature, moisture content, stamping density, etc.)
- The frequency of measurements and the number of controlled ovens per campaign

ArcelorMittal Florange used to follow the CPM measuring method to study Internal Gas Pressure. Indeed, AMAL did not regularly measure the IGP and so, during each campaign, AMAL used to measure to measure IGP in collaboration with CPM.

In order to obtain IGP, three tubes side by side in common steel are introduced inside the charge through adequate holes drilled in the coke oven door. These tubes are connected with pressure sensors which give a continuous and electrical signal (Figure 1.1.1).

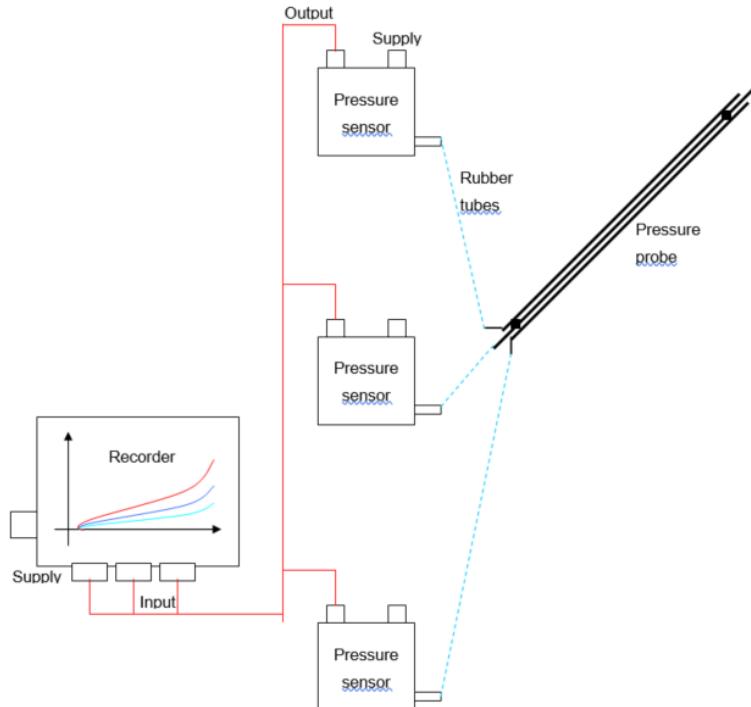


Figure 1.1.1: Principle of the Internal Gas Pressure measurement

The pressure probes are closed at one end (on the coal side) and they have two splits, 1 mm wide and 30 mm long, diametrically opposed and arranged in a vertical plan. This arrangement has two advantages:

- the introduction of the probe inside the charge is safely accomplished, without plugging by the coal,
- the explored area is well defined, because it corresponds with a 1 mm wide plan parallel to the plastic layers.

CPM generally used three tubes side by side, instead of only one, for the two following reasons:

- Firstly, despite the taken care, the probe may be not exactly in the middle of the chamber,
- Secondly, even if the probe is very well centered in the middle of the chamber, it may happen that the two heating walls do not have the same temperature. In that case, the thermal centre of the charge is not equal to the geometric centre.



Figure 1.1.2: End of the pressure probe



Figure 1.1.3: Slits of the pressure probe

At the beginning of the project, ArcelorMittal Ostrava measurement of IGP is provided by 2.0 meters long probe from fire resistant steel. Probe is made similarly to the one standardly used at pilot scale (Figure 1.1.4).



Figure 1.1.4: Probe with pressure and temperature transmitter

For pressure and temperature signal transmission, wireless system made by Honeywell was installed. Base scheme of measuring arrangement is shown in Figure 1.1.5.



Figure 1.1.5: Scheme of arrangement for internal gas and temperature measurement

Location of the probe is shown in Figure 1.1.6. Probe is standardly placed in the oven door through guide-way tube located in the door lining, signal transmitters are isolated to protect equipment against the heat from adjacent oven doors opening.



Figure 1.1.6: Placement of the probe at coke oven doors and its isolation

Following principle has been set up at Ostrava batteries in frame of first trials with the new 2m long single probe. As was mentioned above, probe is made from fire resistant steel. One end is equipped by embranchment with threads for pressure and temperature transmitters. Temperature transmitter is connected with a K-type thermocouple placed through the whole length of probe. Another end of probe is simply squeezed to avoid a coal penetration into the tube during insertion of the probe. Both endings of industrial probe are shown in Figure 1.1.7.



Figure 1.1.7: Detail of the probe's endings used by ArcelorMittal Ostrava

One coke oven of the coke plant of voestalpine Stahl in Linz is equipped with a measurement system for the IGP. This equipment was installed in the year 2009 and consists of a probe, which has the following characteristics:

- The probe is a pipe with a pointed tip and four holes (each 5mm in diameter) at the end of the tip
- The probe has a length of appr. 2 meters, and is 35 mm in diameter, it is inserted in the center of the oven (average chamber width: 455 mm, cf. Figure 1.1.8)
- The probe measures the IGP, additionally a thermocouple (Type K, NiCr-Ni) is installed inside this probe
- The IGP is evaluated by a pressure transmitter from company ABB
- The data are sent every minute to the control unit from company B&R (Bernecker + Rainer)

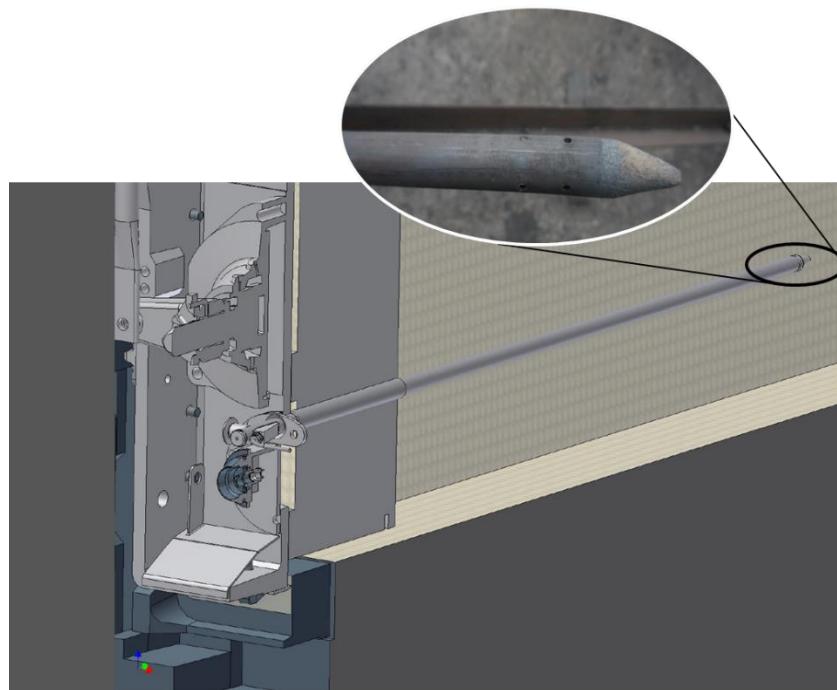


Figure 1.1.8: Schematic drawing of the installed IGP probe in the coke oven, and a detail of the tip of the probe (4 holes)

At coke plant Schwelgern, single lance Internal Gas Pressure probe is used for industrial measurements. The measured IGP data represents the performance of ca. 78 tons of coal charged into the oven. A single coke oven has a height of 8.43 m and a width of 0.59 m and is charged with coal blend having an average coal bulk density of 855 kg/m<sup>3</sup>(wet). The coal is carbonized with approximately 25 h coking time corresponding to a coke rate of 12.4 mm/h.



Figure 1.1.9: IGP probe tip

The lance is inserted into coal charge through the oven door on the coke side of the battery (Figure 1.1.9). The coke side has been chosen as there is always larger volume of coal present and flue gas temperatures are on the coke side higher as well. The length of the lance is ca 2.8 m so that its measuring slot is located directly under the geometrical center of the charging hole and is resting in coal on the level of 0.65 m above the oven sole. The highest bulk density is foreseen in this part of the oven chamber. The opening of the lance is a single slot which is facing the oven sole. As many as five oven doors at a battery, i.e. ten oven doors in total, are retrofitted and ready to accommodate IGP probe. The ovens, on which is the IGP measurement executed, are located close to the coal tower. Terminal oven at a battery is not used for IGP measurement because these ovens tend to show uneven temperature profiles.

The following table summarizes the main characteristics of measurements of Internal Gas Pressure by project partners, in top charging operation.

Table 1.1.1: Methodology used in gravity charging at the beginning of the project

	<b>CPM/AMAL</b>	<b>CPM (pilot oven)</b>	<b>AMO- CCL (pilot oven)</b>	<b>AMO</b>	<b>DMT (pilot oven)</b>	<b>tkSE (pilot oven)</b>	<b>tkSE</b>	<b>voestalpine</b>
<b>Design of the probe</b>	Triple probes 2 slits	Triple probes 2 slits	Single probe Squeezed tube ending	Single probe Squeezed tube ending	2 x Single probes 3 holes ( $\varnothing$ 3mm)	Single probe 1 hole ( $\varnothing$ 3mm)	Single probe 1 slit	Single probe 4 holes ( $\varnothing$ 5mm)
<b>Length / Diameter</b>	2.6m $\varnothing$ 10mm	Center of the pilot oven $\varnothing$ 10mm	Length 1.5 m Center of the pilot oven $\varnothing$ 12mm	2m $\varnothing$ 12mm	Center of the pilot oven $\varnothing$ 18mm	Center of the pilot oven $\varnothing$ 10mm	2.8m $\varnothing$ 16mm	2m Inner $\varnothing$ :28.5mm Outer $\varnothing$ :33.7mm
<b>Position</b>	Coke Side 0.5m above the oven sole	Coke Side 0.5m above the oven sole	Coke Side 0.435m above the oven sole	Pusher Side 1.2m above the oven sole	Charging hole	Pusher Side 0.25 m above the oven sole	Coke Side 0.65m above the oven sole	Pusher Side 0.25m above the oven sole
<b>Insertion / Removal</b>	Manually	Manually	Manually	Manually	Manually	Manually	Manually	Manually
<b>Frequency of measurements</b>	According plant	Each charge	Each charge	Not applied	Each blend	Each blend	1 measurement	6 measurements

	request			regularly			per blend	per week
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Modes of operation of the partners involved in this task is described in Annex A of this report.

### ***Task 1.2 - Measuring methods used in stamp charging [AGDH, AMO]***

This task was devoted to the description of the methodology used in case of stamp charging batteries with the same items as presented in the Task 1.1. Moreover, there are two special points to be considered for a stamp charging system: One is the higher moisture content of the coal blend up to 12% H<sub>2</sub>O; the other one is the higher bulk density of more than 1050 kg/m<sup>3</sup> (dry). Attentions should be paid to put the probe into a right position either manually or by means of a suitable drilling equipment. Thus, description of IGP measurement will be divided in two parts: measuring equipment and placement of the probe.

#### **Measuring equipment and methodology**

##### Aktiengesellschaft der Dillinger Hüttenwerke (AGDH)

First probe of AGDH was designed as a single heat-resistant steel tube with two slits on the tip (Figure 1.2.1). The length of the measuring probe was 2m and the length of the slits was around 7cm. For a better placement into the coal cake and the avoidance of coal penetration into the tube the tip was peaked.

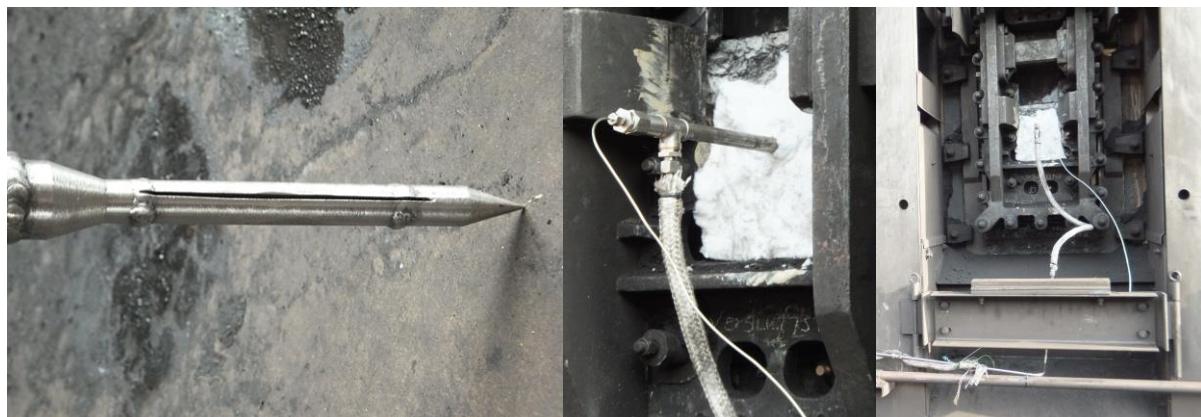


Figure 1.2.1: First measuring setup for internal gas pressure determination by AGDH

At the end of the probe there were two flanges (Figure 1.2.2), one connected to the pipe for the pressure measurement with a manometer on the other end of the tube and the other flange to the K-type thermocouple. The thermocouple is integrated into the tip of the steel tube via the hole of the flange.



Figure 1.2.2: Connection of manometer and thermocouple to the probe by tube + external manometer and Receiver with hard drive

The connection to the manometer and the hard drive for recording the data was classical through a steel tube and by wires. But the arrangement with the steel tube had serious risks respectively to pressure leakages or damage through the standard oven operations around it. Therefore, the setup was changed by connecting the manometer directly onto the probe. The new designed probe is still build up from a single heat-resistant steel tube with two slits on the tip (Figure 1.2.3). Length of the measuring probe and of the slits remains the same.



Figure 1.2.3: Current measuring setup for internal gas pressure determination

At the end of the probe now there are three flanges, one connected to the manometer on top, one equipped with a reservoir for condensed water on the bottom and the third one to the K-type thermocouple at the end. In this way the electrical signals for pressure and temperature measurements are transmitted by wire to the receiver with a hard drive. The optimization of the measuring setup is presented on Task 1.4.

ArcelorMittal Ostrava (AMO):

Measurement of IGP is provided by 2.0 meters long probe from fire resistant steel. Probe is made similarly to the one standardly used at pilot scale (Figure 1.2.4).



Figure 1.2.4: Probe with pressure and temperature transmitter

For pressure and temperature signal transmission, wireless system made by Honeywell is installed. Base scheme of measuring arrangement is shown in Figure 1.2.5.

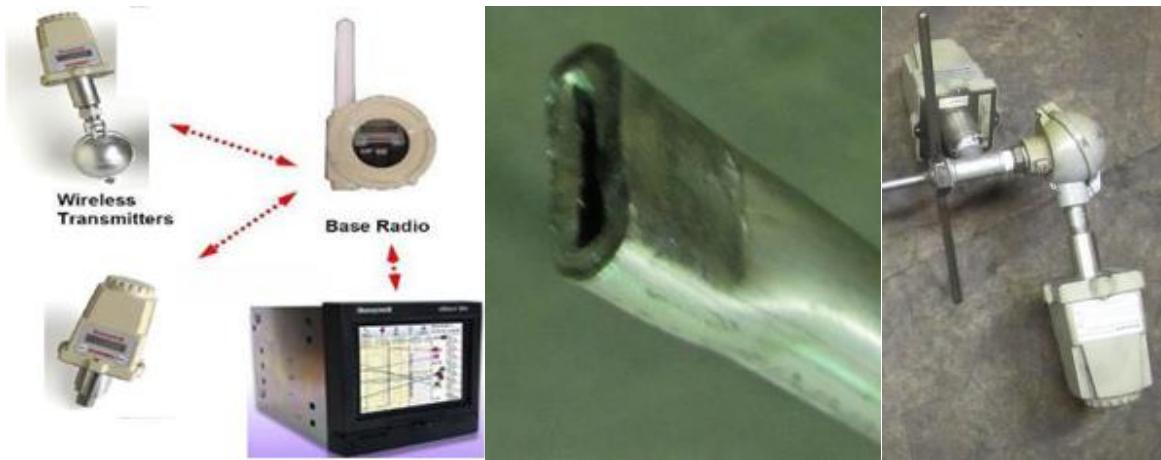


Figure 1.2.5: Scheme of arrangement for internal gas and temperature measurement

Following principle has been set up at Ostrava stamp charged batteries in frame of first trials with the new 2m long single probe. As mentioned above, probe is made from fire resistant steel. One end is equipped by embranchment with threads for pressure and temperature transmitters. Temperature transmitter is connected with a K-type thermocouple placed through the whole length of probe. Another end of probe is simply squeezed to avoid a coal penetration into the tube during insertion of the probe.

#### **Placement and position of the probe**

##### Aktiengesellschaft der Dillinger Hüttenwerke (AGDH)

A schematic sketch of the current position of the probe inside the coke oven at AGDH is shown in Figures 1.2.6. The placement is in the middle of the coal cake at a height of around 0,8m. The depth of the drilling hole is around 2,25 m so that the afterwards installed measuring probe is located at a depth of 2m. The probe is inserted with an angle of around 8-10° to avoid interruptions by condensed water.

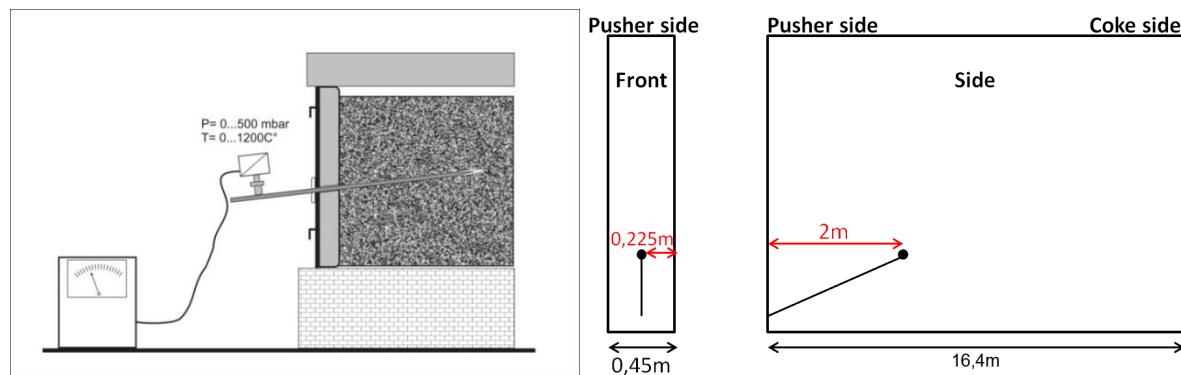


Figure 1.2.6: Placement of the measuring probe within the coke oven

The insertion of the probe is managed through a customized oven door with a perforated hole on the bottom end (Figure 1.2.7). The associated wires are running underneath the oven doors along the oven framework.

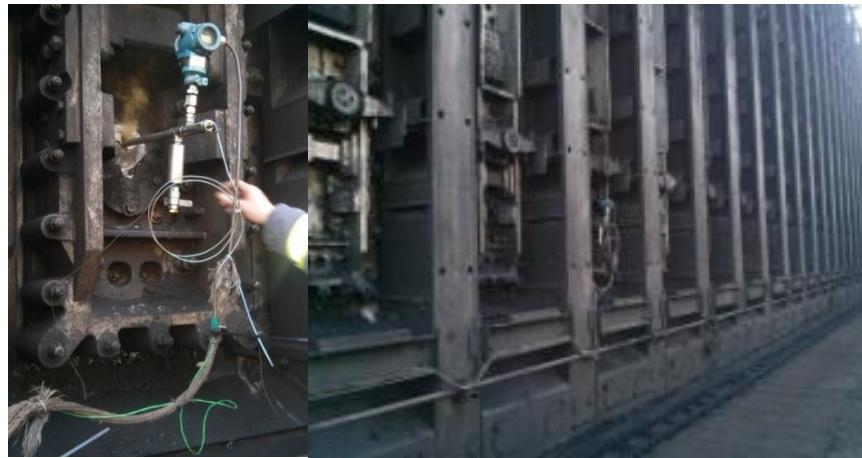


Figure 1.2.7: Perforated oven door with inserted probe and look onto the placed probe along the oven battery

As mentioned before, the current measuring setup has been improved and optimized during the project.

ArcelorMittal Ostrava:

Location of the probe at AMO is made from pusher side, as well from coke side and is shown in Figure 1.2.8.

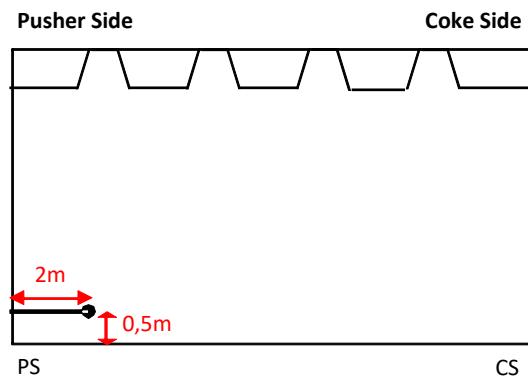


Figure 1.2.8: Placement of the measuring probe within the coke oven

Probe is standardly placed in the oven door through guide-way tube located in the door lining, signal transmitters are isolated to protect equipment against the heat from adjacent oven doors opening.



Figure 1.2.9: Placement of the probe at coke oven doors and its isolation

Principle of placement of the probe was tested during whole project phase and possible improvements are expected from other trials at Ostrava as well as on the basis of other partner's recommendations.

Thus, the following table summarizes the main characteristics of measurements of Internal Gas Pressure in stamp charging.

Table 1.2.1: Methodology used in stamp charging

	<b>AGDH</b>	<b>CPM (pilot oven)</b>	<b>AMO-CCL (pilot oven)</b>	<b>AMO</b>
<b>Design of the probe</b>	Single probe Squeezed tube ending 2 slits	2 x Single probe 2 slits	Single probe Squeezed tube ending	Single probe Squeezed tube ending
<b>Length / Diameter</b>	2m	1m Ø 10mm	1.5m Ø 12mm	2m Ø 12mm
<b>Position</b>	Pusher Side Probe is inclined (~0.8m above the oven sole)	Coke Side + Pusher Side 0.5m above the oven sole	Coke Side 0.435m above the oven sole	Pusher Side 1.1m above the oven sole
<b>Insertion</b>	Drilling equipment	Manually	Steel tube in stamping box during stamping	Drilling
<b>Frequency of measurements</b>	1 measurement per week	Each charge	Each charge	Not applied regularly

As mentioned in Task 1.1, modes of operation of the partners involved in this task is described in Annex A of this report.

### ***Task 1.3 - Organization of a dedicated workshop [all]***

Workshop that should be organized in Q2 2016 was postponed to September 2017. All the European coke makers involved in measurements of Internal Gas Pressure were invited to participate to a technical meeting in order to exchange procedures, to define possible ways of improvements and to discuss the use of the results for preventing the degradation of coke oven batteries.

Due to cancellation of ECC meeting, the number of external participants was below expectations. The final program was the following one:

- Industrial measuring techniques of IGP in wet gravity charging (CPM)
- Industrial measuring techniques of IGP in stamp charging (AGDH)
- Studies on coal dangerousness at INCAR-CSIC (INCAR-CSIC) (cancelled)
- Comparison of IGP measurements at pilot scale (ArcelorMittal Ostrava)
- Measurements of IGP in a coking retort (voestalpine)
- Influence of Parameters on IGP values (DMT)
- Comparison between industrial and pilot oven measurements (tkSE)
- Industrial measurements at Gent coke plant (AM Gent) (cancelled)
- Industrial measurements at Bottrop coke plant (AM Bremen) (cancelled)

Despite the weak number of participants, this workshop allowed an exchange between the different partners and to present common results in different topics.

#### **Task 1.4 - Ways of improvements [AMMR, AMAL, CPM]**

This task was initially devoted to improving the measurements of Internal Gas Pressure and specially the installation of triple probes. During the project, due to the important numbers of industrial measurements, some improvements have also emerged. Hereafter, description of main improvements in terms of probe installation, design or recording are presented.

#### **Installation and probe design**

AMMR/CPM worked to improve the IGP measurements in terms of safety, installation of the probes and accuracy of the measurements. Brainstorming was performed in October 2016 with Florange coke plant and AMMR team about modification of probe design but also for insertion and removal of the probes.

The main specifications for this new probe were the following ones:

- Keep the three tubes in order to check the centering of the measurement,
- Keep the same principle for positioning of slits,
- Keep the same connector technology for pressure and temperature sensors,
- Strengthen the mechanical resistance of the probe,
- Study the possibility to re-use the same probe several times,
- Improve the safety procedure for insertion and removal of the probe.

First drawing was discussed at CPM in January 2017. Main modifications deal with the insertion and removal of the probes as illustrated on Figure 1.4.1.

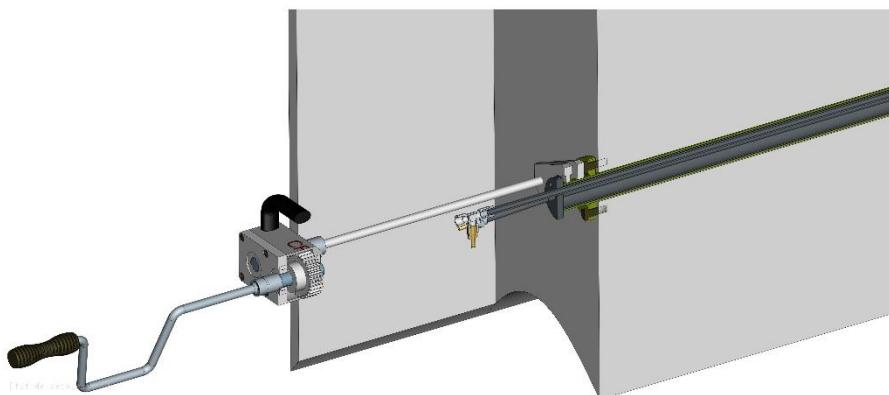


Figure 1.4.1: New system for inserting Internal Gas Pressure probe

Validation of drawings was done in March 2017; manufacture of new system was completed in May 2017.

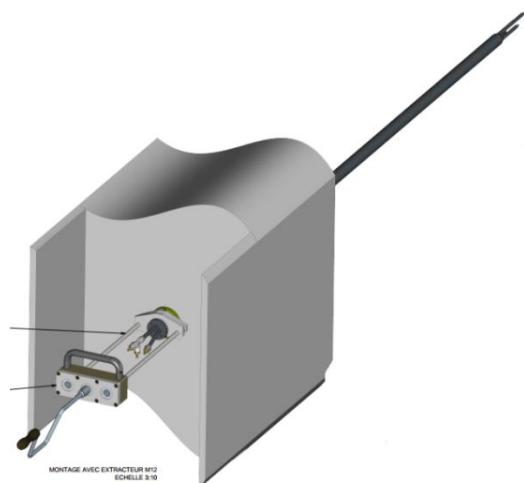


Figure 1.4.2: New system and new design of triple probe

Tests to validate the new design were initially planned in July, then in September 2017, but due to industrial issues at AMAL coke plant, the tests were finally performed in February 2018. During this campaign, four successive tests were carried out: one test with classical triple probe and three tests with new design. Hereafter some pictures taken during the campaign of measurements:



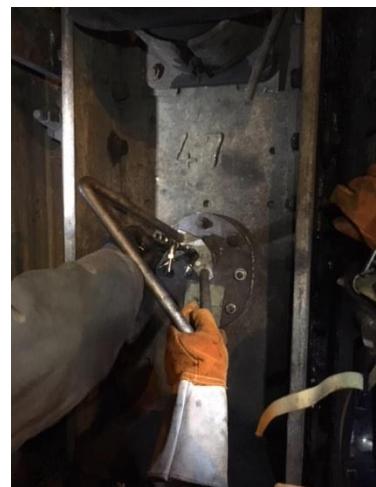
Classical triple probe of CPM after one test: deformations are too important to be re-used



Insertion of the new probe



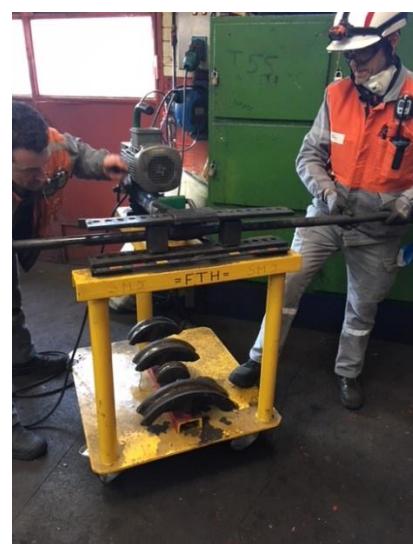
Removal of the wheel



Manually removal of the probe



New triple probe after 1 test in the industrial oven



Small rectification of the probe before the next measurement

Figure 1.4.3: Pictures illustrating the validation campaign of the new probe

Results of the different measurements are presented on the following Figures. Results obtained are encouraging since it can be noticed on Figures 1.4.5, 1.4.6 & 1.4.7 that this new probe allows to have a good centering (the 3 curves of IGP are overlapped). Indeed, in the test with classical probe, the 3 curves of IGP are not superimposed and curve of temperature presents two inflection points which is the consequence of a bad centering (Figure 1.4.4). On the other hand, the curves with new probe design do not present this issue. Dealing with results, the new triple probe respectively gives 23.7/20.4/19.5 kPa in comparison with 14.4kPa obtained with previous triple probe.

These results are in agreement with the range of IGP obtained by other partners (cf. Task 2.2), and confirms that a good positioning allows to have a better accuracy for determination of IGP.

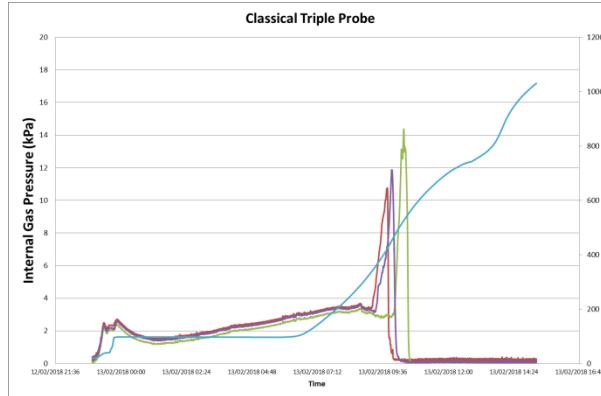


Figure 1.4.4: IGP measurement with classical triple probe of CPM

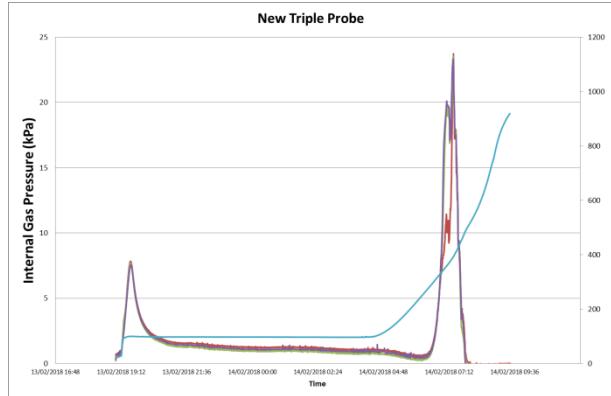


Figure 1.4.5: 1<sup>st</sup> measurement with new triple probe designed by CPM/AMMR/AMAL

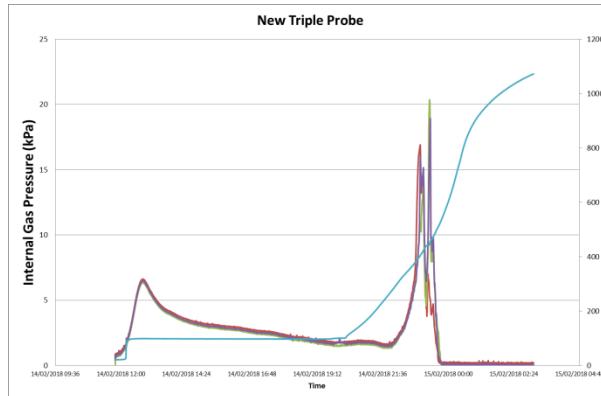


Figure 1.4.6: 2<sup>nd</sup> measurement with new triple probe designed by CPM/AMMR/AMAL

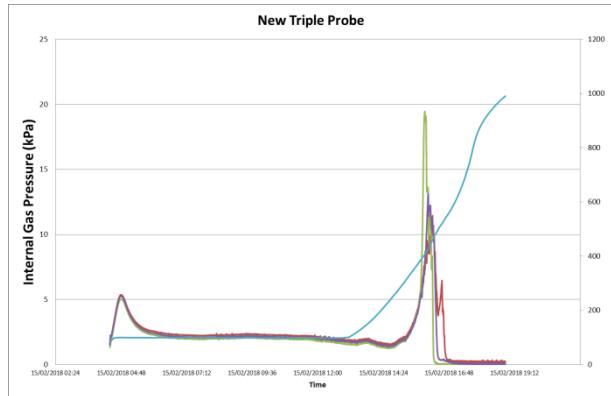


Figure 1.4.7: 3<sup>rd</sup> measurement with the same triple probe than 1st test

The other good point for this new system is the handling. Indeed, insertion/removal of this probe was manually done without using hammer or a hand winch and the most important, without disturbing the coke production. This new solution will be implemented in all ArcelorMittal coke plant with the assistance of AMMR/CPM team for the next series of tests. This new design of probe was proposed to be patented.

### Sensors and data recording

In frame of BINGO project, more than 40 industrial measurements at stamp charged Battery no.2 and 5 measurements at gravity charged Battery no.11 were carried on at ArcelorMittal Ostrava. Due to constant low internal gas pressure at gravity charging conditions, other measurements were cancelled in order to focus on stamp charging conditions, where higher variability of IGP is giving more information about influencing parameters.

Design of industrial probe was significantly changed during the project. While single probe equipped by wireless sensors, shown in Figure 1.4.8, was used in the first half of the project, new

triple probe equipped by MadgeTech combined pressure and temperature sensors was launched in the beginning of 2017.

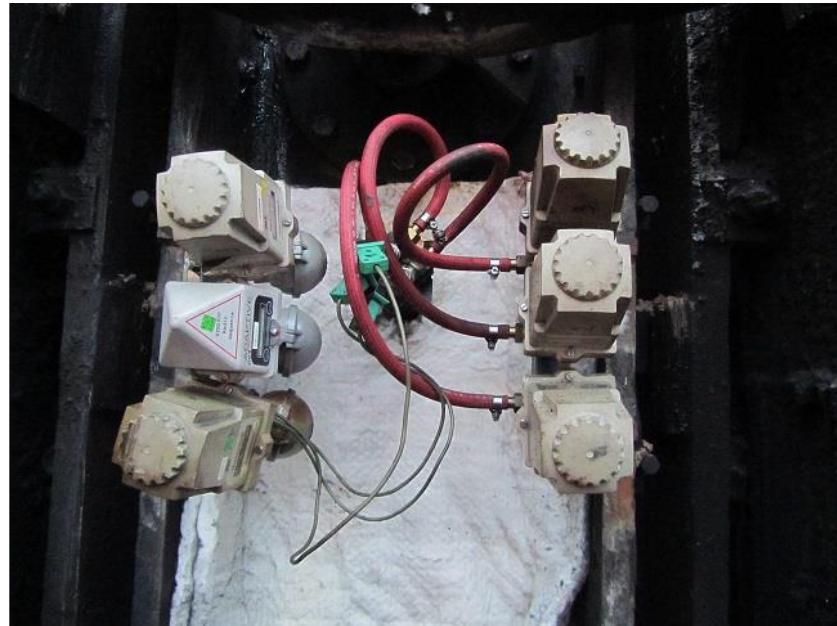


Figure 1.4.8: Wireless pressure and temperature sensors installed on coke oven at AMO

New triple probe equipped by MadgeTech combines pressure and temperature sensors as shown in Figure 1.4.9. Madgetech sensors with synchronous detection of pressure and temperature together do not have any wireless transmission into data recorder, but own data recording that can be downloaded after trial.



Figure 1.4.9: MadgeTech combined pressure and temperature sensor and industrial installation at stamp charged battery No.2

In frame of BINGO project, 20 new triple probes were manufactured in AM Ostrava maintenance workshop. Stainless steel heat resistant steel was applied in order to achieve multiple re-usage of probes at industrial conditions. Triple probe immediately after withdrawal from coke oven is shown in Figure 1.4.10. It can be noticed that deformations seem to be less important than CPM probe (Figure 1.4.3), it is due to the non-existence of welding point between tubes in AMO probes. With

regards to the success rate of IGP measurement, mentioned in Figure 1.4.11, proper renewal of tubes for another use is another success rate influencing factor.



Figure 1.4.10: Triple probe immediately after withdrawal from coke oven

The importance of successful renewal of tubes is indirectly confirmed by new probe's measurement success rate which is also shown in Figure 1.4.11. Blue points representing totally new probes are showing 100% success in terms of tar seam position of probe's peak.

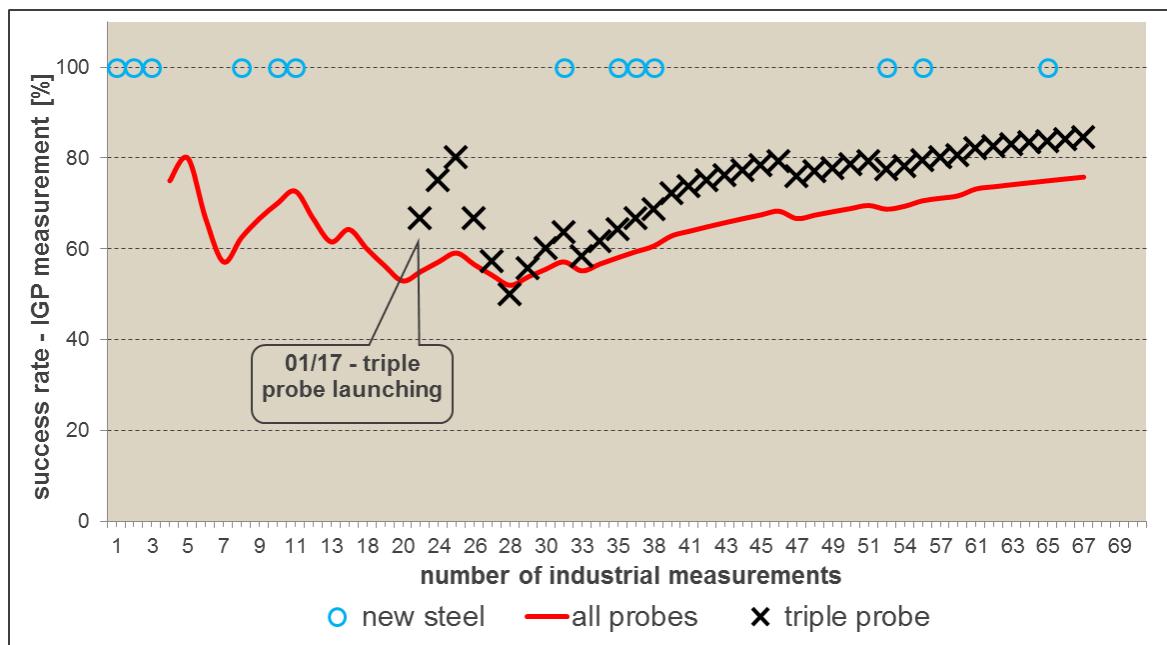


Figure 1.4.11: Success rate of industrial measurements in stamp charging conditions

During the project BINGO, AGDH worked also on improvement on the measurement. Thus, they established a new wireless system to measure IGP and temperature. The new system is composed of different elements as illustrated on Figure 1.4.12. The repeaters are installed on the side of the battery (red circles) to send the signal of temperature and pressure to the top of the battery to avoid the signal lost due to the Pushing Machine standing between data receiver (end of Battery 3, left) and sender (oven of IGP measurement).

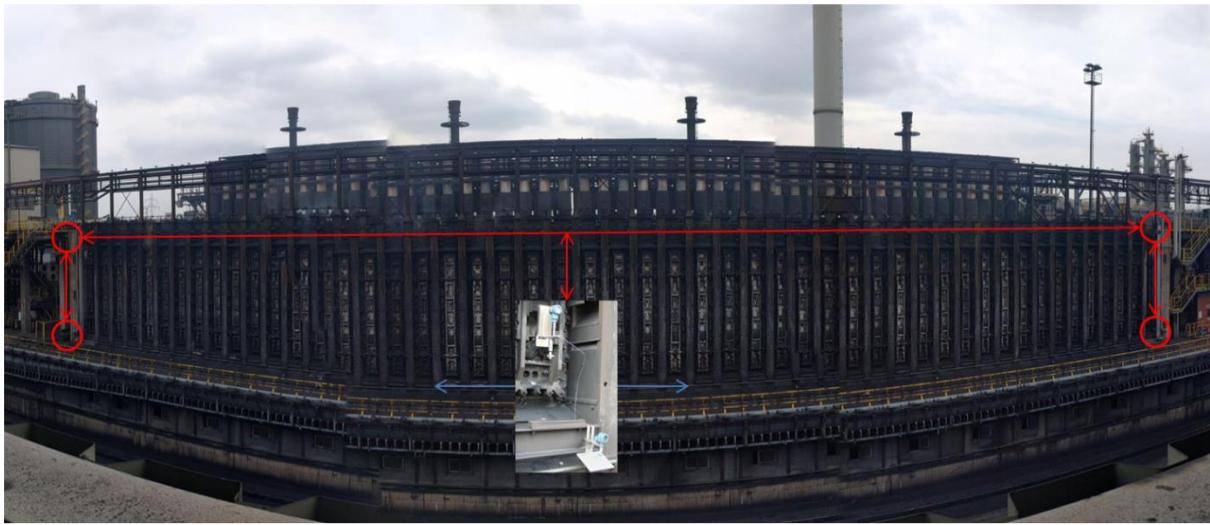


Figure 1.4.12: New wireless system established by AGDH

#### **Task 1.5 - Analysis of best practices [all]**

AGDH started to improve the design of the IGP probe by modifying the tip. The classical design with slits was replaced by one with a squeezed ending. This cheap and easy to build version of the probe was tested in different geometrical arrangements in the oven. Altogether there have been studied three different angles  $0^\circ$ ,  $45^\circ$  and  $90^\circ$  which are illustrated in Figure 1.5.1.

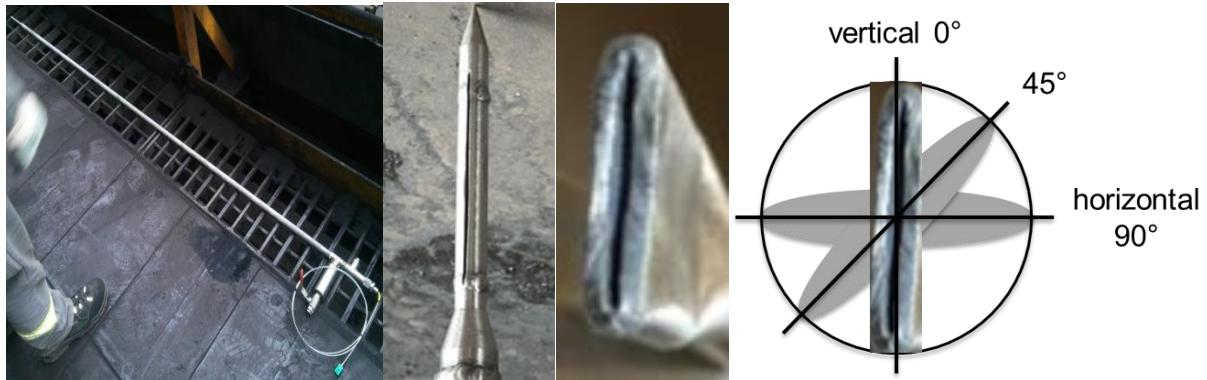


Figure 1.5.1: IGP probe with modified probe design at AGDH for the project period

The aim was to maximize the accuracy to hit the junction of the plastic layers in the coking process. By changing the angle from  $0^\circ$ (vertical) to  $45^\circ$  or  $90^\circ$ (horizontal) the range of the opening of the probe was raised. Therefore, the chance to hit the conjunction of the plastic layers with the front of the probe and the possibility to measure the best possible value for the internal gas pressure should be increased. In Figure 1.5.2 there are the determined peak values for the IGP measurements given. The data has been revised by the correlation with the position of the Soprecco valve. In the figure is only the data shown with a good or a very good Soprecco correlation. The different arrangements of the squeezed tip are shaded in different colors. Furthermore, on the bottom of the graph there is listed the mean value of the peak pressure for the various types.

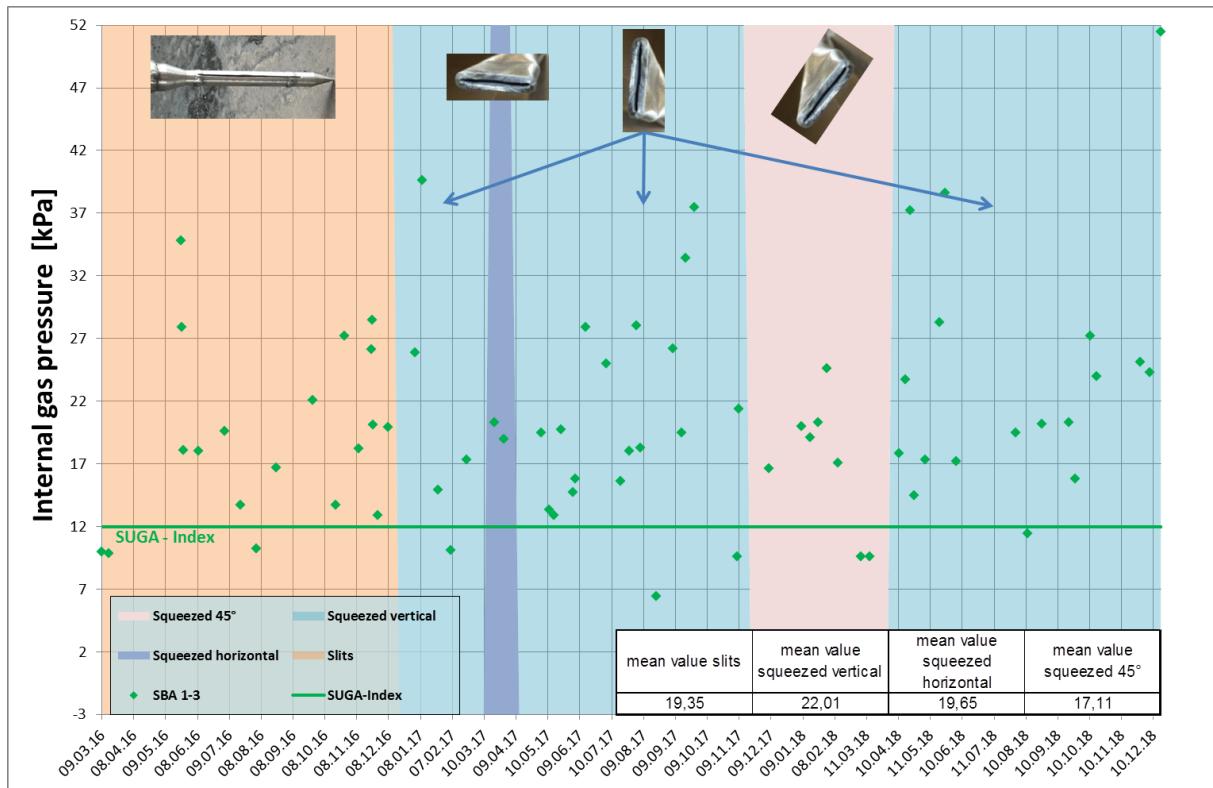


Figure 1.5.2: IGP measurements with different probe design at AGDH

For the detected IGP peaks it is obvious most of them range between 15-25kPa. There are upward outliers in every variation of the ending, by what the mean values don't show a significant difference. The lowest mean value was obtained with the 45° squeezed design with 17,11kPa, highest mean value with the vertical (0°) squeezed ending at 22,01kPa. From this perspective, regarding only the nature of the tip the best values could be obtained with the squeezed vertical probe design.

In the next figure there is given a comparison of the peak shape of all executed IGP measurements, as well as a comparison of the correlation of the IGP peak with the position of the Soprecco valve. Furthermore, the graph on the right side the obtained IGP trials into the different probe design. In total there are 83% of acceptable IGP curves for a reliable evaluation and only 17% are insufficient. In regard of the probe ending the best yield gave the squeezed one with vertical insertion angle (97%), followed by the squeezed one with 45° insertion angle (88%) and the classical design with slits (71%). The worst yield gave the squeezed one with horizontal insertion angle (50%).

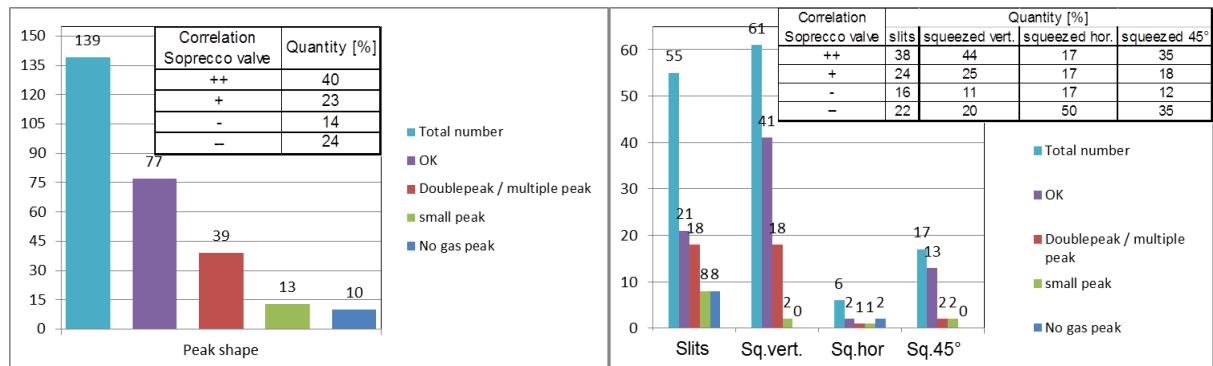


Figure 1.5.3: Comparing statics of IGP in regard of peak shape and Soprecco correlation

A similar trend can be observed in respect of the correlation of the IGP peak with the position of the Soprecco valve. 63% of the executed pressure recordings gave a good or a very good correlation with the oven valve. Differed into the various designs of the probe tip, the squeezed vertical one gave the best yield for a good or a very good Soprecco correlation with 71%. The

worst yield again goes along with the measurements with the squeezed ending with 90° insertion angle, 34% with a good or a very good coincidence.

In the next figure there is a comparison of the measured IGP values divided into the determined reliability statuses through the correlation with the position of the Soprecco valve of the oven. For a better estimation the statuses are highlighted in different colors.

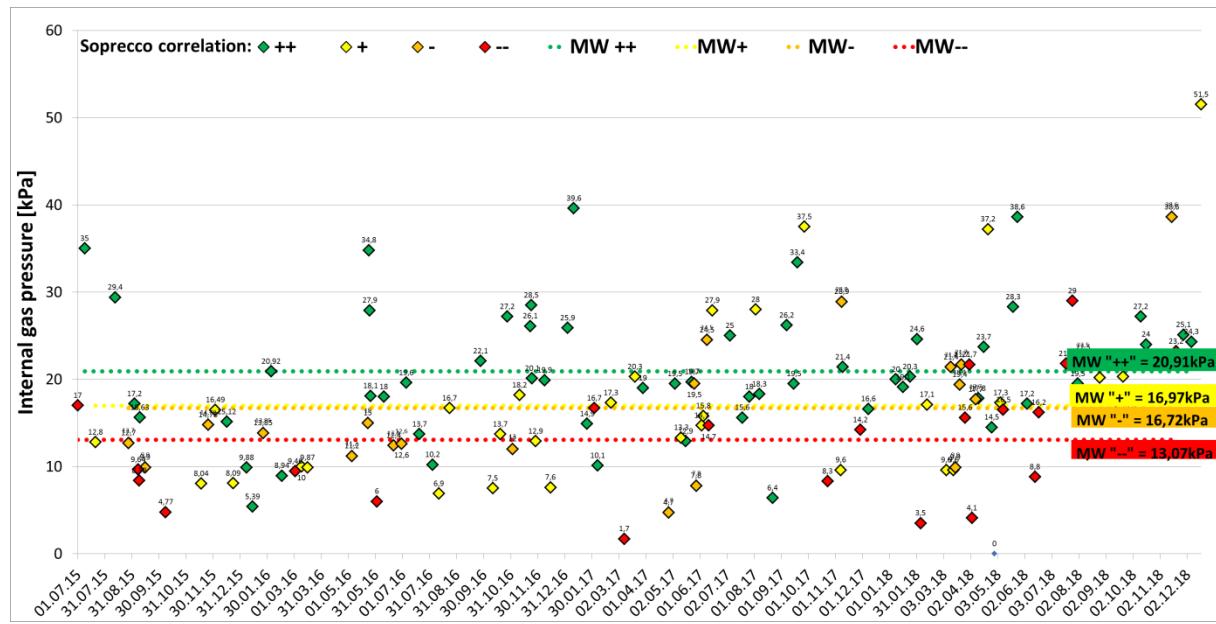


Figure 1.5.4: Comparison of IGP mean values divided into the status of the Soprecco correlation

It is obvious the better the correlation between the gas pressure peak and the position of the Soprecco valve is, the higher the mean value of the IGP peak. This is a good approval of the evaluation technique at AGDH and confirms the plausibility of the correlation of IGP peak with the position of the Soprecco valve.

Thus, from the different experience and improvement highlighted during the BINGO project, a guideline gathering the best practices used by the different partners is proposed in the following table.

Table 1.5.1.: Proposed guideline for measurement of IGP

	<b>Best Practices In Gravity Charging</b>	<b>Best Practices In Stamp Charging</b>
<b>Design of the probe</b>	Triple probe Vertical squeezed ending	Simple probe Vertical Squeezed tube ending
<b>Probe Length</b>	Under the 1 <sup>st</sup> charging hole to catch the higher bulk density	2m
<b>Probe Diameter</b>	Ø 10mm	Ø 10mm
<b>Probe Position</b>	Pusher Side	Pusher Side Probe can be inclined to prevent clogging due to water
<b>Temperature measurement in the middle of the charge</b>	X	X
<b>Probe Insertion</b>	Manually	Drilling
<b>Probe Removal</b>	Manually, specific system can be used in case of sticking	Manually
<b>Frequency of measurements</b>	1 measurement per week	1 measurement per week
<b>Recording of the data</b>	Wireless system to transmit gas pressure and temperature to the operations control room	
<b>Evaluation of the measured data</b>	<ul style="list-style-type: none"> <li>• Keep the maximal peak of Internal Gas Pressure</li> <li>• Control the shape of temperature profile in the middle of the charge</li> <li>• Measurement of heating flues temperature around the probe ending to control the gradient temperature in the charge</li> <li>• Correlation with the opening positions of the SOPRECO-valve when available</li> <li>• Correlation with the temperature of the gas inside the ascension pipe when available</li> </ul>	

## **WP 2 Investigation of parameters influencing IGP by industrial measurements**

### ***Task 2.1 - Configuration of the measurements [AMAL, AMMR, CPM, AMO]***

In the frame of BINGO, AMMR/CPM and AMAL studied the influence of the position of the probes (depth, height, Pusher Side, Coke Side) in wet gravity charging techniques at industrial scale. This investigation was completed by a comparison of different design of probe endings.

CPM and AMAL performed four successive coking tests in oven n°6 during two weeks in November 2015. Measurements were carried out through Pusher Side and Coke Side doors in Florange coke plant, at different heights above the oven sole and at different depths of insertion as presented on Figure 2.1.1. Two different lengths of probes (2.6m and 4.2m) were tested in order to study influence of the density (under or between the charging hole).

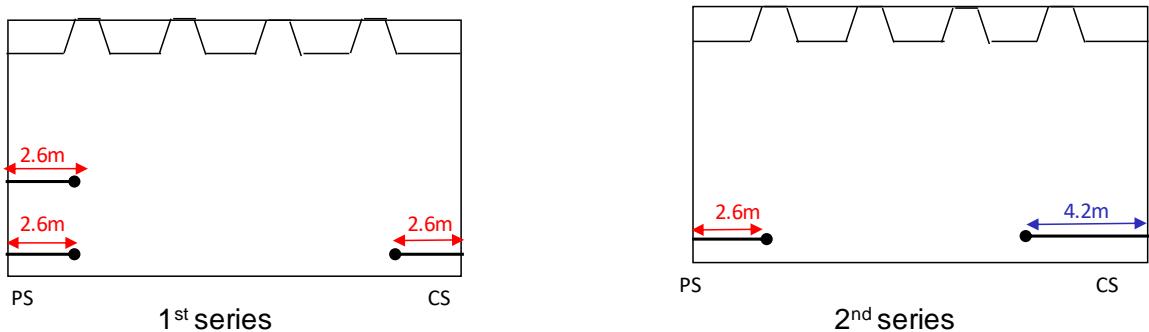


Figure 2.1.1: Principle of 2 series of measurements (1 series per week)

During the 1<sup>st</sup> series, no difficulty appeared with 2.6m long probes. Probes were generally easy to insert in the charge. Examples of obtained results are presented on Figure 2.1.2.

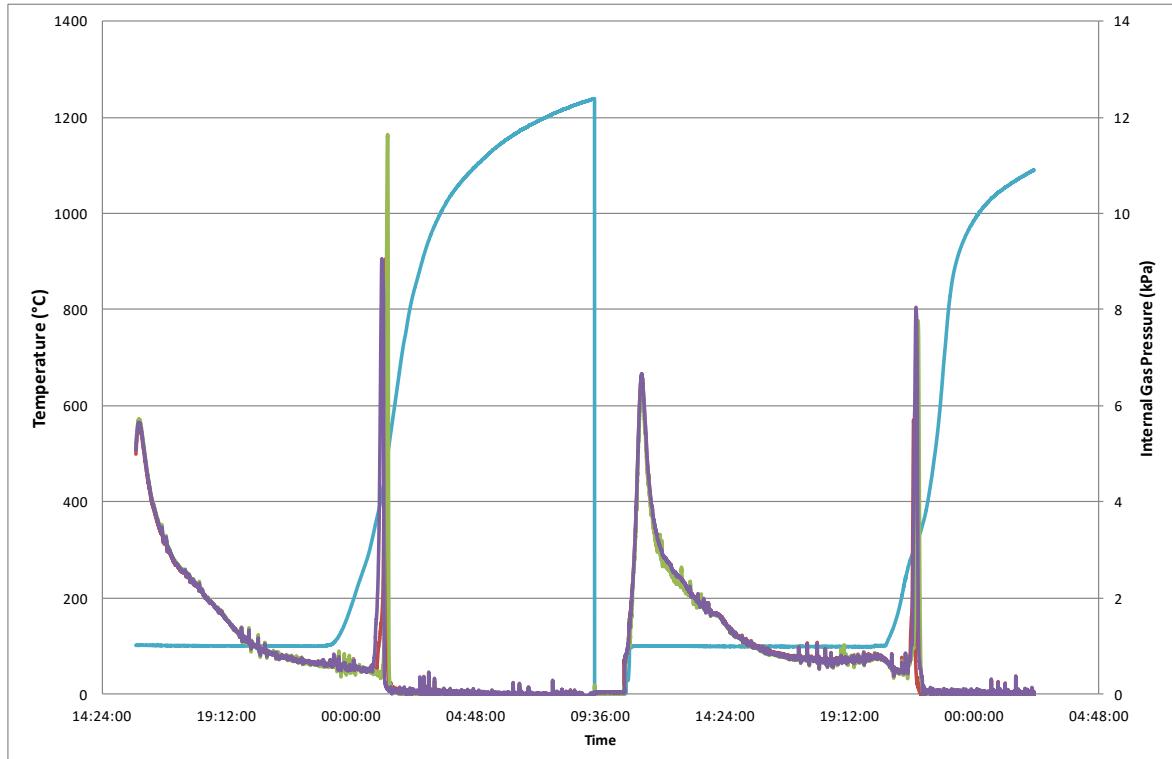


Figure 2.1.2: Internal Gas Pressure measurement at Florange coke plant during 2 cycles (50 cm of oven sole on Coke Side)

During the 2<sup>nd</sup> series, results of measurements between charging holes with 4.2m long probes were not useable since it was very difficult to insert and center such long probes in the charge. As described on Figure 2.1.3, the shape of temperature in the middle of the charge is not characteristic of a good positioning in the middle of the charge.

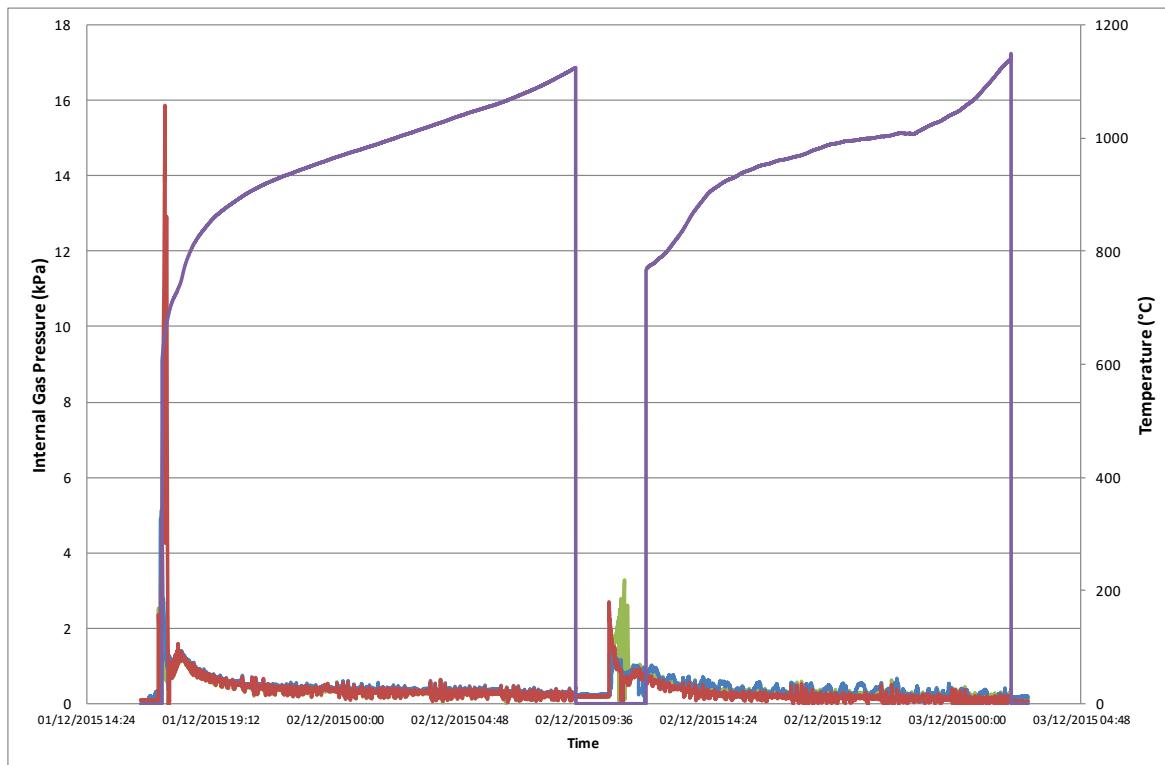


Figure 2.1.3: Internal Gas Pressure measurement during 2 cycles with probes of 4.2 m length (50 cm of oven sole on Coke Side)

After these series of measurements, results showed that values are more scattered on Coke Side without clear explanation. It is not a centering problem since pressure peaks appear at the same time. Values obtained on Pusher Side show very good reproducibility at 0.5m from oven sole (average pressure = 15.1 kPa) as presented on the following Table 2.1.1. As presented in Task 1.4, the new design of CPM probe improves the centering of the probes in the charge and allows to maintain the reproducibility.

Table 2.1.1: Maximum Internal Gas Pressure at Florange coke plant with 2.6m long probes

Probe of 2.6m	Coke Side (0.5m) kPa	Pusher Side (0.5m) kPa	Pusher Side (1.5m) kPa
Coking Test 1	11.6	13.1	8.8
Coking Test 2	8.0	13.4	10.9
Coking Test 3	19.1	17.1	-
Coking Test 4	6.9	17.9	14.3
Coking Test 5	14.5	14.0	-
Coking Test 6	-	13.5	-
Coking Test 7	-	15.2	-
Coking Test 8	7.4	16.7	-
Average	<b>11.3</b>	<b>15.1</b>	<b>11.3</b>
Standard deviation	4.8	1.9	2.8

Corporate Coke lab Ostrava completed comparative tests of probes for internal gas pressure measurement with various design. The principle of comparative testing was suggested for pilot scale when always two different probes with different design were inserted into the pilot oven during same pilot oven test from both sides of pilot oven.

Basic parameters, that were tested, were the designs of the probe's ending and comparison of single or multiple probes. Openings at the end of probe, that allow pressure transmission to the sensor, have various designs varying mainly in complexity with impact on manufacturing costs.

Various shapes of probes openings were tested in frame of Task 2.1, three tested types are shown on Figure 2.1.4.



Figure 2.1.4: Probes design tested in frame of task 2.1 – holes openings, slot opening and squeezed point of the probe

In Table 2.1.2, additional parameter was added when horizontal and vertical position of the probe's ending was compared.

Table 2.1.2: Comparison of investigated probes design

		squeeze probe		triple probe (kPa)	classical slot design (kPa)	holes endings (kPa)	delta (%)	standard deviation
		vertical (kPa)	horizontal (kPa)					
probes design								
2.1 a	MV coal	20.9		22.9			8.7	1.4
	MV coal	42.5		41.0			3.7	1.1
	LV coal	112.2		112.3			0.1	0.1
2.1 b	MV coal	11.2			11.5		2.6	0.2
	LV coal	27.5			27.7		0.7	0.1
2.1 c	MV coal	35.4				38.9	9.0	2.5
	MV coal	34.4				34.3	0.3	0.1
	LV coal	122.7				115.5	6.2	5.1
2.1 d	LV coal	95.6	91.9				4.0	2.6
	MV coal	25.2	25.4				0.8	0.1
	LV/MV coal	85.0	86.6				1.8	1.1
average							3.5	1.3

In pilot scale, value of internal gas pressure was always detected by two probes inserted into the oven doors from both sides. Development of internal gas pressure is shown in Figure 2.1.5 for MV coal tested in pilot scale under stamp charging condition, in Figure 2.1.6 for LV/MV coal and in Figure 2.1.7 for LV coal also tested in movable wall oven under stamp charging conditions.

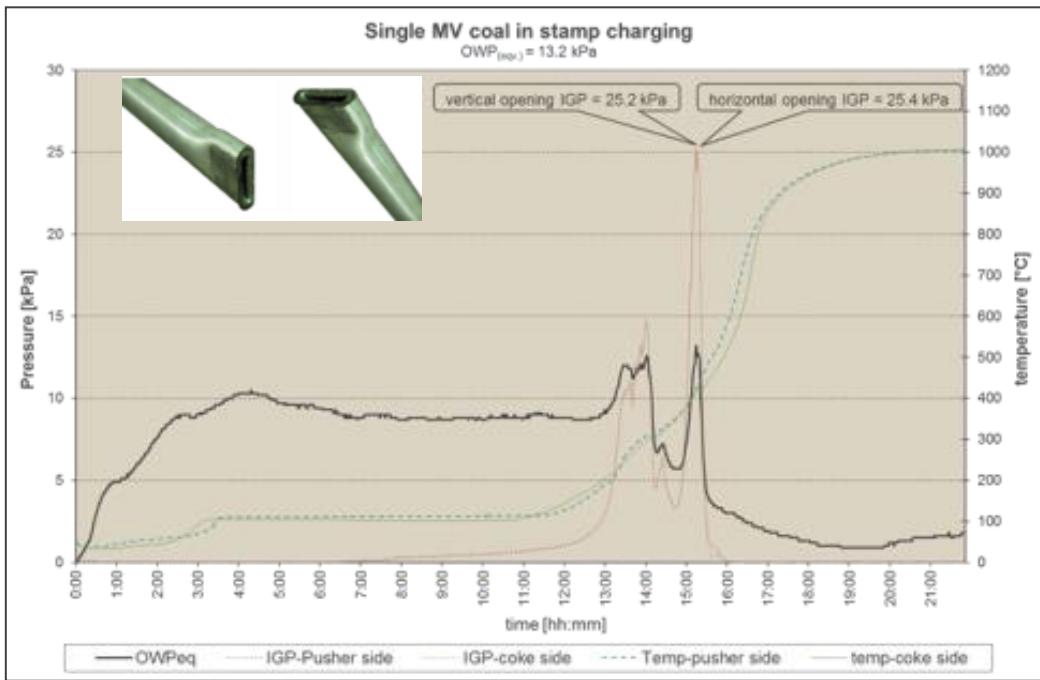


Figure 2.1.5: IGP generated by medium volatile coal in stamp charging conditions

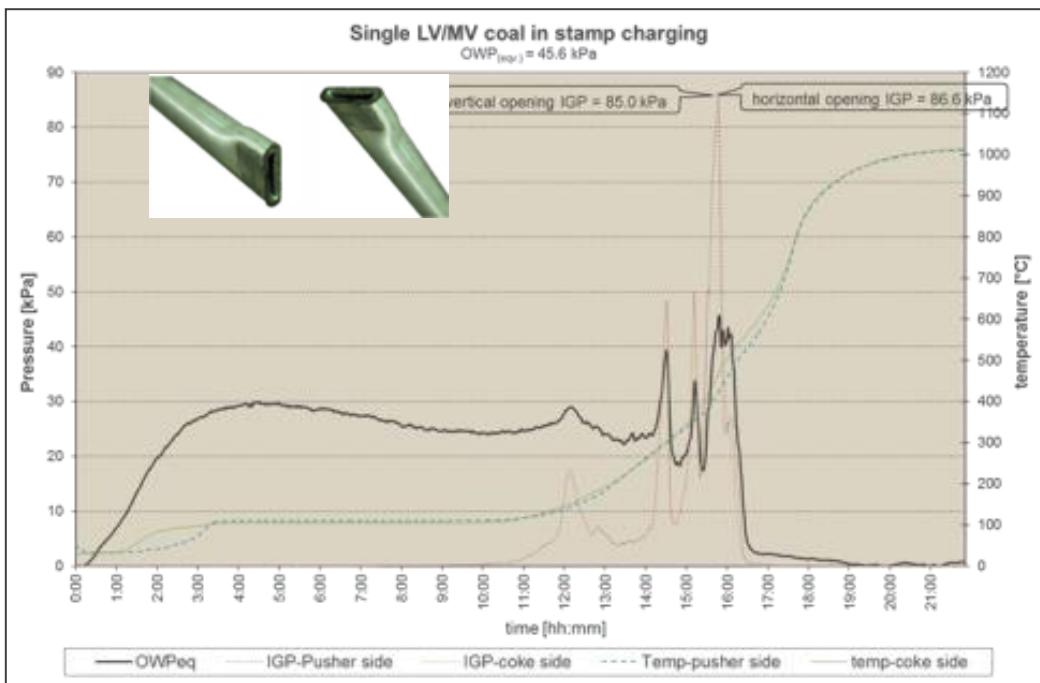


Figure 2.1.6: IGP generated by LV/MV in stamp charging conditions

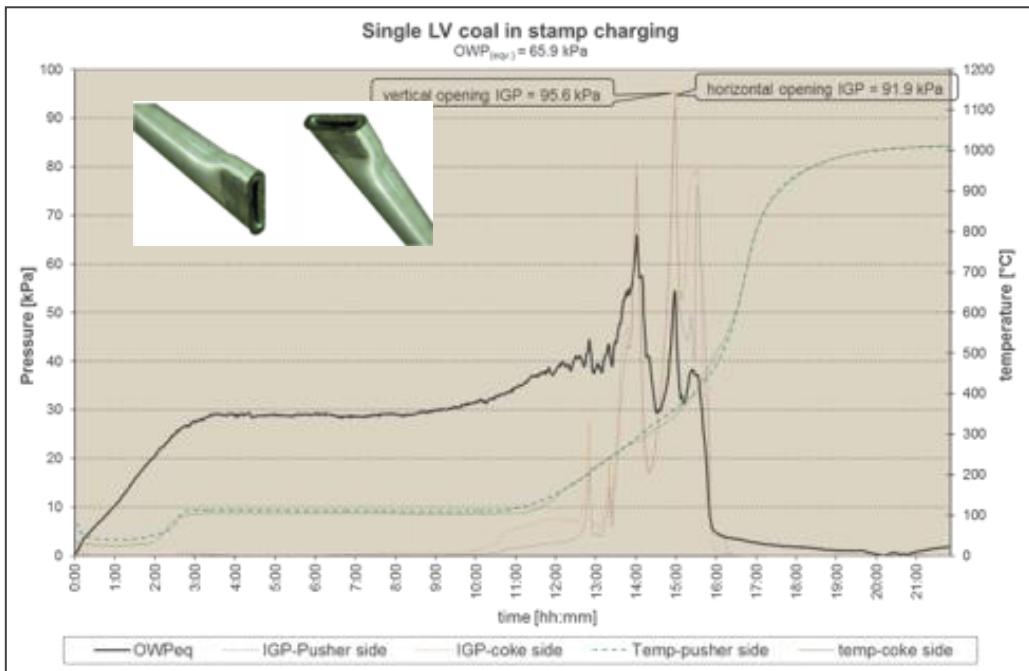


Figure 2.1.7: IGP generated by LV in stamp charging conditions

Another parameter that was considered in this investigation, was comparison of stainless steel material with commercial steel tubes which also allows evaluation of financial benefits from possible multiply re-using of the same tube. In the following Figure 2.1.8, success rate of IGP measurement in industrial scale is shown.

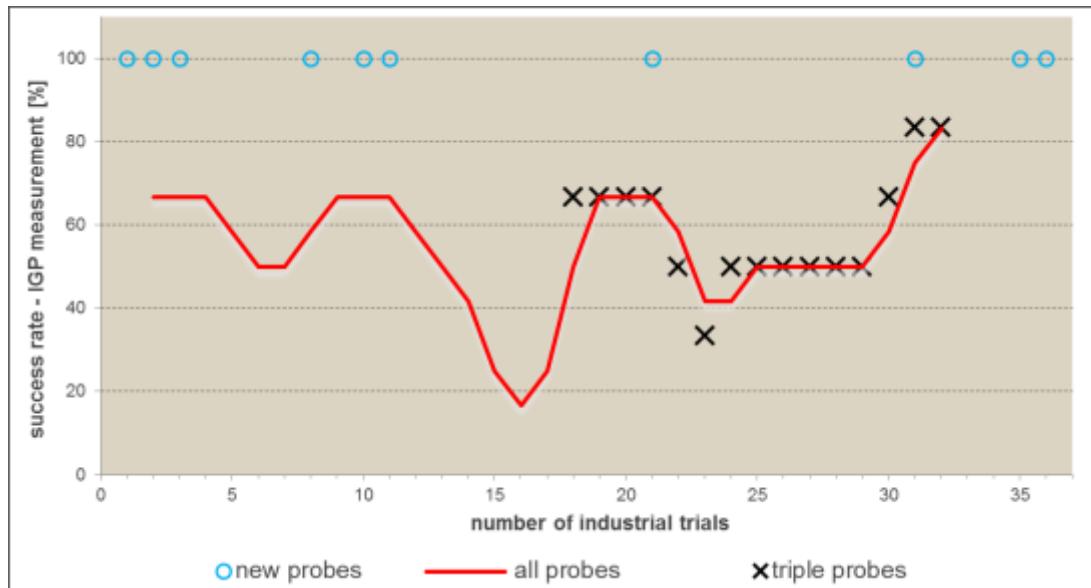


Figure 2.1.8: Success rate of IGP measurement at industrial scale

Red line represents running average from last six measurements, black daggers represent insertion of triple probe and blue circle points represent application of probe made from new tubes. A very important finding is 100% success rate of new tubes. It means that corrections of probe – welding, straightening and sealing work significantly increase the probability of wrong insertion of the probe at industrial scale. However, lower success rate of reused triple probe can be given by development of optimal probe insertion procedure, especially in case of stamp charging conditions. Growing success rate during last measurements is promising for future common application of overhauled triple probes. In aim to define the best design of the probe with a reliable procedure for probe insertion, it is necessary to carry out more trials with recently developed triple probe.

To summarize, various probes designs were tested and compared in movable wall oven. Design of probes openings does not have any significant effect on the internal gas pressure value and the

simplest design represented by squeezed probe can be used for gas pressure detection. Also, the position of probe's ending was tested in pilot scale when especially horizontal position of squeezed ending can be influenced by leakage through the semicoke beside the tar seam. Pilot oven tests comparing both horizontal and "ideal" vertical position of the probe's ending didn't prove any influence on the value of internal gas pressure.

### **Task 2.2 - Properties of coal charge [tkSE, AGDH, voestalpine]**

The following task describes different properties that influence internal gas pressure (IGP) of coal blend. Besides focusing on blend performance, the study also covers single coal properties and blending methods as variables. Indirect influences of both blending and charging techniques are discussed as well.

#### **Differences between top charge and stamp charge blends**

In the course of the project, two partners – thyssenkrupp Steel Europe (tkSE) and Aktien-Gesellschaft der Dillinger Hütte (AGDH) – conducted long-term investigations on factors influencing IGPs of coal blends. The main difference between the two partners, in terms of their coke plant facilities, was the employed charging technique. Coke plant Schwelgern (KBS), which is linked with tkSE, operates conventional top charge ovens whereas Zentralkokerei Saar coke plant (ZKS) operates stamp charge batteries. This difference has major impact on coal blending strategies of the two partners.

Table 2.2.1 outlines the ranges in selected coal quality parameters. It reflects a fairly low volatile matter (VM) target for the blends used at KBS. Low VM targets are typical for top charge batteries as compared to stamp charge batteries. The data analysis also proves that many of the borderline values are erroneous and biased by much higher than planned proportions of petroleum coke (petcoke) in the blends as can be seen from the results of volatile matter, ash, sulfur, fluidity, and vitrinite content in the analyzed samples.

Table 2.2.1: Quality variations

Parameter	
<b>Volatile matter</b>	18.1% – 21.7%
<b>Ash</b>	6.73% – 9.91%
<b>Sulfur</b>	0.62% – 1.30%
<b>Phosphorus</b>	0.03% – 0.05%
<b>&lt; 3.15</b>	80.7% – 92.5%
<b>Cone bulk density (db)</b>	560 kg/m <sup>3</sup> – 676 kg/m <sup>3</sup>
<b>Fluidity</b>	16 ddpm – 252 ddpm
<b>MRR</b>	1.20 – 1.29
<b>Vitrinite</b>	59.2% – 72.0%

Figure 2.2.1 illustrates the trend of mean maximum reflectance (MMR) of coal blends at ZKS over the project period and the corresponding IGP values. MMR of ZKS blend, which is calculated from a datasheet based MMR of single coals and their percentage, is significantly lower than MRR of KBS blends. This demonstrates that plants employing stamp charging process are typically using blends with higher amount of high-volatile coals. The reason behind is the difference in achievable charge bulk density of the two charging techniques. Typical bulk densities for top charge operations spread between 750 kg/m<sup>3</sup> and 800 kg/m<sup>3</sup> (db) whereas charge bulk density in stamp charging process can be as high as 1050 kg/m<sup>3</sup> (db), thus making it possible that even lower quality coals, when densified to very high levels, can produce requested quality of coke. Such increase in bulk density has a direct influence on blend design practice because higher bulk densities produce higher IGP values.

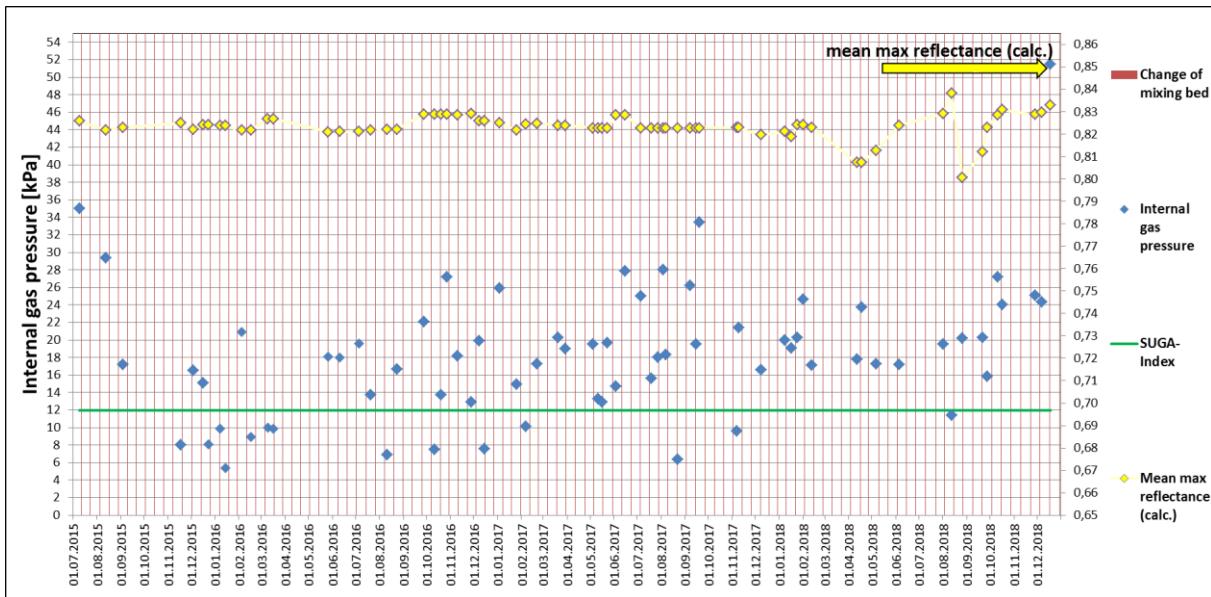


Figure 2.2.1: MMR and IGP trend at ZKS

Figure 2.2.2 illustrates IGP values of three different blends when carbonized under different bulk densities. Besides the two blends from KBS and ZKS, a third top charge blend from ArcelorMittal Ostrava (AMO) was tested. All carbonization tests were carried out in a 10-kg carbonization retort when having the charge bulk density as the only variable. The graph shows that both top charge blends produce IGP higher than 20 kPa, which is at many plants considered critical value, already at the time when they are carbonized with bulk density of 900 kg/m<sup>3</sup> (db). As a consequence, such blends would have to be modified in composition for stamp charging which is featuring with even higher charge bulk densities in coke ovens. These results were industrially confirmed in Task 2.3 at ArcelorMittal Ostrava. The easiest way of how to adapt the blend is to increase the percentage of high-volatile coals at the expense of low-volatile coals. The influence of volatile matter content on IGP performance is for single coals analyzed later in the report.

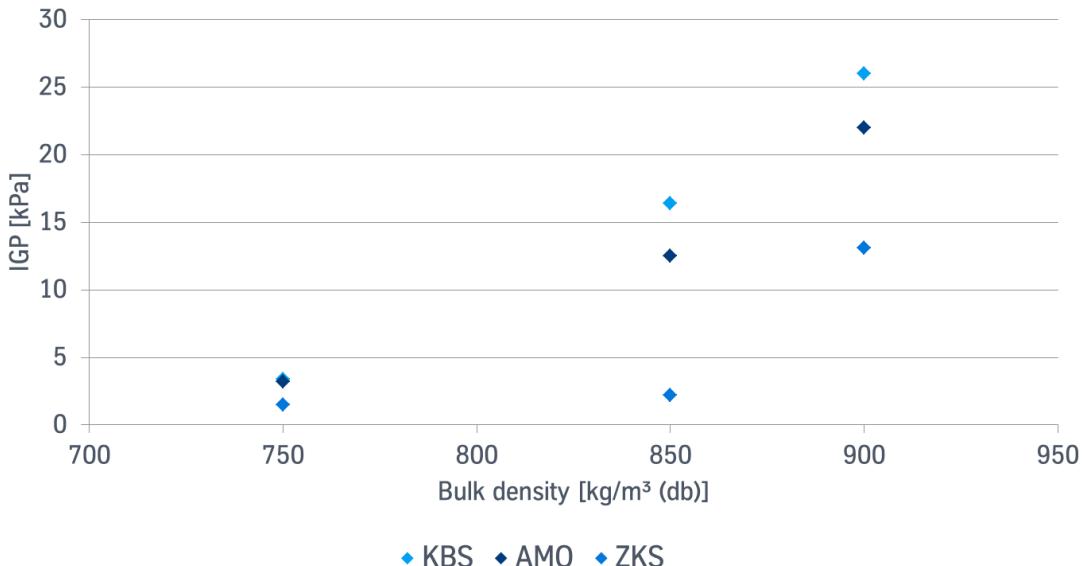


Figure 2.2.2: IGP of different blends as influenced by bulk density

Figure 2.2.3 compares IGP values obtained from investigations conducted at KBS and ZKS. It is noticeable that both plants have comparable IGP magnitude although they have different blend targets for the content of volatile matter. It shows at first that IGP targets are at these two particular plants comparable and at second, that higher bulk density negates pressure benefits gained from using higher percentage of coals with low IGP.

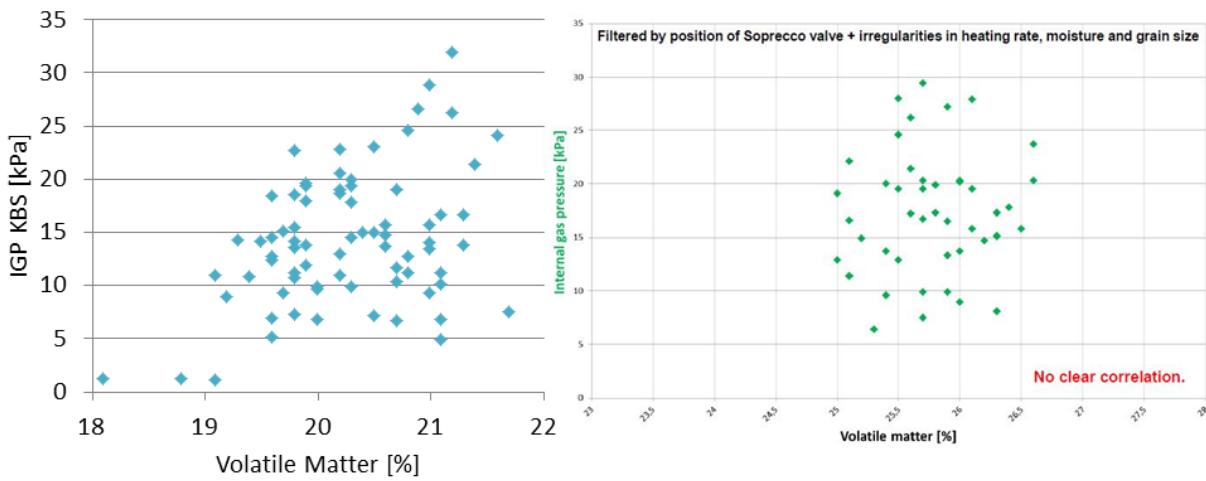


Figure 2.2.3: IGP versus VM in coal blend at KBS and ZKS

### Influence of blend preparation method on homogeneity of coal charge

There are several concepts used in preparing a coal blend from single coals but in general, majority fits to two framework concepts. In the first one a blending pile (known as mixing bed as well) is built from single coals which are stockpiled upon each other in corresponding number of layers representing the percentage of single coals in the blend, e.g. Chevron method. The second method is so called bunker blending: single coals are after pre- and/or final crushing stored in bunkers out of which they are dozed using weighing cells on a conveyor belt underneath.

tkSE has carried out an investigation to identify parameters that might influence IGP levels as measured at Schwelgern coke plant. The first measurement used for the investigation was the routine IGP measurement, which is by rule executed in the middle of the 7-to-8 day period during which a particular blend is charged into coke ovens. The second measurement was carried out approximately one day before the end of the period. In 2017, 25 mixing beds (blends) were tested, which in combination with tests executed in 2016 produced a total set of 44 tested blends. IGP results obtained from both sets of measurements are illustrated in Figure 2.2.4, in which blue bars represent IGP readings taken in the middle of the blend-use periods and yellow bars represent the readings taken shortly before every particular mixing bed was completely used.

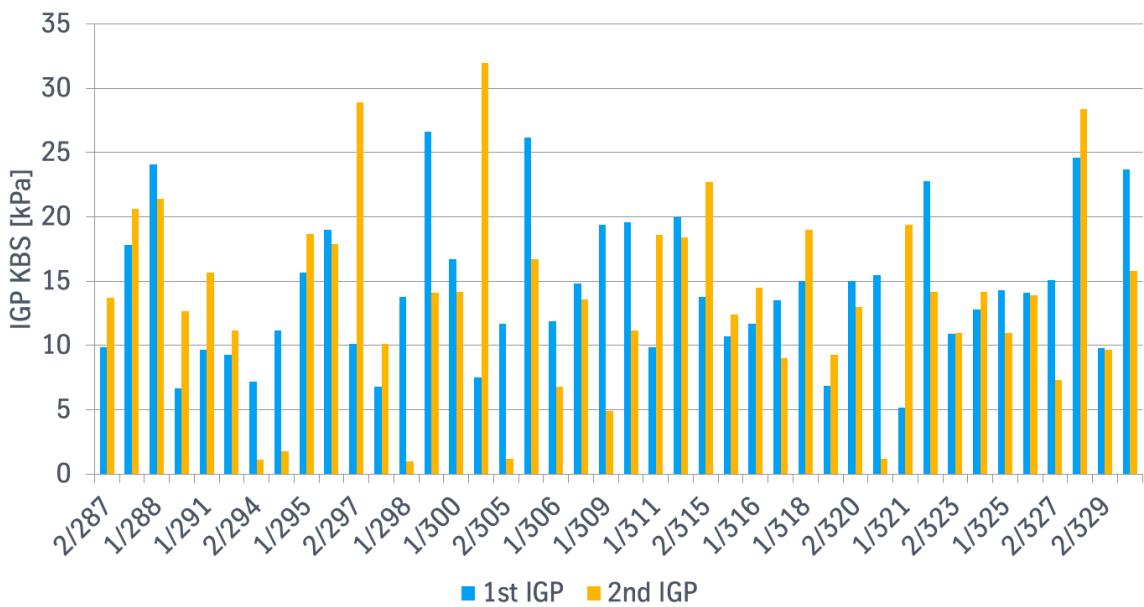


Figure 2.2.4: IGP measured at KBS

The bar chart shows that there are sometimes very significant fluctuations in IGP values for a given blend. Such findings are in majority of cases supported by chemical analysis that also shows measurable blend quality variations. Figure 2.2.5 illustrates the difference between the first and the second measurement for each of tested blends denominated by blend number on x-axis. The figure clearly demonstrates that blending piles which are built using Chevron method are subject to

indefinable quality (thus IGP) variations without any remarkable trend applicable to the end, and hence expectably to the beginning, of a piling period. At the end of a blend-use period, both positive and negative outliers can be identified. The maximum difference between the readings taken in the two sets of measurements was 39 kPa with the absolute mean difference of 6.3 kPa. Such observations support the conclusion that a single IGP measurement per blending pile (mixing bed) is not sufficient to describe the IGP potential of a certain blend.

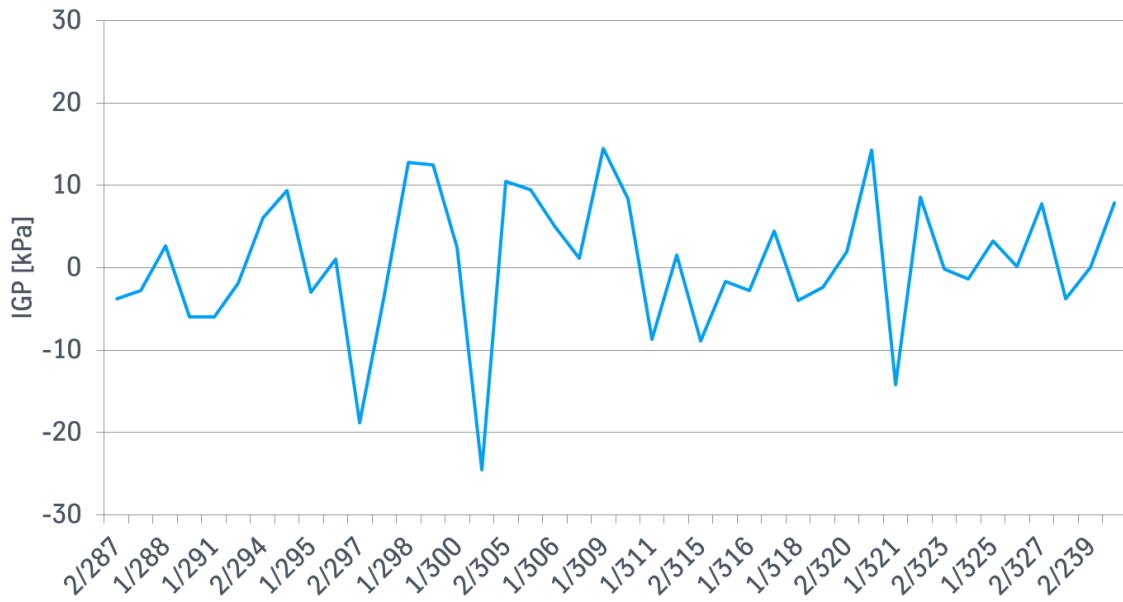


Figure 2.2.5: Differences between the first and the second measurement per blend

Besides the measured values and results, the recorded IGP curves were also analyzed in detail in order to identify biased IGP readings caused by plugged IGP probes. This analysis delivered two to three IGP measurements featuring a barely identifiable IGP peak as well as low IGP values of around 10 kPa. Given the blend composition, the IGP potential for the blends in subject was much higher.

An example of a well representative IGP pattern is shown in Figure 2.2.6 which depicts the curve derived from the measurement taken in the middle of the blend period 1/29/1 at KBS.

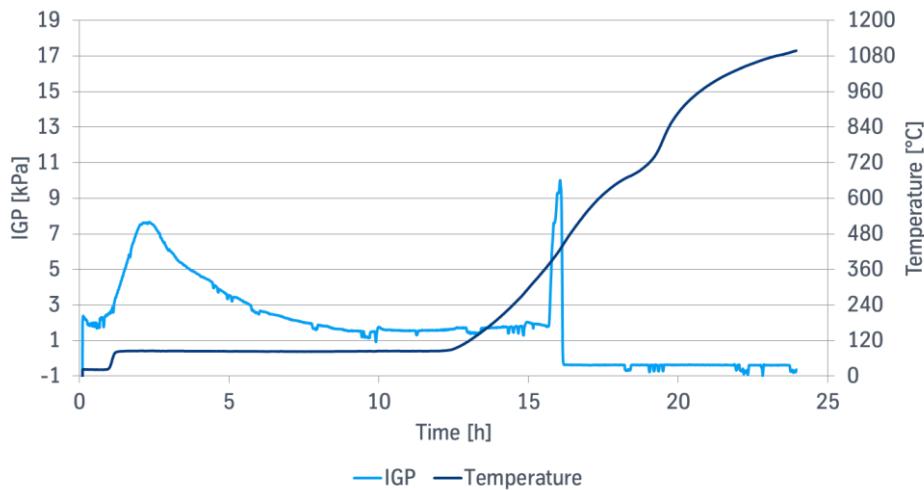


Figure 2.2.6: Proper IGP pattern

On the contrary, Figure 2.2.7 shows an example of a pressure reading indicating issues with partially plugged IGP probe.

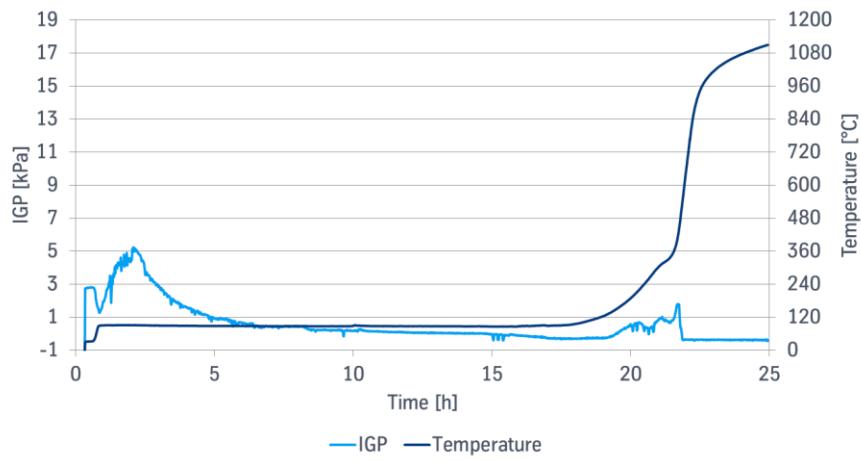


Figure 2.2.7: IGP curve with barely identifiable pressure peak

Such observations lead to a simple conclusion that one IGP analysis per mixing bed is not sufficient to identify the IGP potential of the blend.

Not only Chevron method is causing blending irregularities. The system of blending by employing bunkers can also produce biased IGP values resulting from typical bunker discharging problems that have an impact on blend homogeneity. As with other bulk materials, discharging of coal from the bunker can be affected by so called core flow inside the bunker with resulting negative influence on the quality of discharged coal. Mechanical and cohesive arches can have similar negative influence on material flow. Another factor to be taken into consideration is the performance and accuracy of weighing cells underneath the bunkers. Figure 2.2.8 demonstrates the fluctuation of IGP results obtained in certain period of time with a constant blend composition at the coke plant of voestalpine where bunker blending is used for blend preparation. Apparently, inhomogeneity of the blend can be considered an influencing factor contributing to other conditions producing such remarkable IGP fluctuation. For both blending methods, regular homogeneity checks should be applied to avoid large number of ovens being charged with potentially critical blend compositions.

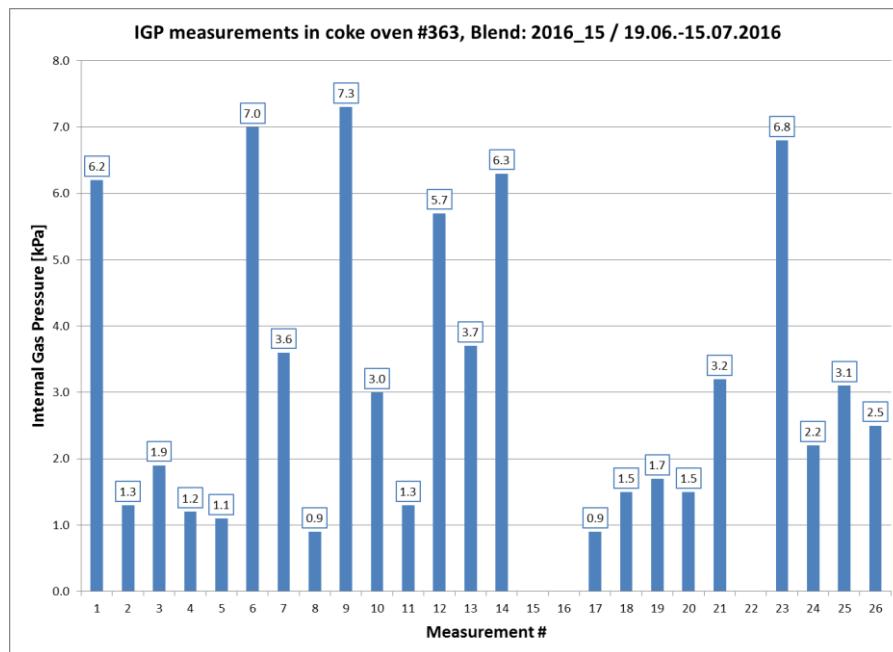


Figure 2.2.8: IGP results from a single blend at voestalpine

### Single coal characteristics

Besides being dependent on operational conditions, IGP of a coal blend is also significantly dependent on the characteristics of single coals used, irrespective of the fact that IGP is not an additive parameter. Therefore, in parallel with designing and/or reviewing the performance of a coal blend, an investigation should also focus on certain quality characteristics serving as IGP

indicator. The first parameter to look at is volatile matter content in coal. The assumption is that the lower volatile matter content, the higher IGP (and vice versa). This can be observed from the measurements carried out at voestalpine using their 10-kg carbonization retort. Figure 2.2.9 demonstrates that with volatile matter content above ca 20%, any critical IGP should be expected. Lower volatile matter contents typically led to a significant increase in IGP. Statistical modeling shows a high impact of volatile matter content on IGP results. This could imply that a very solid and direct relationship between volatile matter content and IGP exists. However, Figure 10 presents a bit different picture. Figure 2.2.10 plots volatile matter contents with corresponding IGP values which were already presented in Figure 2.2.4. In this particular case the figure shows the results obtained from KBS blends and it indicates that the rule of "the higher volatile matter content, the lower IGP" does not entirely work and cannot be as such directly applied to coke plant operations. Under KBS conditions there are many readings where higher IGP value corresponds to higher volatile matter content in the blend. What is also noticeable is that the largest spread of IGP can be observed around 21% volatile matter content. The occurrence of a wide range of IGP values at comparable content of volatile matter suggests there is another coal quality parameter having direct influence on IGP.

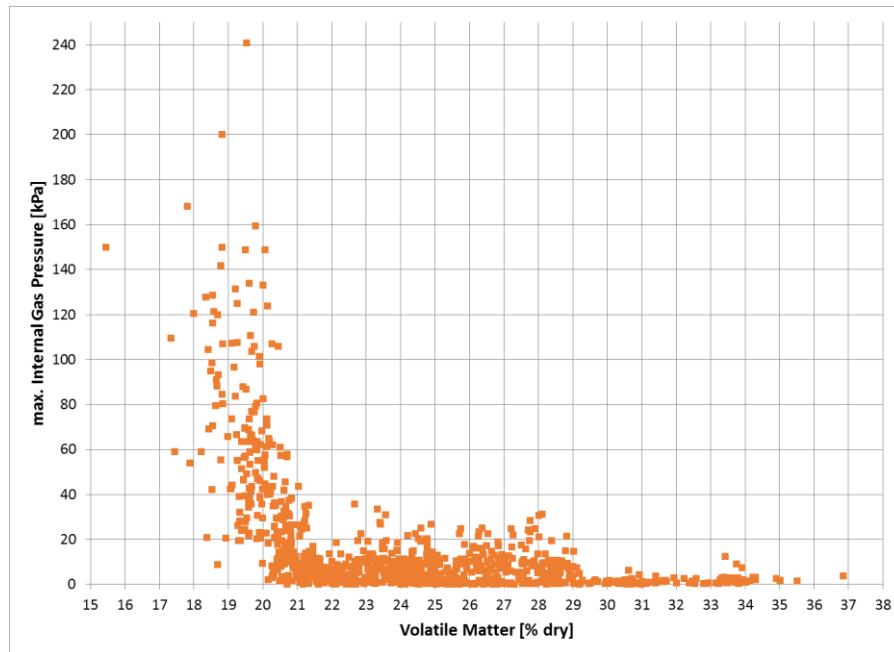


Figure 2.2.9: IGP versus VM in coal for single coals

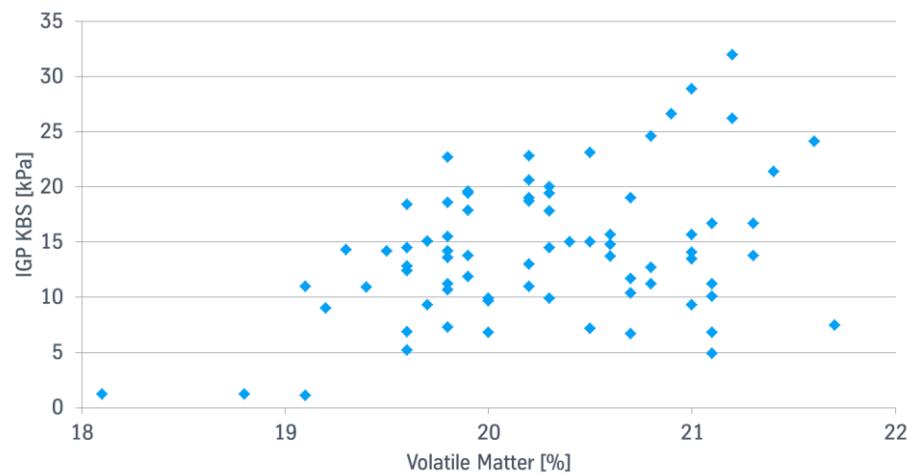


Figure 2.2.10: IGP versus VM in coal blend at Schwelgern coke plant

Having this in mind, tkSE studied the results achieved from carbonization tests of single coals in 10-kg carbonization retort. Before any detailed analysis of the results is executed it should be pointed out that the most remarkable difference between test conditions used at voestalpine and tkSE was the bulk density of the analyzed coal. While the lab at voestalpine tested the coal at about  $750 \text{ kg/m}^3$  (db), tkSE lab compacted the coal to ca.  $850 \text{ kg/m}^3$  (db). The difference has its

origin in different dimensions of industrial ovens at parent coke plants to which the laboratories serve. However, this should have no impact on interpretation of the results as far as IGP-influencing coal parameters are concerned.

Evaluation of the results at tkSE showed the influence of a second parameter. The one which does not belong to chemical data; instead, it describes the content of inert in coal, i.e. the content of inert macerals viewed from the perspective of petrographic analysis. The value itself represents the sum of all types of inert macerals and the non-reactive portion of semifusinite in coal. The influence of inerts on IGP is shown in Figure 2.2.11. With inert content higher than 20% there is any IGP value exceeding 50 kPa. The figure thus illustrates that with comparable content of inerts the volatile matter content can indicate whether a coal may produce elevated IGP or not. Or similarly, with comparable volatile matter content the content of inerts can become the indicator. The studies have made it clear that in addition to chemical analysis the maceral composition of coal should be tested on a regular basis. Even marginal change in the inert content can lead to noticeable IGP change irrespective of consistent chemical composition.

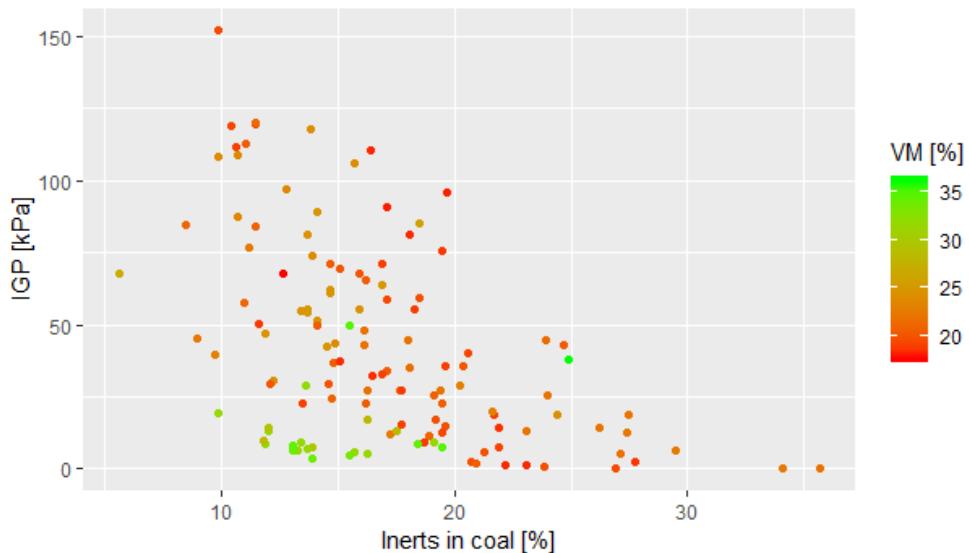


Figure 2.2.11: IGP versus inert content in single coals with reference to VM content

To combine these findings with the influence of bulk density, which was already discussed during the comparison of top and stamp charging, three coals with nearly the same content of volatile matter but with different percentage of inert constituents were tested at four different bulk density levels in a 10-kg carbonization retort. The results are plotted in Figure 2.2.12. The figure demonstrates that lower amount of inerts can lead to significant increase in IGP, and as a result, blends with very comparable volatile matter content can produce different IGPs. Such findings emphasize that especially for stamp charge batteries as well as top charge operations with tall ovens the inert content of low volatile components in a blend can significantly influence IGP performance.

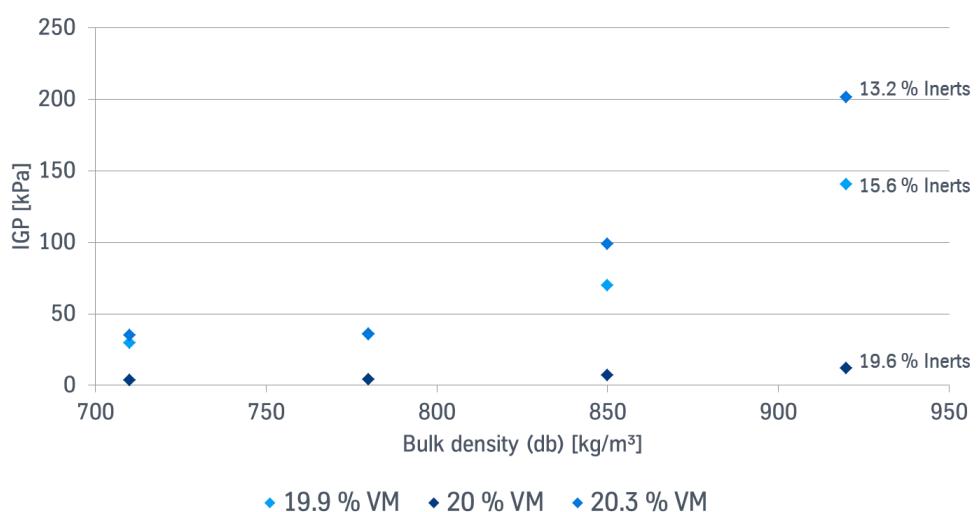


Figure 2.2.12: IGP versus inert contents at different bulk densities

Given the knowledge that chemical and petrographic parameters can influence IGP, tkSE tried to develop a multiple linear regression model for prediction of IGP of single coals. The first approach was to calculate IGP from the results of carbonization tests in 10-kg retort using coal chemical and petrography analysis data. They used a dataset containing the results of 130 single coal tests that were carried out under standard operating conditions. It was assumed therefore that the only influence on IGP came from the tested coal. The developed model was based on employing linear regression and at this stage it included only numeric parameters. Categorical parameters such as coal origin were not captured. The first results presumed that the origin factors can be covered by chemical parameters. Table 2.2.2 shows all relevant parameters of the model. The model included the parameters of volatile matter content and inert content, both of them being already addressed. It also included multiple V-types. The results supported the idea of considering volatile matter content as an influencing factor because of a direct relationship between volatile matter content and V-types. Sometimes though, the V-type distribution added additional information, in particular when already-blended coal products were used at the coke plant.

Table 2.2.2: Relevant parameters of single coal IGP prediction model

Parameter	Factor
<b>Intercept</b>	-185.28
<b>Volatile Matter</b>	3.32
<b>Sulfur</b>	91.78
<b>Phosphorus</b>	285.71
<b>V2O5 in ash</b>	223.19
<b>Reactive/Inert</b>	2.09
<b>V9</b>	-1.16
<b>V10</b>	0.76
<b>V11</b>	0.27
<b>V12</b>	1.02
<b>V13</b>	1.75
<b>V14</b>	0.53

It is conspicuous that besides the expected parameters, some chemical elements also seem to have influence on IGP. However, this could be spurious correlation and, the influence comes from the origin of the coal. Figure 2.2.13 shows a diagram of sulfur content in coal sorted out by coal origin. The figure illustrates that tested coals from Mozambique and USA have high sulfur contents. These coals are also the coals developing high IGP values. Further studies should therefore include suitable categorical factors in order to cover the influence of single elements in a more specific way.

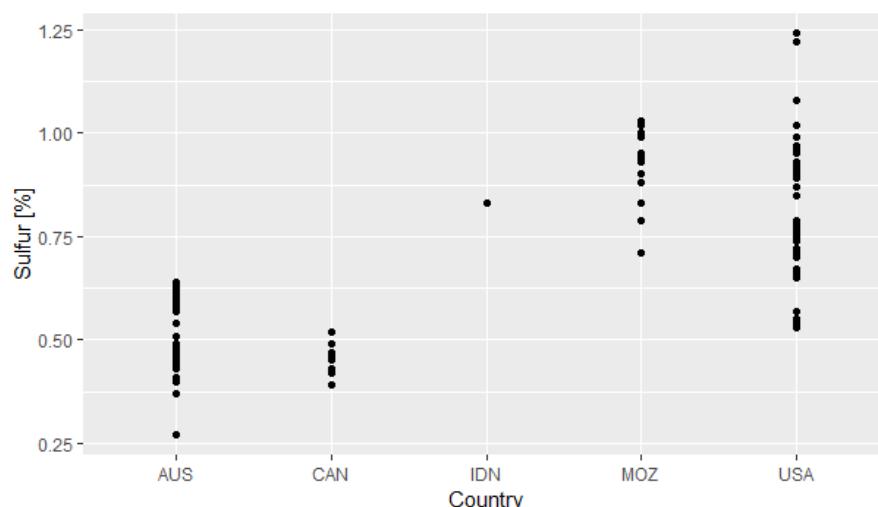


Figure 2.2.13: Sulfur content in single coals sorted out by country of origin

Figure 2.2.14 gives a graphical interpretation of correlation between the measured and predicted IGP values. The model delivers a clear trend; hence, it can be used for initial predictions at tkSE. The  $R^2$  of the model is 0.84 so that at this stage the model can be considered good starting point for further development. It is important to mention that the model is developed from the set of data collected solely at tkSE and obtained under test conditions specific to tkSE.

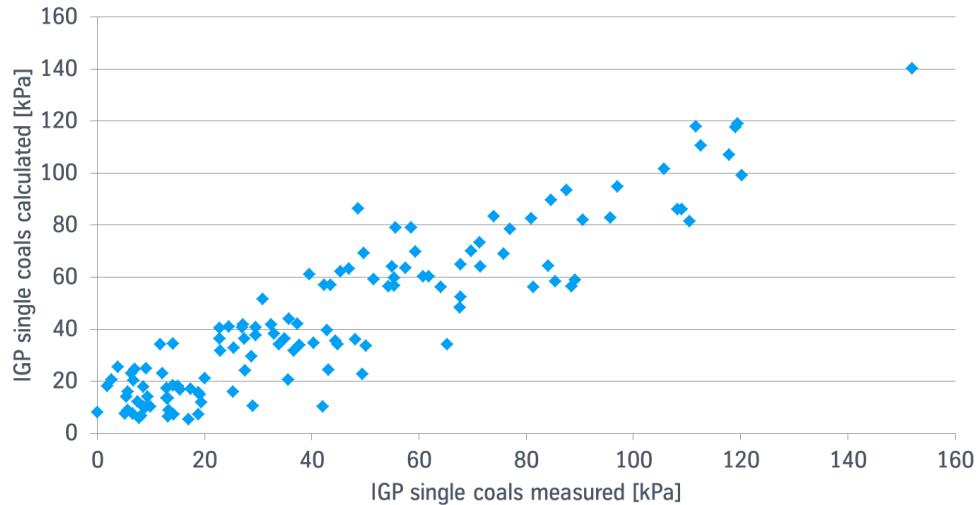


Figure 2.2.14: Measured and calculated IGP of single coals

Model optimization will continue with a test phase after which additional parameters will be included providing the test phase delivers positive results. Particular focus will be laid on including rheological and carbonization parameters, the former being represented as for instance by dilatation and fluidity of coal. Different combinations of analysis data coming out of the two standardized laboratory test methods will be investigated. Furthermore, the shrinkage of coal as measured in the sole heated oven will be tested in the future as additional IGP-influencing factor. The company voestalpine can already demonstrate that the shrinkage of coal belongs in their dataset to remarkably high impact factors influencing IGP values. Therefore, further investigation of the relationship between the two parameters should be considered.

#### Influence of inert material on IGP

To support the conclusions regarding single coal characteristics such as the influence of inert content, the evaluated KBS blends were also analyzed by petrographic methods. Maceral analysis can be used as a tool suitable for providing evidence on the content of petcoke in the analyzed sample. Maceral analysis was carried out once per blend and the results obtained are shown in the form of the percentage of petcoke in the blend plotted against IGP results in Figure 2.2.15.

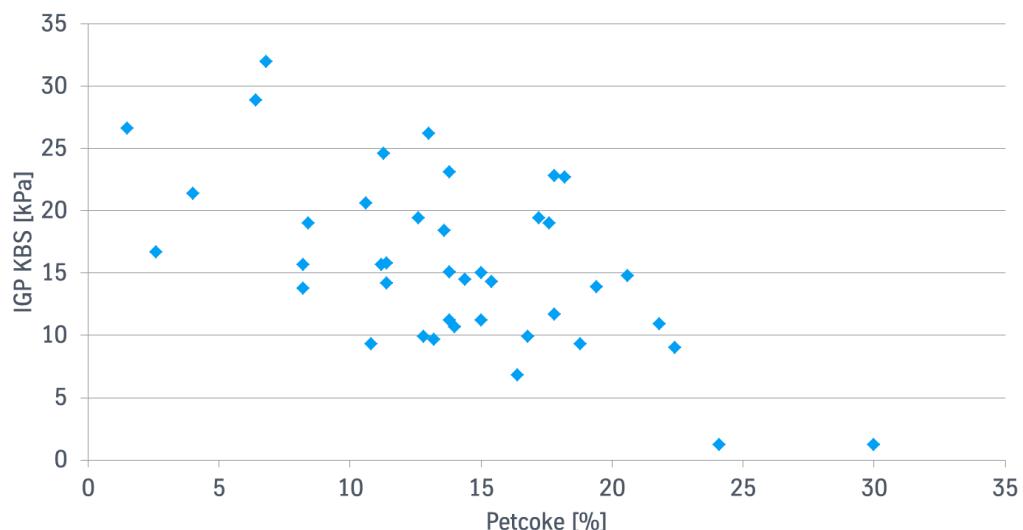


Figure 2.2.15: Relationship between IGP and petcoke content

The graph shows certain relationship between petcoke content in the blend and IGP. Over the investigated period there was a target of 10% to 14% petcoke in the KBS blend. Apparently, many IGP results fall outside this range due to several reasons such as logistics and segregation. What also becomes clear from the graph is that with a lower percentage of petcoke in a blend one can observe IGP values above 20 kPa. Such results exceed the limit value which has been set for IGP at Schwegern coke plant. On the other hand, much higher content of petcoke in the blend was found in blends producing IGP levels of around 10 kPa and lower. As a conclusion, the petcoke content in a coal blend can also be considered significant factor influencing IGP.

Besides petrography based verification, the petcoke content can also be determined by chemical analysis of vanadium content in the blend. In this particular case, vanadium serves as tracer for petcoke in a blend, which is only possible due to differences in the genesis of coal and crude oil, the latter being raw material for petcoke. The relationship is demonstrated in Figure 2.2.16.

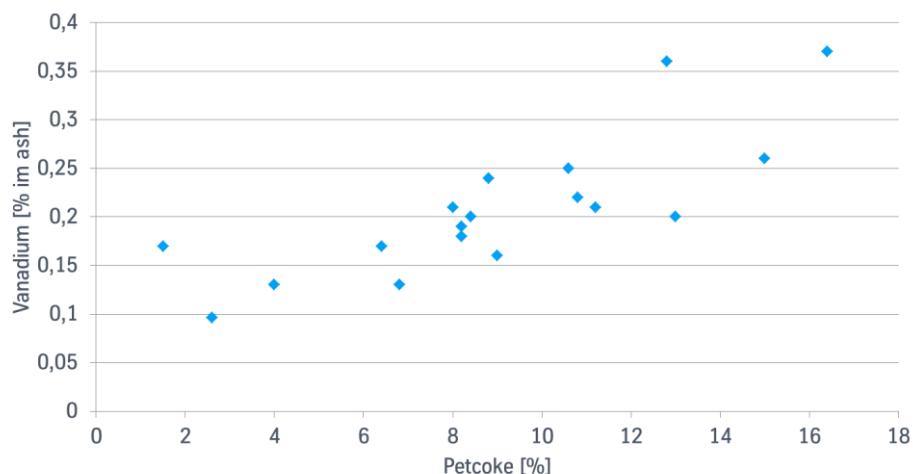


Figure 2.2.16: Relationship between vanadium and petcoke content in KBS blend

Figure 2.2.15 includes many data points in which the petcoke content falls outside the targeted blending range of 10 – 14%. Such outliers clearly represent blending deviations. However, it is also obvious that even within targeted petcoke content in the blend the variation of IGP ranges between 10 kPa to 25 kPa. It leads, in combination with the above discussed findings for single coals, to the assumption that the total content of inert material in the blend influences its IGP performance. Therefore, the contents of coal reactives, coal inerts, and petcoke in various KBS blends are plotted in a ternary diagram (Figure 2.2.17) in order to visualize their interdependencies.

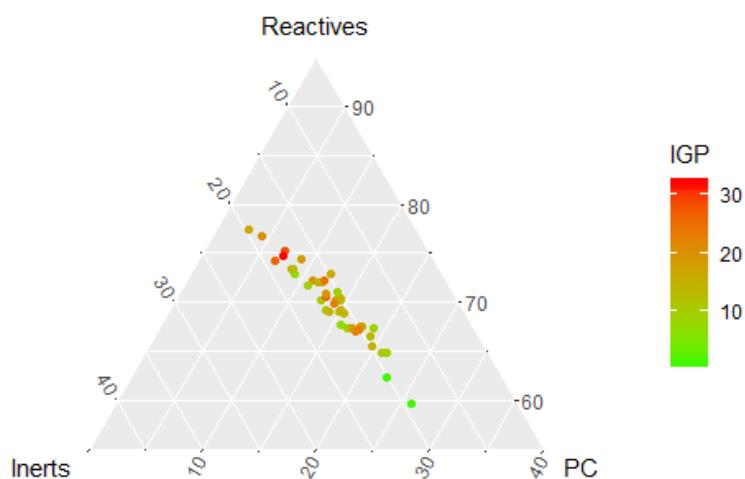


Figure 2.2.17: Ternary diagram for coal reactives, inert, and petcoke content related to IGP of KBS blends

Figure 2.2.17 supports the obtained observations concerning the influence of petcoke on IGP but the figure also shows that blends with significantly high IGP values also have high contents of reactive components. Therefore, the missing inert content was not substituted by inert content from coal that elevated the IGP. The diagram only covers maceral data and does not give any direct indication of volatile matter content, which could also have an influence on IGP. Based on this, V-type distribution should also be considered as petrographic data and taken into account in order to describe the influence on IGP. The V-type distribution can be correlated, as mentioned before, to volatile matter content. Therefore, tkSE tried to carry out a multiple linear regression having solely petrographic data on macerals and V-type distribution included. The decisive factors for this model are summarized in Table 2.2.3.

Table 2.2.3: Relevant parameters of KBS blend IGP prediction model

Parameter	Factor
<b>Intercept</b>	-88.80
<b>Vitrinite</b>	1.17
<b>V 6.0-6.5</b>	-36.49
<b>V 9.0-9.5</b>	5.25
<b>V 9.5-10.0</b>	-1.91
<b>V 11.5-12.0</b>	0.98
<b>V 13.0-13.5</b>	1.57

The table shows that the most significant parameters are vitrinite content and various V-types, which can be interpreted as a distribution of coals with different ranks. The model has at this stage rather low  $R^2$  of 0.53 (Figure 2.2.18), nevertheless, it can definitely be optimized by integrating additional parameters as much as database enlargement. However, even at this stage the prediction model demonstrates how important the control of petrographic factors is for the purpose of controlling IGP in industrial ovens. IGP predictive models based on operation parameters was also studied in Task 3.3 by voestalpine and AMO.

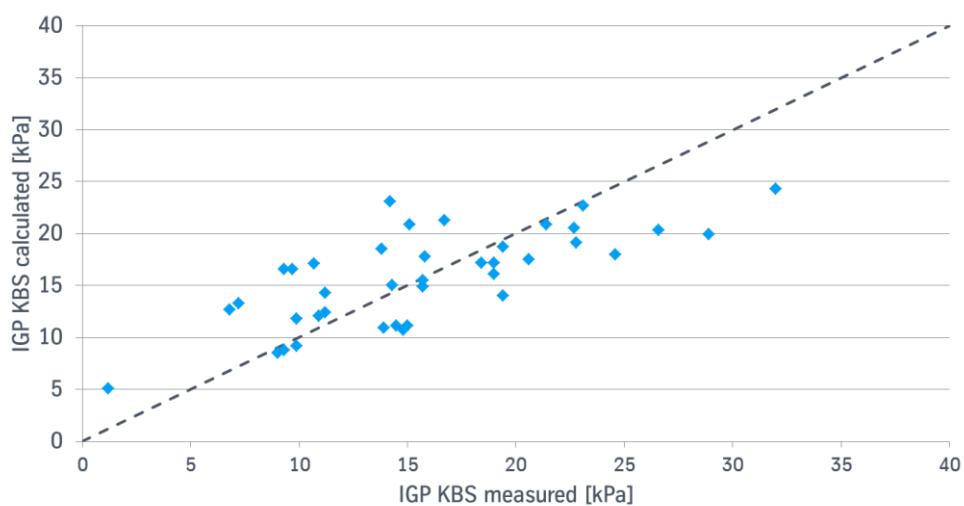


Figure 2.2.18: Measured and calculated IGP of KBS coal blends

#### **Task 2.3 - Coke oven operations & design [AGDH, AMMR, AMAL, CPM]**

This Task 2.3 was devoted to study influence of coking rate, moisture, grain size as well as charging methods on Internal Gas Pressure results during the whole project.

#### **Operating factors**

In order to achieve target grain size of the coal blend, ZKS operates normally two coal grinders in a row. However, if one of these two grinders is defective or in maintenance and only one is in operation, the grain size of the coal blend becomes coarser. Influences of a coarser grain size on the stamping density and consequently on the IGP has been investigated over the period of the BINGO project. Figure 2.3.1 exhibits the correlation between the grain size and the IGP measurements. Furthermore, the data is matched with the downtimes of the two hammer mills. Influencing parameters on IGP like heating rate, moisture or differences in the heating wall temperatures have been excluded. There are two encircled periods in which only one grinder was in operation. Therefore, the grain size of the blend got coarser and the measured gas pressure rose. The grain sizes of the used blends vary in a typical range of  $\pm 5\%$  and the resulting internal gas pressure was recorded with maximum values up to 25kPa.

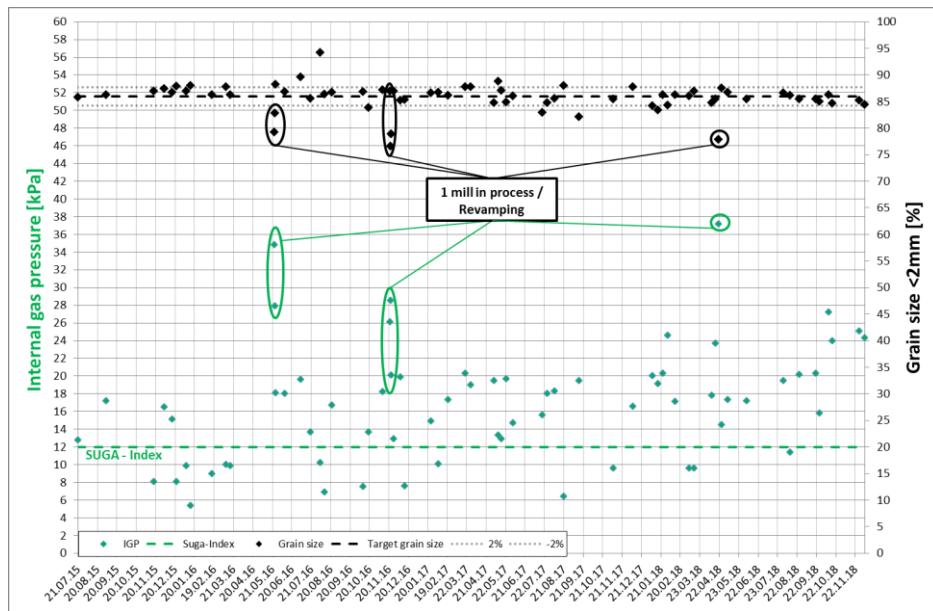


Figure 2.3.1: Correlation of IGP and grain size of the blend from Jul 2015 - Dec 2018

In Figure 2.3.2, there is the direct correlation between internal gas pressure and grain size of the used blend given. The influence of a low grain size and the resulting higher gas pressure is seen very well. Furthermore, the higher gas pressure values can easily be associated with the downtimes of the hammer mills.

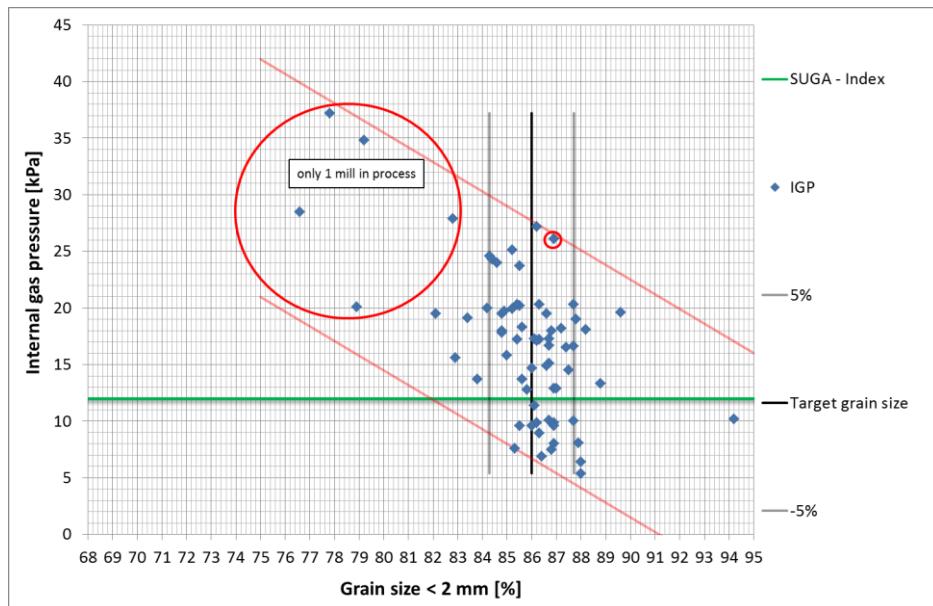


Figure 2.3.2: Correlation of IGP and grain size of the blend from Jul 2015 - Dec 2018

A detailed look onto the relationship between the internal gas pressure and the grain size is taken in Figure 2.3.3. Especially the impact of the operational deviation of the grain size in coke plant operation on the measured gas pressure values is considered. The variation of the used size of the coal is limited in stamp charge operation. To coarse blends show a high risk of wall pressure as an effect of the higher coal cake density. To fine coal blends bare the risk of a lack of stability of the coal cake. Therefore, the grain size is kept in a certain operation limit. As highlighted even this moderate variation of  $\pm 2\%$  of the target grain size ( $86\% < 2\text{mm}$ ) results already in a difference in IGP of 5kPa.

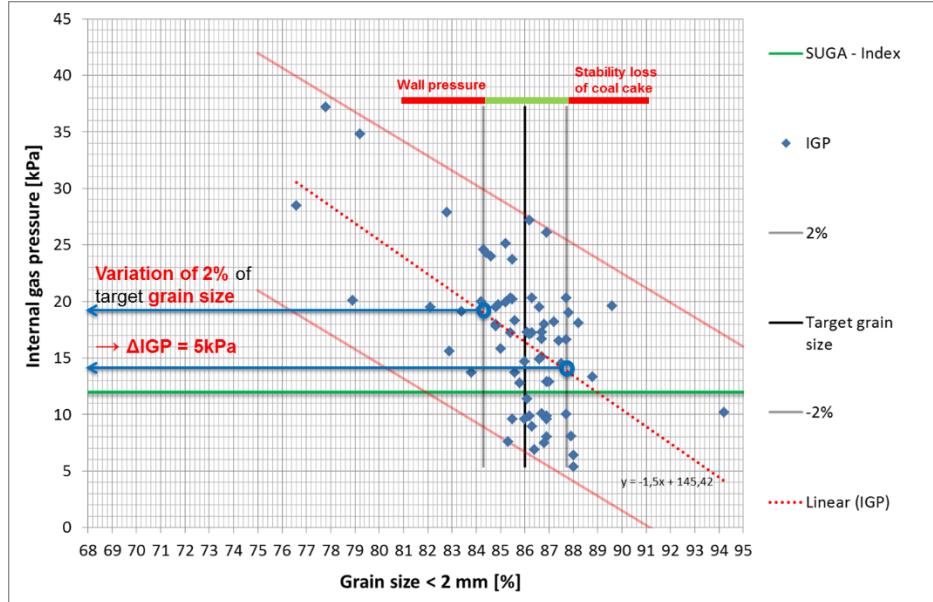


Figure 2.3.3: Variation of IGP with grain size of the blend from Jul 2015 - Dec 2018

Another obeyed coke oven operation parameter was the heating rate (Figure 2.3.4). There the relationship between IGP and heating amount gave an expected behavior. With rising speed of the coking process caused for example by a higher heating wall temperature the internal gas pressure values were higher.

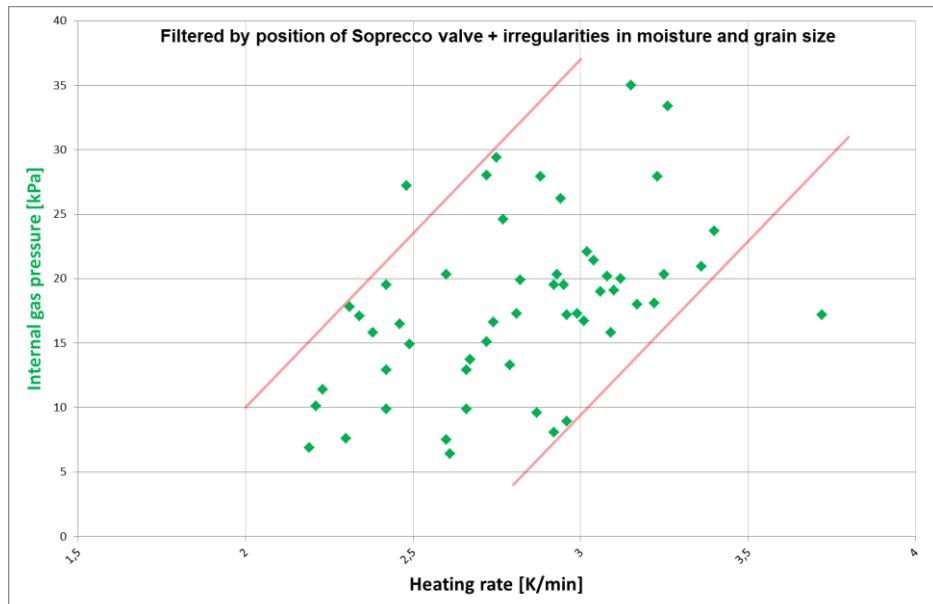


Figure 2.3.4: Correlation of IGP and heating rate from Jul 2015 - Dec 2018

As well as the recorded data of the IGP measurements gave a good relationship with the heating rate, the comparison of the heating rate with the time of the internal gas peak also did. Due to the higher heat transfer into the coal cake the coking front moves faster and as a consequence the meeting of the plastic layers starts earlier. The time for the peak maximum of the internal gas pressure therefore decreases with raising heating rate and is shown very good in Figure 2.3.5.

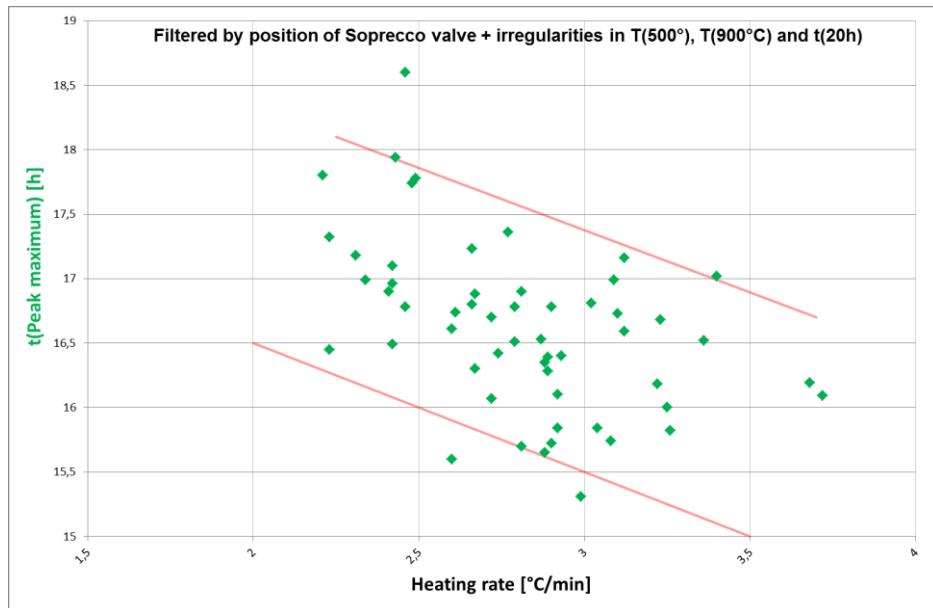


Figure 2.3.5: Correlation of time of IGP peak and heating rate from Jul 2015 - Dec 2018

### Charging Method and Coke plant design

To quantify effect of bulk density at industrial scale, ArcelorMittal Ostrava prepared a coal blend for stamp charging battery and this blend was used in gravity charging battery with the aim to check influence of charge density of the same blend on the value of IGP. Indeed, ArcelorMittal Ostrava operates three coke oven batteries, when two batteries no. 1&2 with Koppers design are equipped by stamp charging, while third battery no.11 with PVR design is equipped by classical gravity charging system. Detail parameters of AM Ostrava coke oven batteries are listed in Table 2.3.1.

Table 2.3.1: Coke oven batteries specifications at AM Ostrava

	STAMP CHARGED BATTERIES Batteries COB 1, COB 2	TOP CHARGED BATTERY Battery COB 11
# of chambers in COB	2 x 72 = 144 ch	2 x 30 = 60 ch
Volume of chamber	19,53 [m <sup>3</sup> ]	41,60 [m <sup>3</sup> ]
Coking time	23,17 [h]	22,68 [h]
Annual capacity [dry coke]	590 kt	490kt
Size of chamber (hot)		
- mean width	442 [mm]	410 [mm]
- height	3 645 [mm]	7 000 [mm]
- length	13 120 [mm]	16 000 [mm]
Heating system	KOPPERS	PVR

The experiment consists of three steps:

- Preparation of safe coal blend for stamp charging system that can be consequently charged at gravity charged battery;
- Measurement of internal gas pressure (IGP) values at the same time at both spots;
- Evaluation of the trial – identity of coal blend at both batteries, the influence of the charge density of the same blend on IGP.

Homogeneity of tested blend at both batteries is extremely important factor that can have a significant impact on IGP value. From both processes, large portion of coal sample was taken in order to make comprehensive analyses and also to carry out pilot oven tests in sole heated oven and movable wall pilot oven. Detailed output analyses are listed in Table 2.3.2.

Table 2.3.2: Industrial blend quality from both sides

	Stamp charging	Gravity charging
Date of sampling	26. 11. 2018	26. 11. 2018
Proximate Analysis (dry)		
Ash [%]	7,0	6,4
Volatile Matter [%]	28,5	27,5
Total Sulphur [%]	0,53	0,54
Rheological Properties		
Free Swelling Index [1]	7,5	7,5
Audibert-Arnou Dilatometer		
Maximum Contraction [%]	31	32
Maximum Dilatation [%]	48	57
Gieseler Plastometer		
Fluid Range [°C]	73	70
Maximum Fluidity [ddpm]	720	690
SHO test		
ER (52;2) [%]	-16,5	-15,8
Petrographic Analysis		
Vitrinite Reflectance (Ro) [%]	1,031	1,025
Vitrinite [%]	54,1	54,3
Liptinite [%]	8,8	8,3
Semifusinite [%]	24,0	24,6
Pseudovitrinite [%]	4,0	2,9
Inertinite [%]	9,1	9,9

With respect to the repeatability limit of concerned analyses, both blends, samples at stamp and gravity charged battery at the same time, were comparable. Detailed analyses in terms of fluidity and petrography are shown in Figure 2.3.6. Again, both samples are equal within reproducibility limits of each method.

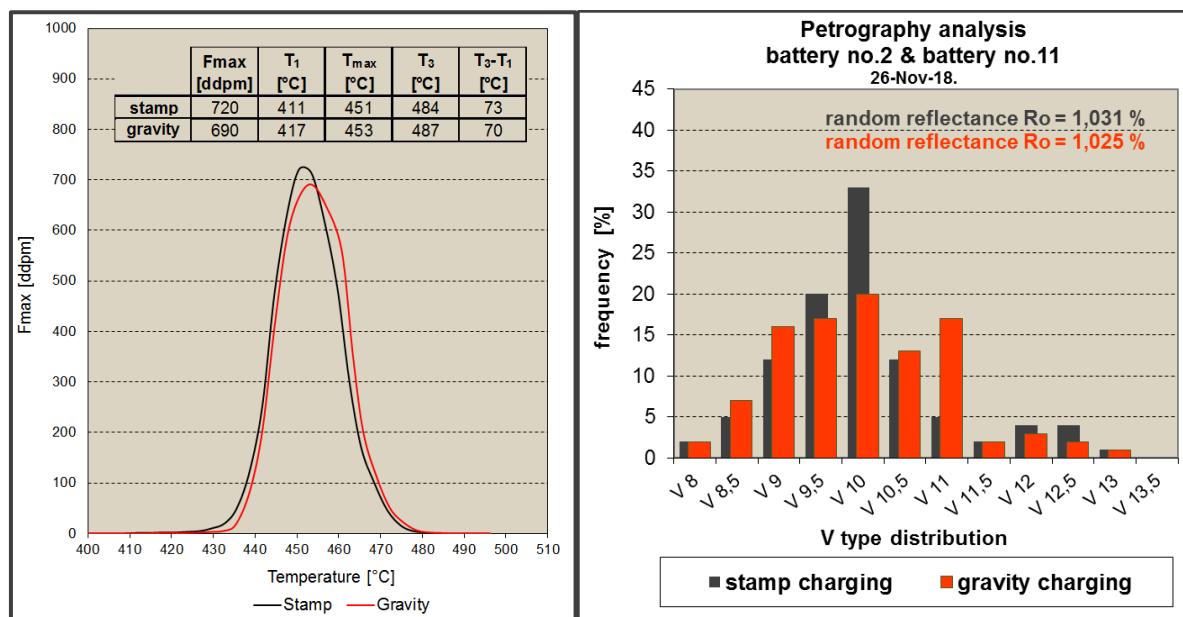


Figure 2.3.6: A-A max fluidity and petrography of industrial blend

During round robin tests that were carried out in frame of Task 3.2, a significant issue with a very low pressure values of Ostrava industrial blend was detected. Industrial blend from gravity charged battery no. 11 was distributed to all partner's pilot ovens in aim to compare pressure values. All pilot ovens detected values only on the level 1-2 kPa in terms of oven wall pressure and also in terms of IGP. Considering very low values of industrial blend for gravity charging conditions as well

as lower rank of industrial blend used for stamp charging, extremely low values of IGP at battery no. 11 were expected.

In following Figures 2.3.7 and 2.3.8, a comparison of both IGP values detected in stamp and gravity charged coke ovens are shown.

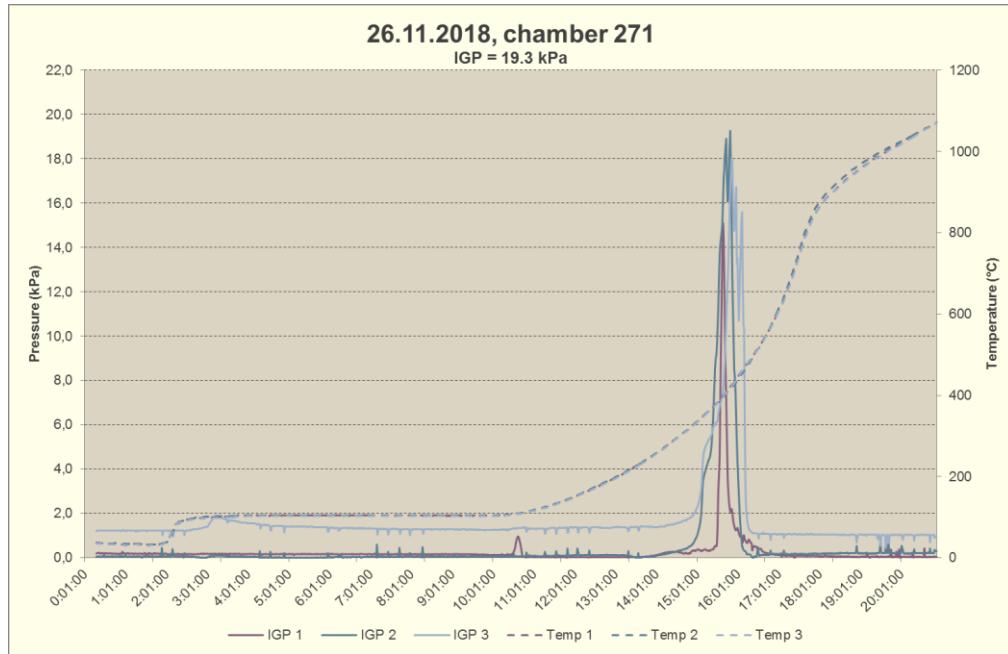


Figure 2.3.7: IGP Industrial measurement of stamp blend in stamp charging conditions

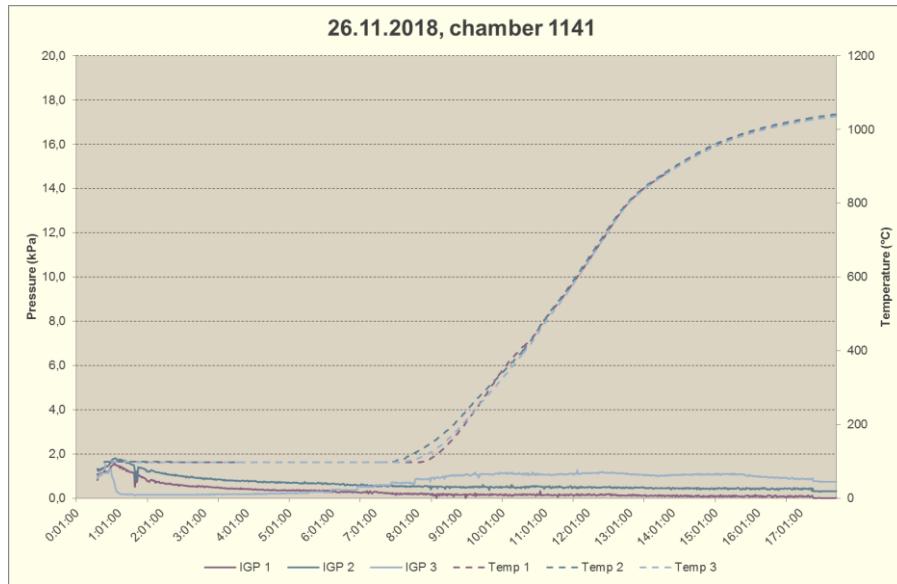


Figure 2.3.8: IGP Industrial measurement of stamp blend in gravity charging conditions

Huge impact of bulk density on Internal Gas Pressure values is obvious. While coal blend in stamp charging system (with density  $\sim 1000 \text{ kg/m}^3$  dry basis) generates maximal Internal Gas Pressure  $IGP = 19.3 \text{ kPa}$ , the same blend with bulk density limited by gravity charging only on  $\sim 700 \text{ kg/m}^3$  dry basis does not generate any significant pressure in the middle of the charge. Detected maximal  $IGP = 1.8 \text{ kPa}$  corresponds very well with outputs from round robin tests that was carried out in frame of Task 3.2. These results are fully in agreement results presented in Figure 2.2.12, and highlight the importance of coal blend strategy according coke plant design and operations.

To complete this study about influence of coke oven operations and design, hereafter a comparison ArcelorMittal Atlantique et Lorraine coke plants (Dunkerque and Florange) was carried out. Dunkerque and Florange are in gravity charging conditions and they use the same coal blend but with different targeted bulk densities (oiling of the blend in Dunkerque) due to difference in the

design if their ovens. The Table 2.3.3 summarizes the preparation and coking conditions used at pilot scale to better fit with the industrial Florange and Dunkirk conditions.

Table 2.3.3: Coking conditions of AMAL plants at Pilot Scale

Testing conditions		Dunkirk coke plant	Florange coke plant
Blend grain size	% < 2 mm	72	75
Moisture	%	7	7
Bulk density	kg/m <sup>3</sup> d.b.	770 (by oiling) <sup>1</sup>	740
Coking time	hours	19 h 30	17 h 15

Once again, the results of Pilot Oven tests highlight the effect of bulk density is preponderant (Figure 2.3.9). Despite a higher heating rate at Florange coke plant, this parameter did not compensate the difference of bulk density.

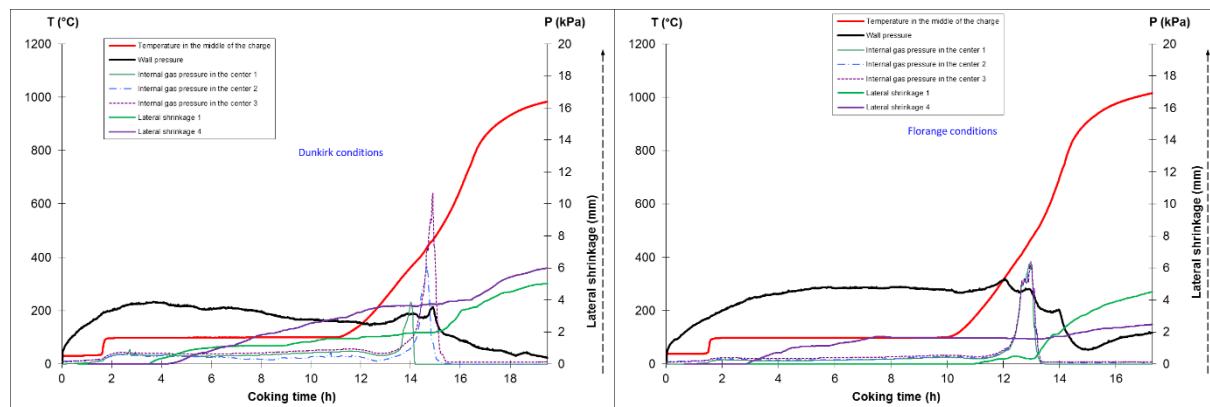


Figure 2.3.9: AMAL blend tested in gravity charging with different operating conditions

### **WP 3 Relationship between IGP and OWP in pilot oven and reflection on industrial IGP measurements with pilot oven results**

#### ***Task 3.1 - Comparison between pilot and industrial measurements [voestalpine, AMMR, AMAL, AGDH, tkSE, DMT, CPM, AMO]***

In this Task 3.1 each project partner has to compare the results of the pilot plant with their industrial coke ovens. Couples of pilot and industrial partners were established in order to compare and correlate Internal Gas Pressure.

Two series of comparison were performed between AMAL and CPM:

- One in November 2015 at the beginning of the project
- Another one in February 2018 after implementation of the new triple probe (cf. Task 1.4)

In both case, industrial blends were sampled at Florange coke plant and sent to CPM. Two series of four tests were carried out at different bulk density in the range 720-800 kg/m<sup>3</sup> db. Dealing with coking conditions, standard procedure was used:

- Coal blend grain size: industrial one
- Moisture content: 8% ± 0.5%
- 17.2 hours coking time with a constant heating wall temperature of 1150°C

Table 3.1.1 summarizes the results of the 1<sup>st</sup> series of four coking tests.

Table 3.1.1: Results of pilot oven tests carried out in November 2015

<b>Density target</b>		<b>750 kg/m<sup>3</sup></b>		<b>790 kg/m<sup>3</sup></b>	
Moisture	%	Test 1	Test 2	Test 1	Test 2
Charge density	kg/m <sup>3</sup> db.	8.4	8.0	8.1	7.8
<b>Internal Gas Pressure</b>	<b>kPa</b>	<b>7.6</b>	<b>7.0</b>	<b>11.3</b>	<b>30.2</b>
Time at maximum gas pressure	h	12.6	12.6	13.5	13.7
<b>Measured wall Pressure</b>	<b>kPa</b>	<b>3.5</b>	<b>4.1</b>	<b>5.3</b>	<b>12.6</b>

Results show a good reproducibility for a charge density of ~750 kg/m<sup>3</sup> whereas results are scattered for a density of ~790kg/m<sup>3</sup>. Lower Internal Gas Pressure values were measured at pilot scale with density of ~750 kg/m<sup>3</sup> than measurements in industrial ovens. Higher IGP values were measured at 790 kg/m<sup>3</sup>, but results are scattered without explanation. Additional tests were performed on blend (petrographic analysis, rheological properties) and the four blends have the same properties. Carbonization curves are presented on Figure 3.1.1.

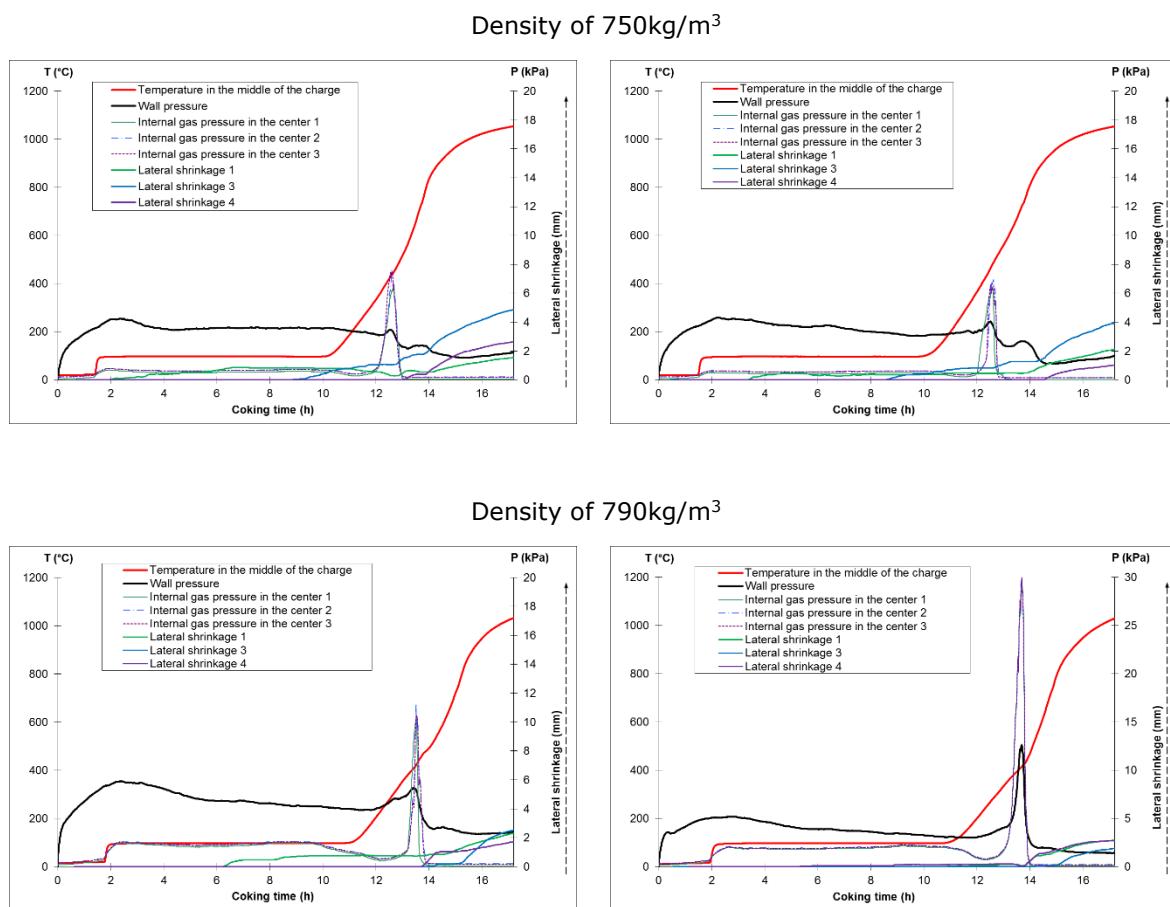


Figure 3.1.1: Internal Gas Pressure and Oven Wall Pressure at Pilot Scale

The 2<sup>nd</sup> series of tests was performed at the end of project, and so according results of the different partners dealing with effects of bulk density, CPM performed 4 industrial blends to point out the effect on bulk density, but mainly to define the operating conditions to use at Pilot scale to obtain the same range of IGP than AMAL coke plant. Thus 4 tests were performed in the range 720-800 kg/m<sup>3</sup> db. and results in terms of IGP were compared with industrial measurements performed in the same period (results were presented in Task 1.4). Table 3.1.2 summarizes the results obtained during this series.

Table 3.1.2: Results of pilot oven tests

Coking Tests		Test 1	Test 2	Test 3	Test 4
Moisture	%	8.3	8.4	8.3	7.8
Charge density	kg/m <sup>3</sup> db.	724	747	759	792
<b>Internal Gas Pressure</b>	<b>kPa</b>	<b>4.2</b>	<b>5.0</b>	<b>10.5</b>	<b>21.6</b>
Time at maximum gas pressure	h	12.2	12.5	12.7	13.4
<b>Measured wall Pressure</b>	<b>kPa</b>	<b>2.1</b>	<b>5.5</b>	<b>6.4</b>	<b>9.6</b>

Figure 3.1.2 represents evolution of Internal Gas Pressure according density. Results confirm that a direct comparison of IGP between Pilot and Industrial measurements is possible if and only if the Pilot oven is setup with coke plant conditions. Thus, in order to simulate the exact coking condition of AMAL Florange at the location of IGP measurement, CPM has to adjust the bulk density to 790 kg/m<sup>3</sup> db.

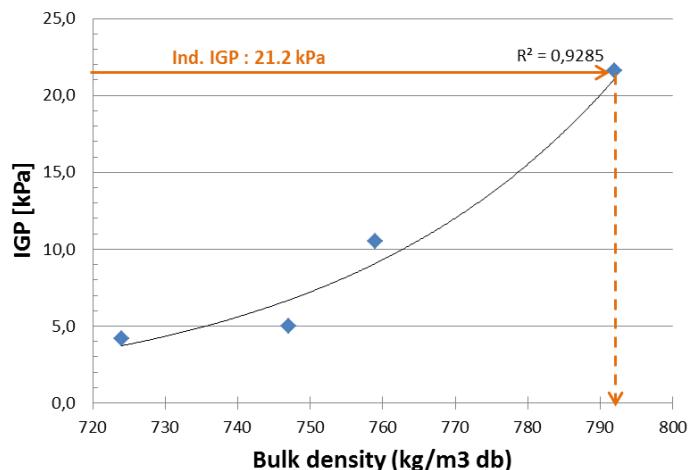


Figure 3.1.2: Comparison between IGP at Industrial and Pilot scale

AGDH has collected a representative 2t sample of the industrial blend of ZKS. The sample was directly taken from the conveyance belt while the mixing bed was in use. The sample was then delivered to the test facilities of CPM. Within the use of the concerning mixing bed there were three IGP measurements at oven 136 of ZKS. In Figure 3.1.3 the recorded temperature and pressure profiles are pictured. Next to them the correlation of the IGP peak with the position of the Soprecco valve is also shown.

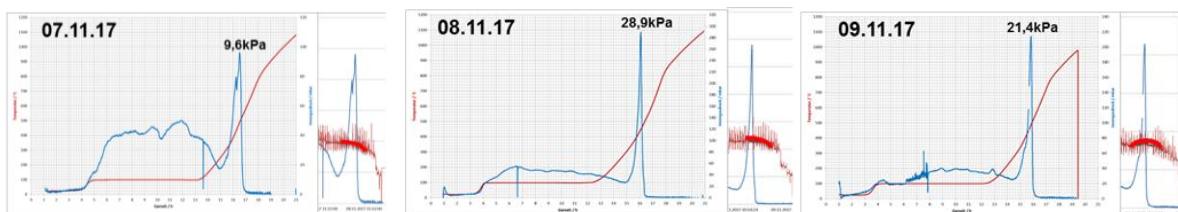


Figure 3.1.3: IGP measurements at AGDH for WP3 Task 3.1

In the frame of BINGO and other exchanges with ZKS, blends of ZKS were tested several times according the past months. Unlike gravity charging, in stamp charging, density between Pilot and Industrial scale is similar since at pilot scale, density of 1000 kg/m<sup>3</sup> could be reached. The Figure 3.1.4 gathers the results obtained in the frame of BINGO project. More than ten tests were performed and the conclusions are similar: in stamp charging, despite similar density than industrial measurements, IGP was not comparable between Pilot and Industrial scale. Industrial average (with squeezed vertical) is around 21kPa whereas at Pilot scale, Pilot average is around 7kPa. Coal cake width are similar to industrial cake (450mm).

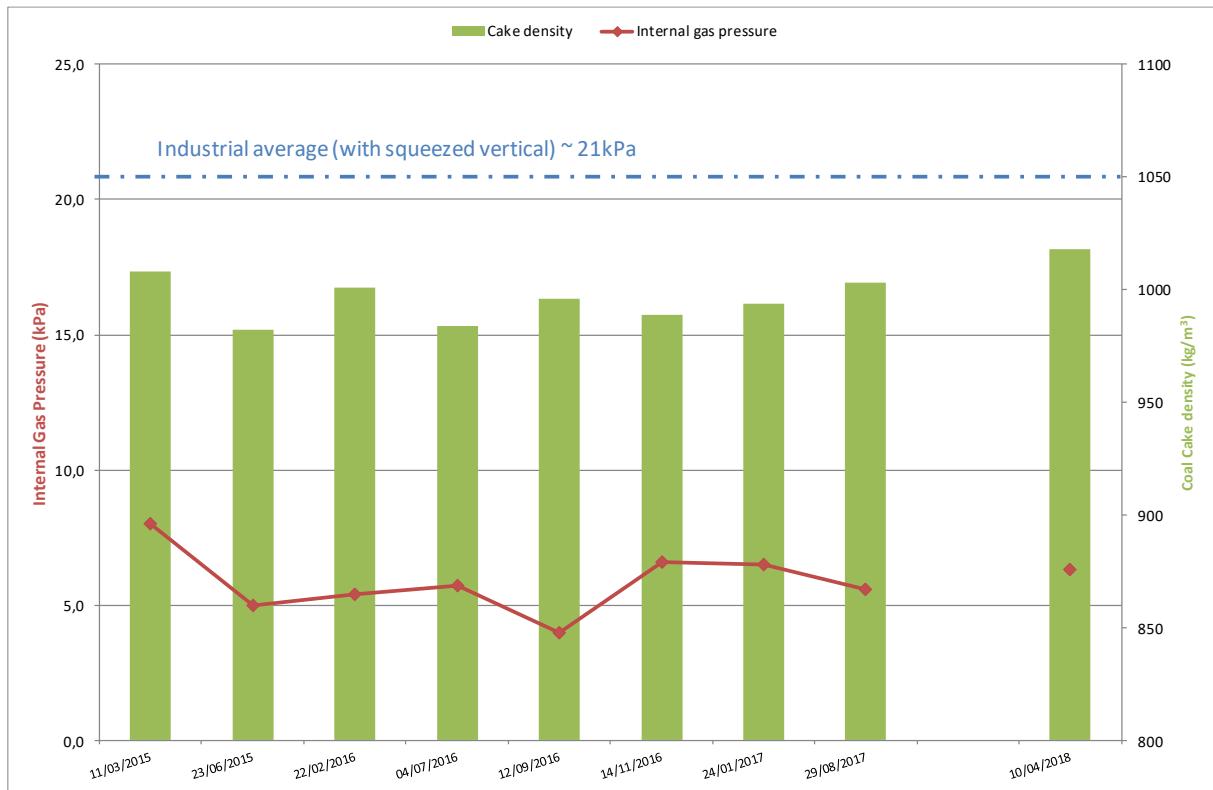


Figure 3.1.4: IGP Comparison between Pilot and Industrial IGP between CPM and ZKS

A possible explanation could be the difference of height of coal cake, indeed at pilot scale, the coal cake height is around 1m whereas the industrial coal is around 6m. This difference could influence the rate of gas release in plastic layer, and so internal gas pressure. In top charging, as mentioned in Task 2.1, a decreasing of IGP is observed at industrial scale according the height due decreasing of density, but in stamp charging, due to the same density according the height, similar IGP is expected. Nevertheless, no measurement has validated this point due to the difficulty to drill and insert probe in stamp charging in high position.

AMO carried out also pilot oven tests of industrial blends for each charging system, stamp as well as gravity charging batteries of Ostrava coke plant. Pilot oven tests of industrial blends as well as every industrial IGP trial were supplemented by coal charge contraction/expansion determination in Sole Heated Oven.

Three comparative trials were carried out in frame of gravity charging conditions at industrial scale. Two consecutive IGP measurements were carried out in chamber 1367 when comparable pressure values on the level 5 kPa were recorded. Internal gas pressure development during both trials in chamber 1367 are shown in Figure 3.1.5 while pilot oven test of same blend, sampled from the larry-car at the time of trials, is shown in Figure 3.1.6. Pressure value recorded in pilot scale is significantly lower compared to industrial scale (5 kPa vs 1.2 kPa). In the frame of this comparison, AMO uses to maintain their operating condition of their Pilot oven, and so no correction of bulk density was applied. This point explains why there is an undervaluation of IGP during the comparisons.

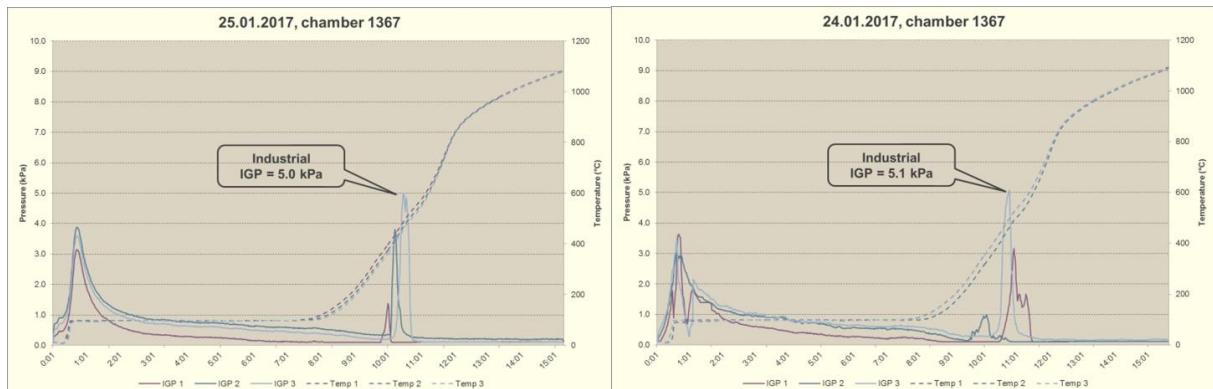


Figure 3.1.5: 2 consecutive IGP industrial measurements at gravity charged battery no. 11

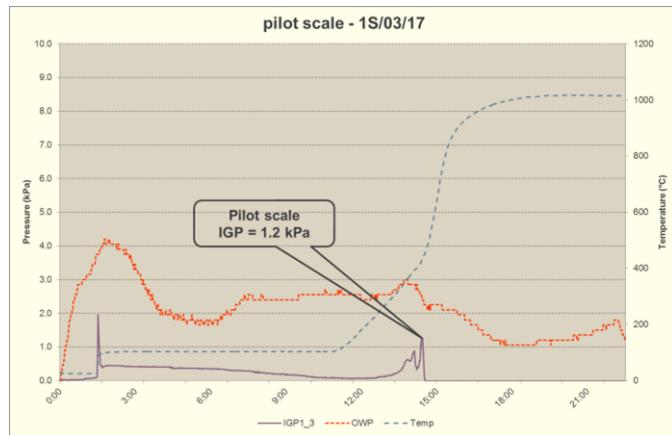


Figure 3.1.6: Pilot oven test of industrial blend sampled in aim to complete chamber 1367 trials

In frame of Task 3.2, where round robins of pilot oven tests were carried out, other comparative trials were executed. Industrial and pilot scale IGP values are shown in Figure 3.1.7. Industrial value of IGP is again significantly lower compared to pilot scale where only small values were recorded (3.5 kPa vs 1.2 kPa).

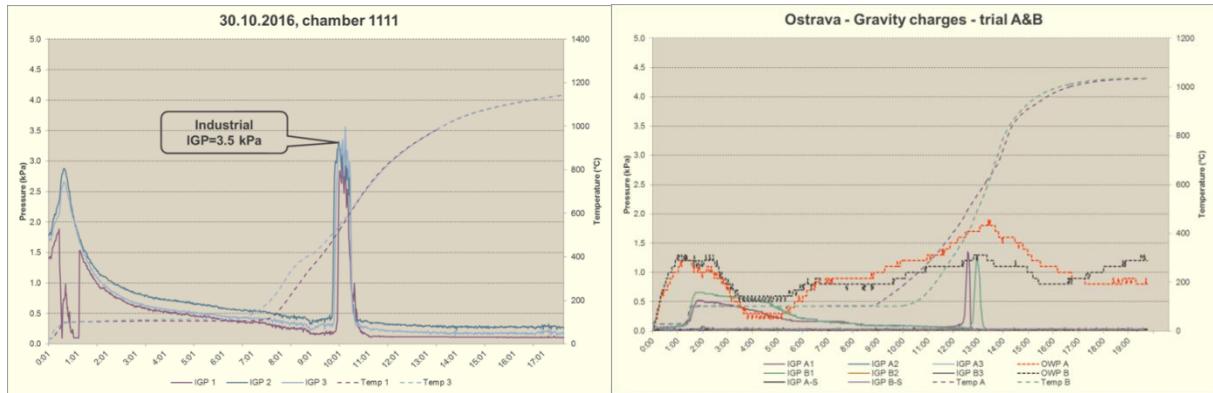


Figure 3.1.7: Industrial and pilot oven measurement in gravity charging conditions

Comparison was also performed in stamp charging conditions. Figure 3.1.8 shows another result between industrial and pilot scale where pilot scale values are also lower compared to the industrial scale. As mentioned during the comparison between CPM and ZKS, despite similar bulk density, pilot oven is not able to correlate with industrial measurements.

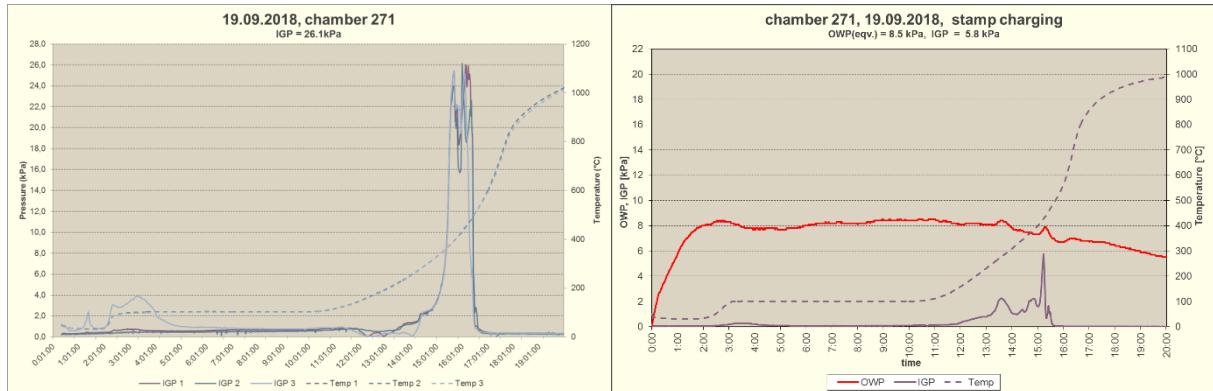


Figure 3.1.8: Industrial and pilot oven measurement in stamp charging conditions

All above mentioned recorded values of IGP in both pilot and industrial scale are summarized in following Figure 3.1.9. Experimental measurements confirmed very low values of IGP from pilot scale that cannot be compared with industrial ovens. Any significant relationship cannot be set up to predict industrial internal gas pressure.

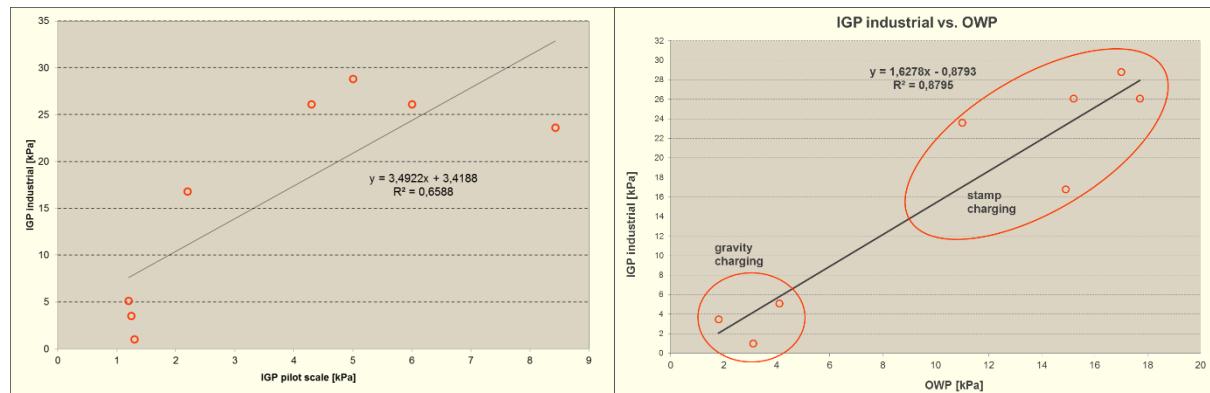


Figure 3.1.9: Comparison between pilot and industrial measurements by AMO

To conclude about these comparisons between CPM/AMAL, CPM/ZKS or AMO, it was highlighted that without any modification of Pilot Oven setup (heating rate, bulk density, etc..), a direct comparison between Pilot and Industrial measurement is not possible. Thus, according the context of Pilot Oven use, two approaches can be considered:

- Development of setup which is not in full correlation with 1 plant, but allows to perform comparison between different coke plants;
- Standardization of the setup to be in perfect correlation with 1 plant.

The 1<sup>st</sup> approach is used by CPM and AMO. Indeed, AMO and a part of CPM belongs to ArcelorMittal, and so they oversee evaluation of new coals for Western and Eastern ArcelorMittal coke plants. To optimize the tests, standard conditions were developed to easily share the results between the coke plants.

It is not the case of tkSE. Implementation of their 60kg oven is to deliver directly applicable results for Schwegern coke plant. Table 3.1.3 shows the measures that have been implemented at tkSE in order to develop and standardize MWO setup and test conditions. Figure 3.1.10 illustrates the relationship between industrial and pilot oven IGP results; it displays a noticeable trend confirming that the measures have been efficient and the setup can be applied for carbonization tests the results of which deliver an indication of a corresponding IGP to be obtained in industrial ovens.

Table 3.1.3: Modifications to tkSE MWO relevant to IGP measurement

Parameter	Beginning of the project	Actual status
<b>Bulk density</b>	750 kg/m <sup>3</sup> (db)	900 kg/m <sup>3</sup> (db)
<b>Heating</b>	Constant temperature	Heating ramp
<b>Probe diameter</b>	20 mm	10 mm
<b>Probe opening</b>	3 holes	Squeezed end
<b>Probe guiding</b>	No guiding	Guiding with screw connections
<b>Probe hose material</b>	Plastic	Stainless steel corrugated

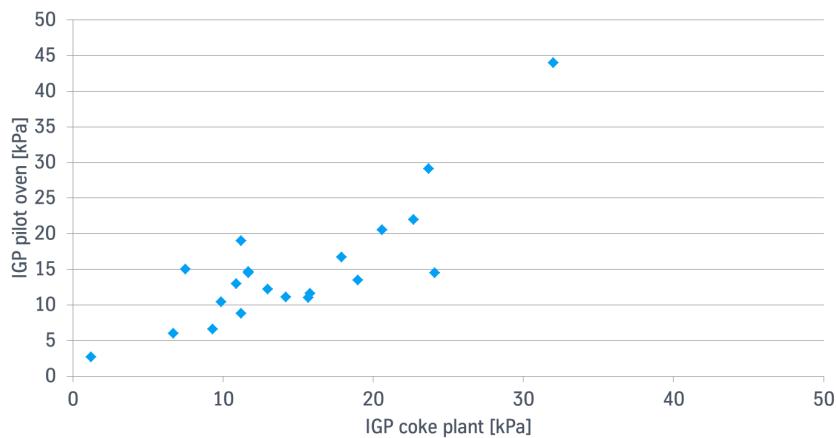


Figure 3.1.10: Relationship between industrial and pilot scale IGP results at tkSE

Another valuable parameter which is typically in focus during carbonization tests in movable wall pilot ovens is the oven wall pressure (OWP) indicating the pressure of the charge, caused by the pressure of gas in the plastic layer (IGP), exerted on the heating walls in the course of carbonization process. For coke operations, OWP is a very critical factor as it cannot be measured directly at the plant but it is as such directly linked with a maximum load the oven heating walls can bear in real conditions. Figure 3.1.11 illustrates the correlation between IGP and OWP as measured in MWO. To obtain OWP results, the measured force was re-calculated to a pressure value considering the whole coal cake surface as the contact area between the charge and the heating wall. To gain additional data, the MWO data set was enlarged by blends that were used at tkSE. The correlation shows that a critical IGP value of 20 kPa should not be exceeded in order to keep OWP values under 10 kPa.

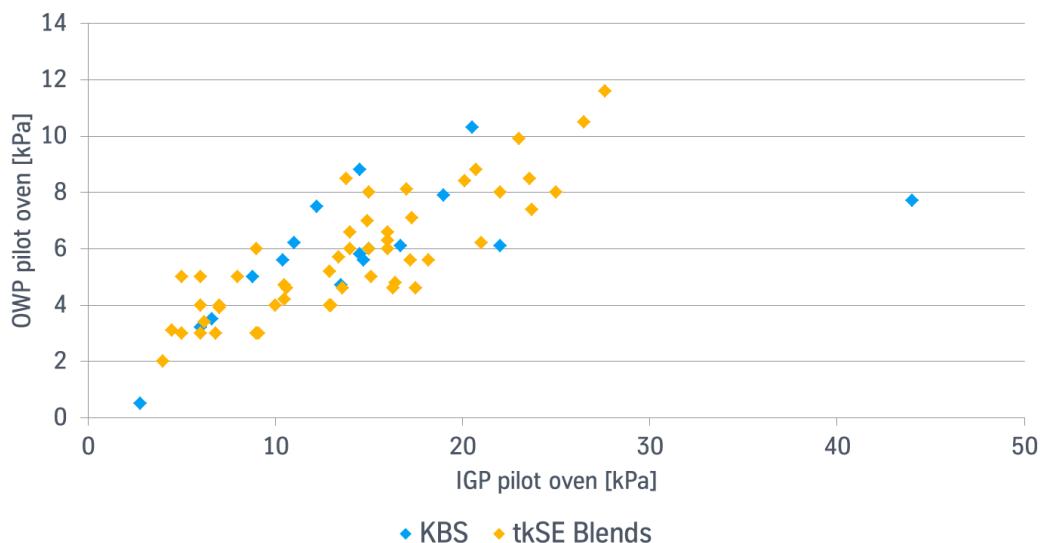


Figure 3.1.11: Relationship between IGP and OWP in pilot scale at tkSE

### **Task 3.2 - Round robin on pilot ovens on two industrial blends [AMMR, tkSE, AGDH, DMT, CPM, AMO]**

Various project partners operate different coking test facilities like coking retorts or pilot coke ovens with fixed or movable wall. The aim of this task is to find the accordance or the functionality between the internal gas pressures measured in pilot scale and in industrial scale at coke oven batteries.

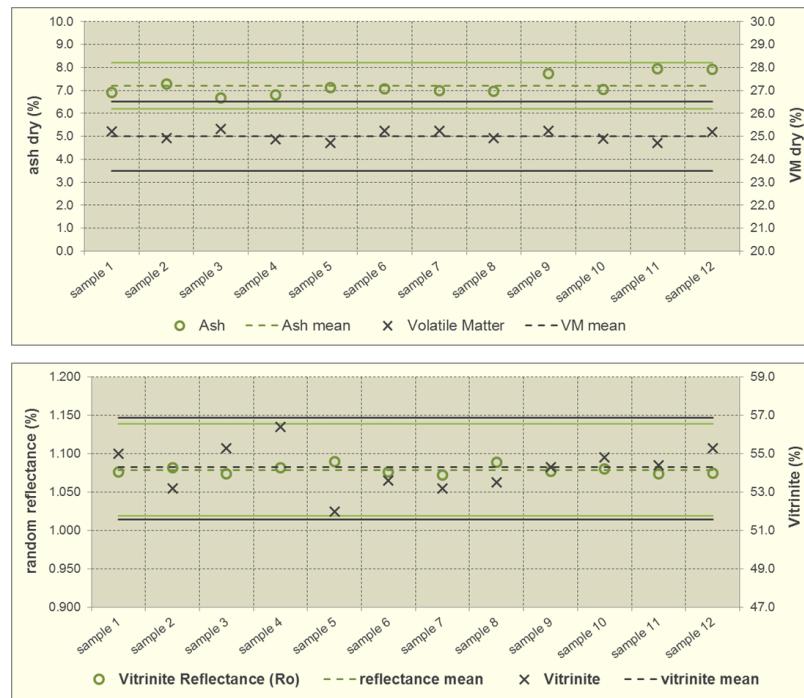
In order to correctly analyze values of internal gas pressures (IGP) from different pilot facilities, round robin tests were organized, when two different blends representing gravity and stamp charging conditions were prepared and distributed among the partners. The project partners get one coal blend representing gravity charging and one blend representing stamp charging. The goal was to carry out carbonization tests at the project partner's facility and compare firstly the results

of the internal gas pressure measurement. Within the round robins, also oven wall pressure (OWP) values and coke quality were compared.

Two coking conditions sets were defined for gravity and stamped charging:

- Gravity charging
  - Moisture: 8%
  - Coal grain size: 82% < 3.15-mm; 42% < 0.5-mm
  - Bulk density: 750 kg/m<sup>3</sup> db
  - Global Coking Time: 18h for 410-mm oven width (corresponding to 11.6 mm/h coking rate)
  - Final temperature: 1025°C
  
- Stamped charging
  - Moisture: 11%
  - Coal grain size: 90% < 3.15-mm; 47% < 0.5-mm
  - Bulk density: 1000 kg/m<sup>3</sup> db
  - Global Coking Time: 24h for 500-mm oven width (corresponding to 10.4 mm/h coking rate)
  - Final temperature: 1025°C

ArcelorMittal Ostrava prepared a coal blend for gravity charging, and AGDH prepared a coal blend used for stamp charging. These two blends were distributed to the project partners which are equipped with devices to measure Internal Gas pressure and Oven Wall Pressure. After preparation of six tons of industrial coal blend for gravity charging, ArcelorMittal Ostrava performed detailed analyses to check proper homogenization. Homogeneity of basic parameters like A<sup>d</sup>, V<sup>d</sup>, R<sub>Random</sub>, Vitrinite, FSI, dilatation, Gieseler fluidity and fluid range of each sample is shown on Figure 3.3.1.



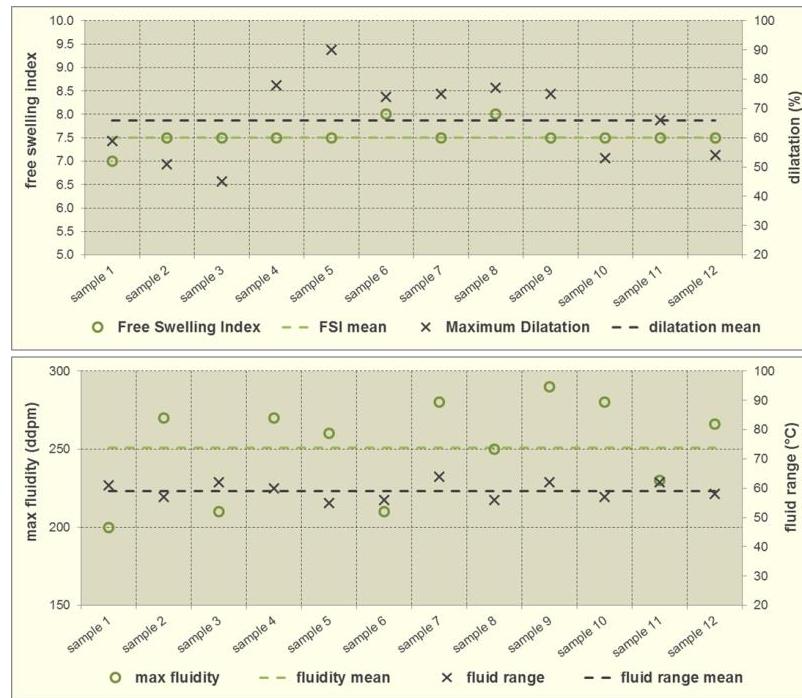


Figure 3.3.1: Homogeneity of the distributed ArcelorMittal Ostrava samples

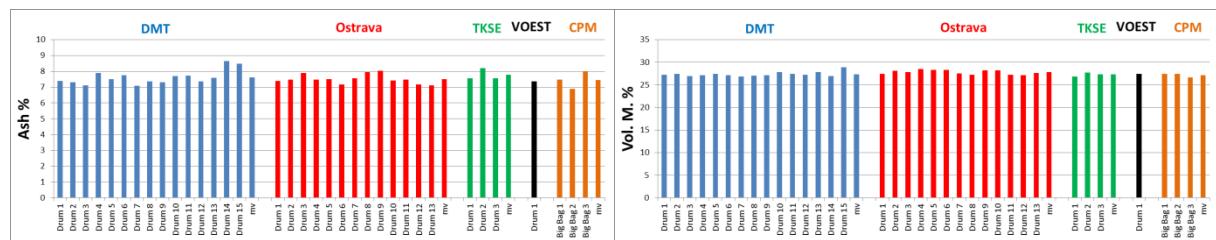
Green and black lines are representing reproducibility limits if are available while dash lines are representing average of all results. All basic parameters from 12 samples showing good homogeneity that is necessary condition for success comparative study.

AGDH sampled uncrushed industrial blend from outdoor stock yard which is used for its stamp charged batteries. The collecting of the coal samples for the project partners took place directly from the mixing bed. The following table summarizes the different quantities of drums and big bags for each partner.

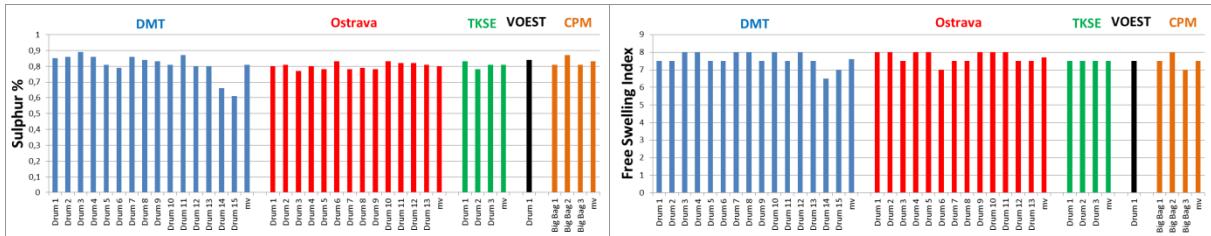
Table 3.2.1: Quantity of the distributed samples from AGDH

	DMT	AMO	TKSE	VOEST	CPM
Drum	15	13	3	1	-
Big Bag	-	-	-	-	3

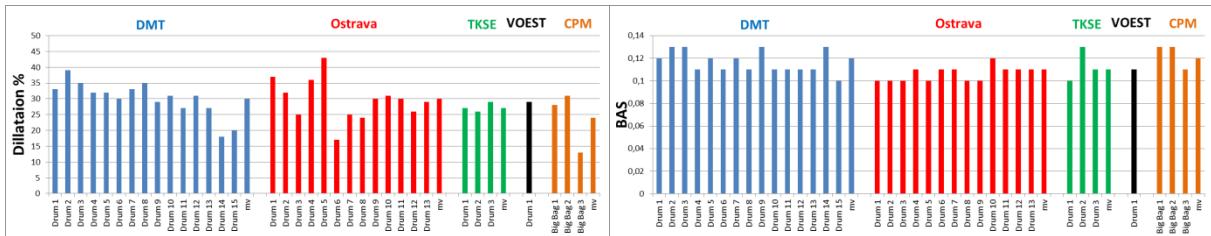
From every drum or bag a representative sample was taken and analyzed at AGDH main lab. The results are shown in the following figures and the mean values of the determined parameters are additionally listed in table 2 underneath.



Figures 3.2.2: Determined ash and volatile matter contents of the distributed AGDH samples



Figures 3.2.3: Determined Sulphur contents and free swelling indices of the distributed AGDH samples



Figures 3.2.4: Determined dilatation and basicity of the distributed AGDH samples

Table 3.2.2: Mean values of the determined parameters of the samples

		DMT	AMO	TKSE	VOEST	CPM	all
<b>mean ash</b>	%	7,6	7,5	7,8	7,4	7,5	7,6
<b>mean volatile matter</b>	%	27,4	27,8	27,3	27,4	27,2	27,5
<b>mean sulphur</b>	%	0,8	0,8	0,8	0,8	0,8	0,8
<b>mean free swelling index</b>	-	7,6	7,7	7,5	7,5	7,5	7,6
<b>mean dilatation</b>	%	30,0	30,0	28,0	29,0	29,0	29,0
<b>mean basicity</b>	%	0,1	0,1	0,1	0,1	0,1	0,1

The mean values were very similar between the different project partners and the deviations of the parameters have also been very low.

All results are divided into two paragraphs according to the test conditions and an additional third part is covering results from retorts where required carbonization rate for pilot ovens could not be kept.

Blends prepared by AGDH and AMO were tested in movable wall ovens at CPM, DMT, Ostrava and tkSE. Each lab carried out two pilot oven tests to ensure reliable results. Main pilot oven characteristics and the testing conditions are presented below:

Table 3.2.3: Pilot oven parameters

Pilot oven parameters for STAMP CHARGING		CPM	DMT	Thyssen	Ostrava
Chamber length (door to door)	mm	1000	~ 1300	540	1520
Chamber height	mm	1200	1000	670	980
Chamber width	mm	470	470	300	450
Charge Volume	m <sup>3</sup>	~0.54	~0.56	0.068	0.6
Charge length	mm	1000	1250	480	1520
Charge height	mm	1095	1000	510	800
Charge width	mm	450	430	280	430
Standard stamp density	kg/m <sup>3</sup> d.b	~ 1000	1000	1000	1000
Standard charge weight	kg	~550	~ 600	75,5	500
type of heating/ energy input		Electrical heating	natural gas	Electricity/ 47 kW	Electricity/ 115 kW
Nb of heat. elements per wall		7	-	3	6
number of IGP probes		3	2	2	3
Temperature measurement in the tar seam	Yes/no	yes	yes	yes	yes

## Results from stamp charging conditions

Main variations in pilot ovens parameters are the volume of each oven and especially the width of the oven with consequent various initial weight of coal charge. Parameters of pilot oven tests from each facility together with recorded values of oven wall pressures and internal gas pressures are indicated in following Table 3.2.4.

Table 3.2.4: Pilot oven tests output - stamp charging

Pilot oven parameters for STAMP CHARGING		CPM		DMT		Thyssen		Ostrava	
Coal charge preparation before the test		Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
< 3.15 mm	%	90.2	90.2	92.0	91.0	92.7	92.7	91.0	91.2
< 0.5 mm	%	42.3	41.7	48.0	46.0	42.6	42.6	37.7	38.2
Type of sieves	Square/round	square	square	square	square	square	square	square	square
Charge moisture	%	10.9	11.1	11.0	11.0	10.8	10.8	11.0	11.0
Charge density	kg/m <sup>3</sup> d.b.	1008	1008	1000	1000	990	990	1001	1000
carbonization parameters									
Coking time	h:mm	22:48	22:56	22:36	22:29	14:30	14:30	21:50	21:50
Final coke temperature	°C	1102	1076	1102	1112	1000	993	1032	1033
real coking rate	mm/h	9.9	9.8	10.4	10.4	10.3	10.3	9.8	9.8
Max oven wall pressure As recorded	kPa	12.0	10.6	7.4	7.1	6.7	7.5	9.1	11.1
Time at OWP max	h:mm	3:42	3:54	14:42	13:36	11:26	11:40	3:16	2:45
Max oven wall pressure during the junction of the plastic layer	kPa	6.6	6.8	6.5	5.6	6.7	7.5	5.1	5.1
Max Internal gas pressure	kPa	8.2	10.1	7.4	11.0	13.4	15.9	9.7	10.6
Time at IGP max	h:mm	14:30	15:24	18:00	18:21	11:25	11:32	14:30	14:37
% of rank time during the junction of the plastic layer	%	66	67	80	81	78	79	66	67

Except tkSE' oven, coking time was similarly set up on 22 hours or more to achieve planned coking rate. Time of maximal wall pressure was different at each oven because of higher water peak at CPM and Ostrava oven compared to the wall pressure value during the junction of plastic layer. Location of maximal internal gas pressure is also significantly varying from 11.5 to more than 18 hours which is given by different coking time. Better awareness of IGP location is given when relative coking time is compared in the last line of table 3.2.4. Absolute values of detected internal gas pressure were recorded within the range 7.4 kPa (DMT) and 15.9 kPa (tkSE). Such variation must be considered during future comparison of results of IGP in pilot scale.

## Results from gravity charging conditions

Parameters of pilot oven tests from each facility together with recorded values of oven wall pressures and internal gas pressures are indicated in following Table 3.2.5.

Table 3.2.5: Pilot oven tests output - gravity charging

Pilot oven parameters for GRAVITY CHARGING		CPM		DMT		Thyssen		Ostrava	
Coal charge preparation before the test		Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2
< 3.15 mm	%	82.5	82.5	88.1	88.1	83.5	83.5	81.4	82
< 0.5 mm	%	38	38	40.8	40.8	42	42	38.8	39
Type of sieves	Square/round	square	square	square	square	square	square	square	square
Charge moisture	%	8.1	8.4	8.0	8.0	8.0	8.0	8.0	8.0
Bulk density	kg/m <sup>3</sup> d.b.	751	740	750	750	747	747	748	748
Oil addition	%	0.1	0.1	0.45	0.5	0.56	0.56	0.25	0.25
carbonization parameters									
Coking time	h:mm	20:37	20:16	20:30	20:36	13:00	13:00	19:45	19:45
Final coke temperature	°C	992	1014	1037	1037	1023	1025	1034	1036
real coking rate	mm/h	11.4	11.6	11.5	11.4	11.5	11.5	11.4	11.4
Max oven wall pressure As recorded	kPa	1.7	1.7	2.7	2.5	2.0	1.9	1.8	0.8
Time at OWP max	h:mm	14:00	13:48	17:12	17:48	9:51	9:48	13:17	12:59
Max Internal gas pressure	kPa	1.2	0.4	0.4	1.1	2.1	1.6	1.2	1.3
Time at IGP max	h:mm	15:06	15:25	17:12	17:48	9:55	9:47	12:35	12:59

Despite of different pilot facilities design, all carbonization tests were well adjusted since planned coking rate ~11.6 mm/h was kept among all partners. No significant oven wall pressure or internal gas pressure was detected on any participant pilot facility. Detected internal gas pressures were in the range 0.4 – 1.6 kPa which can be considered as negligible compared to previously presented

values in stamp charging conditions. With such small values a procedure of IGP measurement cannot be evaluated and there is no point in making any comparison of recorded IGP values for each pilot.

## Results from retort tests

Small retorts that can carbonize several kilograms of coal have always significantly different carbonization conditions compared to semi-industrial pilot ovens. All parameters like wall temperature, carbonization rate, final coke temperature and initial properties of coal charge are optimized for physical possibilities of such small equipment.

In frame of Task 3.2, two different retorts from DMT and voestalpine participated in round robins. Both distributed samples were charged into the retorts despite the gravity/stamp density was not precisely adjusted. In following Table 3.2.6, all retort parameters are indicated together with IGP values.

Table 3.2.6: Data from retort test

retort's parameters		DMT retort		voestalpine retort	
		gravity	stamp	gravity	stamp
diameter	mm	180 diameter		199 diameter	
Chamber height	mm	505		500 (charging height of coal blend)	
Chamber width	mm	-		-	
Charge Volume	m <sup>3</sup>	0.012		0.016	
Standard bulk density	kg/m <sup>3</sup> d.b.	750	1000	800 (wet) 720-740 (dry)	
Standard charge weight	kg	9.64		12.5 (wet)	
type of heating/ energy input		-		Electricity/ 3 x 5 kW	
		-			
number of IGP probes		1		1	
Temperature measurement in the tar seam	Yes/no	yes		yes	
< 3.15 mm	%	-	-	81.4	91.1
< 0.5 mm	%	-	-	30.6	36.7
Type of sieves	Square/round	square		-	
Charge moisture	%	8.0	11.0	7.2	11.6
Bulk density	kg/m <sup>3</sup> d.b.	750	1000	742	707
<b>carbonization parameters</b>					
Coking time	h:mm	4:00	4:00	4:09	4:06
Final coke temperature	°C	1018	1020	1000	1005 max. 1025
real coking rate	mm/h	22.5	22.5	24.9	24.9
Max Internal gas pressure	kPa	2.8	4.6	1.1	1.1

Regarding DMT retort, detected IGP value of gravity charge were approximately three times higher compared to pilot ovens but only 50% value regarding stamp blend compared to the pilot oven. On the other hand, voestalpine retort detected the same value of internal gas pressure during the carbonization of both stamp as well as gravity charge test. One of the reasons could be the identical bulk density of both charges compared to DMT retort where same density as for pilot oven tests can be adjusted.

One value for each carbonization condition is not enough to make any correlation for IGP values interpretation from DMT retort. Same values of IGP from voestalpine retort do not allow to use this equipment for evaluation of minor changes of pressure generation of industrial coal blends. With respect to the completely different values of IGP from both retorts compared to pilot ovens, such small devices cannot be useful to discriminate dangerous coals. Of course, evaluation of dangerous single coals is another situation and higher values of IGP could be evaluated by testing in retort tests.

In the frame of this round robin, coke quality was also compared. All recorded coke quality parameters related to both tested charges are shown in following Figure 3.3.2 and 3.3.3. Regarding coke produced in stamp charging conditions, only CSR parameter can be considered as comparable within all pilot ovens and DMT retort when the value from voestalpine retort is significantly lower.

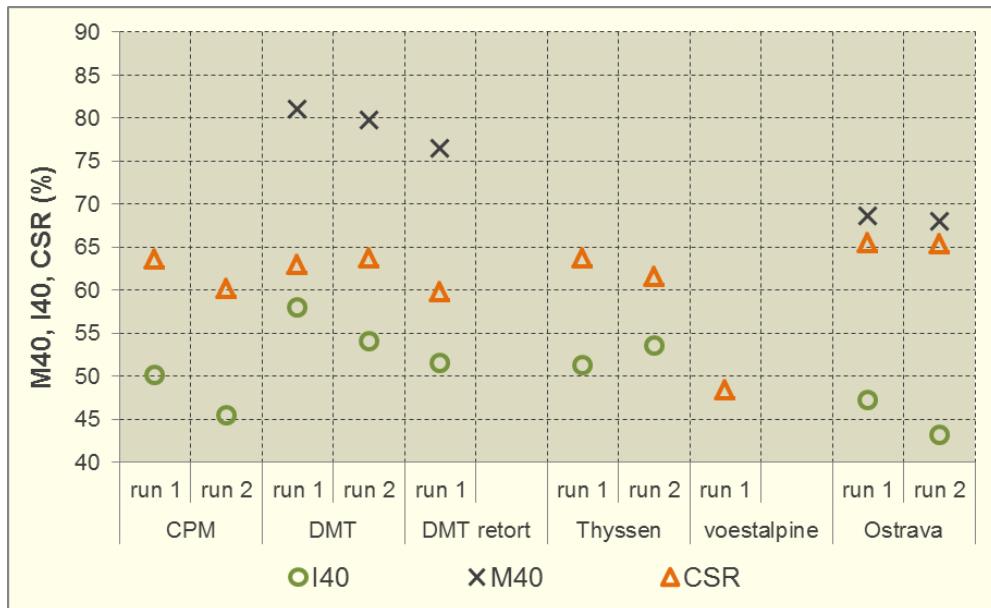


Figure 3.3.2: Coke quality from stamp blend

Significantly worse comparison is offered by coke samples from gravity charging conditions. Both CSR and I40 strength is varying from 45 to 60 percent points, only available M40 values are slightly comparable for coke samples from Ostrava and DMT.

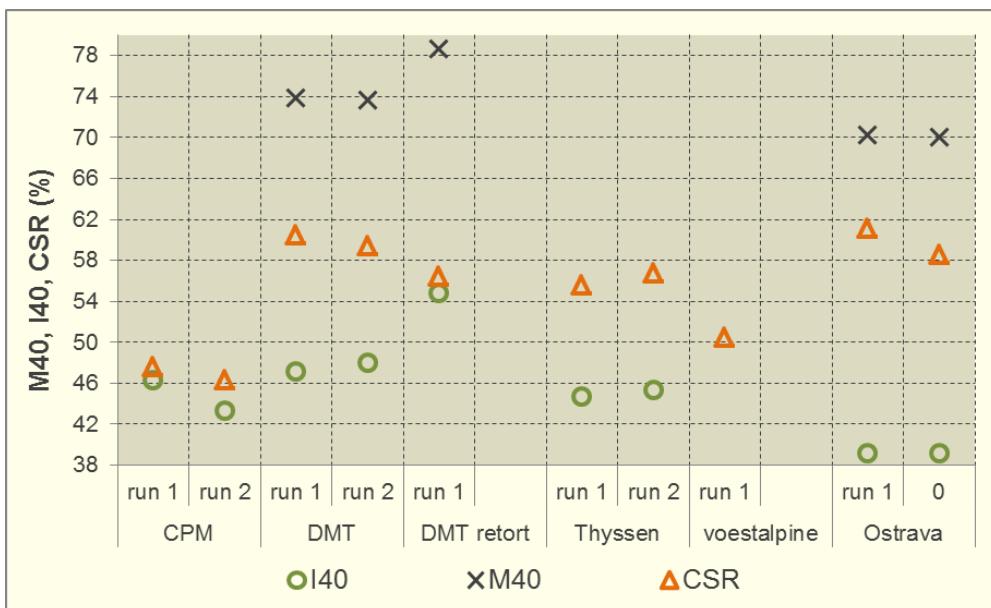


Figure 3.3.3: Coke quality from gravity blend

Determination of coke quality was not the prior purpose of the round robins and thus all carbonization parameters were carefully adjusted to reach similar coking rate and similar internal gas pressure values. Moreover, because of significantly different coke stabilization procedure, various shape of sieves and other parameters like initial coke sample size, any similar coke quality has not been achieved. This part dedicated to comparison of Coke Quality is under investigation in the frame of RFCS project ESTIVAL.

Round robin tests of internal gas pressure measurement at project partners pilot equipment were successfully executed, nevertheless, especially in stamp charging conditions a significant variation in IGP values were detected. Regarding stamp charging, values of IGP from 7.4 to 15.9 kPa were detected while in gravity charging system, values from of IGP 0.4 to 2.1 kPa were detected. Variation of internal gas pressure values derived from round robins must be considered during future comparison of results of IGP in pilot scale.

### ***Task 3.3 - Investigations of influence of parameters under defined operation conditions in pilot oven [AMMR, DMT, CPM]***

In Task 2.2 and 2.3, influences of properties of coal charge and coke oven operation & design on the internal gas pressure are investigated in the industrial scale. Nevertheless, at industrial scale, operating windows is small and so influencing parameters were not clearly pointed out. However, wall pressures which are generated through the IGP and transmitted to the coking oven walls cannot be directly measured industrially. Therefore, in this task carbonization tests were performed, at which there will be a variation of the following parameters moisture, grain size distribution, bulk density, coking rate and rank of the blend.

In 2017, DMT studied the influence of the previous parameters in wet gravity charging whereas AMMR/CPM carried out the same investigation in stamp charging technique in 2018. Influencing parameters were also analyzed by mathematical point of view by AMO and voestalpine, in order to develop predictive model. All these points are discussed hereafter.

#### **Investigation in Top charging**

##### Methodology

DMT create a coal blend with four components to investigate the influences on internal gas pressure (IGP) and wall pressure (WP). Table 3.3.1 displays the coals and their share in the coal blend.

Table 3.3.1: Coal blend

Coal	%
AUS LV	15
US LV	20
AUS MV	40
US HV	25

The share of the US LV has changed from 20 % to 10 % and 30 % for the investigation of the influence of coal rank on the internal gas pressure and wall pressure. The reducing and raising of the US HV coal has composited the decrease and increase of the US LV. The coal blend as shown in Table 3.3.1 was used for the investigation of the influence of coking rate, bulk density, grain size and moisture content on internal gas pressure and wall pressure. The test conditions are shown in Table 3.3.2.

Table 3.3.2: Coking conditions

Parameters			Reference	
Heating flue temperature	°C	1200	1250	1300
Bulk density	kg/m <sup>3</sup> db.	700	750	800
Moisture content	%	7.0	9.0	12.0
Grain size < 2 mm	%	65	75	85
Coal blend rank Ror	%	1.10	1.15	1.20

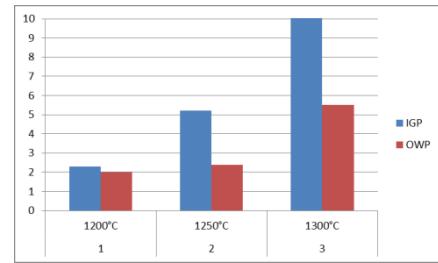
DMT charged each coal blends twice in the semi-industrial movable coke oven (MWO oven described in Task 1.1).

##### Results and discussion

The variation of the heating flue temperature has adjusted different coking rates. Table 3.3.3 shows the resulted internal gas pressure and wall pressure by different heating flue temperatures.

Table 3.3.3: Influence of coking rate on IGP and WP

Temperature	°C	1200	1250	1300
IGP	kPa	2.3	5.2	10.4
WP	kPa	2.0	2.4	5.5

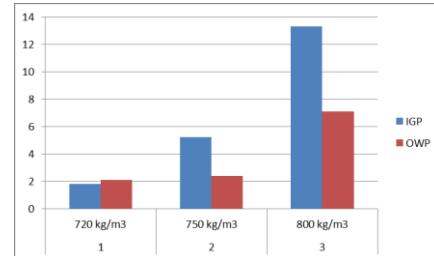


The results of this test series have indicated that an increase of 50°C of the reference temperature (1250°C) double the internal gas pressure. The decrease of 50°C has resulted in a halving of the internal gas pressure. An increase in heating flue temperature has accelerated the release of the gas in the plastic layer in a shorter coking time that result in an increase internal gas pressure.

A stronger influence on internal gas pressure and wall pressure was observed by variation of the bulk density of the coal blend charge. The effect of bulk density on IGP and WP is well known and an increase of bulk density results in most cases in an exponential increase of IGP and WP. Table 3.3.4 displays the impact of bulk density variation on the IGP and WP of the charged coal blend.

Table 3.3.4: Influence of bulk density on IGP and WP

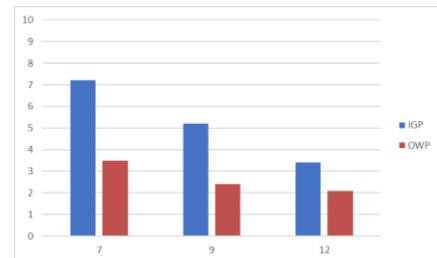
Bulk density	kg/m³	700	750	800
IGP	kPa	1.8	5.2	13.3
WP	kPa	2.1	2.4	7.1



Another effect that is strongly linked to the bulk density is the moisture content of the charge. It is well known that the internal gas pressure of coal blends exponentially increases with decreasing moisture content due to the increase of gas evolved and resistance of gas flow in the plastic layer by the increase of bulk density. Therefore, DMT has adjusted the same bulk density at each moisture content to eliminate the effect of bulk density. Table 3.3.5 indicates the reducing effect of higher moisture content on IGP and WP most likely caused by increased permeability of the plastic layer.

Table 3.3.5: Influence of moisture content on IGP and WP

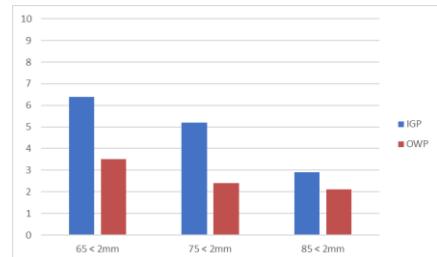
Moisture content	%	7	9	12
IGP	kPa	7.2	5.2	3.4
WP	kPa	3.5	2.4	2.1



DMT has adjusted the same bulk density as well for the investigation of the effect of grain size because the fineness of a coal blend has huge impact on the bulk density. Table 3.3.6 displays that an increase in fineness reduces the IGP and WP.

Table 3.3.6: Influence of grain size < 2 mm on IGP and WP

Grain size < 2 mm	%	65	75	85

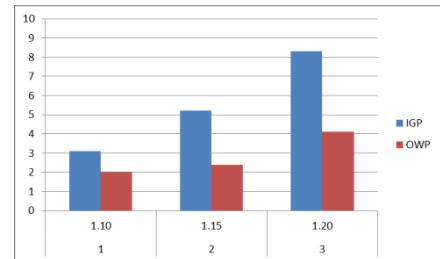


IGP	kPa	6.4	5.2	2.9
WP	kPa	3.5	2.4	2.1

The effect of coal rank on internal gas pressure and wall pressure at the coal blend charge is shown in Table 3.3.7. As can be seen, internal gas pressure increases with increasing coal rank. Results of this test series for internal gas pressure and wall pressure seem to tend an agreement with other works. Coal blends with higher portion of high volatile matter coals are not dangerous at normal bulk density. But, with increasing of a dangerous low volatile US coking coal the IGP and WP increase clear.

Table 3.3.7: Influence of coal blend rank on IGP and WP

Coal blend rank Ror	%	1.10	1.15	1.20
IGP	kPa	3.1	5.2	8.3
WP	kPa	2.0	2.4	4.1



### Investigation in Stamp charging

#### Methodology

CPM directly uses an industrial blend of AGDH coke plant to perform this study. This coal blend was used for the investigation of the influence of coking rate, bulk density, grain size and moisture content on internal gas pressure and wall pressure. The test conditions are shown in Table 3.3.8.

Table 3.3.8: Coking conditions

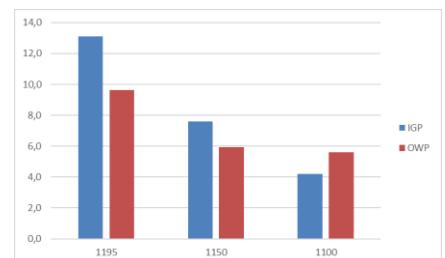
Parameters		CPM Standard	CPM
Coking rate	°C	1195 (pushing 1020°C)	1150 + 1050
Density	kg/m <sup>3</sup> (obtained not controlled)	1005	1055
=> Stamping energy was modified		Normal process	High energy process
Grain Size	% <2mm	87	79 + 83
Moisture	%	11	9 + 13

#### Results and discussion

Like DMT, the variation of the heating flue temperature has adjusted different coking rates. Table 3.3.9 shows the resulted internal gas pressure and wall pressure by different heating flue temperatures.

Table 3.3.9: Influence of coking rate on IGP and WP

Temperature	°C	1195	1150	1100
Density	kg/m <sup>3</sup>	1024	1018	1018
IGP	kPa	13.1	7.6	4.2
WP	kPa	20.7	13	11.2

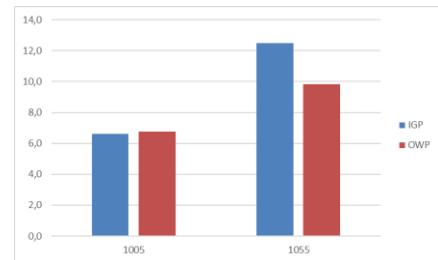


Like in gravity charging, similar conclusions were observed: an increase of 50°C has a consequence to double IGP. Effect on Oven Wall Pressure is less important, but an increase is also present. It can be noticed that during these different tests, bulk density is similar.

At CPM, bulk density is not controlled in stamp charging, indeed they control stamping energy like in industrial charging machine. Thus, to investigate impact of density, stamping energy was modified: high energy process was applied, i.e. energy to stamp the coal cake energy was doubled. This modification allows to obtain very high density as shown on Table 3.3.10.

Table 3.3.10: Influence of stamping energy on IGP and WP

Energy Stamping		Normal process	High energy process
Density	kg/m <sup>3</sup>	1005	1055
IGP	kPa	6.6	12.5
WP	kPa	6.8	9.8



The effect of bulk density on IGP and WP like in gravity charging was confirmed: an increase of 50kg/m<sup>3</sup> doubles the IGP. Effect on Oven Wall Pressure is less important, since during this test, maximum of IGP and OWP was not simultaneous.

Investigation of moisture and grain size was also performed in this task. Results are presented in Tables 3.3.11 and 3.3.12. As discussed previously, during this investigation of these two operational parameters, stamping energy was kept constant. Thus, like in industrial ovens, effects of grain size or moisture is linked with bulk density. The influence of a low grain size and the resulting higher gas pressure is seen very well, as well as the effect on Oven Wall Pressure. These results are fully in agreement with observations obtained in Task 2.3.

Table 3.3.11: Influence of moisture on IGP and WP

Moisture	%	9	11	13
Density	kg/m <sup>3</sup>	1001	1005	1024
IGP	kPa	7.0	6.6	13.1
WP	kPa	5.1	6.8	9.6

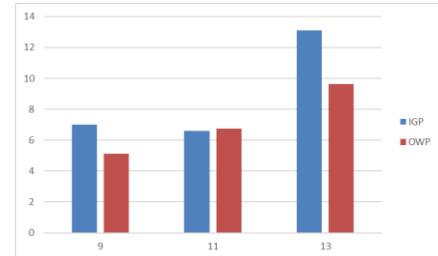
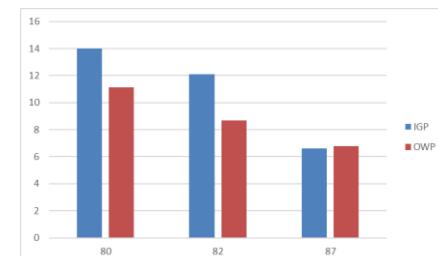


Table 3.3.12: Influence of grain size on IGP and WP

Grain size	% < 2mm	79	82	87
Density	kg/m <sup>3</sup>	1038	1029	1005
IGP	kPa	14	12.1	6.6
WP	kPa	11.2	8.7	6.8



### Factors influencing coal bulk density

The previous benchmarking studies on the two different charging methods demonstrate the influence of coal bulk density on IGP. These findings can be supported by various carbonization tests. Figure 3.3.1 shows, for example, the increase in IGP of KBS blend caused by an increase in the charge bulk density. The tests were conducted in a 10-kg carbonization retort when keeping all parameters constant and only varying the charge bulk densities. The question remains whether such increase is generally caused only by the increased bulk density or whether it is also supported by other factors contributing to that increase in the charge bulk density such as coal grain size or

moisture content, i.e. the factors playing their significant roles in top charge operations where the coal charge is not densified by service machines.

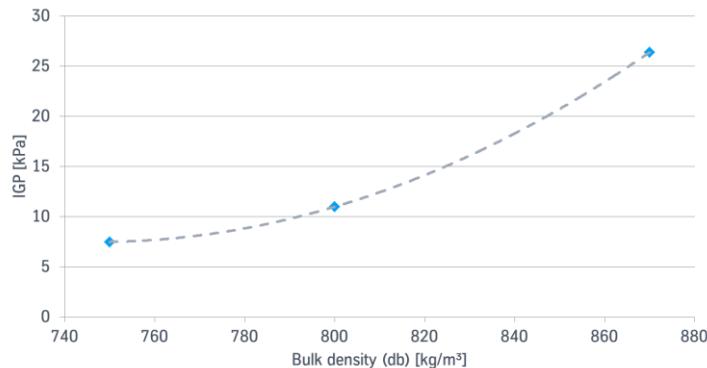


Figure 3.3.1: Relationship between IGP and bulk density

Based on the results obtained from KBS blend sampled at the end of the pile-use period and delivering IGP value of 32 kPa, several investigations were executed to identify whether it was either coarser grain size or an increase in coal bulk density which caused the increase in IGP. The measurement at the plant was taken at the time when emergency reclaiming of the blend from the blending pile had to be organized due to temporary malfunction of blend preparation facilities. Emergency reclaiming resulted in a much coarser grain size of the blend. The arithmetic mean grain size of the blend was 2.1 mm, which was remarkably larger than the target of 1.3 mm to 1.5 mm. Figure 3.3.2 shows the results from 10-kg carbonization tests conducted with two different grain size levels at two different bulk density levels. The results indicate that the charge bulk density has significantly greater influence on IGP than the grain size does. However, it must be kept mind that the grain size influences the bulk density and therefore does have as such an indirect influence on IGP.

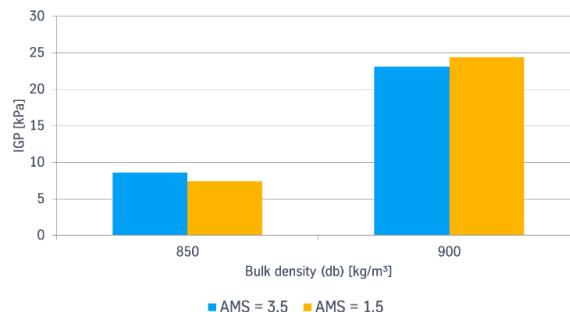


Figure 3.3.2: Influence of coal grain size on IGP

In addition to coal grain size, coal moisture also has significant impact on the charge bulk density. The company AGDH investigated the influence of moisture on IGP during the project (Task 2.2). Figure 3.3.3 shows that AGDH could not find any correlation between moisture content and IGP. These results demonstrate that the charge bulk density is the determinant factor dominating over other factors that influence the charge bulk density.

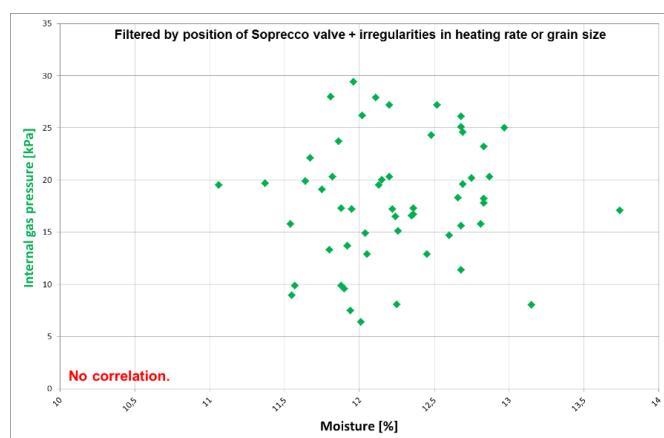


Figure 3.3.3: Relationship between moisture and IGP at ZKS

### Prediction and pre-calculation of the IGP

In parallel, voestalpine and AM Ostrava developed an approach to predict and pre-calculate the IGP based on the most influencing parameters.

In order to develop IGP prediction formula reflecting the operation conditions in stamp charge ovens, several modelling steps were conducted in ArcelorMittal Ostrava. In the first phase the results of IGP measurements executed in industrial size ovens of battery No. 2 were compared in pair correlation with operational parameters. Correlation matrix is listed in Table 3.3.13.

Table 3.3.13: Correlation matrix for operational parameters at AMO

Variable	coking time	Tpmax	coking rate	wall temp	IGPmax
coking time	1,00	-0,30	0,28	-0,35	-0,32
Tpmax	-0,30	1,00	0,24	0,42	0,33
coking rate	0,28	0,24	1,00	0,28	-0,01
wall temp	-0,35	0,42	0,28	1,00	0,32
IGPmax	-0,32	0,33	-0,01	0,32	1,00

The relationships are very poor; the highest correlation coefficient for IGP is R = 0.3 with Tpmax (temperature in the middle of the charge at IGP max).

In the second phase, IGP results were correlated with the quality of the charge with respect to its rheology, coal rank and blend preparation procedure. Correlation coefficients between IGP and coal quality parameters are also very low; the highest relationship was determined for dilatation b, fluidity Fmax, and grain size distribution. An overview of mutual interdependencies is listed in Table 3.3.14.

Table 3.3.14: Correlation matrix for coal charge quality parameters at AMO

	Wtr	Ad	Vd	SI	b	Fmax	FR	Inertinite	Ro	<3.15	<0.5	IGPmax
Wtr	1,00	0,06	0,31	0,22	0,00	-0,17	-0,16	-0,26	-0,09	0,14	0,05	0,09
Ad	0,06	1,00	0,27	0,51	0,07	-0,14	-0,06	-0,33	0,02	0,00	0,21	-0,17
Vd	0,31	0,27	1,00	0,00	0,04	-0,25	-0,20	-0,49	0,03	0,09	-0,02	0,18
SI	0,22	0,51	0,00	1,00	0,42	-0,13	0,16	-0,47	0,12	-0,02	0,09	-0,20
b	0,00	0,07	0,04	0,42	1,00	0,22	0,22	-0,18	0,39	-0,20	-0,12	0,38
Fmax	-0,17	-0,14	-0,25	-0,13	0,22	1,00	0,49	0,35	0,27	-0,32	-0,07	0,37
FR	-0,16	-0,06	-0,20	0,16	0,22	0,49	1,00	-0,18	0,14	-0,09	0,11	0,11
Inertinite	-0,26	-0,33	-0,49	-0,47	-0,18	0,35	-0,18	1,00	0,13	-0,07	-0,02	0,00
Ro	-0,09	0,02	0,03	0,12	0,39	0,27	0,14	0,13	1,00	0,11	0,06	0,10
<3.15	0,14	0,00	0,09	-0,02	-0,20	-0,32	-0,09	-0,07	0,11	1,00	0,68	-0,36
<0.5	0,05	0,21	-0,02	0,09	-0,12	-0,07	0,11	-0,02	0,06	0,68	1,00	-0,32
IGPmax	0,09	-0,17	0,18	-0,20	0,38	0,37	0,11	0,00	0,10	-0,36	-0,32	1,00

Aiming at achieving the most reliable IGP prediction model, regression analysis was executed in the third phase using the variables with the highest relationship to IGP. Pairwise correlation coefficients of all investigated parameters are arranged in order and illustrated in Figure 3.3.4.

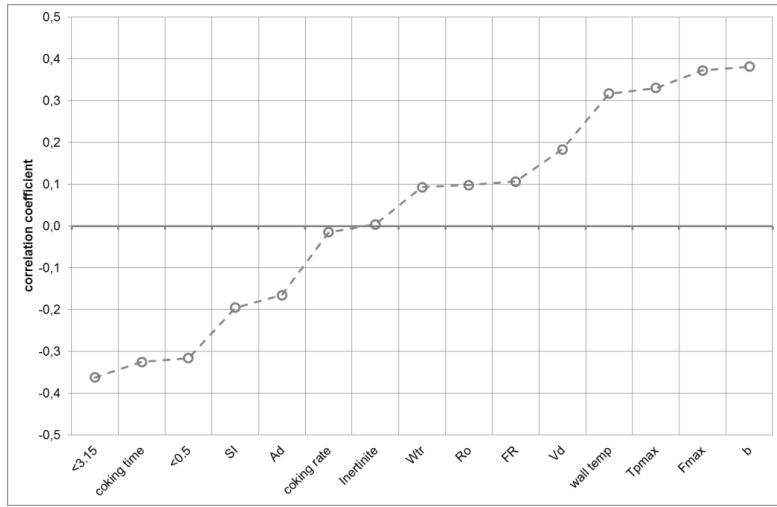


Figure 3.3.4: Pairwise correlation coefficients (R) for coal operational & quality characteristics at AMO

The best parameters were chosen according to their correlation coefficients shown in Table 4 and Table 5, specifically: grain size distribution represented by fractions <3.15 mm and <0.5 mm; carbonization conditions represented by oven wall temperature, temperature at IGPmax, coking rate, and coking time; coal charge rheology represented by dilatation b, fluid range, and maximal fluidity Fmax; coal rank represented by mean random reflectance of vitrinite Ro, and volatile matter content Vd. Using the selected parameters, a few multiple regression models were built. Five multiple linear regression models 1-5 are complemented by five models 1B-5B calculated on the same principle but with limited number of recorded data. In total, 4 lines were not used in B-models calculation with the aim of testing these models for a new data entry. The coefficients of all models are listed in Table 3.3.15.

Table 3.3.15: Calculated coefficients and constants Abs for the prediction of IGP at AMO

	Model	Abs	coking time	Tpmax	coking rate	wall temp	Vd	b	Fmax	FR	Ro	<3.15	<0.5
all available measurements	model 1	-170,9	7,977	0,052	0,568	0,058	4,461	0,308	0,021	-0,197	-25,274	-0,026	-0,567
	model 2	-51,309	-6,185	0,064		0,045		0,250	0,014			0,073	-0,713
	model 3	-438,65	8,138	0,028	3,041	0,069	10,550	0,260	0,030	0,157	11,514	0,212	-0,878
	model 4	-467,65	10,948	0,023	3,593	0,069	11,263	0,274	0,031	0,203	12,401	0,212	-0,909
	model 5	-399,13	5,598	0,042	2,242	0,067	9,665	0,308	0,031	0,001	14,223	0,053	-0,606
with external data	model 1B	-149,95	6,755	0,060	0,512	0,055	3,992	0,322	0,021	-0,200	-40,764	0,104	-0,614
	model 2B	-51,162	-4,412	0,072		0,041		0,243	0,013			0,061	-0,688
	model 3B	-508,89	1,407	0,022	3,542	0,076	11,974	0,175	0,027	0,349	31,207	0,251	-0,930
	model 4B	-542,76	3,581	0,011	4,329	0,077	12,913	0,186	0,027	0,409	35,316	0,179	-0,978
	model 5B	-520,4	-1,718	0,056	1,910	0,074	11,686	0,179	0,026	0,228	37,875	0,440	-0,630

Qualitative indicators of multiple regression analysis for all models are listed in Table 3.3.16 where the item "no of parameters-columns" quantifies the number of independent variables used in respective model, and MEP is mean quadratic error of prediction calculated as follows,

$$\text{MEP} = \sum_{i=1}^n [y_i - \mathbf{x}_i^T \mathbf{b}(i)]^2 / n$$

where  $\mathbf{b}(i)$  is the estimation of regression model parameters when all points except the  $i^{\text{th}}$  ( $i^{\text{th}}$  row  $\mathbf{x}_i$  of matrix  $\mathbf{X}$ ) are used; AIC – the Akaike Information Criterion as common estimator of the relative quality of statistical models for a given set of data.

Table 3.3.16: Indicators of investigated IGP prediction models at AMO

evaluation parameter	model 1	model 2	model 3	model 4	model 5
calculation method	Least squares	Least squares	Robust IRWLS	Robust IRWLS	Robust Welsch
no of parameters-columns	11	7	11	11	11
Multiple correlation coeff. R :	0,756	0,714	0,944	0,957	0,963
MEP	63,35	55,07	4178,12	65,19	71,67
AIC	170,40	168,07	112,78	101,20	94,98
Residual sum of squares :	1371,1	1569,2	347,7	269,5	231,6
Mean absolute residual	4,727	5,051	4,5	4,0	4,1
Variability explained by model	57,12%	50,92%	89,12%	91,56%	92,75%
	model 1B	model 2B	model 3B	model 4B	model 5B
correlation coeff. R with external data	0,757	0,713	0,664	0,653	0,653
Mean absolute residual	4,72	5,10	4,83	4,90	4,97
R different	0,001	0,001	0,280	0,304	0,310
Max absolute residual	12,31	13,64	29,47	31,74	25,60

The models differ in the number of used parameters and calculation method. In the bottom part of the table, qualitative parameters of similar models, marked as B-models, are listed after withdrawal of 4 lines. Such limited datasets were used in order to test the stability of the model for a new data entry. Minimum difference between correlation coefficients with and without new data is between Model 1 and Model 2. Model 1, which is calculated by least square method, has also the smallest mean absolute residuals and minimum maximal value of absolute residual. Considering Model 1 as the best available IGP predictor, Figure 3.3.5 shows regression plot between predicted and measured IGP.

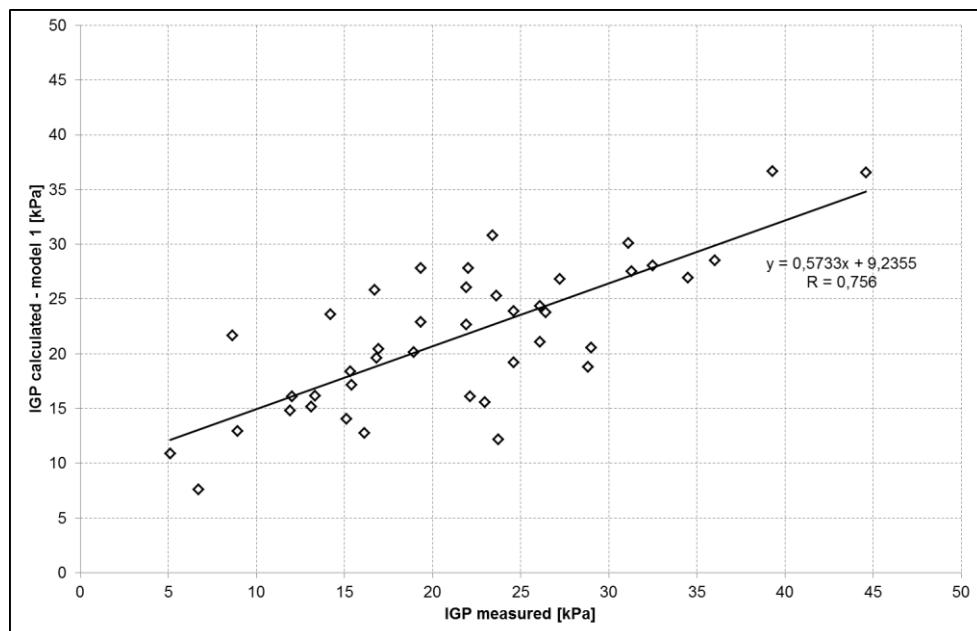


Figure 3.3.5: Measured and calculated IGP of AMO coal blends

At the beginning of the project, voestalpine developed a formula for the IGP, which is based on the contraction/expansion of the coal cake. This parameter is measured in the Sole Heated Oven (SHO), the measurements itself were carried out in lab in the USA according to ASTM D2014-97

(2010). Figure 3.3.6 shows the correlation between the SHO contraction and the IGP for single coals (tested in the coking retort).

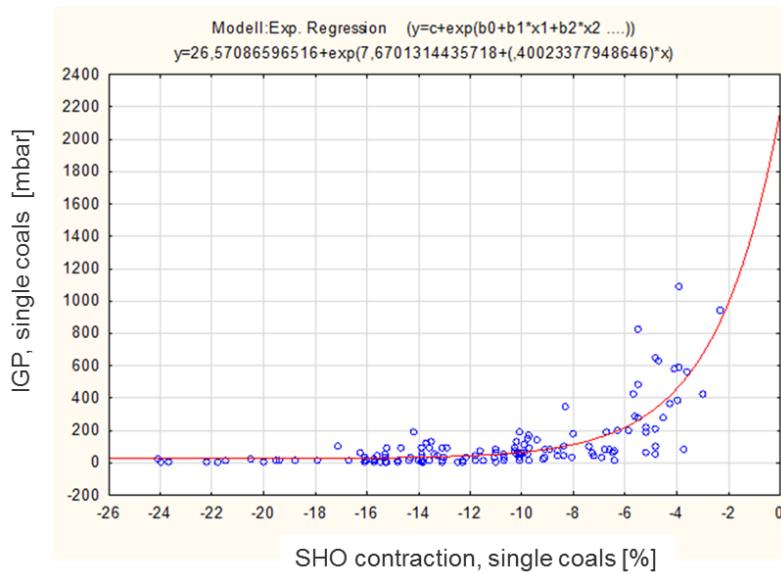


Figure 3.3.6: Correlation between the SHO contraction and IGP for single coals

This correlation between the internal gas pressure and the contraction of single coals can be described as follows:

$$IGP = a + e^{(b + c * \text{Contraction})}$$

a, b, c ... absolute terms, Contraction ... Contraction/Expansion measured in the SHO [%]

The shrinkage of a coal blend can be calculated (additive calculation on basis of the amount and contraction of the single coals used in the blend). This calculation gives at least a good indication for the IGP. The main drawback of this method is, that the lab of voestalpine is not equipped with a SHO device. Therefore, these tests are carried out in a lab in the USA. This means, that the test results are not available on time, it takes some months between sampling and receiving the results. Thus, a co-operation with the "Heuristic Lab" of the "Fachhochschule" (Technical college) Hagenberg was started to develop a heuristic algorithm to predict the IGP. This institute is specialized in data mining. The constraint for the development of this model is to use coal characteristics which are available every month from all single coals which are charged in the industrial coke ovens. These single coals and coal blends are also used for carbonization tests in the coking retort including the measurement of the IGP.

Heuristic algorithms are non-linear models. Single coals are separated in a training and in a test set. The goal was to create a global model, so there is no separation in "harmless" and "dangerous" coals. The final model, which delivers the best results, is introduced here. Most important impact factors are the content of volatile matter and the dilatation. The other parameters: content of oxygen of the coal, and amount of vitrinite reflection classes > 1,60 (> V 16) are of minor importance. These impact factors are in agreement with investigation of parameters in top charging, specially Table 3.3.17.

The impact factors were developed by the FH Hagenberg, and show the importance of variables (they are no coefficients). Table 3.3.17 shows the variables, which were used for this model and the impact factors.

Table 3.3.17: Variables and impact factors for the best model, created with heuristic algorithms for single coals

Variable	Impact Factor
Volatile matter, [%dry]	0.77
Dilatation d, [%]	0.20
Oxygen content, [%]	0.12
Vitrinite reflection classes > 1.60 (V16), [%]	0.02

Figure 3.3.7 shows the correlation for the training data (on the left with the green background), in blue color the measured IGP values (target values), in yellow the estimated values; on the right side (red background) the correlation for the test data is shown, in blue color the measured IGP values, in red color the estimated ones.

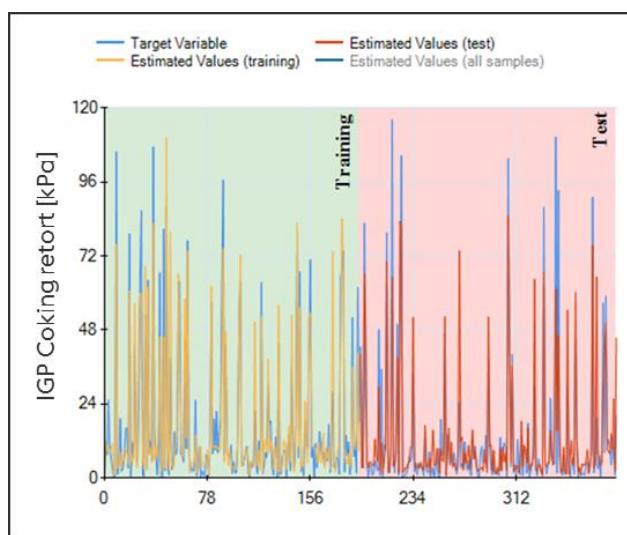


Figure 3.3.7: Best model for the prediction of the IGP of single coals, training and test set

Figure 3.3.8 presents the same data as a scatterplot:

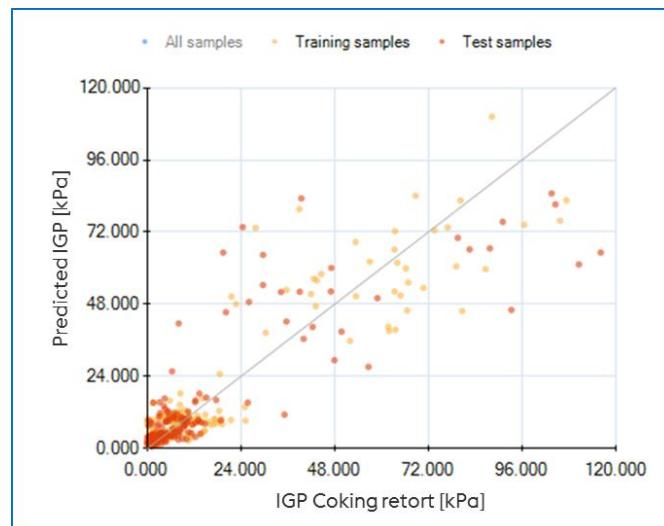


Figure 3.3.8: Scatterplot for the prediction of the IGP of single coals, separated in a training and a test set

Although it seems, that the statistical spread is rather high, the correlation between the measured and the predicted IGP is not that bad. Table 3.3.18 shows the absolute error and the correlation coefficient  $R^2$ .

Table 3.3.18: Absolute error and correlation coefficient for the prediction of the IGP of single coals

	Abs. Error [kPa]	$R^2$
Training	6.0	0.84
Test	6.0	0.73

It seems, that this method is suitable to predict the IGP for single coals with just a few parameters quite properly, except for very high IGP values. These high values are all from the same coal, which shows a rather high fluctuation of the IGP. For coal blends it was tried to find a prediction with the same method, but it was not possible to find a suitable formula, as the variation of the volatile matter content, and of the IGP is rather small.

#### **WP 4 Wall deformation and force transmitted to the chamber walls**

##### ***Task 4.1 - Measurements of wall deformation [AMMR, AMAL, AGDH, CPM]***

The previous work packages deal with measurements of Internal Gas Pressure. In this task, the objective is to measure the heating wall deformation generated by IGP. AMMR/CPM with the support of ArcelorMittal Florange coke plant and AGDH will measure heating wall displacement during the pressure peak.

In order to estimate the maximum wall deformation, two different systems were studied: a mechanical and an optical device. The mechanical measuring device allows [4][5]:

- To know wall deformations during a complete coking cycle,
- To measure their amplitude versus the dimensions of the ovens and the nature of the blend,
- To determine if the deformations are reversible or not,
- To find some correlations between IGP and max deformations

The aim of this measurement is to obtain maximum displacement of a heating wall during the junction of plastic layers. To obtain this value, measurements should be carried out in an empty oven during the maximum peak of Internal Gas Pressure of the neighbor oven (Figure 4.1.1).

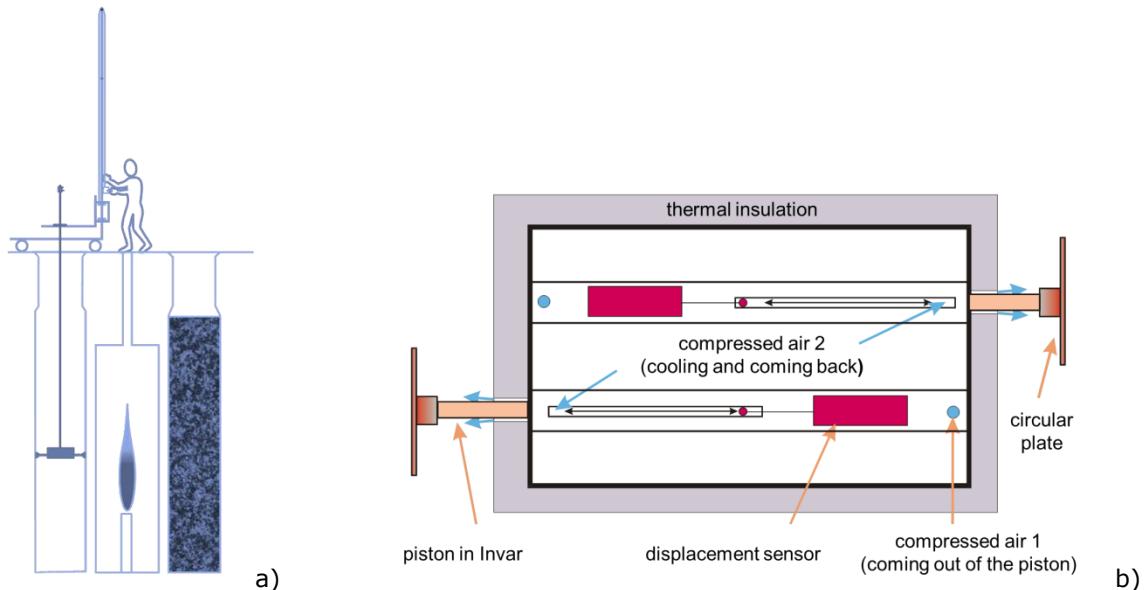


Figure 4.1.1: a) Handling of the mechanical sensor; Introduction inside an empty oven at mid-height of the oven and at different coking times of the adjacent ovens b) Principle of sensor

In 2018, this sensor was revamped by CPM with a special lift to handle the probe in a safe way (cf. Figure 4.1.2). Indeed, a safe methodology had to be established before to use this 5m long probe on the battery top. Despite this validation, the tool was not authorized to be tested at industrial scale at AMAL Florange due to safety reason, but also due to operational constraints at the coke plant.



Box with mechanical sensors inside



Upper part of the probe, with tubes for compressed air



Figure 4.1.2: Revamping of mechanical sensor

In parallel, another tool was developed by CPM to measure chamber wall dimensions at high temperature thanks to Laser 3D technology. CPM with the support of AGDH measured heating wall displacement during the pressure peak.

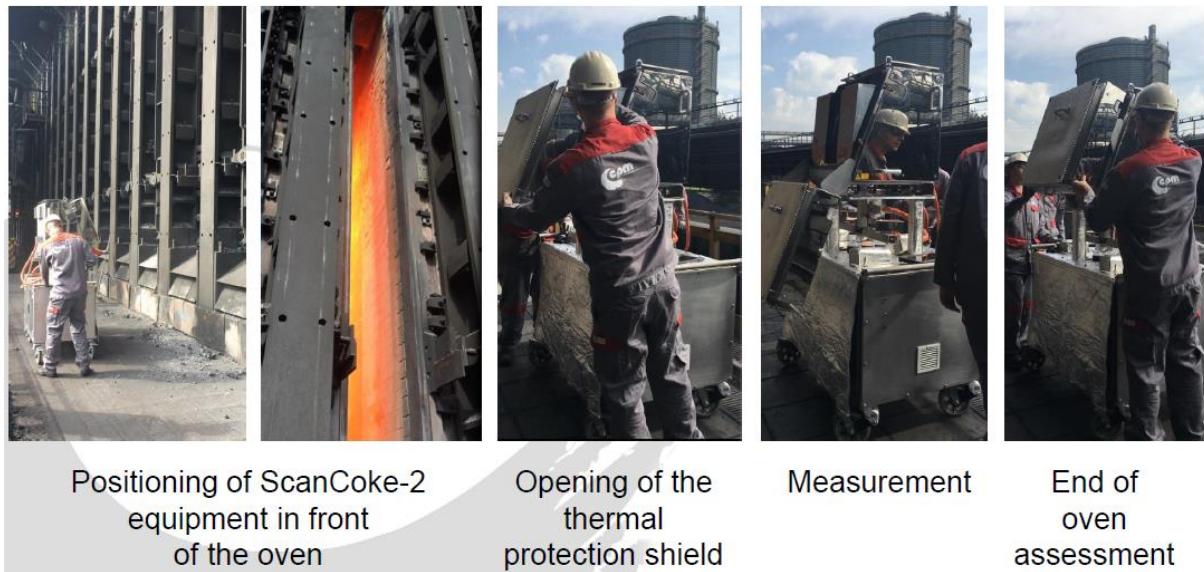


Figure 4.1.3: Main steps to install and measure wall deformation at AGDH

This equipment is based on Laser Technology which is capable to scan a whole coke oven within a few minutes. This equipment is a completely autarkic system and able to scan whole working day without any electrical support (Figure 4.1.3). In the frame of BINGO, the aim of this measurement was to obtain maximum displacement of a heating wall during the junction of plastic layers. To obtain this value, measurements were carried out in an empty oven during the maximum peak of Internal Gas Pressure of the neighbor oven (Figure 4.1.4). Due to high temperature condition, a continuous measurement of wall displacement is not possible, and so eight scans were performed during all the coking cycle (green arrows on Figure 4.1.5).

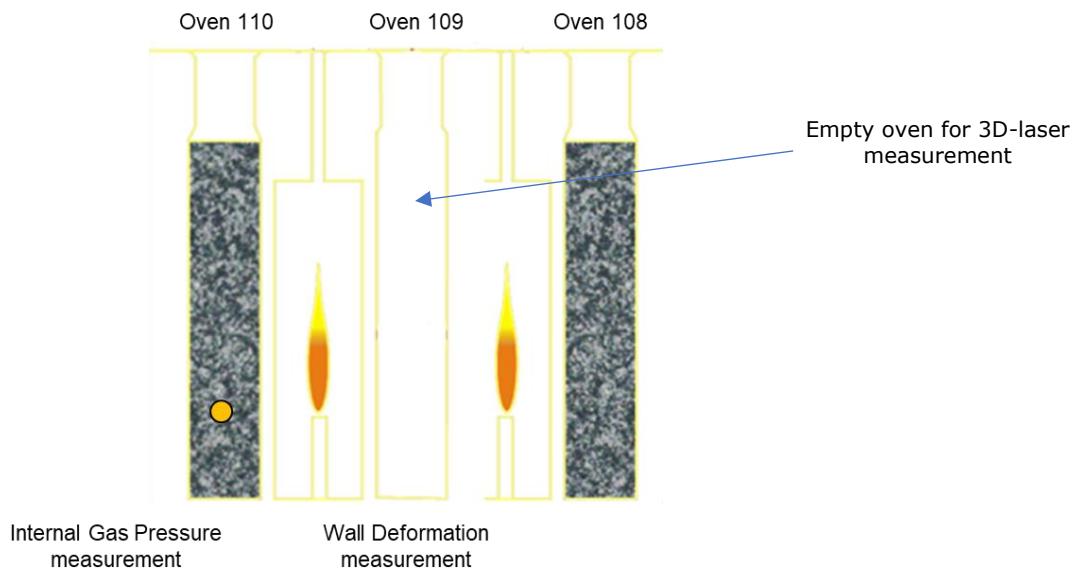


Figure 4.1.4: 3D-Laser measuring times based on a coking cycle and the related IGP curve

These eight measurements are planned to follow the main steps of coking cycle: just after charging, during water peak, after end of water plateau and 5 measurements during the junction of plastic players in order to catch the maximal wall displacement.

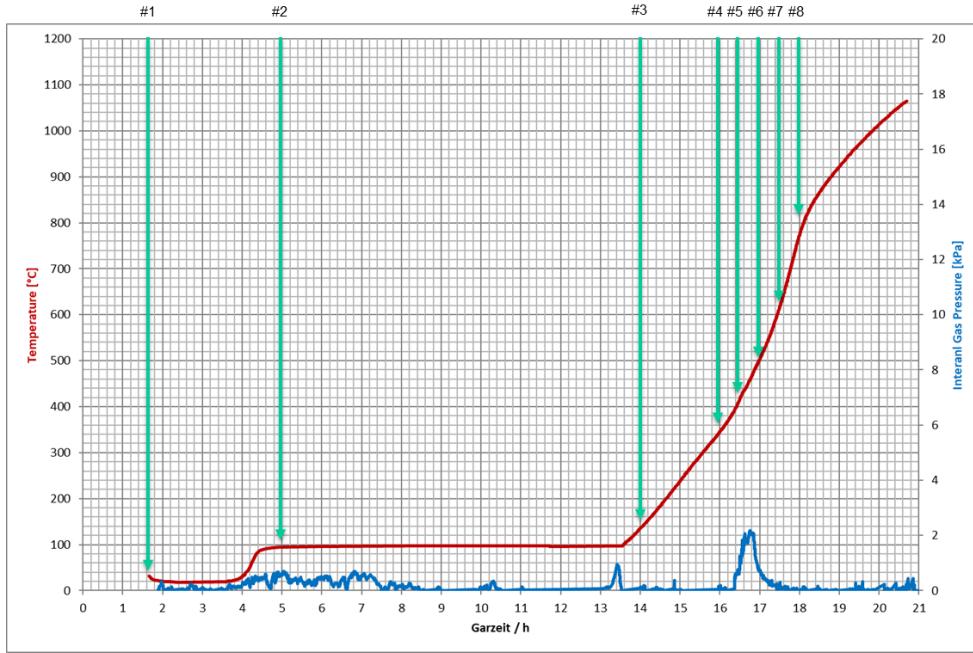


Figure 4.1.5: 3D-Laser measuring times based on a coking cycle and the related IGP curve

The measurements were planned for December 2018, but had to be postponed into January of 2019 due to some operational issues of the coke plant. They took place on the 16<sup>th</sup> of January at the oven 109 of ZKS coke plant. In parallel at oven 110 an internal gas pressure measurement was installed. Table 4.1.1 summarizes the timeline of the whole test run.

Table 4.1.1: 3D-Laser measuring times based on a coking cycle and the related IGP curve

Oven 110 IGP / Oven 109 empty for 3D-Laser	
	Time
Charging oven 110	16.01.2019 11:34
3D-Laser measurement after charging	16.01.2019 11:45
Drilling for IGP and placement of IGP probe in oven 110	16.01.2019 13:00
3D-Laser measurement after 5h	16.01.2019 16:34
3D-Laser measurement after 14h	17.01.2019 01:40
3D-Laser measurement after 16h	17.01.2019 03:30
3D-Laser measurement after 16h30min	17.01.2019 04:00
3D-Laser measurement after 17h	17.01.2019 04:30
3D-Laser measurement after 17h30min	17.01.2019 05:00
3D-Laser measurement after 18h	17.01.2019 05:30

In Figure 4.1.6 the recorded temperature and pressure profile is given. Next to it the correlation of the IGP peak with the position of the Soprecco valve is shown. Owing to a blocking problem of the probe the pressure curve showed no typical IGP behavior. The suspected IGP peak is in good correlation with the position of the Soprecco valve, but the entire graph is underestimated. Also, the temperature curve indicates a good positioning of the probe, as well as a stable heating rate.

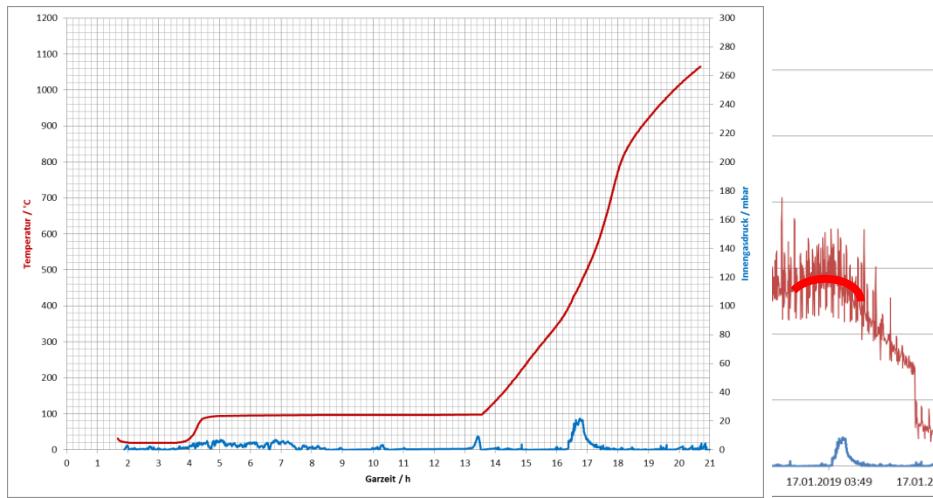


Figure 4.1.6: Recorded temperature and pressure curve of oven 110

In Table 4.1.2 there are selected important influencing parameters of the heating management or the summarized coke oven operation.

Table 4.1.2: Influencing parameter of the IGP measurement

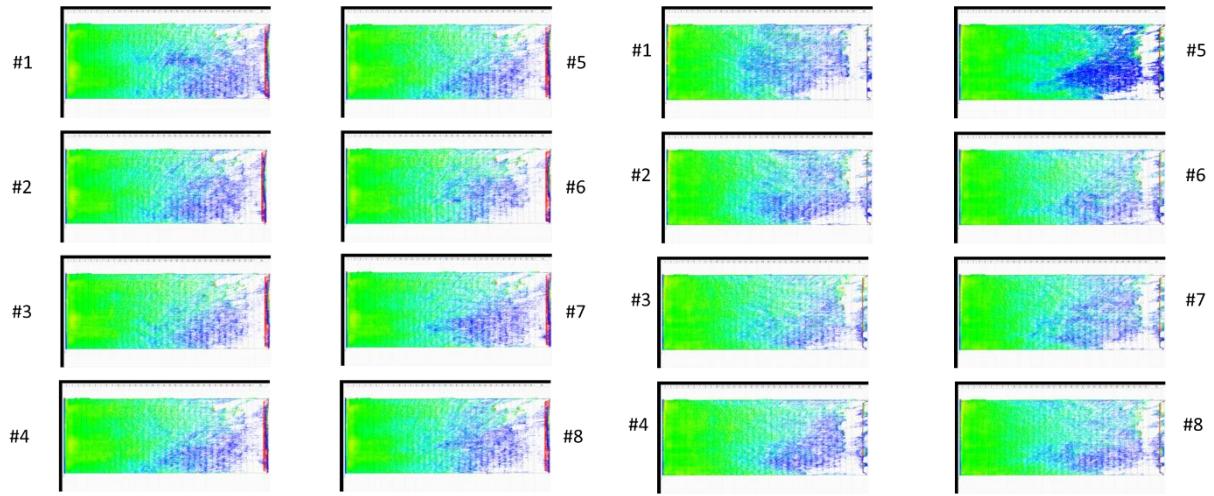
Date Oven	16.01.2019 110					
Pressure	Full area	mbar * h	31	H2O-Peak	Peak time	h
	Area peak	mbar * h	11		Peak intensity	mbar
	Peak time	h	16,76	Pushing force	Peak time	m
	Peak intensity	mbar	22		Peak intensity	kN
	Peak intensity	kPa	2,2	CTC-Temp	Coking time	hh:mm:ss
	FWHM	h	-		MV top	°C
Temperature	Heat Rate	K / min	3,01		MV bottom	°C
	t (500 °C)	h	16,99		MV complete	°C
	t (900 °C)	h	18,80	Grain size	H2O	%
	T (20 h)	°C	1015		< 2 mm	%
Heating flue measurements	1st measurement ΔT Heating flues 4	°C	32		< 1 mm	%
	2nd measurement ΔT Heating flues 4	°C	-46		< 0,5 mm	%
	3rd measurement ΔT Heating flues 4	°C	-6	Coal quality	H2O	%
	4th measurement ΔT Heating flues 4	°C	8		Ash wf	%
SOPRECO Correlation	Peak time (IGP)	h	16,76		VM wf	%
	Soprecco peak time	h	15,84		CSN	5
	Δt	h	0,92		Dilatation	%
	Subjective Soprecco correlation		+		MMR calculated	%
						0,832399

On the basis of the recorded process data there are no significant value given for a plausible explanation of the low pressure curve. The heating of the oven walls was in a regular way, as well as the quality of the used coal blend and the Soprecco positioning indicates a good correlation. During the test trial there has been twice a proof on gas leakages of the connections and fittings of the probe with no impairment. As a consequence, the underestimation could be caused by a blocking of the probe.

In terms of wall displacement, Figure 4.1.7 summarizes the 8 measurements in terms of wall deformations. These different maps display the deformations the wall in comparison with the theoretical dimensions of the oven.

Displacement of wall 109

Displacement of wall 110



Scale for wall deformation: from the chamber wall, red means outward deformation, whereas blue means deformation inward. Green means that there is no deformation

15.0mm  -15.0mm

Figure 4.1.7: Results of wall deformation measurement thanks to 3D laser Technology

It can be noticed that accuracy of measurement is decreasing with length of the oven as illustrated on next Figure. Indeed, scanning was performed from Pusher Side, and so density of points on Coke Side is less important.

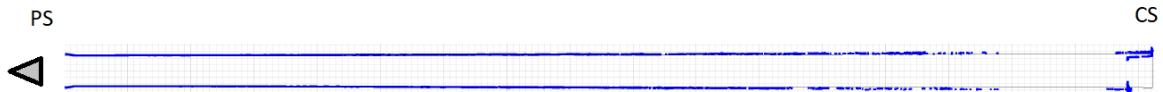


Figure 4.1.8: Section according the height highlighting that points are missing at the end of the oven

This loss of accuracy is visible by apparition blue color on the maps of Figure 4.1.7. In green color, there is a perfect agreement between theoretical and measured wall. Red color means that deformations exceed 15mm inside the chamber wall. Blue color means that there is a deformation in direction of heating flues. To study wall deformation during coking time, the 1<sup>st</sup> scan (#1) was used as reference since no wall deformation is expected just after charging in stamp charging. Thus, the next scans were analyzed relative to the 1<sup>st</sup> one as presented on Figure 4.1.9.

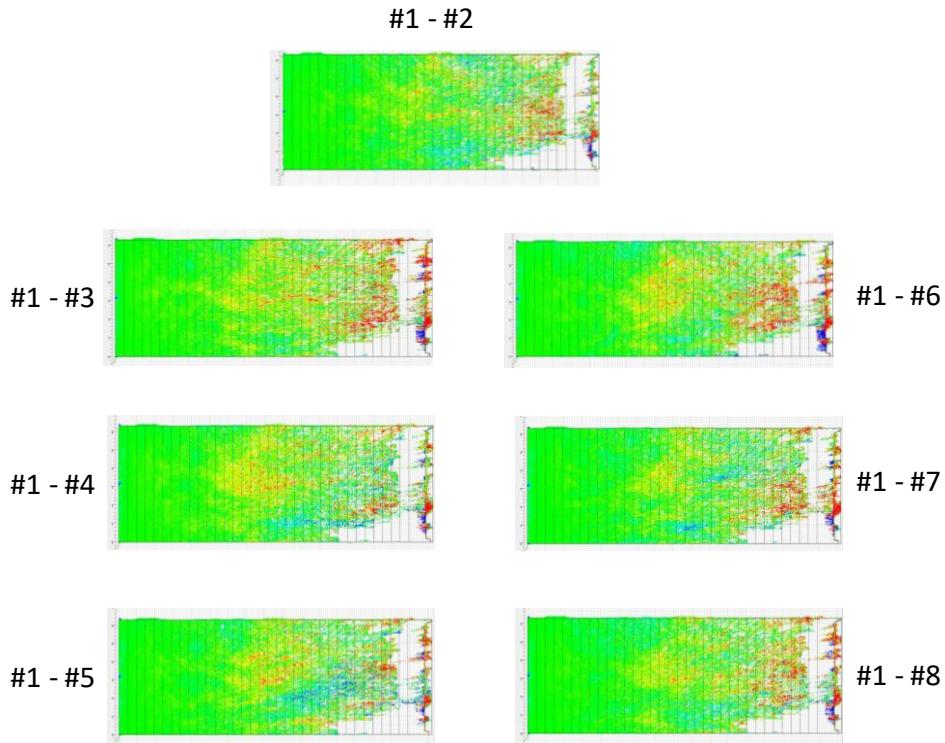


Figure 4.1.9: Map of wall deformation relative to the 1st scan

Results are in agreement with expected results, since obtained maps are in green-yellow color, which means that deformations in the range 0-5mm. The maximal deformations occur in the middle of oven, which is in agreement with expected results. Indeed, after several years of operating, it is not rare to observe deformed chamber walls like a "banana". In this measurement, it is difficult to explain the evolution of yellow deformation, since measurement of IGP was not operating, and so a doubt remains for timing of maximal IGP.

Moreover, in October 2000 at Dunkerque coke plant, similar measurements were performed with the mechanical sensor described in Figure 4.1.2 [4]. Results of wall deformation are outlined on Figure 4.1.10. The width of oven ranges from 448.5mm to 450.5mm, i.e. 2 mm of maximal deformation.

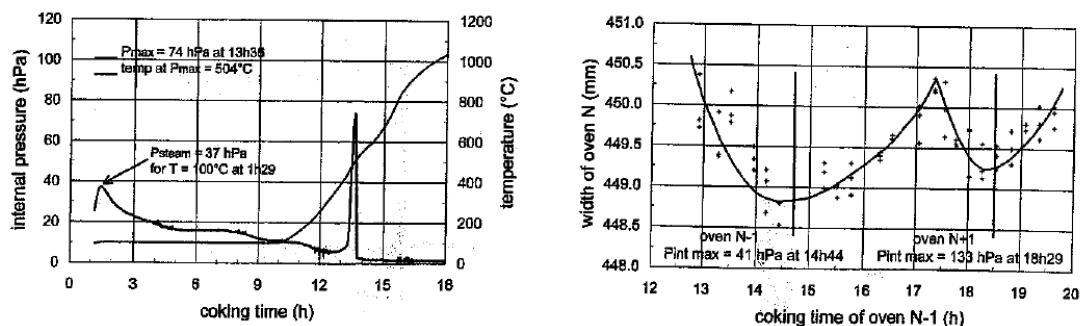


Figure 4.1.10: Wall deformation obtained at Dunkerque coke plant in October 2000 [4,5]

To conclude this Task 4.1, new equipment was used to quantify wall deformation in parallel of IGP measurement. Despite the weak value obtained of IGP owing to a blocking problem of the probe, it can be observed and measured that there is no important wall deformation with industrial blend used at ZKS coke plant. Thus, SUGA index for ZKS plant, underestimates the maximal limit.

#### **Task 4.2 - Use of heating wall model with different sets of boundary conditions. Comparison with industrial observations [AMMR, AMAL, AGDH, CPM]**

This task is devoted to developing and running calculation from the global 3D model of a complete heating wall which was established in the frame of RFCS SPRITCO [1] and COOL [2,3] projects.

This complete model takes into account both assembly of bricks and joints, material properties and also loads applied on the structure. Different parameters have to be used to build the model based on Finite Element Method:

- the complexity of the structure: heating walls include flues and are made of masonries with various types of bricks (with various shapes and materials). Moreover, the presence of joints has a strong influence on wall behavior since they can open in tension and close in compression;
- the bricks and joint material behaviors;
- the knowledge of thermomechanical loading: temperature field, weights of walls, roof and larry-car, pre-stresses (anchoring system), lateral pressure due to coal swelling.

### **Modelling Approach**

To build a 3D thermo mechanical model taking all parameters into account (bricks shape, brick and joint material behaviors and thermo-mechanical loads), a method based on periodic homogenization was used [1,2,3]. This method requires three steps: the first one is the identification of material parameters; the second one is the identification of representative elementary cell and finally, the last one is the determination of equivalent parameters. The Figure 1 illustrates the used method. For the heating wall two elementary cells were chosen: one for the liner wall and a second for the binder wall.

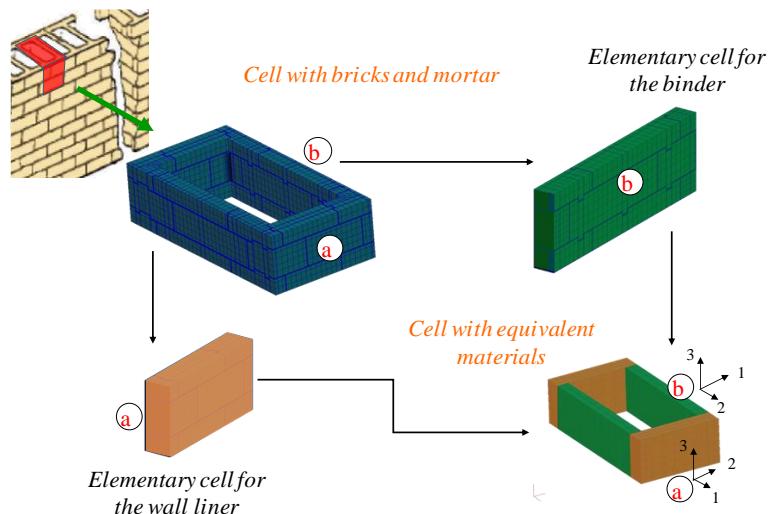


Figure 4.2.1: Representative elementary cell for heating wall modelling

During operating conditions, thermal and mechanical loads applied on the heating wall could generate tension regions where joints would be able to open and thus, the masonry behavior would be different. That is why it is very important to take into account the behavior of the brick-joint interface. In this study and as presented in numerous approaches, it can be assumed that fractures can develop only in the brick/mortar interface due to the small thickness of mortar.

In order to build an accurate thermo-mechanical model, a good knowledge of materials used for its design is essential. Tables 4.2.1 and Table 4.2.2 summarize properties of these materials used in the models.

Table 4.2.1: Thermo mechanical properties of Silica brick

KD Silica		
Density (kg.m <sup>-3</sup> )	1802	
35°C	1.36	
Thermal conductivity (W.m <sup>-1.K<sup>-1</sup></sup> )	890°C	1.83
	1180°C	2.04

	200°C	$0.97 \cdot 10^3$
<b>Specific Heat (<math>\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}</math>)</b>	800°C	$1.19 \cdot 10^3$
	1200°C	$1.28 \cdot 10^3$
	800°C	$1.51 \cdot 10^{-5}$
<b>Coefficient of thermal expansion (K-1)</b>	1080°C	$1.12 \cdot 10^{-5}$
	1350°C	$8.70 \cdot 10^{-6}$
	800°C	3880
<b>Young's modulus (MPa)</b>	1080°C	2280
	1350°C	1410

Table 4.2.1: Thermo mechanical properties of mortar

<b>Mortar KS-94</b>		
<b>Density (<math>\text{kg} \cdot \text{m}^{-3}</math>)</b>		1503
	25°C	0.68
<b>Thermal conductivity (<math>\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}</math>)</b>	891°C	0.56
	1197°C	1.24
	200°C	$0.95 \cdot 10^3$
<b>Specific Heat (<math>\text{J} \cdot \text{kg}^{-1} \cdot \text{K}^{-1}</math>)</b>	800°C	$1.13 \cdot 10^3$
	1200°C	$1.15 \cdot 10^3$
	800°C	$1.68 \cdot 10^{-5}$
<b>Coefficient of thermal expansion (K-1)</b>	1080°C	$9.09 \cdot 10^{-6}$
	1350°C	$7.57 \cdot 10^{-6}$
	800°C	360
<b>Young's modulus (MPa)</b>	1080°C	340
	1350°C	270

Calculation is performed in two steps: thermal, then mechanical loads. In the mechanical step, all loads (weight of roof, anchoring system, lateral pressure) are applied on the structure. Thus, a vertical pressure of 0.1 MPa is applied on the heating wall to represent the weight of roof. The efforts due to anchoring system are applied on the protection plate and are equal to constructor values. Dealing with lateral pressure, the distribution of swelling pressure along the height of the wall was introduced as nearly triangular shaped. An average swelling pressure of 10 kPa was chosen, with a value of 6 kPa at 6.4m and a maximum pressure at the bottom of the charge (15 kPa). Moreover, a shift in pressure peak from neighboring ovens is applied to take into account charging sequence as illustrated on Figure 4.2.2.

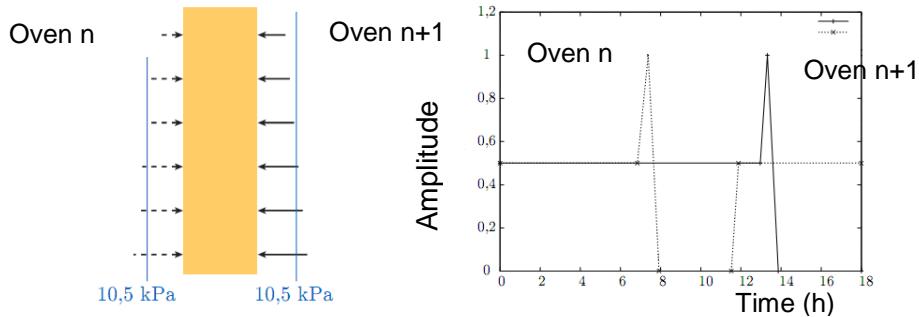


Figure 4.2.2: Coke swelling pressure

### Results

In this Task 4.2, AMMR in collaboration with University of Orléans has implemented a new heating wall geometry based on ArcelorMittal Gijon design instead of Florange as initially planned. Modelling of Gijon coke plant was done in 2016. Design of Gijon coke plants was sent by the coke plant in order to accurately model the heating wall masonry. At present, model is operational as presented on Figure 4.2.3.

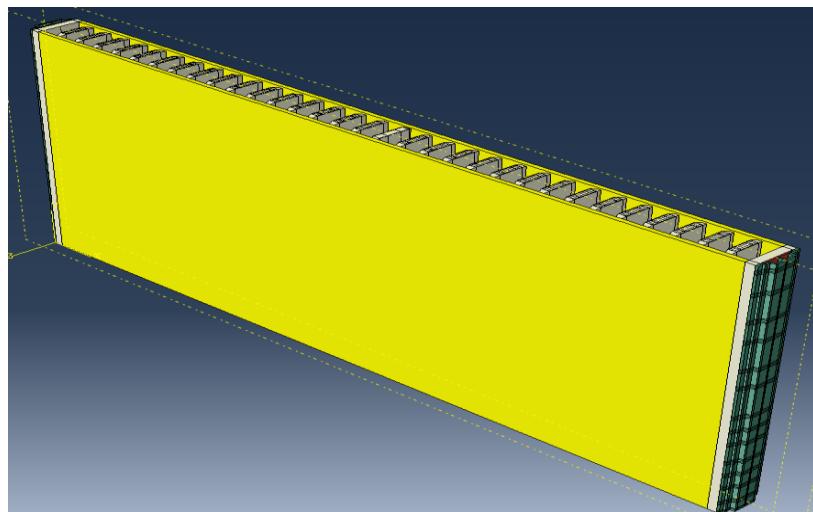


Figure 4.2.3: Modelling of Gijon heating wall

First calculations were performed without taking into account joint opening. The aim of these calculations was to study effect of chamber wall thickness on mechanical behavior of the heating wall. Two wall thicknesses were studied: 85 and 95mm. The Figure 4.2.4 presents the comparison of wall thickness in terms of wall displacement in the 2 cases.

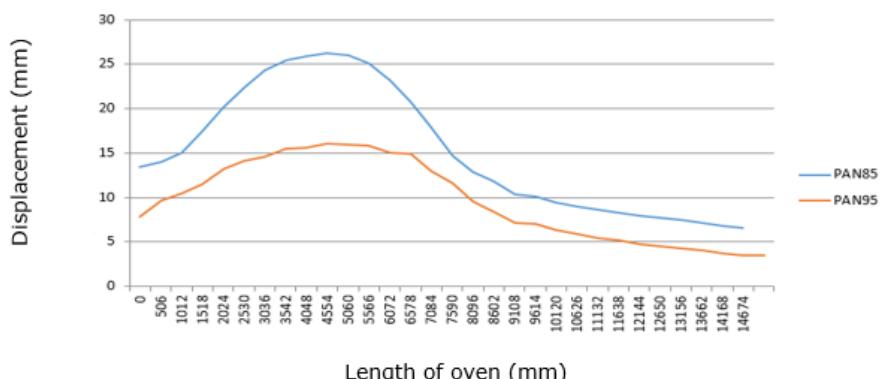


Figure 4.2.4: Influence of wall thickness in terms of wall displacements during plastic phase junction

These calculations give two interesting results: the 1<sup>st</sup> one which is quite intuitive, show that a thick heating wall has a higher stiffness, and so can absorb more important wall pressure; the 2<sup>nd</sup> one is the good agreement between magnitude of displacement obtained by the model and by equipment used to measure wall deformation during Task 4.1.

In 2017, this is the design of AGDH coke plants which was studied. At present, model is operational as presented on Figure 4.2.5.

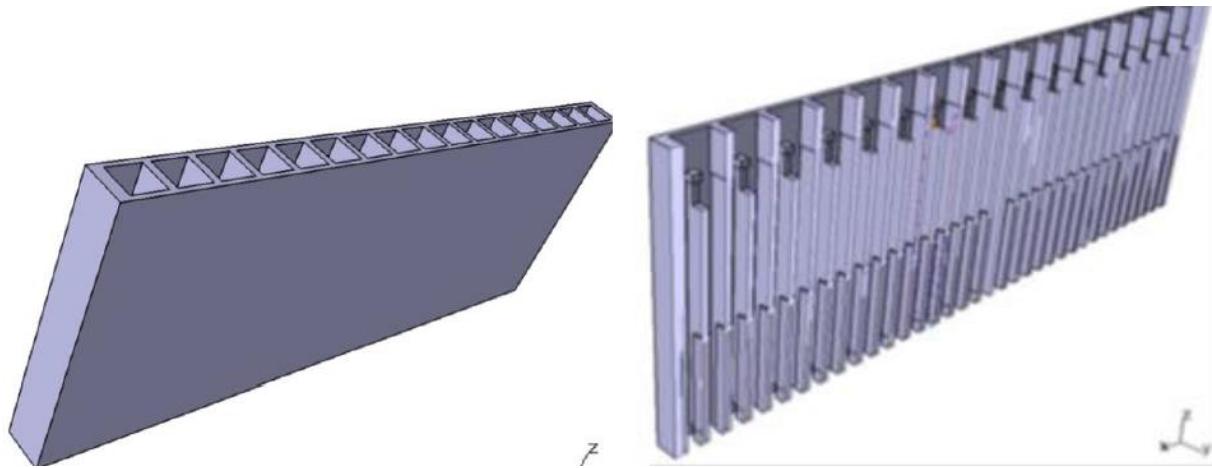


Figure 4.2.5: Modelling of AGDH heating wall

The simulations were also performed in 2 steps: thermal simulation, then mechanical simulation using the mechanical loads and the stress due to the thermal calculations. The Figure 4.2.6 presents the thermal field obtained on this geometry. Some hypothesis were performed like same vertical profile in the different heating flues, the average temperature at the interface between protection plate and the masonry is around 300°C [3], etc.

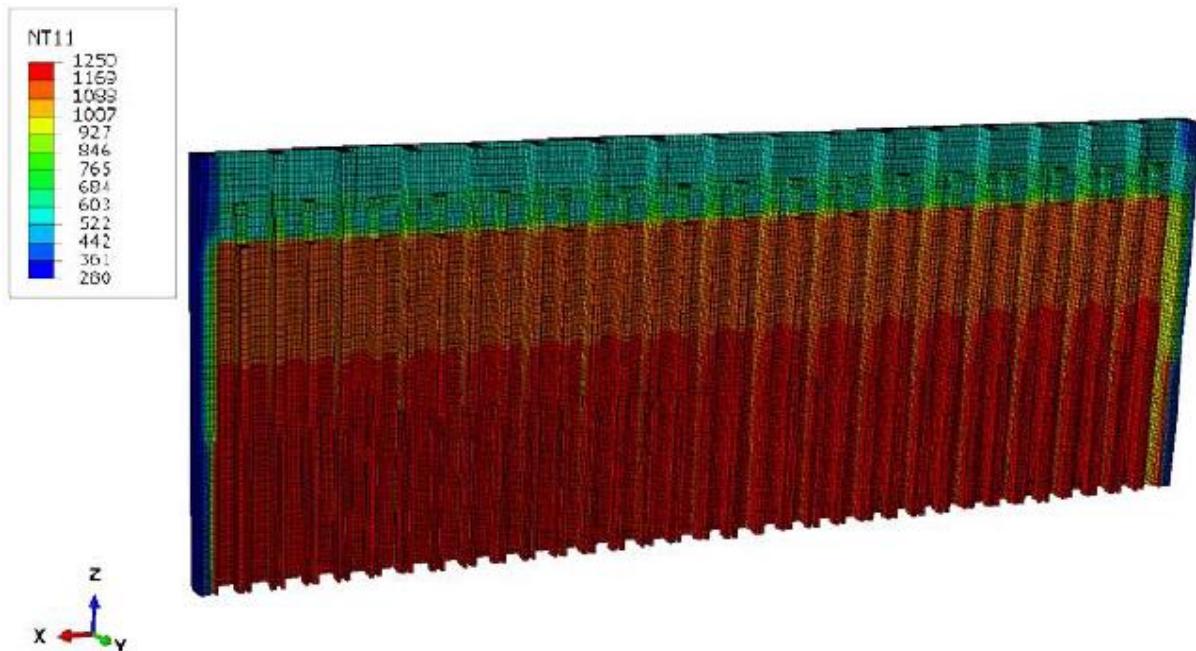


Figure 4.2.6: Thermal field of AGDH heating wall

The Figure 4.2.7 presents the stress field obtained after thermal and mechanical simulations. The results obtained are promising since thanks to this model, it is possible to obtain numerically the

location of weak zones on the masonry. Indeed, the red and yellow parts of the following figures indicate the tension area. As presented in the literature and other results obtained in the frame of previous RFCS report [1, 2], the tensile strength of coke oven masonry is very low, and so, it means that cracks should appear in this area.

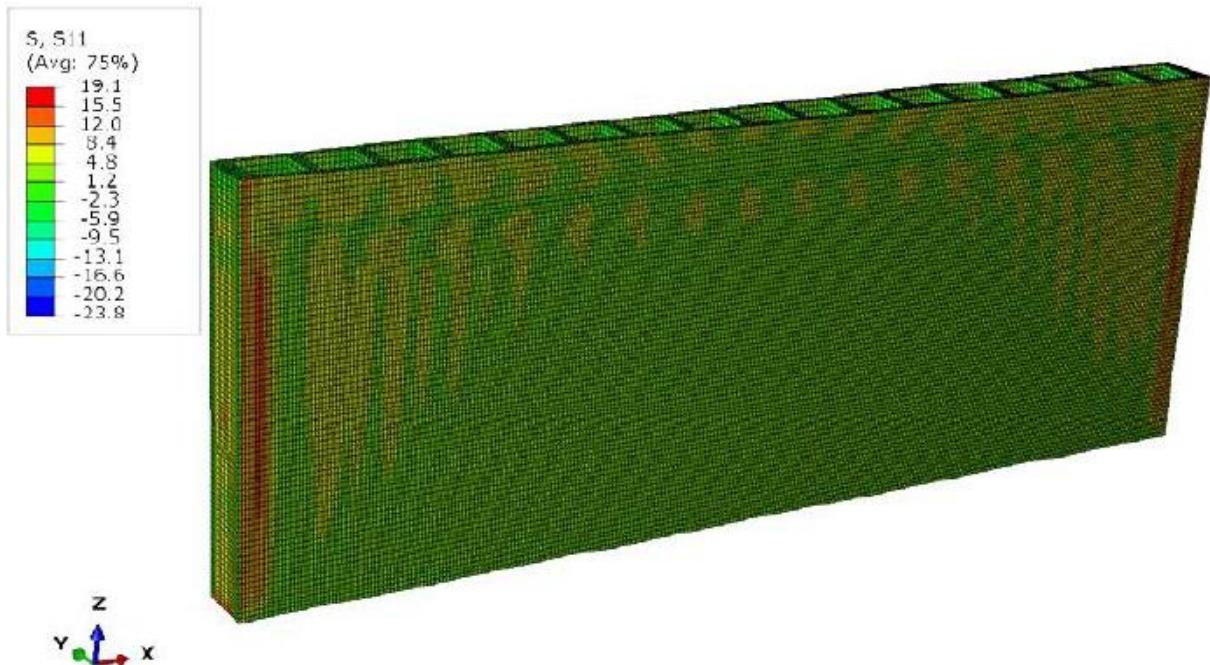


Figure 4.2.7: Stress field in direction 1 of AGDH heating wall

The weak areas are located at the entrance of heating wall and the upper part which is in agreement with industrial observations. On Figure 4.2.8, the drawing is an extract of industrial observations of damages in a coke oven. The colored drawings indicate the damages, and it is clear that cracks appear at the entrance of oven, and are vertical. This is similar to the obtained results of Figure 4.2.8.

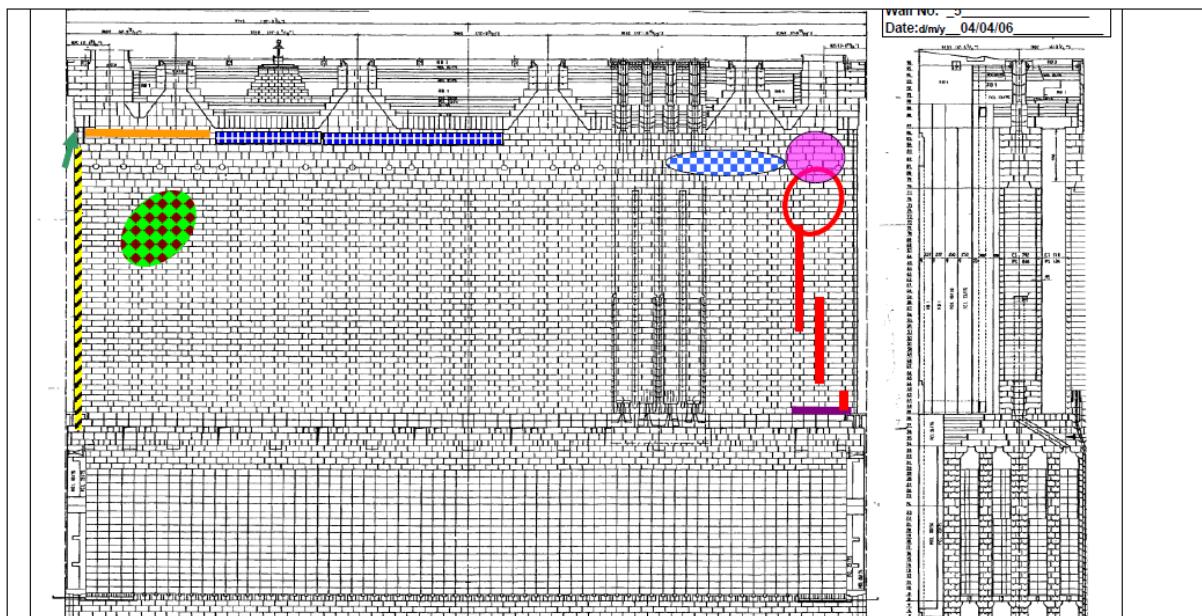


Figure 4.2.8: Observed damages at industrial scale

To conclude, the results of heating model could allow to estimate the deformation of heating wall and the maximum acceptable Oven Wall pressure and consequently to define threshold values for Internal Gas Pressure measured at industrial scale. The maximum deformation was compared with measurements obtained in Task 4.1, and the range of value is fully in agreement with experimental results. Nevertheless, such model based on Finite Element Method is time consuming in terms of

calculation and requires specific knowledge in modelling, and so cannot be deployed in industrial coke plants.

### **WP 5 Coordination meetings and reporting of the project**

Seven coordination meetings were organized during the period of the project to exchange the results and discuss the methodology to measure internal gas pressure in a coke oven but also to discuss the influencing parameters.

Meetings took place in each partner's laboratories to see the specific equipments used or developed to measure Internal Gas Pressure or Oven Wall Pressure.

## Conclusion

Various probe designs for measuring the internal gas pressure were tested and compared in industrial coke oven. Shapes of probes openings do not have a significant effect on the Internal Gas Pressure value. The simplest design represented by squeezed probe can be used for gas pressure measurement with a good reliability. On the other hand, the insertion angle has an impact on the estimation of the gas pressure. The best result is obtained with the vertical variant of the squeezed ending. Due to successfully testing, the squeezed vertical design has replaced the classical probe with slits and is becoming a new standard. To verify the correct position of the IGP probe in the coal cake at the junction of the plastic layers the correlation with the position of the Soprecco valve at AGDH showed satisfying results. In combination with observation of the heating management of the corresponding coke oven as well as the temperature profile of the IGP measurement, the accurate position in the oven can be assured.

The investigations on coal properties demonstrate that for a single coke plant, because of very tight coal blend specifications and blend design targets producing rather condensed set of data, the isolation of single factors influencing IGP can be very difficult from a general applicability point of view. Data comparison among several coke plants executed on larger sets of results has a better chance to reveal critical factors or boundary conditions providing that corresponding parameters are monitored at single plants on a routine basis.

The studies focusing on the charging methods have confirmed that the charge bulk density has a significant influence on IGP whereas later investigations demonstrated that the bulk density influencing factors have no important impact on IGP.

There are multiple coal characteristics that have an influence on IGP. Which characteristics are the most important ones, depends on the blend strategies applied at respective coke plant operations. The research conducted within the framework of this project on top charge blends has determined the volatile matter content and various petrographic data as the most decisive factors. However, blend modifications such as the use of inert additives have additional impact on IGP and shall be as such considered in both IGP predictions and limits which can be set by the operations based on the analysis of available data.

Similar statement concerning variability of input conditions and the resulting accuracy of model calculations also applies to the operating conditions. It was demonstrated by presented IGP predictions for stamp charge blends in which several process parameters were used and correlated with coal quality characteristics. However, under conditions of stamp charge operation one has to always consider the impact of extremely high densification of the charge which is intensifying the blend performance in terms of its IGP.

Effects of grain size, properties of the coal charge or influence of the heating rate were highlighted during industrial measurements. It was observed when grain size of the blend got coarser, the measured gas pressure rose significantly. But as mentioned before, these parameters are bulk density influencing factors than IGP factors. Furthermore, with an increase of the heating rate through a variation of the heating flue temperatures the IGP values were enhanced. Due to stamp charging conditions, the deviations of coal blend ranks were moderate, thus, their properties affecting the wall or gas pressure were very low, as a consequence the observation of any effects on IGP similarly.

A round robin was performed between the pilot ovens on gravity and stamp charging method in order to compare the results of the Internal Gas Pressure measured on the same blend in different pilot facilities. Results of partners are all in good agreement. Comparisons of pilot and industrial measurements were carried out in order to find the accordance or the functionality between the internal gas pressures, measured in pilot facilities with the industrial coal blend. A specific setup has to be implemented at pilot scale in order to be closed of the industrial measurements.

Measurement of deformation of the coke oven wall during a coking cycle was performed. In adjacent ovens there have been scans with a 3D-Laser of an empty oven and a regular IGP run. The IGP measurement showed a correct position of the probe and no significant value for the pressure influencing parameters. Results of the scans show that no wall deformation was noticed in normal operating conditions. Development of 3D model of heating wall was done and results are promising since a good agreement with industrial observations was found.

## **Exploitation and impact of the research results**

Numerous experimental campaigns were carried out in this project. Their aim was to better measure internal gas pressure. BINGO project allowed to define the best practices to measure IGP at industrial scale. Most of improvements can be used for daily industrial control of dangerous coals and there are also research tools to better understand the phenomena of IGP. At present, all industrial partners involved in the project have integrated in a routine way the measurement of internal gas pressure. The defined guideline could be used for all global coke plants. Participation to American and European conference in 2019, will allow to disseminate the results. Moreover, since AMAL and AMMR are partners of the project, developed equipment will be part of the Best Available Technology and so will be shared in all ArcelorMittal coke plants.

Industrial measurements of internal gas pressure should be done regularly, especially when the blend composition is modified. Measurements should be done with a probe inserted through oven doors at 0.5 to 1 m from the oven sole. The technique is available both for top charging and for stamp charging battery. Pressure in stamp charging must particularly be checked as the very high bulk density increases the pressure compared to conventional top charging. Operating a battery with a dangerous blend has dramatic influence on the service life. The cracks which appear in the brickwork cannot always be detected early enough. When pushing problems occur, the battery is generally damaged and repair work is necessary. Reconstructing headflues costs a lot of money (~300k€ for headflue repair on one heating wall) but is technically feasible. If cracks are in the middle of the wall, ceramic welding can still be a solution to the problem. But a damaged wall remains more fragile than walls in good condition. Thus, regular IGP measurement in industrial conditions remains as important routine activity in the portfolio of the best coke oven battery life prolongation practice.

Models are not enough mature to be used industrially on a routine basis. Thus, this work was also devoted to a heuristic model which is easy to use but needs to be extend to different ranges of coals. The 3D model of heating wall behavior is now representative and applicable to study the oven wall ageing. The limitation is that it requires long computational time and a specific powerful computer cluster (presently 6 days of calculation for 40 processors). Furthermore, to run the model with Abaqus software, many licenses of this software are necessary. For the moment only, a specialized laboratory can run it. Results obtained in this project could help the plants to better know the acceptable wall pressure limit for tall and small ovens.

### **Patents**

A patent is under examination about the new design of triple probe proposed by AMMR/CPM.

### **Publications / Conference presentations resulting from the project**

- VDKF Fachtagung 2017, 19.05.2017, Haus der Technik – Essen
- First Results of Comparison Between Industrial and Test Oven IGP Measurements at thyssenkrupp Steel Europe AISTECH 2018
- Innengasdruckmessungen im Kokslabor im Vergleich zur industriellen Messung an der Kokerei Schwelgern, Stahl und Eisen 138 2018 Nr. 8
- Měření vnitřního tlaku plynu v koksovací komoře v provozním měřítku, Czech Cokemaking Seminar, October 2018

Following papers will be presented in 2019 conferences:

- Bulk Density and Internal Gas Pressure in Coke Ovens, M. Hoven, AISTech 2019
- Internal gas pressure in coke ovens, M. Landreau, ESTAD 2019
- Comparison between industrial and laboratory IGP measurements, M.A. Schulten, ESTAD 2019

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## List of acronyms and abbreviations

AGDH	Aktien-Gesellschaft der Dillinger Hüttenwerke
AMAL	ArcelorMittal Atlantique et Lorraine
AMMR	ArcelorMittal Maizières Research
AMO	ArcelorMittal Ostrava
tkSE	ThyssenKrupp Steel Europe AG
AIC	Akaike Information Criterion
AMS	Arithmetic Mean grain Size
FEM	Finite Element Method
HV	High Volatile
IGP	Internal Gas Pressure
KBS	Kokereibetriebsgesellschaft Schwegern GmbH
LV	Low Volatile
MEP	Mean quadratic Error of Prediction
MMR	Mean Maximum Reflectance
MRR	Maximum Random Reflectance
MV	Mid Volatile
PS	Pusher Side
CS	Coke Side
OWP	Oven Wall Pressure
PCI	Pulverized Coal Injection
SHO	Sole Heated Oven
VM	Volatile Matter
WP	Wall Pressure

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## **Annex A: Mode of Operation of IGP measurement in Gravity and Top Charging**

### **A. CPM / ArcelorMittal Atlantique et Lorraine (Gravity Charging)**

The probe guides have to be installed on one of the coke oven doors. It is easier to work on pusher side near the battery end (avoid the two last ovens).

The probe guides can be installed at different levels on a same door. CPM generally installs a probe guide near the oven sole, in order to measure the highest Internal Gas Pressure developed during the carbonization of coal.

To install a probe guide, the instructions below have to be followed:

- A hole of a 60 mm diameter has to be drilled on the whole thickness of the door. This hole has to be adjusted in the door axis and perpendicular to the front face of the door.
- The probe guide is then introduced inside this hole and placed
- Before to be fixed, the guide has to be placed very carefully in the axis of the door and perpendicular to the front face of the door.
- The fixing can be performed either by bolts or by welding on the front face of the door.
- After fixing, it is necessary to control the position of the guide on the door (in the vertical axis and perpendicular to the front face).
- Just after putting again the door in its place, it is necessary to close the openings of the guides by some plugs to avoid gas leakages. The plugs have the same design than the probes used; they are only shorter than the probes used for the measurements. Another solution is to install the equipped door just before starting the tests.

The probes are installed just after charging, in order to avoid any problem of insertion. The probes are connected with the pressure sensors by means of rubber tubes. In order to avoid any loop that can create some water accumulation, the rubbers tubes have to be laid out as straight as possible and the pressure sensors have to be placed into the waste gas gallery, right under the equipped ovens. Figures 10, 11 & 12 illustrate the methodology.

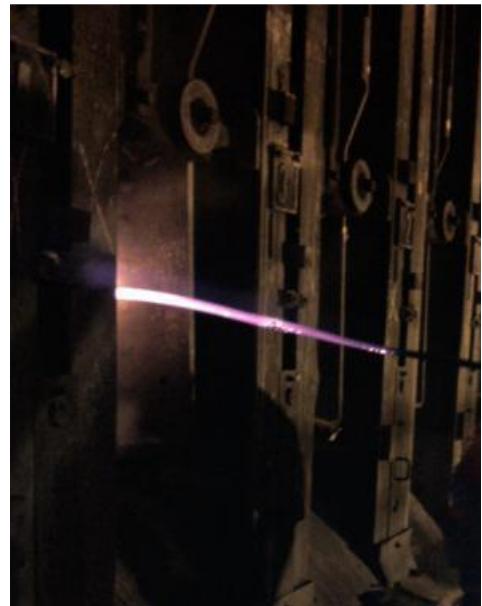
The electrical signals delivered by the sensors are recorded on a classical recording device. It is better to display in real time the pressure and the temperature measurements during the whole coking cycle in order to react rapidly in case of failure. Just before removing the door for pushing the coke, the probes have to be removed manually or by means of a winch attached in one solid fixed point.



*Figure 1: Probe insertion*



*Figure 2: Connection to sensors*



*Figure 3: Probe withdrawal*

With the three tubes, CPM/AMAL generally observed three peaks of pressure, nearly always different and slightly shifted in time. Only the highest pressure peak is taken into account.

#### **B. ArcelorMittal Ostrava (Gravity Charging)**

Preparation, probe placement and IGP measurement together with probe restoration is described in detail as follows:

- Before probe placing into the certain oven x, operator downloads heating flue temperatures of heating walls x and x+1;
- Before each new campaign at certain battery, operator is going to check a signal transmission between transmitters, which are located in front of the oven, and data recorder, which is located in indoor room close to the bench. Signal must be detected also during pushing car movement over coke oven with inserted probe;
- Operator checks temperature profile in the area of 2-4th heating flue. Similar temperature profile on both heating walls is necessary for successful placement - reaching of plastic layer;
- During whole procedure of probe placement and its withdrawal, operator is in permanent contact with pushing machine operator;
- Before probe placement and plug removal, liquor injection is launched in aim to avoid raw coke oven gas leakage from the oven;
- All actions related to the placement and withdrawal of the probe are managed from pushing machine platform;
- Probe is inserted through the oven door when last 10-15 centimeters must be strongly infixed into the coal charge;
- Both transmitters are isolated by insulation fabric to avoid sensor damage by heat during the opening of adjacent doors. Also, a slot between door plate and probe is isolated by insulation rope to avoid raw coke oven gas leakage;
- Both transmitters and data recorder with signal receiver are turned on, pressure and temperature data transmission is checked;
- Just before pushing, steel probe is removed from the oven by door opener placed at pushing machine when a steel part of the probe is fixed by steel rope;
- After its cool down, both transmitters are disassembled and probe is taken into the workshop for alignment and reattachment of the sensors;
- Recorded data of temperature and internal gas pressure are downloaded from data recorder via USB;

- Based on received data, decision about successful placement of the probe into the tar seam is made.

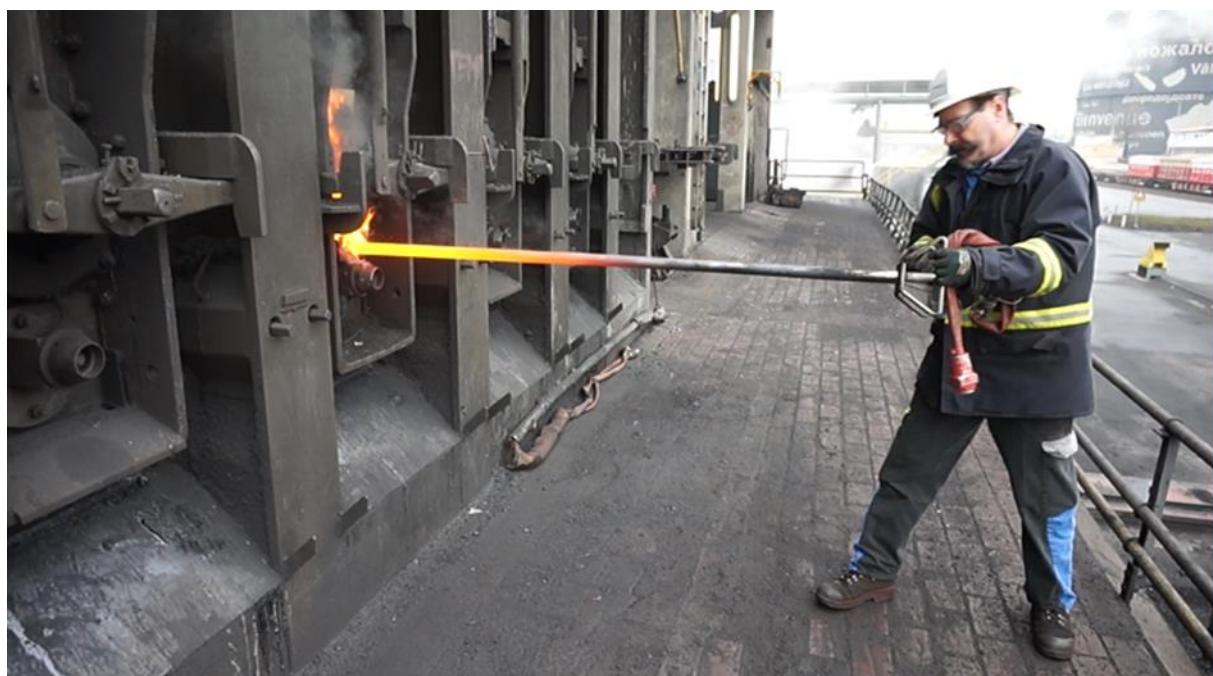
### **C. voestalpine Stahl (Gravity Charging)**

In voestalpine plant, the measurement always takes place in the same oven (Battery 10, Oven 363). The probe is inserted through the oven door (Figure 13). The position is appr. 250 mm above the oven sole and is close to the position of the first charging hole. Therefore, the position is inside the zone of the highest bulk density, and therefore it is expected to measure the highest IGP which occur in this coke oven.



*Figure 4: Front view of the oven door equipped with the IGP measurement*

The measurements take place in only one coke oven. Every coking cycle, which is between Monday and Friday is measured, therefore appr. 6 measurements per week are carried out. The IPG probe is inserted manually (without drilling a hole, or using a hammer). The probe is manually removed before the coke is pushed. Figure 14 shows the removal of the probe before the coke is pushed:



*Figure 5: Removal of the IGP probe before the coke is pushed*

After every measurement, the pipe has to be cleaned from coking residues by beating with a hammer. The IGP probe is a simple and robust device, and has a lifetime of appr. one month. Sometimes faulty measurements take place. This may have several reasons:

- the probe is not in the center of the oven,
- the holes of the probe are blocked by coal particles or tar,
- there is no sufficient contact between the probe and the coal, etc.

#### D. tkSE (*Gravity Charging*)

At Schwelgern coke plant, IGP measurement is typically carried out once per coal blend mixing bed consisting of ca. 80.000 t of coal which is consumed in a battery in approximately eight days. Additionally, extra measurements are performed during changes in operating parameters such as adjustments in blend oiling or blend grain size. The inserting procedure of the lance can be described by the following procedure (Figure 15):

- Unscrewing the blind plug at the door
- Predrilling the coal charge with the guiding tube
- Manual insertion of the lance into the oven until reaching the point when the resistance of coal mass is too high
- Final positioning of the lance using pneumatic driller
- Aligning the measuring slot with oven sole
- Fastening the lance to the guiding tube by screwing
- Inserting the thermocouple
- Screwing the detector head on the lance
- Connecting the measuring system with data receiver

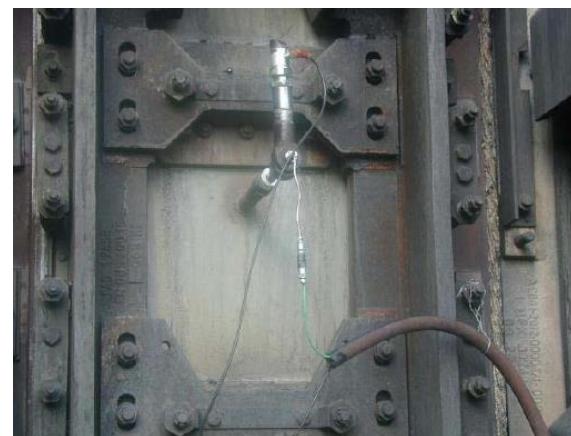
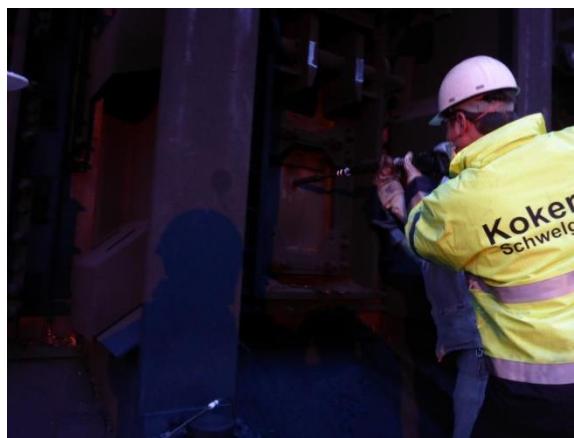


Figure 6: Inserting IGP probe

Problems during the procedure may occur with heavily compacted coal deposits and impurities at the door. In such cases an easy insertion of the lance is not possible. Under normal circumstances the operation as described above can be executed in a time frame of 15 minutes. The lance is inserted under slight inclination so that deposition of the condensates is avoided. The IGP probe is inserted into the oven with the help of three staff members, typically ten minutes after the oven has been charged.

Besides internal gas pressure, the temperature inside the coal charge is measured by thermocouple during the entire measuring phase. Both gas pressure and temperature data is transmitted wireless to the operations control room. In addition to these measurements the coke shrinkage and temperature of the gas inside the ascension pipe are recorded for the particular oven. In order to get additional information a possibility exists to sample certain amount of coal blend shortly before it is charged into the oven (Figure 16). The sample is taken from oscillating conveyor channels delivering the blend to the larry car.

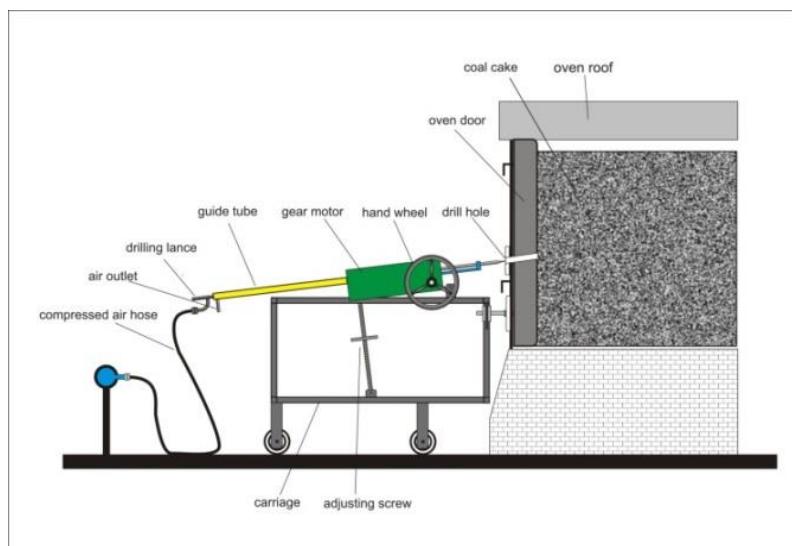


*Figure 7: Coal sampling point at KBS*

The lance is pulled out of the oven one hour before the scheduled oven pushing time in order to avoid any interference with pushing schedule and to maintain occupational safety. The withdrawal of the probe is realized by means of a chain hoist. Every lance is used only once and then disposed of after the measurement has been finished. The collected data can be used for optimization of the bulk density and heating parameters. Providing that any distinctive IGP peak is observed the measurement is repeated on the following day.

#### **E. AG der Dillinger Hüttenwerke (Stamp Charging)**

The internal gas pressure measurements are generally carried out once per week and are coordinated with the competent workers on the oven battery as well as the electrical technicians. In figure 12 the current used drilling device with its components is shown.



**1. Figure 12:** Placement of the drilling machine at the coke oven

- Shortly before the stamping, charging and pushing machine (SCP) reaches the oven foreseen for the IGP measurement, the operator approaches contact with the SCP-driver. Coordination of changing the standard oven door of this oven through the perforated one is done.
- When the oven before the one for the measurement is charged, the operator takes a specimen of the coal blend out of the coal feed hopper.
- When the desired oven is reached, the SCP-driver changes the door and in parallel the coal cake for charging the oven gets stamped.
- After pushing the coke out of the oven from the coking cycle the day before the straightforward stamped coal cake gets charged and the SCP-driver closes the oven with the perforated oven door.
- After getting enough space, the cleaned drilling platform (figure 13) is placed right in front of the oven. The positioning is made by hand with the help of marks on the oven framework.
- When the targeted position of the drilling platform is reached, the device is fixed with the help of clamps. Thereby the platform always stays in the same orientation while drilling and furthermore the position is the same over different drilling operations.
- Afterwards the operator fastens the drill into the chuck and starts to bore the channel into the coal cake (figure 14). The feed rate while drilling is made by hand through a threaded rod.



2. **Figure 13:** Drilling platform placed in front of the oven door and clamps for fixation of the device onto the oven framework



3. **Figure 14:** Interior of the drilling device

- As soon as the drill reaches the desired depth, indicated via a mark on the drill bit, he can remove the borer and install the measuring probe.
- Then the operator connects the cables from the receiver to the probe, secures the probe against movement with a steel wire and seals as good as possible the space between the probe and the oven door.
- After checking if the measurement is running well the operator has to wait for the end of the coking time of the coke.
- Enough time before pushing the finished coke cake the operator removes the measuring probe and the SCP-driver replaces after pushing the coke the prepared oven door through a regular one.

- The recorded data is downloaded via a portable device.

#### **F. ArcelorMittal Ostrava (Stamp Charging)**

Preparation, probe placement and IGP measurement together with probe restoration is described in detail as follows:

- Before probe placing into the certain oven no. x, operator downloads heating flue temperatures of heating walls no. x and x+1.
- Before each new campaign at certain battery, operator is going to check a signal transmission between transmitters, which are located in front of the oven, and data recorder, which is located in indoor room close to the bench. Signal must be detected also during pushing car movement over coke oven with inserted probe.
- Operator checks temperature profile in the area of 2-4<sup>th</sup> heating flue. Similar temperature profile on both heating walls is necessary for successful placement - reaching of plastic layer.
- During whole procedure of probe placement and its withdrawal, operator is in permanent contact with pushing machine operator.
- Before probe placement and plug removal, liquor injection is launched in aim to avoid raw coke oven gas leakage from the oven.
- All actions related to the placement and withdrawal of the probe are managed from pushing machine platform.
- After removal of the plug, a straight hole is made by drilling machine when its length is approximately 1.9 m. Drilling of the hole into stamp charge through the door is shown in figure 15.



4. **Figure 15:** Example of drilling into the stamp charge

- Probe is inserted through the oven door when last 10-15 centimeters must be strongly infixed into the coal charge.
- Both transmitters are isolated by insulation fabric to avoid sensor damage by heat during the opening of adjacent doors. Also a slot between door plate and probe is isolated by insulation rope to avoid raw coke oven gas leakage.
- Both transmitters and data recorder with signal receiver are turned on, pressure and temperature data transmission is checked.
- Just before pushing, steel probe is removed from the oven by door opener placed at pushing machine when a steel part of the probe is fixed by steel rope.
- After its cool down, both transmitters are disassembled and probe is taken into the workshop for alignment and reattachment of the sensors.
- Recorded data of temperature and internal gas pressure are downloaded from data recorder via USB.

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The measurement of Internal Gas Pressure in industrial coke ovens is necessary to obtain experimental data on the expansion behavior of the coal charge and the risk of potential oven wall damage. The completed project was devoted to gather and analyze the different methodologies used by many industrial European partners to measure Internal Gas Pressure.

Another objective of this project was to investigate the influence of rank, volatile matter, inert content on Internal Gas Pressure from industrial measurements. Effects of grain size, percentage of High Vol coals or petcoke were highlighted during these industrial measurements. Influence of charging method was also estimated.

To obtain a comparison between pilot and industrial scale, samples of industrial blends were prepared and delivered to the pilot facilities of the project partners. Furthermore, a round robin was performed between pilot ovens on gravity and stamp charging method in order to compare the results of the Internal Gas Pressure measured on the same blend in different pilot facilities. Results of partners were all in good agreement. Results showed the major influence of density. Investigations of influencing parameters under defined operation conditions in pilot oven were carried by two partners in top and stamp charging conditions.

Laser technology was used to measure wall deformation during a coking cycle, and especially during junction of plastic layers. Models of heating walls were also developed based on AM Gijon and ZKS coke plants design.

