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Abstract

VDZ, IKN and HeidelbergCement (HC) have successfully managed to cool clinker under oxyfuel technology conditions. The innovative oxyfuel clinker cooler prototype has successfully been tested in a HC plant in Hannover. The overall aim of this experimental work was the testing of an oxyfuel clinker cooler in an industrial environment in order to assess its cooling performance (efficiency), as well as the potential impacts of CO_2–rich cooling gas on clinker quality. The oxyfuel clinker cooler prototype was designed with a capacity of 80 t/d. During the trials a maximum of 47 t/d clinker has been extracted, which corresponds to 1.5% (1:64 scale) of the clinker production of a reference cement plant (3000 t/d). Despite the experimental challenges, CO_2 concentration levels of the cooling medium higher than 70 vol.% have been achieved repeatedly by continuous CO_2 supply. No leakages of CO_2–rich gas occurred during the trials, which proved the efficiency of the sealings. The testing process parameters were recorded continuously and clinker samples from the cement plant main cooler and from the oxyfuel clinker cooler were taken periodically for further analysis. The lessons learned during the trials will be a valuable input for the design of future industrial scale oxyfuel clinker coolers. The operation of the oxyfuel clinker cooler prototype indicates that boundary zones, such as the cold clinker discharge system, demand special attention regarding minimisation of false air ingress also in industrial scale projects. Moisture content of the recirculated cooling gas was much higher than expected. This was attributed to the experiment conditions and the lack of a condenser in the prototype setup. Unusual layers of up to 2 µm thickness around alite crystals in contact with pores, probably resulting from up to 4% alite decomposition, have been observed in a few analysed clinker samples. However, layer formation cannot be correlated with the high CO_2 concentrations in the cooling medium alone. Decomposition of alite caused by water (moisture) combined with high CO_2 concentration in the cooling gas shall be further investigated. Also, the absence of significant effects on cement quality shall be confirmed by additional laboratory testing .

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1 INTRODUCTION

The overall aim of the oxyfuel clinker cooler prototype work package (CEMCAP WP9) was the testing of the world's first oxyfuel clinker cooler prototype constructed and operated in an industrial environment, with the objectives to:

1. Determine the influence of cooling gas composition on clinker chemistry
2. Determine the gas leaking rate to ambient
3. Determine false air in-leakage
4. Determine the clinker cooling curves and efficiency with CO_2 rich gas

Based on the existing designs for single-stage and two-stage gas-tight clinker coolers, which have been developed in ECRA's CCS project phase III [1], a choice for a layout of the prototype has been made taking into account the potential for up-scaling, as well as technical feasibility to be operated in prototype scale. The oxyfuel clinker cooler prototype was operated in parallel to the existing clinker cooler in the HeidelbergCement (HC) plant in Hanover.

To evaluate the testing of the oxyfuel clinker cooler prototype a few process parameters (e.g. clinker mass flow, pressures, temperatures, volume flows, cooling gas composition etc.) have been recorded continuously at different points of the pilot plant. To assess the impact of changed cooling rate and cooling gas chemical composition on clinker chemistry, several clinker samples have been taken from the HC plant main clinker cooler and from the oxyfuel clinker cooler prototype for further analysis in VDZ's laboratories. To derive information about the process reliability, cooling curves and sealing efficiency, the comprehensive data gathered during the trials has been evaluated and discussed by IKN and VDZ.

The conclusions from WP9 provide relevant input data to the simulation in a process model of an oxyfuel cement plant (WP6 - Oxyfuel Modelling and Optimization). Moreover, a comprehensive list of lessons learned during the trials will help to provide guidance in future design of an industrial scale oxyfuel clinker cooler.

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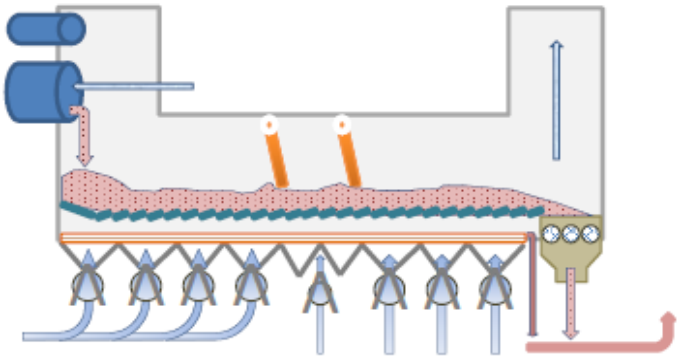
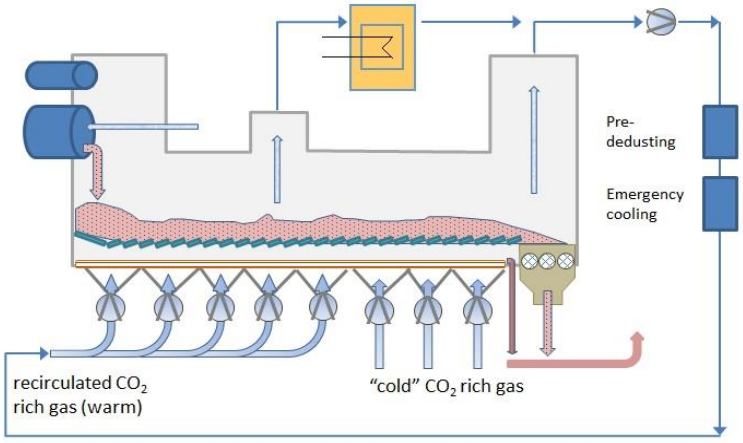
2 OXYFUEL CLINKER COOLER PROTOTYPE DESIGN

2.1 Selection of the clinker cooler prototype design

Within ECRA's CCS project phase III different oxyfuel cooler designs have been developed by IKN [1]. Whereas the focus of the first two designs was on the task of gas separation, the advanced designs aim at a minimization of movable parts in the hot zone of the cooler especially with regard to lower recirculation rates (Table 2.1).

All the designs should be able to fulfill the task of gas separation and appropriate cooling. However, each design offers advantages and disadvantages (Table 2.2). Based on this evaluation the basic preference in the ECRA CCS project has been laid on the continuous bed cooler either with heat shields or recirculation as these seem more likely for potential retrofit (e.g. no additional height) and due the higher flexibility as well as lower operational risk.

Table 2.1: Concepts for an oxyfuel clinker cooler prototype

Name	Description	Sketch
Continuous Clinker Bed (CCB)	- Gas separation by heat shields and pressure adjustment between the stages. - Minor leakages in case of big lumps. - Optimal efficiency	
Continuous bed cooler with gas recirculation	- Complete cooler aerated with CO_2-rich gas from internal and external recirculation. - Cooler mid- and exhaust gas recirculated (pre-dedusted and cooled) - Highest cooler efficiency - High flexibility in regard to recuperation gas amount.	

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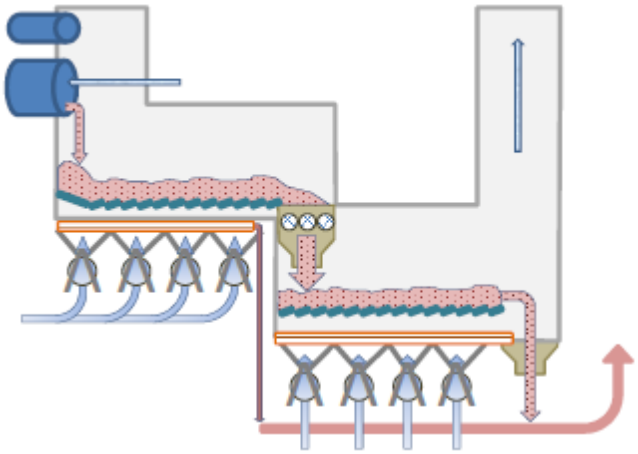
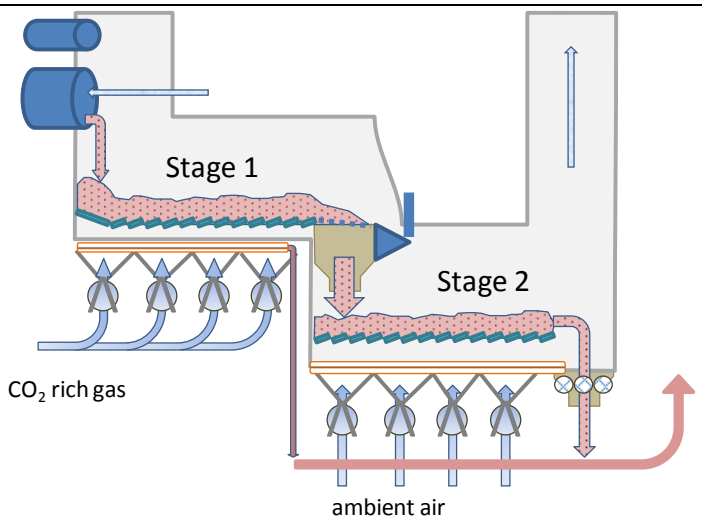
Intermediate Clinker Bin (ICB)	- Gas separation by intermediate crusher - Reduced efficiency due to bed disturbance - Sufficient sealing even in case of bigger lumps.	
Intermediate connection chute	- Gas separation by the clinker bed controlled by lower grate speed and gas pressure - Small chute allows only normal clinker to pass, bigger particle will bypass - Lower risk of leakage compared to ICB	

Table 2.2: Advantages and disadvantages of the cooler options

Cooler type	Advantage	Disadvantage
Continuous Clinker Bed (CCB)	- feasible to be retrofitted - gas separation possible	- movable parts in the hot zone - risk of leakage - less flexible
Continuous bed with gas recirculation	- no leakage risk within the cooler itself - high flexibility	- influences the overall layout as cooler exhaust air is not available for raw material drying - additional heat exchanger is required
Intermediate crusher (ICB)	- low risk of leakage - improved cooling of big lumps	- movable parts in the hot zone - less flexible - risk of overheating the crusher - additional height
Intermediate connection chute	- gas separation possible - more energy available for raw material drying	- still requires movable part for safety reason - risk of leakage - additional height - less flexible towards flow changes

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The prototype design has been selected based on the above shown continuous bed with full gas recirculation layout taking into account the potential for up-scaling as well as technical feasibility to be operated in prototype scale. For a proper up-scale of the prototype results the use of hot clinker and the size of the facility are essential. Therefore, the prototype has either to be operated in an existing low capacity kiln line or supplied by an operating kiln in parallel to the existing cooler. Based on an optimal test capacity and the availability of plants in which the prototype could be installed, the option to operate in parallel was chosen. The details are listed in the following sections.

2.2 Oxyfuel clinker cooler pilot system general concept

Based on these considerations the entire cooler prototype was aerated with the same cooling medium, a CO_2-rich gas, which would be available as flue gas from the oxyfuel process. For the cooler prototype different gases should be supplied from bottles. Main difference beside the production capacity compared to a full scale plant is that no recuperation gas is routed to the kiln and the heat is not utilized. This requires a large heat exchanger.

The general concept of the oxyfuel clinker cooler pilot plant is shown in Figure 2.1. A low percentage of hot clinker (up to 3 % of the usual clinker production of the main pyro line) is extracted from the kiln hood of the pyro line and is then charged into the oxyfuel clinker cooler prototype. As the hot clinker drops onto the static clinker inlet, the hot clinker distributes evenly across the clinker cooler width and forms a clinker bed. The grate speed of the clinker cooler will be adjusted to form a bed depth of the bulk clinker inside the cooler suitable for best cooling efficiency.

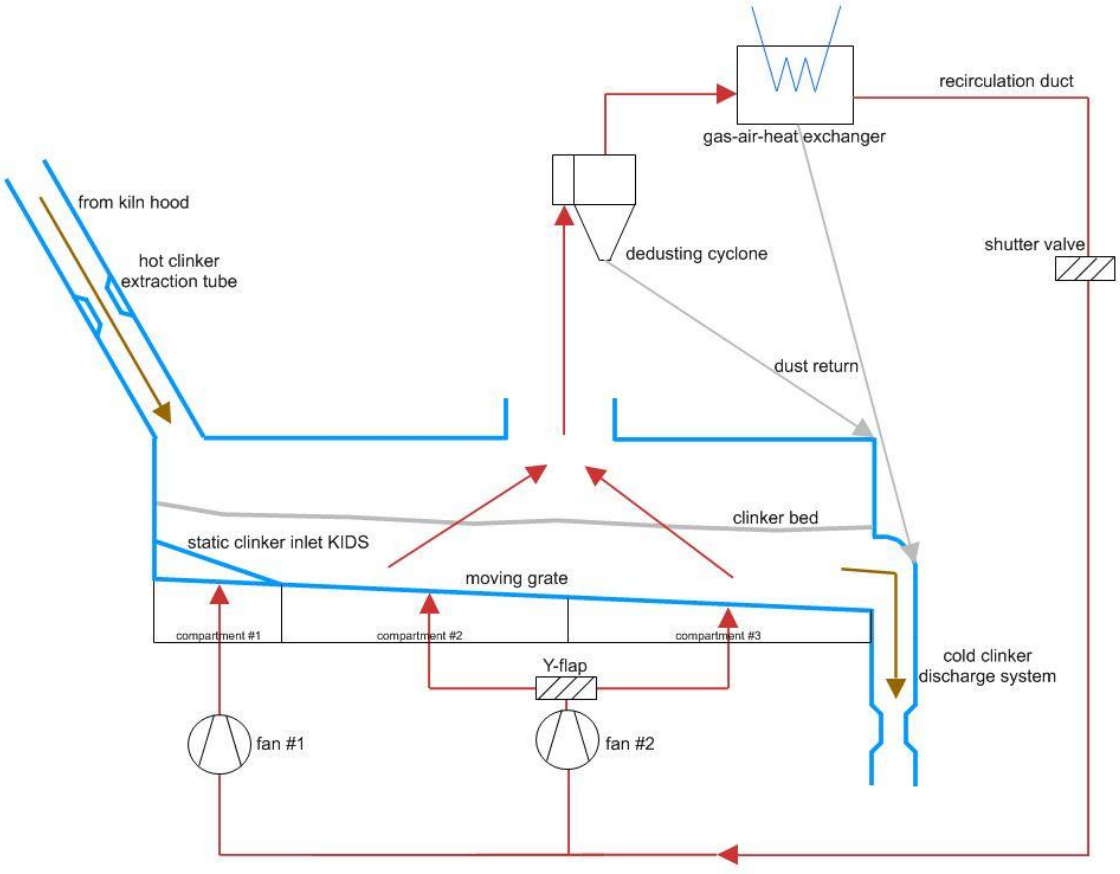

Figure 2.1: General oxyfuel clinker cooler pilot system concept

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The hot clinker bed is cooled with the CO_2-rich gas which is taken from the suction line behind a gas/air heat exchanger and is transported by two cooling fans to the pressurized compartments underneath the oxyfuel clinker cooler. From the cooler compartments the cooling gas is distributed to the bottom of the clinker bed by passing through the grate surface of the oxyfuel clinker cooler. The grate surface consists of grate plates with COANDA effect, which are permeable for cooling gas but not for clinker. From the bottom of the clinker bed, the cooling gas passes through the bed towards the surface and, by this, cools the clinker (cross flow principle). While the clinker temperature decreases the cooling gas temperature increases. After passing the clinker bed the cooling gas is leaving the oxyfuel clinker cooler as cooler exhaust gas through an exhaust duct located at the cooler roof. The calculated exhaust gas temperature is approximately 500 °C. Subsequent it is de-dusted in a cyclone and cooled by the gas/air heat exchanger to approximately 100°C. At the heat exchanger outlet, the gas is routed again to the cooling fans, which closes the recirculation loop. At the same time the material inside the oxyfuel clinker cooler is transported by the moving grate all the way through the cooler and is finally discharged at the end of the cooler via a clinker hopper. With the help of a discharge screw conveyor system the clinker is then carried to the main cold clinker extraction system or, for chemical analysis of the clinker, to a manual clinker extraction point.

2.2.1 Concept for filling and draining procedure (original plan)

CO_2 is available from a filling station equipped with CO_2 bottles, 50-liter, 200 bars. The pressure from the bottles is reduced to 0.5 bars and the gas is supplied to the oxyfuel clinker cooler pilot system at three different entering points as per Figure 2.2. To minimize the mixing of CO_2 with ambient air two of the entering points have a ring pipe with 4 tapping points at each ring pipe. Three tapping points for CO_2 filling are planned at the cooler backside. For CO_2 filling the flap inside the recirculating duct after heat exchanger will be closed as shown in Figure 2.2. The cooling fans should initially be stopped. Opening of the CO_2 supply leads to an influx of CO_2 into the system caused by the higher pressure of the CO_2 supply system (0.5 bars).

The CO_2 displaces the ambient air which leaves the pilot system through the hot clinker extraction system into kiln hood. The filling procedure can be supported by operating the cooling fans with low speed if required and after opening the flap. As CO_2 can dissolve into ambient air and skip zones can occur, purging of the system may become necessary which means that actually a higher CO_2 volume compared to system volume is required for filling. It can be expected that the CO_2 volume has to be 3-5 times higher than the systems volume for entirely filling of the oxyfuel clinker cooler pilot system. The filling procedure has to be repeated whenever the actual CO_2 concentration fells below accepted limits. The total volume of the oxyfuel clinker cooler pilot system is approximately 46 m^3, which can be covered by four CO_2 bottles, 50 liters, 200 bars. In addition to the CO_2 supply, a readily mixed gas with 70.5% CO_2, 17.8% O_2 and 10% N_2 could be introduced to simulate oxyfuel flue gas by using the same filling procedure.

Comparatively easier is the draining of the oxyfuel clinker cooler pilot system with ambient air. While the cooling fans are running, throttling of the flap in the recirculating duct creates sufficient suction inside the duct after the flap, which allows fresh air to enter into the system, e.g. through the opening where the gas-composition probe can be installed. The fresh air will partly displace the gas in the system which will be exhausted through the clinker extraction pipe into the kiln hood, similar as per the CO_2 filling procedure.

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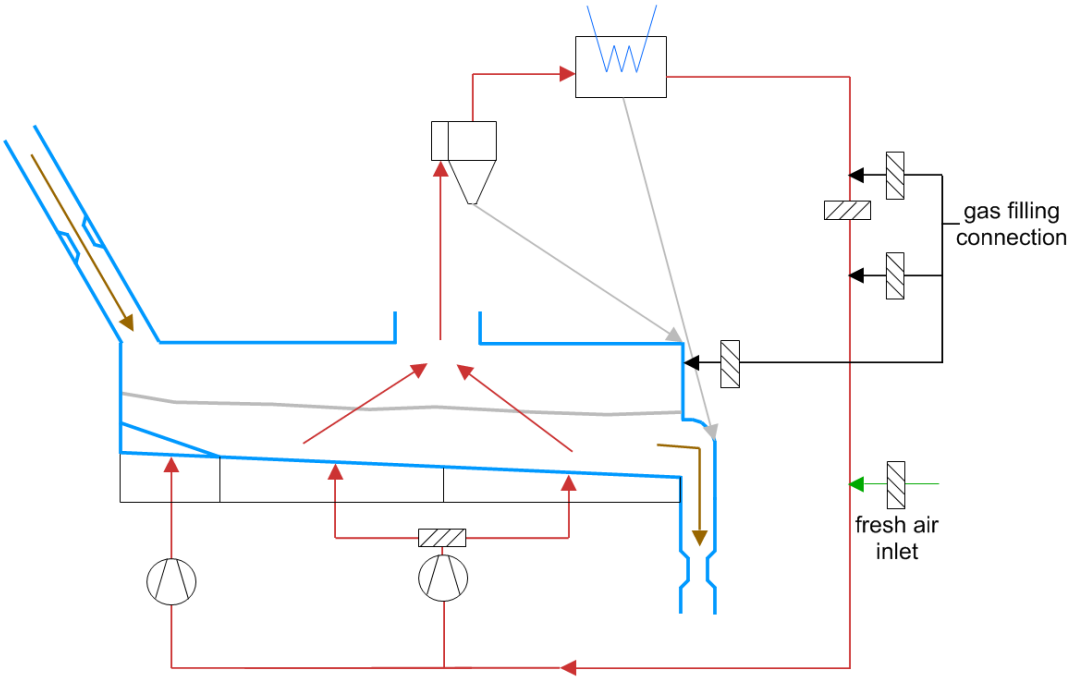

Figure 2.2: Basic arrangement of the oxyfuel clinker cooler pilot system for CO_2 – Filling/Draining

2.2.2 Instrumentation concept

The instrumentation concept is very important for the analysis of the oxyfuel clinker cooler pilot system. Once in operation pressure, temperature, flow and gas composition are measured at different locations as shown in Figure 2.3.

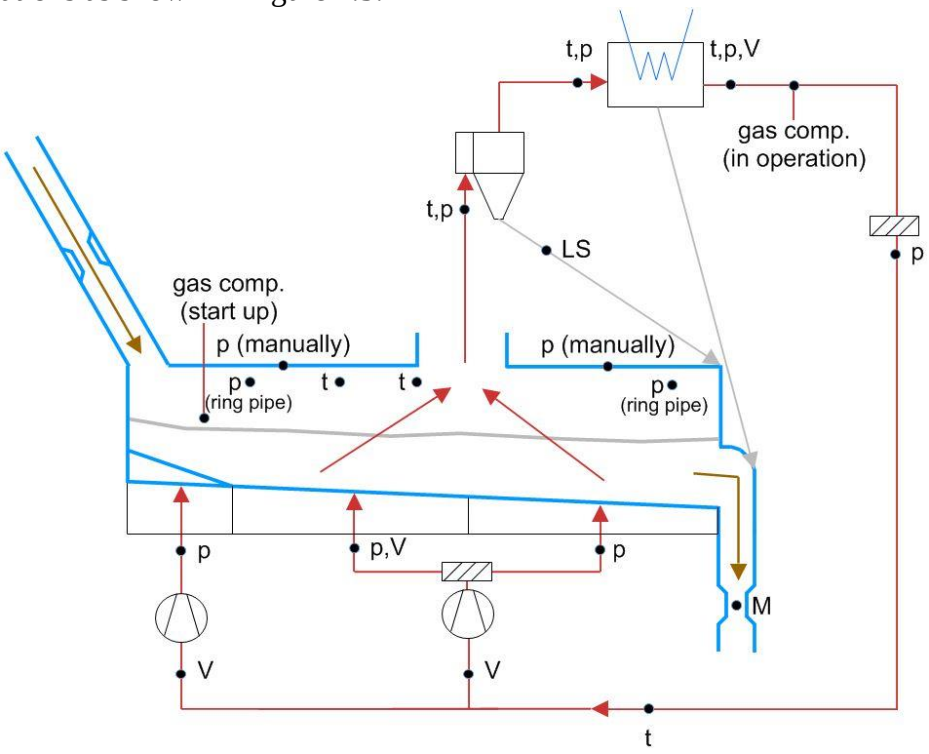

Figure 2.3: Instrumentation plan of the oxyfuel clinker cooler pilot system

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Similar to the usual clinker cooler aeration procedure, the volume flow at fan inlet and static pressure at fan outlet are measured, which is mandatory to control required cooling gas amount. As fan #2 provides cooling gas for two cooler compartments pressure/flow distribution is controlled by a so-called Y-flap. For that purpose, the static pressure in both gas ducts and in addition the flow in one of the gas ducts behind the fan is measured. Furthermore, pressure conditions inside the oxyfuel clinker cooler prototype are measured with the help of two ring pipes with pressure transducers next to the cooler inlet and outlet. Cooler outlet gas is monitored by one temperature and one pressure measuring probe in front of the de-dusting cyclone. This enables the determination of heat in terms of gas flow leaving the cooler. After de-dusting of the clinker exhaust gas the temperature and pressure are again measured for estimation of the cyclones pressure and temperature drop and as initial parameters of the gas-air-heat exchanger which then is cooling down the gas to the desired temperature. Inside the recirculating duct leading to the fans inlet static pressure, temperature and volume flow is measured again at different locations as measured values can differ when flap is closed during the filling or draining procedure. In order to identify the gas composition with regard to the estimation of leakage a gas analyzer is used. During the filling of the oxyfuel clinker cooler pilot system the gas composition is measured at cooler inlet in order to identify the complete fill-up of the system based on the rapid increase of CO_2 concentration. During operation the gas composition is measured behind the gas-air-heat exchanger for structural reasons. Clinker mass flow is measured at the cooler outlet by a mass flow probe. Extraction point for cold clinker sampling is included at the screw conveyors.

Control devices will not be connected to the control room. Only a release switch is installed in the existing control room to allow emergency shut-downs and grant the permission for start-up.

2.2.3 Health and safety concept

With regard to the operation of the prototype system different health and safety issues could occur, which were carefully assessed and countermeasures identified.

Handling CO_2

As CO_2 has a higher density compared to air, and the risk of accumulation in sublevel areas, special safety measures had to be implemented on-site, like the installation of a static CO_2 warning system at ground level (Figure 2.4) and mandatory usage of individual mobile CO_2 detectors by each member of the team (Figure 2.5). The CO_2 detection systems had been calibrated and tested at VDZ headquarters before on-site installation and usage. Moreover, all personnel had to be instructed not to inhale flue gas e.g. by opening any maintenance openings. Since the oxyfuel clinker cooler prototype was located outside at plain concrete ground, the installation of special ventilation was not necessary. Moreover, the pressurized bottles for filling of the pilot system were protected against falling by approved cluster carrier. After filling, the bottles were stored at a secure place.

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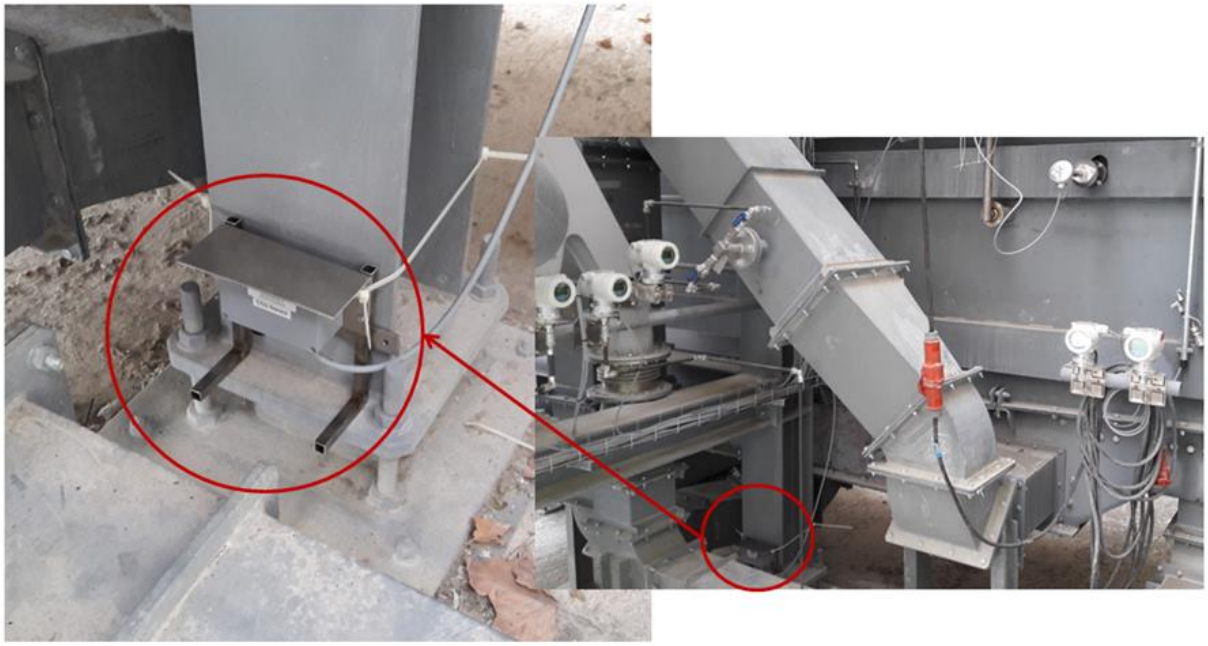

Figure 2.4: Static CO_2 warning system installed at the ground level

Figure 2.5: Mobile CO_2 warning system

Working in hot areas and handling samples

Working in hot areas and handling hot samples requires certain personal safety equipment (gloves, jackets, helmet, safety shoes etc.). Respective HSE assessment and the VDZ code of practice VT 12 "Health and safety in hot areas of cement plants" provided information of the necessary equipment.

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2.3 Equipment overview

When describing the pilot system, a distinction needs to be drawn between the clinker and the gas flow. A fundamental challenge was stable and continuous extraction of hot clinker from the running kiln to the oxyfuel prototype. This was solved with a revolving, refractory-lined pipe that trapped a small part of the hot clinker falling from the kiln towards the main clinker cooler static grate. Material supply to the oxyfuel clinker cooler prototype was controlled through the axial pipe position in combination with variable rotational speed. The hot clinker was then entering the cooler and was spread evenly across the width of the grate system by the static inlet system, the so-called KIDS. The clinker was then moved through the cooler while at the same time being cooled by aeration with cooling gas from the under-grate fans. The cooled clinker then left the cooler and was routed back to the plant's clinker conveying system via a series of screw conveyors. In the meantime, the cooling gas after absorbing the heat of the hot clinker was routed to a cyclone for de-dusting. Separated dust was reverted back to the cooler prototype while the now de-dusted gas stream was routed to a gas-to-air axial heat exchanger. In the pilot system, the heat exchanger was necessary to allow recirculation of the gas stream by cooling the gas stream to a lower temperature, enabling it to be used again as a cooling gas in the oxyfuel clinker cooler prototype. Recirculation was ensured by two radial fans, one fan providing cooling gas to the static inlet section and another one providing cooling gas to the two cooler compartments below the movable grate. With those two material streams (clinker and gas) in mind the pilot system construction could be broken down to the following main equipment (Figure 2.6):

- Oxyfuel clinker cooler prototype
- Two fans
- Gas-air heat exchanger (HEX)
- Dedusting cyclone
- Hot clinker extraction system (extraction via tube at existing cooler front wall)
- Cold clinker conveying system (incl. screws for transport back to original plant clinker transport system)
- CO_2 filling station
- Two rotary valves for dust return from cyclone/HEX to the cooler
- Sliding damper for recirculation valve

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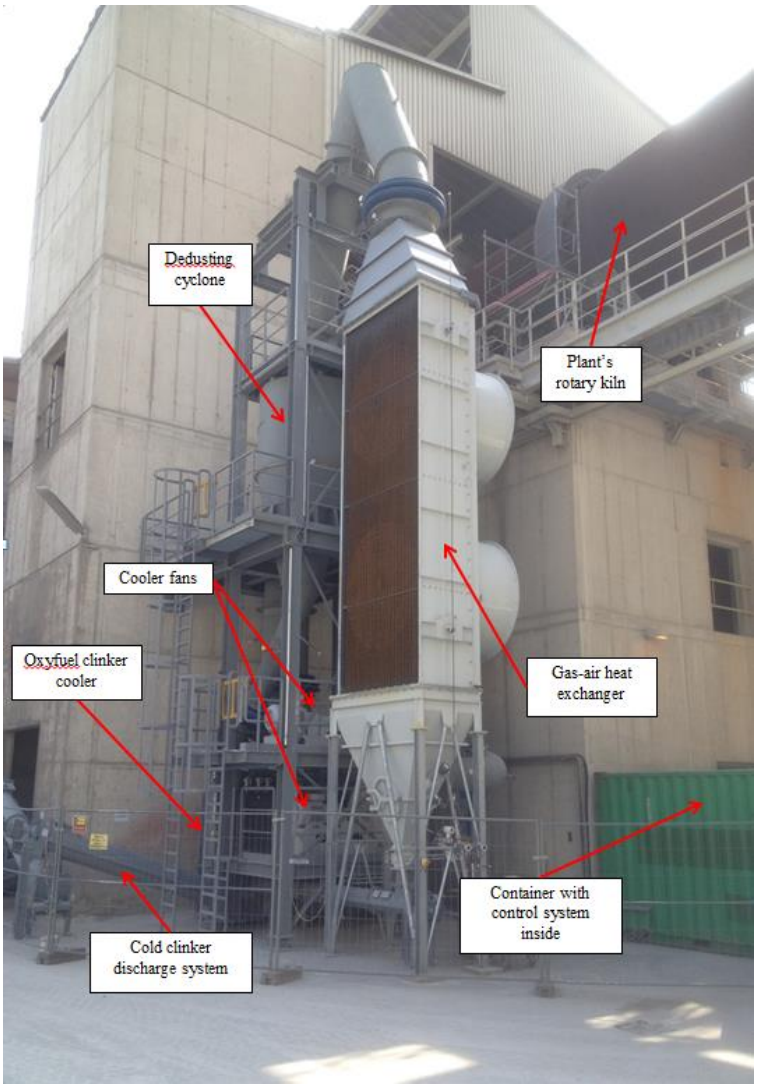

Figure 2.6: Oxyfuel clinker cooler pilot system

2.4 Specifications of the oxyfuel clinker cooler prototype

The cooler prototype has been dimensioned based on a hot clinker temperature of 1450 °C and gas compositions as given by ECRA. Up to 3 % of hot clinker taken from the plant's pyro process accounts for roughly 80 tons per day, which requires a downscaling of the clinker cooler compared to industrial applications with 1000 to 12000 tons per day. Main specifications of the cooler prototype are as follows:

- Dimensions including refractory, housing and supporting structure: height 2.1 m, length 4.6 m and width 1.2 m
- Aerated area: 2 m^2
- Nominal production capacity: 80 t/d
- 35 grate plates in 11 rows (2 rows form the aerated static inlet and 9 the movable grates)
- Two cooling gas chambers
- Total equipment volume approx. 45 m^3
- Electro mechanical drive for 1.2 strokes per minute (depending on clinker bed height)

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The fixed clinker inlet distribution system (KIDS) is aligned with two rows with four grate plates each. The grate has nine rows with 3 grate plates each of which 3 rows are movable. The movable rows push the clinker bed to the discharge system. The movable rows are driven by an electromechanical drive, which is aligned below the grate.

The upper cooler housing above the grate system consists mainly of the walls and roof, which is aligned with refractory. Special attention was paid to the supply of hot clinker through a revolving pipe. The hot clinker is directly fed to the KIDS area through the upper cooler housing. The parts of the drive equipment, which are in direct contact to the gas, have been designed and purchased to withstand these conditions and to ensure safe operation. All equipment has been designed to ensure best possible gas tightness.

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3 COOLER PILOT SYSTEM CONSTRUCTION, PRE-ASSEMBLY, ASSEMBLY AND COMMISSIONING

Time until trial period can be broken down into three different phases starting with the oxyfuel clinker cooler prototype construction phase, which had already begun in previous CCS research project conducted by ECRA. Construction phase in general begins with brainstorming, followed by the basic and detail engineering and ends with manufacturing of the components and pre-assembly. On completion of first phase there comes the assembly-phase where the pilot system is erected on-site. Finally, there is the commissioning phase which can be further subdivided into cold and hot commissioning. These phases are described in much more detail in this chapter.

3.1 Construction and pre-assembly (May 2015 – July 2016)

On May 1st 2015 IKN started the construction phase of the oxyfuel clinker cooler prototype system based on already established oxyfuel clinker cooler designs from the ECRA CCS research project. Based on IKN drawings of the cooler prototype (Figure 3.1), needed components were purchased, manufactured and delivered to the IKN warehouse.

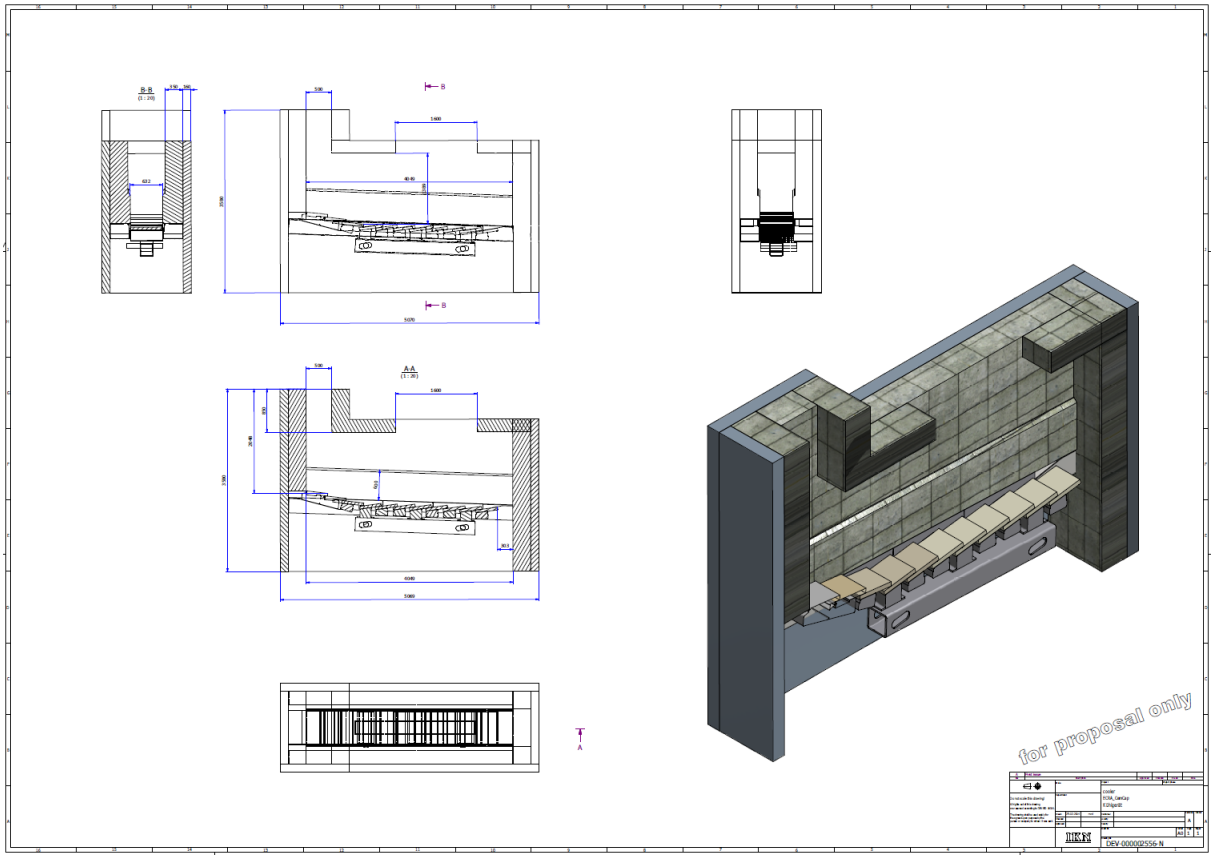

Figure 3.1: Oxyfuel clinker cooler prototype drawing (draft version)

Here, the components were aligned by IKN supervisors and engineers (Figure 3.2; Figure 3.3; Figure 3.4). The construction was fulfilled without major issues. The electrical drive was connected and first tests showed promising results for a smooth and trouble-free operation. The modular design and pre-assembly in the IKN warehouse enabled an easy transport and quick assembly on site.

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Figure 3.2: Oxyfuel clinker cooler prototype upper cooler housing pre-assembly.

Figure 3.3: Oxyfuel clinker cooler prototype view into upper cooler housing

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Figure 3.4: Oxyfuel clinker cooler prototype grate on lower cooler housing pre-assembled at IKN warehouse

Together with the peripheral equipment (heat exchanger, screw conveyors, de-dusting cyclone, etc.) a 3D-CAD-model from the whole oxyfuel pilot system was created (Figure 3.5 and Figure 3.6). For the next stage, and based on that 3D model, technical drawings were derived. The process of designing, manufacture and procurement of the pilot clinker cooler was finished at mid of June 2016. Main equipment like the clinker cooler itself, the gas/air heat exchanger, the clinker extraction device and fans were first delivered to IKN warehouse or directly to the plant site.

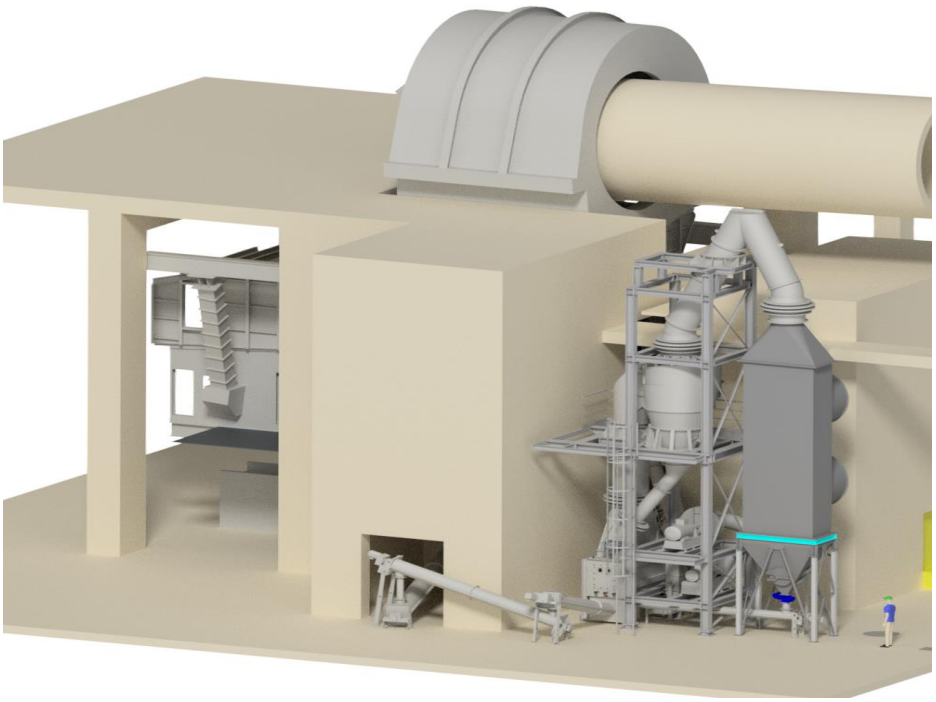

Figure 3.5: 3D-CAD model of the oxyfuel pilot plant 1/2

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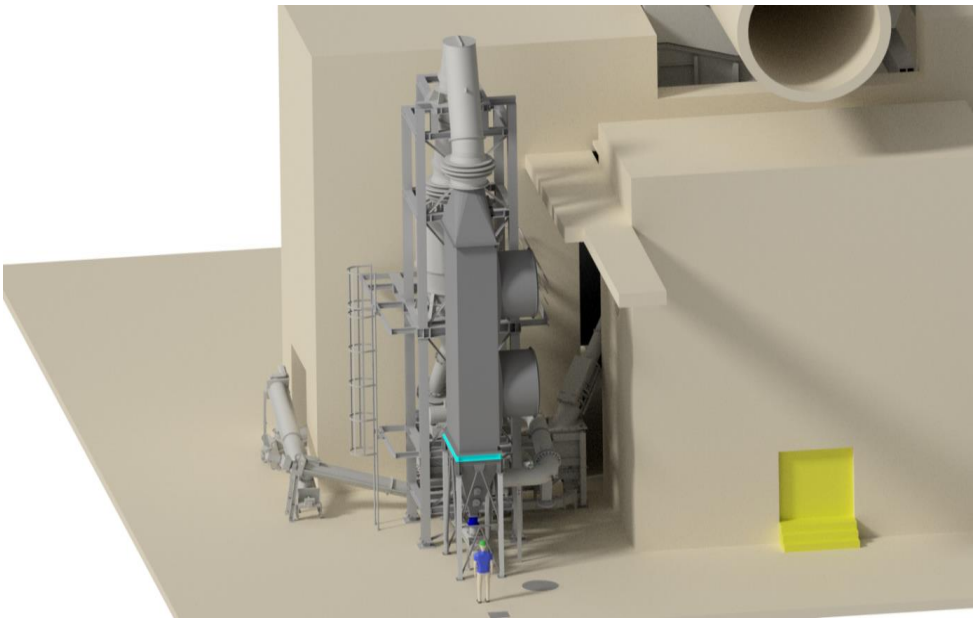

Figure 3.6: 3D-CAD model of the oxyfuel pilot plant 2/2.

3.2 Assembly (February & August – September 2016)

To guarantee a swift and smooth integration of the clinker extraction device to the plant process in summer 2016, IKN already prepared the plant during its regular winter shut-down in February 2016. Thus, loss of production in HeidelbergCement's everyday business could be avoided. An adaption piece was welded into the clinker cooler front wall. The revolving clinker extraction tube was later connected to the plant process through this adaption piece (Figure 3.7).

Figure 3.7: Installation of the adaption piece into the existing cooler front wall

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After the finalization of the pre-assembly and testing in the IKN warehouse, the pilot clinker cooler was delivered to the plant site and installed along with the hot clinker extraction device (Figure 3.8, Figure 3.9).

Figure 3.8: Installation of the pilot cooler

Figure 3.9: Installation of the hot clinker extraction device.

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Afterwards, the upper cooler housing was aligned with refractory in order to withstand the hot clinker temperatures of up to 1450°C during operation (Figure 3.10).

Figure 3.10: Oxyfuel clinker cooler prototype from the inside after refractory works

Afterwards, the peripheral equipment was installed and electrically connected to round off the overall pilot plant configuration (Figure 3.11). Finally, process control system (DCS) of the pilot system was established with a so-called HMI panel allowing individuals to interact with the prototype system and to run into different operation points (Figure 3.12). The assembly of the pilot plant on-site was initiated in August and finished in September 2016. Everything went to plan and the pilot plant integrated nicely into the existing HeidelbergCement process.

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Figure 3.11: Installation of the peripheral equipment

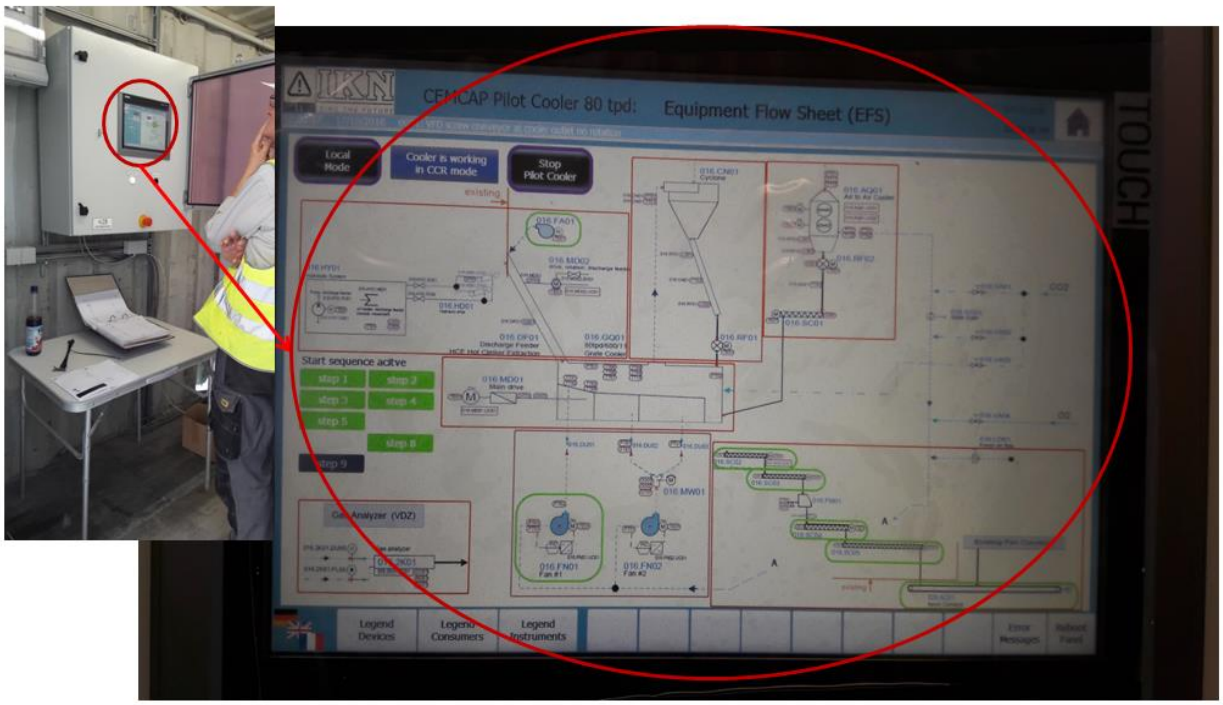

Figure 3.12: Pilot plant process control system (DCS) with HMI panel

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3.3 Commissioning (October 2016 – January 2017)

Directly after the assembly of the pilot plant IKN started the cold and hot commissioning tests. At the same time and as preparation for the trial period and operation with CO_2 VDZ installed a CO_2 static safety sensor as addition to the mobile units carried by the trial attendees.

The commissioning of the pilot plant began in October 2016 and included functional tests of the hot clinker extraction device and cold clinker discharge screw conveyor system, check of tightness and leakage detection as well as operation of the pilot plant with and without clinker. Due to some unforeseen circumstances during the commissioning tests described in more detail on the following pages, stable operation was not possible to reach until the end of 2016.

3.3.1 Cold Commissioning (October 2016)

First functional tests were already performed during erection and electrical assembly of the pilot system. Thus, major functions could already be tested before hot operation. This includes testing of the cooling fans, hot clinker extraction tube, cold clinker discharge screw conveyors and many more. Moreover, the connection to the HeidelbergCement process control system could be established successfully while security-related software interlocks as well as an emergency-stoppage system were installed and tested successfully during that phase. To sum up, cold-commissioning in October 2016 turned out positively and without special incidents. Figure 3.13 shows the functional testing of the screw conveyors. Clinker from the HeidelbergCement clinker silo was used to test the cold clinker discharge system, which routes the cold clinker from the oxyfuel pilot cooler back to the plant's clinker conveying system.

Figure 3.13: Functional test and cold commissioning of the cold clinker discharge screw conveyors

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3.3.2 Leakage tests (November 2016)

For the pilot scale clinker cooler, a design providing high gas tightness was developed. Tightness is important for the oxyfuel process, as CO_2 may not be able to escape and air may not penetrate the cooling system. After installation, tightness would have to be tested on-site. Different testing methods are described in norms. An ordinary method is shown in DIN EN 1593 "non-destructive testing – leak testing – bubble emission techniques". A gas filled object is dipped into water, but the procedure cannot be used for large units. Tightness tests for the described scale can be performed for example with a tracer gas method or a pressure change method described in DIN EN 13184 and DIN EN 13185. As pressurized gas is already available in the plant it was opted for the latter option. The pressure change method is used to determine mass change in a closed object, here as gas flow through leakages. This method is based on the ideal gas law. If most of the parameters can be set constant the change in mass is deducible from pressure changes. In the case of the pilot scale clinker cooler, a closed system was prepared by shutting the clinker inlet and outlet as first step. Afterwards the system was connected to the compressed air system of the cement plant. The system's pressure gauge showed a lower pressure than pressure provided by the used pressurized air network. After stopping the pressure air feed, the pilot system pressure dropped immediately. This induced the presence of leakages. To locate these leakages a tracer testing method was used, as described in DIN EN 131835. This method can show air flow through leakages from the inside to the outside. A pressure difference is produced, a tracer is injected and a leakage proof is given, resulting in a local change of color. To observe leakages of the pilot clinker cooler, a tracer is added to the system together with pressurized air. Signal Orange Smoke CF3 was used as a tracer gas. The bottle was placed and activated in the closed pilot cooler system (1). To spread the smoke signal the fans inside the cooling system were started (2). Afterwards pressurized air was turned on (3). The orange smoke could be detected outside the pilot plant visually by observing the pilot plant where leakages occurred (4). This procedure was performed three times and is shown in Figure 3.14. During these repetitions of the experiment all the main leakages were detected.

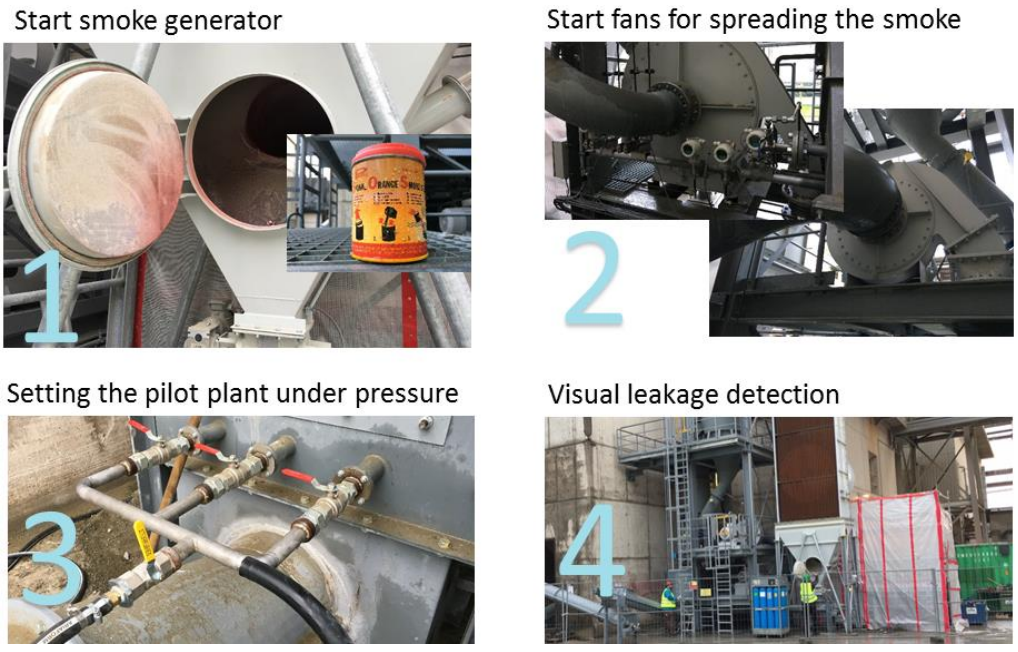

Figure 3.14: Tightness measurement with pressurized gas and smoke generator

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Main leakage points were located at component part connections like screw or flange connections (Figure 3.15). It was therefore decided to use silicon as additional sealing medium at affected areas. Also, already installed measurement ports were found not to be gas tight. One of the biggest leakage points found was the sealing from the clinker extraction device where the revolving pipe enters the static oxyfuel pilot cooler roof. For the sealing of the hot clinker extraction device soap water was used to detect leakages supporting the leakage detection in addition to the smoke indicator (Figure 3.16). With the lasting occurrence of air bubbles, it was decided to renew the sealing tape. After that, also that critical part of the pilot system became nearly gas tight.

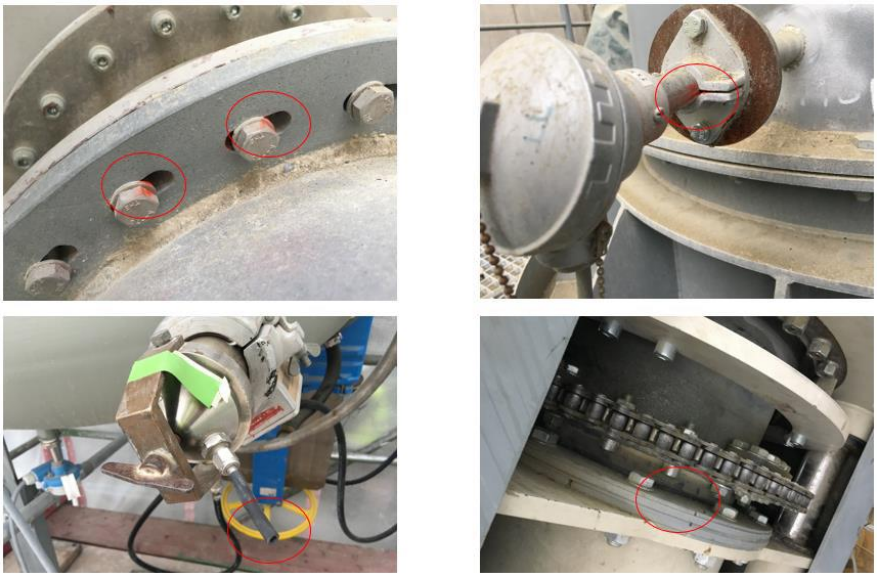

Figure 3.15: Leakages indicated by red residue from the smoke generator

Figure 3.16: Leakage detection with soap water between rotating clinker extraction device and static pilot cooler housing

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After replacing sealing ropes, tightening of screws and sealing with silicone the pressure change method was retried (Figure 3.17 and Figure 3.18). Here, the pressure was kept constant even if the pressurized air inlet was closed. Nevertheless, it must be pointed out that at that time the cold clinker extraction system (screw conveyors behind the pilot cooler) and the connection to the existing cooler front wall for the hot clinker extraction were fully closed for tightness testing. These two system boundaries turned out to be very important as found out during continuous operation.

Figure 3.17: Fixed leakages observed during the tightness tests1/2

Figure 3.18: Fixed leakages observed during the tightness tests 2/2

3.3.3 Commissioning of the hot clinker extraction tube and first clinker (November/December 2016)

One of the main challenges from the beginning was the extraction of a small percentage of hot clinker falling from the kiln while routing it into the pilot system. For that purpose a so-called hot clinker extraction tube was designed by IKN. A hydraulic drive was used to move the tube in and out of the falling stream of material. For an undisturbed sliding of the hot clinker through the extraction tube into the pilot system a critical angle of 65° was defined during basic engineering but due to local constraints this angle could not be reached in practice. That is why it was decided to install a drive for rotational movement of the extraction tube in addition. During commissioning this concept has proved to be successful and thus represents world's first

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continuous hot clinker extraction device. Although a scavenging blower was installed, it turned out that as time went by the sealing of the extraction tube slowly filled up with clinker dust leading to sluggishness. Particularly at start-up this has led to overload of the existing mechanical drive. It was then decided to install an identical second mechanical drive for the rotational movement of the extraction tube. With the second mechanical drive installed the extraction tube could be operated much more reliably. Unfortunately, shortly after commissioning the hot clinker extraction tube's tip was hit by large clinker lumps falling from the rotary kiln and hence mechanically destroyed. Broken refractory pieces were clogging the extraction tube preventing henceforth hot clinker reaching the prototype system.

3.3.4 CO_2 filling/draining and CO_2 tightness tests (December 2016)

Contrary to the future oxyfuel operation, where CO_2 in the recirculation gas is coming from calcination and fuel combustion, the CO_2 during the pilot plant trials was supplied from gas bottles. Twelve CO_2 bottles make one CO_2 bundle. Three bundles were used to fill the pilot plant and to maintain CO_2 percentage in the recirculation gas during operation at three different locations. One injection point was located directly at the cooler outlet while the other two injection points were located between heat exchanger outlet and fan inlets (Figure 2.2). Latter injection points were separated by a shut-off gate, which was intended to be used while flooding different parts of the pilot plant with CO_2. As injection of CO_2 directly into the cooler shows impact on cooling behaviour this injection point was only used for initial filling of the plant while the other two injection points were used during operation to guarantee a constant high level of CO_2 concentration. The period up to the required repairs of the hot clinker extraction tube's tip during the plant's winter shutdown could be used properly by performing some first CO_2 filling/draining as well as CO_2 tightness tests. During these tests, it quickly became obvious that the pre-assigned filling procedure should be reconsidered as filling with CO_2 during complete shutdown of the pilot system proved to be very difficult. Initially, filling of the pilot system was stopped ~15 min after gas analysers showed ~100 vol.% CO_2. As soon as the cooling fans were started the CO_2 content more than halved to less than 50 vol.%. That phenomenon could be theoretically explained by the existence of so-called air pockets in the pilot system. It was then decided to fill the pilot system with CO_2 from the bottles (Figure 3.19) while cooling fans were running. On the one hand this led to an increased loss of CO_2-rich gas, as during filling the same amount of gas entering the closed gas recirculation system through the CO_2 bottles had to leave the system through the hot clinker extraction device. On the other hand, this procedure proved to be the only solution. Beside of that the CO_2 tightness tests underlined the results obtained from the smoke tests but it must be pointed out again that at that time the cold clinker extraction system (screw conveyors behind the pilot cooler) was fully closed for tightness testing. As confirmed later the cold clinker extraction system turned out to be the biggest entry point for false air.

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Figure 3.19: CO_2 gas filling station with 3 bundles, 12 bottles each

3.3.5 Hot commissioning (January 2017)

In January 2017, just after the plant's winter shut down, the pilot system became fully operational. The starting procedure consisted of the following:

1. Clearance from HeidelbergCement control room?
2. Cement plant clinker conveyor running?
3. Start of cold clinker discharge screw conveyors
4. Start of cooler grate-drive
5. Start of heat exchanger de-dusting screw conveyor
6. Start of electromechanical and hydraulic drive of hot clinker extraction tube
7. min delay – waiting for first clinker
8. Start of cooling fans
9. Start of heat exchanger axial fans
10. Start of rotary valve behind de-dusting cyclone

This approach has proved to be effective in practice taking about 2 hours of time reaching a steady-state for initial start of the pilot plant. Once fully in operation changing from one operational point to another took approx. 30 min. Against this background and as a precaution after the first defect, it has been deliberately decided not running the pilot plant 24/7. Just like the cold-commissioning, hot-commissioning turned out positively and without special incidents with the pilot plant now ready for trial operation (Figure 3.20).

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Figure 3.20: Pilot system restart and completion of hot commissioning in January 2017

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4 OXYFUEL CLINKER COOLER OPERATION

Before the experiment in HeidelbergCement plant in Hannover has taken place, an experimental plan was carefully designed by IKN and VDZ with focus on the objectives of CEMCAP work package 9 (WP9 – Oxyfuel clinker cooler prototype):

1. Determine the influence of cooling gas composition on clinker chemistry
2. Determine the gas leaking rate to ambient
3. Determine false air in-leakage
4. Determine the clinker cooling curves with CO_2 rich gas

The original experimental plan was ambitious but nevertheless realistic, based on the information available at that time. However, the operation of the oxyfuel clinker cooler prototype revealed some unexpected experimental challenges and the original experimental plan had to be adapted to the new circumstances. Despite the challenges the team kept always the focus on the main objectives of the experiment and was able to operate the oxyfuel clinker cooler pilot plant successfully. The original and adapted experimental plans, as well as the main experimental challenges the team had to overcome, are described in the following subchapters in detail.

4.1 Experimental plan (original)

The cooler operation parameters, such as clinker throughput, bed depth, cooling gas volume flow would be varied by constant gas composition to monitor effects on clinker outlet temperature, clinker chemistry, granulometry and dust formation. The main cooling gas chemical components, which would be varied during the testing phase, were nitrogen, oxygen and CO_2. Because of its low quantity the influence of argon was neglected. Since argon is an inert gas, it could be replaced by nitrogen. The incidental introduction of water vapour was considered extremely challenging. Therefore, the recirculation of dry cooling gas was chosen. In that case, the humidity of the cooling gas would be expectably low and could be neglected (no significant change of the cooling gas heat capacity). Based on these pre-conditions an experimental matrix was set according to Table 3 and Table 4**.**

The oxyfuel cooling medium (CO_2–rich gas) would be supplied on-site through the injection of CO_2 and O_2 into the pilot plant from a filling station equipped with CO_2 and O_2 bottles, 50 litre, 200 bar. Clinker samples would be taken regularly for each operation setting and its temperature would be measured. Those clinker samples would be later analysed by VDZ and the impacts on clinker quality would be assessed. Cooling gas composition would be measured continuously. Moreover, several probes installed in the pilot plant at different points would collect the necessary process data regarding volume flows, pressure levels and temperatures.

Table 3 Experimental settings – cooling gas chemical composition

Cooling gas composition setting	Cooling medium	CO_2 [vol.%]	O_2 [vol.%]	N_2 [vol.%]
a	Ambient air	0	21	79
b	Oxyfuel	~ 100	0	0
c	Oxyfuel (base case)	70	18	12

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Table 4 Experimental settings – pilot cooler control parameters @ 80 tpd clinker load

Cooling gas composition setting	Clinker specific cooling gas volume flow (Nm^3/kg clinker)	Temperature of the cooling gas (°C)	Clinker bed depth (mm)
a	- Reference (2 Nm^3/kg clinker)	- Reference (100 °C)	- Reference (500 mm)
	- Variation	- Reference (100 °C)	- Reference (500 mm)
	- Reference (2 Nm^3/kg clinker)	- Variation	- Reference (500 mm)
	- Reference (2 Nm^3/kg clinker)	- Reference (100 °C)	- Variation
b	- Reference (2 Nm^3/kg clinker)	- Reference (100 °C)	- Reference (500 mm)
c	- Reference (2 Nm^3/kg clinker)	- Reference (100 °C)	- Reference (500 mm)
	- Variation	- Reference (100 °C)	- Reference (500 mm)
	- Reference (2 Nm^3/kg clinker)	- Variation	- Reference (500 mm)
	- Reference (2 Nm^3/kg clinker)	- Reference (100 °C)	- Variation

4.2 Unexpected experimental challenges

As stated before, several unexpected experimental challenges led the team to adapt the experimental plan initially envisaged. Among the most conditioning unexpected challenges were:

1. Damage and blockages of the hot clinker extraction tube
2. Lower clinker production rate than design capacity
3. False air ingress through the cold clinker extraction system

Each one of the above challenges had some other side effects, which conditioned the experiment. All of them are described below in more detail.

4.2.1 Damage and blockages of the hot clinker extraction tube

The hot clinker extraction tube was severely damaged 3 times during the experiment by big material lumps falling from the kiln onto the tip of the hot clinker extraction tube (November 2016, February 2017 and March 2017). Every time stable operation of the pilot plant was then compromised and the tube needed repair (refractory was damaged and blocked clinker entrance - Figure 4.1). Repair of the hot clinker extraction tube demands a kiln stoppage. Kiln stoppages are planned long time before they take place in cement plants (e.g. winter shutdowns). Therefore, HeidelbergCement (HC) could not stop the kiln immediately after the damage and blockage of the hot clinker extraction tube as desired. Every time the hot clinker extraction tube was damaged, the experiment had to be left in standby until the tube had been repaired. The impossibility to run the oxyfuel pilot plant for long periods of time before the hot clinker extraction tube got damaged considerably limited the amount of data collected during the experiment.

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Figure 4.1: Broken refractory pieces clogging the extraction tube

4.2.2 Lower clinker production rate than design capacity

Stable operation of the oxyfuel clinker cooler was reached during some periods of time but with lower clinker production rates than design capacity (up to 46 t/d instead of 80 t/d). Clinker production rates were hard to replicate on a daily basis due to inherent process instabilities (with the identical clinker trapping point different clinker amount could be routed to the pilot plant) and difficulties in operating with the hot clinker extraction tube.

Many of the trials were performed during very cold winter days, with ambient temperatures close and below 0 °C. Lower clinker production rates together with cold false air ingress decreased the temperature of the pilot cooler exhaust gas. For such operation conditions the air-gas heat exchanger became over dimensioned (too large heat exchanging surface). Even with the cooling fans of the air-gas heat exchanger turned off, low ambient temperatures were able to cool down the pilot cooler exhaust gas to temperatures much lower than 100°C. The air-gas heat exchanger had not been designed for such low pilot cooler exhaust gas temperatures and volume flows, which jeopardized its operation and control.

In conclusion, too low clinker production rates hampered the operation and control of the pilot plant as a whole, which in turn challenged the replication of the same experimental settings for different cooling mediums (e.g. temperature and volume flow variation).

4.2.3 False air ingress through the cold clinker extraction system

Filling the pilot plant with CO_2 and keeping CO_2 concentration at levels higher than 70 vol.% revealed to be extremely challenging. The team was able to reach concentration levels close to 100 vol.% CO_2 during the filling stage of the oxyfuel pilot plant. However, whenever the cooling fans were turned on and the injection of CO_2 was stopped, CO_2 concentration started to fall very quickly until reaching values close to 0 vol.% CO_2. Based on those observations the team

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concluded that CO_2 was escaping and air was being pulled through the cold clinker discharge system (screw conveyors) into the pilot plant (Figure 4.2). This conclusion was based on the fact that the pilot plant had already proven to be gas-tight after conduction of several leakage tests.

In order to improve tightness and decrease the amount of false air ingress, the team raised the filling degree of the screw conveyors during trial operation. However, during the trials the team concluded that the natural sealing effect of the material inside the screw conveyors was greatly overestimated. Experiments performed by IKN and VDZ to investigate the sealing efficiency of the cold clinker discharge system showed that even with high filling degrees false air intake could not be avoided. Moreover, during the same trials the team realized that different settings of the pilot cooler recirculation fans did not have a significant impact on false air ingress. Even when the cooling fans were turned off, false air was being pulled into the oxyfuel pilot plant. Based on those observations it was concluded that false air was being pulled into the system by the kiln hood under-pressure of the HC plant. The under-pressure level at the kiln hood was strong enough to pull a considerable amount of false air continuously from the clinker discharge system (through the screw conveyors) into the pilot plant. Therefore, the CO_2 –rich cooling gas was being continuously mixed and diluted by false air ingress and the resulting gas-mix was pulled out of the pilot plant through the hot clinker extraction tube. At that time it was impossible to keep high enough CO_2 concentration levels, even with continuous CO_2 refilling. In order to test that thesis, HC agreed to increase kiln hood pressure as much as possible during the time of the experiments (from -0.9/-0.7 to -0.15/-0.3 mbar). Keeping CO_2 concentration levels higher than 70 vol.% was then possible. Nevertheless continuous injection of CO_2 into the pilot plant was still required.

Figure 4.2: False air ingress circuit

The pilot plant was designed with 3 gas injection points, which had been planned to be used exclusively during filling and/or punctually refilling of the pilot plant with CO_2 and O_2. However, after performing some more tests the team concluded that at least 2 injection points would have to be permanently used for continuous CO_2 injection. Moreover, the team decided to avoid continuous injection of cold gas at the third gas injection point (at the cooler) as it would impact the clinker cooling rate during operation. The necessary injection of high CO_2 volume flows demanded changing of the gas bundles very frequently. Nevertheless, the team was able to manage this situation without completely interrupting CO_2 injection and keeping the decay of CO_2 concentration in an acceptable range during bottles change.

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The amount of false air ingress precluded the production of cooling gas with a chemical composition defined as "gas composition setting c" **(**Table 3). Only the injection of a high O_2 volume flow could compensate the amount of N_2 that was entering into the pilot plant through false air ingress. The control of CO_2 injection to keep the cooling gas with the desired CO_2 concentration was already extremely challenging. The additional injection of O_2 would have made the control of cooling gas composition simply impossible. Moreover, there were not enough O_2 injection points available that could enable the injection of the required O_2 volume flow.

4.3 Experimental plan (adapted)

A total of three measuring campaigns were performed by the team during the operation of the oxyfuel clinker cooler prototype. A set of different parameters, such as different cooling gas compositions and cooling rates were investigated and clinker samples were taken as previously planned (Table 6). Different clinker bed velocities (clinker bed depth) were not possible to test due to the operation of the clinker cooler prototype with much lower clinker production than previously envisaged. A maximum allowable clinker bed velocity had to be ensured during the tests in order to guarantee an acceptable minimum clinker bed height. CO_2 and O_2 concentration levels of the recirculated cooling gas were measured continuously in order to characterize the cooling gas chemical composition.

Due to the unexpected experimental challenges mentioned in chapter 4.2, experimental settings were hard to replicate on a methodical basis. Fixing all main process variables with exception to one of them in order to assess the effect of its variation on cooler performance was not possible anymore (Table 6**)**. Nevertheless, the team proceeded with the experiment and decided to concentrate all its efforts on reaching and keeping a high CO_2 concentration level (> 70 vol.%) of the cooling gas, as well as on collecting clinker samples (Table 5**)**. WP9 partners agreed that CO_2 concentrations higher than 70 vol.% would be already representative of oxyfuel cooling operation and thus a very good indicator regarding eventual impacts of CO_2 on clinker quality and potential recarbonation effects.

Table 5 Adapted experimental settings – cooling gas chemical composition

Gas composition setting	Cooling medium	CO_2 [vol.%]	O_2 [vol.%]	N_2 [vol.%]
d	Ambient air	0	21	79
e	Oxyfuel	> 70%	< 6.3 *	< 23.7 *

*** Note: Only CO_2 was injected. Air was the single source of O_2 and N_2. Therefore, O_2 and N_2 volume percentage is related to each other and their relative volume proportion remained constant.**

Table 6 Adapted experimental settings – pilot cooler control parameters

Cooling gas composition setting	Clinker specific cooling gas volume flow (Nm^3/kg clinker)	Temperature of the cooling gas (°C)	Clinker bed depth (mm)
d	- Variation *	- Variation *	- Variation *
e	- Variation *	- Variation *	- Variation *

*** Note: Detailed information can be found in chapter 5.1 – Experimental results.**

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5 EXPERIMENTAL DATA ASSESSMENT AND RESULTS

In this chapter the assessment of all relevant experimental data is shown. Moreover, experimental results linked to the main experimental objectives of CEMCAP WP9 are presented as well.

5.1 Experimental data

Table 7 to Table 9 show all the relevant process data collected during the trials. Settings 1 and 2 correspond to trials performed in January 2017, with air as cooling medium. Settings 3 and 4 correspond to the first successful trials performed with oxyfuel cooling medium (02/02/2017).

*Table 8*Table 8 shows that in settings 1, 2, 3 and 4 clinker mass flow was well below the design capacity of the pilot plant (80 t/d). The measurement of clinker mass flow by a contactless solid flow sensor was not very reliable (strong fluctuation and inaccuracy) and the team finally decided to cool the clinker with higher clinker specific cooling gas volume flows than previously envisaged in the original experimental plan (2 Nm^3/kg clinker - Table 4). The clinker specific cooling gas volume flow from settings 1, 3 and 4 is too high and thus not representative of normal cooler operation. Therefore, the use of these settings of the pilot cooler performance assessment is limited. The clinker specific cooling gas volume flow from setting 2 was also higher than it should be, but it was the best available data for the calculation of the cooling curves of the pilot cooler with air as cooling medium. Despite the very high clinker specific cooling gas volume flows, settings 3 and 4 were of high importance, as they represent a remarkable milestone in the experiment: the possibility to reach and hold high CO_2 concentration levels in the cooling gas for the first time (Table 7**)**. Moreover, for the first time clinker samples of an industrial kiln cooled with an oxyfuel cooling medium were taken for further analysis in VDZ's laboratories.

Table 7 Experimental data 1

Setting	Cooling medium	CO_2 vol.% (dry) *	CO_2 vol.% (dry) **	O_2 vol.% (dry) **	Cooling medium temperature (°C)	Cold clinker temperature (°C)
1	air	0%	0%	~ 21%	91	51
2	air	0%	0%	~ 21%	57	30
3	CO_2 –rich gas	90%	84%	3.0%	71	25
4	CO_2 –rich gas	77%	67%	6.6%	55	n/a
5	CO_2 –rich gas	81%	75%	4.8%	66	45
6	CO_2 –rich gas	81%	75%	4.9%	65	45
7	CO_2 –rich gas	79%	76%	4.6%	53	67
8	air	0%	0%	21%	63	131
9	air	0%	0%	21%	60	83
10	air	0%	0%	21%	57	92

***Note: estimated after CO_2 injection and based on an energy and mass balance (Appendix c – Pilot plant heat and mass balance model)**
****Note: measured during the trials before CO2 injection.**

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Table 8 Experimental data 2

Setting	Cooling medium	Cooling medium volume flow (Nm^3/h) (wet)	Clinker mass flow (t/d)	Clinker specific volume flow (Nm^3/kg clinker) (wet)	Cooler exhaust gas temperature (°C) (at dedusting cyclone inlet)	Cooler exhaust gas volume flow (Nm^3/h) (wet)
1	air	7914	30*	6.3	225	7914
2	air	2747	23**	2.9	396	2747
3	CO_2 –rich gas	6205 ▪	20**	7.4	218	4012
4	CO_2 –rich gas	5856 ▪	19**	7.4	152	4038
5	CO_2 –rich gas	2774 ▪	47*	1.4	298	2089
6	CO_2 –rich gas	2257 ▪		1.2	352	1674
7	CO_2 –rich gas	1114 ▪		0.6	555	971
8	air	1781		0.9	454	1781
9	air	1120		0.6	405	1120
10	air	1005		0.5	394	1005

***Note: measured by clinker extraction from the pilot plant and weighing. For settings 5 to 10 the clinker weighing was performed only once due to the impossibility of running the experiment and weighing the clinker simultaneously.**

****Note: measured by mass flow sensor.**

▪Note: based on 10 vol.% H_2O in average (according to measurements).

Table 9 Pilot cooler and cold clinker extraction system setpoints

Setting	Cooling medium	Screw conveyor 1 rotation speed (rpm)	Screw conveyor 2 rotation speed (rpm)	Clinker conveying time in screw conveyors (min.)	Cooler grate speed (s.p.m.)
1	air	2,3	25	4	2
2	air	7	25	2	3
3	CO_2 –rich gas	0,50	25	20	2,6
4	CO_2 –rich gas	7	25	2	2,6
5	CO_2 –rich gas	1,63	25	6	5
6	CO_2 –rich gas	1,63	25	6	5
7	CO_2 –rich gas	1,63	25	6	5
8	air	1,63	25	6	5
9	air	1,63	25	6	5
10	air	1,63	25	6	5

Settings 5 to 10 correspond to trials performed on the 28/03/2017, which was the day before the experiment has ended (last day of measurements). The team was aware of the need to collect more process data and clinker samples during oxyfuel operation for the success of the experiment. As the team was running out of time to perform the experiment, the focus was driven to oxyfuel operation as first priority. Air operation became a secondary priority. A total of 9 CO_2 bundles were available to perform the experiment with CO_2–rich cooling gas. Settings 5 to 7 have been performed until the moment the pilot plant ran out of CO_2. Trials with air as

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cooling medium started immediately after the CO_2 concentration in the pilot plant reduced to 0 vol.% (settings 8 to 10).

Table *8* shows that the cooler exhaust gas temperature of settings 8 to 10 decreased as the clinker specific cooling gas volume flow decreased as well. Exactly the opposite would be expected. The lower the clinker specific cooling gas volume flow, the higher the pilot cooler exhaust gas temperature has to be (this trend is shown in settings 5 to 7). The phenomenon observed in settings 8 to 10 can be explained by the fact that the pilot plant with significant thermal mass in its refractory material did not have enough time to cool down, release the energy stored during setting 7 before the tests with air as cooling medium have started. The energy stored in the system ended up to mislead the temperature measurements of the cooler exhaust gas of setting 8 and thus the expected trend of the exhaust gas temperature with the variation of the cooling gas volume flow. Settings 9 and 10 have very similar clinker specific cooling medium volume flows and exhaust gas temperatures. A small fluctuation of the clinker mass flow during settings 9 and 10 would impact exhaust gas temperature and could explain to a certain extent the unexpected exhaust gas temperature trend observed. Therefore, settings 8 to 10 could not be used to assess pilot cooler performance.

Clinker temperature was measured during clinker sampling and was lower than cooling medium temperature in settings 1 to 6. This phenomenon can only be explained by the fact that the clinker was cooled down even further by false air ingress between the pilot cooler outlet and the clinker sampling point.

5.2 Experimental results - clinker quality

During the different phases of the trials, in total nine clinker samples were taken and subsequently analysed. Two clinker samples were taken from the pilot cooler when it was operated with air (S1_air / S2_air). The two samples were homogenized before analysing the resulting mix sample (ØS3_air). Five clinker samples were taken in two different trial phases (02.02.2017 and 28.03.2017) from the pilot cooler, when it was operated with CO_2-rich gas ("S4_oxy", "S5_oxy", "S6_oxy", "S7_oxy", "S8_oxy"). Two clinker samples ("S9_air" and "S10_air") were taken from the HC plant cooler at the same time when other samples were taken from the pilot cooler (02.02.2017 and 28.03.2017).

The samples ØS3_air to S10_air (Table 10) were analysed with regard to their chemical composition (X-ray fluorescence, infrared spectroscopy), their mineralogical composition (X-ray diffraction) and their microstructure (clinker microscopy).

Table 10 Clinker samples

Short name of the clinker sample	Component
S1_air	Pilot plant clinker 16.1.17 14.40 (air)
S2_air	Pilot plant clinker 17.1.17 15.50 (air)
ØS3_air	Ø Pilot plant clinker 16./17.01.2017 (air)
S4_oxy	Pilot plant clinker 02.02.2017 11:20 (oxy)
S5_oxy	Pilot plant clinker 02.02.2017 15:15 (oxy)
S6_oxy	Pilot plant clinker 28.03.2017 16:35 (oxy)
S7_oxy	Pilot plant clinker 28.03.2017 16:55 (oxy)
S8_oxy	Pilot plant clinker 28.03.2017 17:20 (oxy)
S9_air	HC clinker 02.02.2017 11:15
S10_air	HC clinker 28.03.2017 16:35

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5.2.1 Methods - sample preparation

In a first step several granules with a diameter of approx. 1 cm were taken from each clinker sample for the preparation of polished sections. Subsequently the clinker samples were crushed in a jaw crusher. The fraction of 2-4 mm was sieved off. A representative subsample was taken from the fraction for the preparation of a polished section. The residual 2-4 mm fraction was combined with the rest of the respective sample and homogenized. A representative subsample was taken and ground to analytical fineness.

5.2.2 Methods - chemical composition

The chemical analysis were carried out with X-ray fluorescence analysis (XRF) on fused beads acc. EN 196-2. The contents of CO_2 and H_2O were measured with infrared spectroscopy (IR) at 950 °C.

5.2.3 Methods - mineralogical composition

The mineralogical compositions of the clinker samples were examined with X-ray diffraction analysis (XRD). The powder samples were analysed within a diffraction angular range of 5 - 80° (2 Theta, Cu Kα radiation). The minor clinker phases were analysed by measuring X-ray diffraction patterns of the clinker samples after dissolving the silicate phases and the free lime in a methanol/salicylic acid digestion (M/S digestion). The X-ray diffraction patterns were evaluated quantitatively with the Rietveld refinement method.

5.2.4 Methods - microstructure

The microstructure of the clinker samples was evaluated with reflected light microscopy on polished sections. The sections were prepared from granules with ca. 1 cm diameter and from subsamples of the 2-4 mm fraction taken from the crushed clinker samples. The granules and fraction samples were vacuum impregnated with a resin, ground and polished. For the examination of the clinker sample with optical microscopy, 4 granules with diameter of approx. 10 mm of the unprocessed sample were embedded in low viscosity epoxy resin under vacuum. Additionally, a representative subsample of each clinker with a grain size of 2 to 4 mm was obtained by crushing and sieving of the clinker sample and likewise embedded in epoxy resin under vacuum. After curing, polished sections were produced and etched with 10 % solution of KOH as well as alcoholic dimethyl ammonium citrate (DAC) solution. The sections were studied with an optical microscope under reflected light before and after etching.

5.2.5 Results – clinker chemical composition

The chemical compositions of all analysed clinker samples are summarized in Table 11 and Table 12.

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*Table 11 Chemical composition of the clinker samples, analysed with XRF, except: * analysed with IR; # calculated*

Sample short name		**S9_air**	**S10_air**	**ØS3_air**	**S4_oxy**
Component		**HC clinker 02.02.2017 11:15**	**HC clinker 28.03.2017 16:35**	**Ø Pilot plant clinker 16./17.01. 2017 (air)**	**Pilot plant clinker 02.02.2017 11:20 (oxy)**
CO_2*	mass %	0.09	0.10	0.12	0.09
H_2O*	mass %	0.11	0.14	0.28	0.14
Loss on ignition*	mass %	0.20	0.24	0.40	0.23
SiO_2	mass %	22.27	21.87	21.89	21.97
Al_2O_3	mass %	5.66	5.55	5.42	5.51
TiO_2	mass %	0.28	0.31	0.35	0.35
P_2O_5	mass %	0.18	0.17	0.17	0.17
Fe_2O_3	mass %	1.71	1.74	1.71	1.74
Mn_2O_3	mass %	0.04	0.04	0.04	0.04
MgO	mass %	0.99	0.97	1.00	1.00
CaO	mass %	66.56	67.46	66.78	66.96
SO_3	mass %	0.83	0.66	0.73	0.73
K_2O	mass %	0.85	0.73	0.78	0.79
Na_2O	mass %	0.28	0.23	0.31	0.32
Na_2O equivalent	mass %	0.85	0.72	0.82	0.85
$C_3S^{\#}$ (Bogue)	mass %	61.50	68.88	67.19	66.41
$C_2S^{\#}$ (Bogue)	mass %	17.69	10.94	12.62	13.18
$C_3A^{\#}$ (Bogue)	mass %	12.15	11.80	11.55	11.71
$C_4AF^{\#}$ (Bogue)	mass %	5.21	5.30	5.26	5.31
LSF#	-	96.0	99.0	98.1	97.9
Silica ratio#	-	3.02	3.00	3.07	3.03
Alumina ratio#	-	3.32	3.20	3.16	3.17
Degree of sulphatisation#	-	75.6	70.9	68.6	66.5

Due to differences mainly in CaO and SiO_2 contents, the lime saturation factor (LSF) varies in the clinker samples. The lowest LSF in the samples (96.0) was calculated for "S9_air". The LSF values for the other samples varied between 97.9 and 99.3. Accordingly, the Bogue calculation led to varying contents of C_2S and C_3S. The highest (C_2S) and lowest (C_3S) values, respectively, occurred in the in the sample with the lowest LSF ("S9_air": 17.69 mass% C_2S, 61.50 mass % C_3S). In the other samples, C_2S contents of 10.28 to 13.18 mass% and C_3S contents of 66.41 to 69.71 mass% were calculated.

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*Table 12 Chemical composition of the clinker samples, analyzed with XRF, except: * analyzed with IR; # calculated*

Sample short name		**S5_oxy**	**S6_oxy**	**S7_oxy**	**S8_oxy**
Component		**Pilot plant clinker 02.02.2017 15:15 (oxy)**	**Pilot plant clinker 28.03.2017 16:35 (oxy)**	**Pilot plant clinker 28.03.2017 16:55 (oxy)**	**Pilot plant clinker 28.03.2017 17:20 (oxy)**
CO_2*	mass %	0.36	0.44	0.70	0.81
H_2O*	mass %	0.15	0.13	0.14	0.14
Loss on ignition*	mass %	0.51	0.57	0.84	0.95
SiO_2	mass %	21.90	21.84	21.71	21.58
Al_2O_3	mass %	5.50	5.60	5.52	5.56
TiO_2	mass %	0.30	0.31	0.30	0.32
P_2O_5	mass %	0.17	0.16	0.17	0.16
Fe_2O_3	mass %	1.68	1.74	1.71	1.69
Mn_2O_3	mass %	0.04	0.04	0.04	0.04
MgO	mass %	1.00	0.97	0.95	0.94
CaO	mass %	67.11	67.46	67.19	66.91
SO_3	mass %	0.60	0.63	0.55	0.60
K_2O	mass %	0.78	0.71	0.65	0.68
Na_2O	mass %	0.33	0.23	0.23	0.23
Na_2O equivalent	mass %	0.84	0.70	0.67	0.69
$C_3S^{\#}$ (Bogue)	mass %	67.80	68.73	69.71	69.49
$C_2S^{\#}$ (Bogue)	mass %	12.03	10.99	10.29	10.28
$C_3A^{\#}$ (Bogue)	mass %	11.80	11.93	11.87	12.01
$C_4AF^{\#}$ (Bogue)	mass %	5.15	5.31	5.24	5.22
LSF#	-	98.5	99.0	99.3	99.3
Silica ratio#	-	3.05	2.98	3.00	2.98
Alumina ratio#	-	3.27	3.22	3.24	3.28
Degree of sulphatisation#	-	55.1	69.0	63.1	68.0

The silica ratio (SR, 2.98 to 3.07) and the alumina ratio (AR, 3.16 – 3.32) of all samples were relatively stable. Consequently, the Bogue calculation delivered relatively narrow ranges of values for C_3A contents (11.55 to 12.15 mass %) and C_4AF contents (5.15 to 5.31 mass %). Due to the high alumina ratio, the C_3A contents according to Bogue were also high for all samples. The alkali content of the clinker samples ranged between 0.67 and 0.85 mass % Na_2O equivalent, the SO_3 content between 0.55 and 0.83 mass %. This resulted in degrees of sulphatisation (DoS) of 55.1 to 75.6. The two clinker samples from the plant cooler ("S9_air", "S10_air") and the clinker sample cooled with air in the pilot cooler ("ØS3_air") had CO_2 contents of up to 0.12 mass %. Four of the five clinker samples cooled with CO_2 in the pilot cooler had increased CO_2 contents of 0.36 – 0.81 mass %. It can be seen that the CO_2 content of the three clinkers sampled on 28.03.2017 increases as the experiment is running (three trials). The water content is not increased in any of the samples and ranges between 0.11 and 0.28 mass %.

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5.2.6 Results – clinker mineralogical composition

The mineralogical compositions of all analysed clinker samples are summarized in Table 11 and Table 12, together with the respective residue after M/S digestion. The X-ray diffraction patterns are shown in Figure 5.1.

In all analysed clinker samples the alite content ranged between 59.1 and 64.7 mass %. The belite content ranged between 16.4 and 19.0 mass %. A correlation of alite and belite contents with the LSF could not be observed. Three samples contained ≤ 0.8 mass % of free lime ("S9_air", "ØS3_air", "S4_oxy"), the other five samples ≥ 1.9 mass %. The three samples with low free lime contents had the highest contents of alite. Usually this indicates a higher degree of burning.

The samples contained belite in two different modifications (α' + β). The two clinker samples from the plant cooler ("S9_air", "S10_air") mainly contained the β-modification of belite, which typically occurs in most technical clinkers. The presence of α'-belite is not certain in these samples. However, the presence of α'-belite in all samples from the pilot cooler seems certain. The ratio of α'- + β-belite is roughly 1:1 in the pilot cooler samples. α'-belite usually indicates fast cooling.

Table 13 Mineralogical composition of the clinker samples, analysed with XRD

Sample short name		**S9_air**	**S10_air**	**ØS3_air**	**S4_oxy**
Component		**HC clinker 02.02.2017 11:15**	**HC clinker 28.03.2017 16:35**	**Ø Pilot plant clinker 16./17.01. 2017 (air)**	**Pilot plant clinker 02.02.2017 11:20 (oxy)**
alite	mass %	63.4	61.9	64.7	64.3
α'-belite	mass %	1.9	3.2	9.0	9.7
β-belite	mass %	15.3	14.6	8.1	7.7
belite (α' + β)	mass %	17.2	17.8	17.1	17.4
C_3A cub.	mass %	13.6	11.8	10.9	10.7
C_3A orthorh.	mass %	1.3	1.8	2.8	2.9
C3A (cub. + orthorh.)	mass %	14.9	13.6	13.7	13.6
C_4AF	mass %	2.8	2.8	2.3	2.3
free lime	mass %	0.5	2.5	0.7	0.8
periclase	mass %	traces	traces	traces	traces
quartz	mass %	traces	traces	traces	traces
calcite	mass %	traces	traces	traces	traces
arcanite	mass %	0.4	0.4	0.6	0.7
aphthitalite	mass %	0.5	0.3	0.6	0.6
mayenite	mass %		traces		
M/S residue	mass %	18.97	17.90	17.64	17.52

All samples contained relatively high amounts of C_3A (13.6 – 14.9 mass %), mostly in its cubic modification (10.7 – 13.6 mass %). Only in sample "S5_oxy" a lower amount of cubic C_3A (7.8 mass %) and a higher amount of the orthorhombic modification of C_3A (6.3 mass %), which is richer in alkalis, was observed. This correlates with a lower degree of sulphatisation (55.1) of

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this sample compared to the other samples (63.1 to 75.6). The amount of alkalis not bound in sulphates is higher at lower degrees of sulphatisation, and they are mainly bound in C_3A, changing its modification from cubic to orthorhombic. The contents of C_4AF were relatively low (2.3 – 3.1 mass %) in all samples.

Table 14 Mineralogical composition of the clinker samples, analysed with XRD

Sample short name		**S5_oxy**	**S6_oxy**	**S7_oxy**	**S8_oxy**
Component		**Pilot plant clinker 02.02.2017 15:15 (oxy)**	**Pilot plant clinker 28.03.2017 16:35 (oxy)**	**Pilot plant clinker 28.03.2017 16:55 (oxy)**	**Pilot plant clinker 28.03.2017 17:20 (oxy)**
alite	mass %	62.6	62.8	59.6	59.1
α'-belite	mass %	10.3	8.5	10.1	10.4
β-belite	mass %	6.3	7.9	8.6	8.6
belite (α' + β)	mass %	16.6	16.4	18.7	19.0
C_3A cub.	mass %	7.8	12.2	11.6	12.2
C_3A orthorh.	mass %	6.3	2.0	2.9	2.1
C3A (cub. + orthorh.)	mass %	14.1	14.2	14.5	14.3
C_4AF	mass %	3.1	2.7	2.8	2.8
free lime	mass %	1.9	2.3	2.3	2.5
periclase	mass %	traces	traces	traces	traces
quartz	mass %	< 0.5	traces	< 0.5	traces
calcite	mass %	0.6	0.8	1.4	1.6
arcanite	mass %	< 0.5	< 0.5	< 0.5	< 0.5
aphthitalite	mass %	< 0.5	< 0.5	< 0.5	< 0.5
mayenite	mass %	-	-	-	-
M/S residue	mass %	18.90	18.49	19.44	19.48

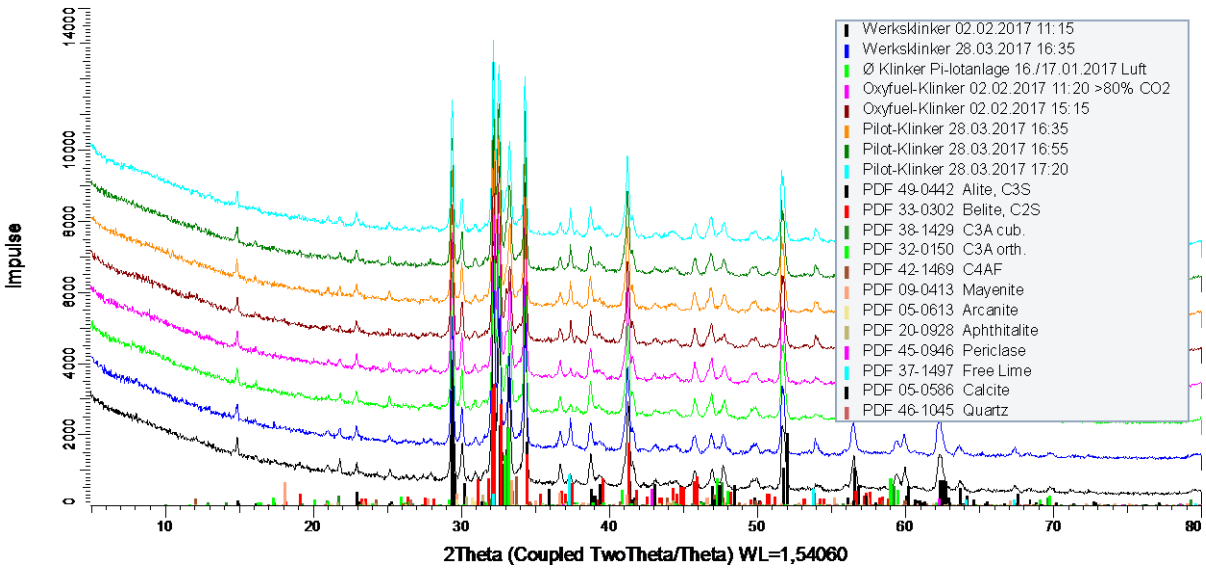

Figure 5.1: X-ray diffraction patterns of all analyzed clinker samples with phase designation

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In one sample ("S10_air") traces of mayenite ($C_{12}A_7$), an intermediate calcium aluminate phase, was found. It can indicate lower burning degrees, but mostly survives the sintering zone in the cores of large granules. Alkali sulphates (arcanite, aphthitalite) were identified in all samples. Unusual for clinker, the samples "S5_oxy", "S6_oxy", "S7_oxy", and "S8_oxy" contained between 0.6 and 1.6 mass % of calcite. This coincides with the relatively high CO_2 contents of these samples. The calcite content of the three clinkers sampled on 28.03.2017 from the pilot cooler increased with increasing duration of the trial. All of these samples were cooled with CO_2. Therefore principally free lime (CaO) from the clinker and CO_2 from the cooling medium might have reacted to $CaCO_3$. However, the sample "S4_oxy" was also cooled with CO_2 and did not show increased contents of calcite or CO_2.

5.2.7 Results – clinker microstructure

Macroscopic pictures of the clinker samples in their original state and their microstructural features can be found in Figure 9.1 to Figure 9.36 in the end of the report (Appendix B – Photos from clinker quality assessment). The most important microstructural features of the clinker sample are listed in Table 15 and Table 16. Using optical microscopy for the analysis of the polished and etched sections, the following clinker phases could be differentiated:

- alite (xenomorphic to idiomorphic, grey to light brown crystals with dark edges)
- belite (round, light grey crystals)
- tricalcium aluminate (C_3A, brownish grey phase in the ground mass)
- ferrite (C_4AF; white phase in the ground mass)
- free lime (round, dark grey or brown crystals)

The analysed clinker samples mostly consisted of alite rich, homogeneous material with a high burning degree (Figure 9.2, Figure 9.7, Figure 9.12, Figure 9.15, Figure 9.19, Figure 9.23, Figure 9.28, Figure 9.33). Only limited portions of the samples had microstructural features (increased porosity, lower homogeneity, increased contents of belite and free lime clusters) indicating lower burning degrees (Figure 9.3, Figure 9.8, Figure 9.16, Figure 9.20, Figure 9.24, Figure 9.29, Figure 9.34). Principally higher contents of domains with these features correlated with increased contents of free lime acc. XRD. This shows that higher free lime contents in the samples were mainly caused by lower average burning degrees. Belite clusters usually had diameters of up to 1000 µm, but single clusters with diameters of several mm were observed. Belite clusters in well burnt clinker are usually caused by coarse siliceous grains in the raw meal or in fuel ashes. Free lime clusters usually had diameters of up to 250 µm, but single clusters with diameters of up to 500 µm were observed. Free lime clusters in well burnt clinker are usually caused by coarse calcium rich grains in the raw meal. The average size of alite crystals in the clinker samples was relatively small (10 to 13 µm). This usually indicates high heating rates of the kiln feed to sintering temperatures in the kiln and a short sintering zone. The belite crystals in the samples had average sizes of 9 to 11 µm. C_3A was the predominant phase in the ground mass of all clinker samples. Its crystal shape was mostly typical for its cubic modification. C_3A crystals with shapes typical for the orthorhombic modification and the ground mass phase C_4AF occurred less frequent. These observations are consistent with the mineralogical data. The fineness of the ground mass crystals differed systematically between the clinkers sampled from the plant cooler and those sampled from the pilot cooler. In the plant cooler samples ("S9_air", "S10_air") the ground mass was mostly fine grained to coarse grained,

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indicating intermediately fast cooling conditions (Figure 9.4, Figure 9.5, Figure 9.9). In the other samples, taken from the pilot cooler, the ground mass was mostly fine grained, which is typical for fast cooling conditions (Figure 9.13, Figure 9.17, Figure 9.20, Figure 9.21, Figure 9.25, Figure 9.30, Figure 9.35). Therefore the cooling rate in the pilot cooler seems to have been higher than in the plant cooler. In clinker samples from the pilot cooler the alite crystals mostly showed signs of corrosion and were surrounded by thin rims of belite (Figure 9.13, Figure 9.17, Figure 9.20, Figure 9.21, Figure 9.25, Figure 9.30, Figure 9.35). The corrosion of alite crystal surfaces is normal and takes place during the crystallisation of the ground mass, caused by lack of calcium in the melt. This leads to a small scale breakdown of alite to belite (secondary belite) and the release of CaO for crystallisation of C_3A and C_4AF. The microstructural features in the pilot cooler samples indicate a short precooling zone of the material after passing the sintering zone in the kiln. A longer precooling zone would result in smoothing of the alite crystal surfaces and in recrystallization of the belite to coarser crystals at high temperatures. These microstructural features were partly observed in the two clinker samples from the plant cooler (Figure 9.5). Therefore the precooling zone seems to have been shorter in the samples taken from the pilot cooler compared to the samples taken from the plant cooler. The signs for a shorter precooling zone and faster cooling in samples from the pilot cooler compared to the plant cooler could result from the experimental setup. The clinker fed into the pilot cooler (Figure 9.10, Figure 9.11, Figure 9.14, Figure 9.18, Figure 9.22, Figure 9.27, Figure 9.32) was systematically enriched in finer granules compared to the usual plant clinker (Figure 9.1, Figure 9.6). Finer granules can be cooled faster due to the higher surface-volume-ratio. Typical signs for reducing burning conditions were not found. In four of the five samples from the pilot cooler which were cooled with CO_2 an unusual microstructural feature occurred ("S5_oxy", "S6_oxy", S7_oxy", "S8_oxy"). In direct contact with pores alite crystals were covered by a very finely crystalline layer of up to 2 µm thickness (Figure 9.21, Figure 9.26, Figure 9.31, Figure 9.36). The layer was often thinner than 2 µm and did not occur in all clinker grains of the samples. The abundance of the layer and its thickness were lowest in the sample "S5_oxy" and highest in the sample "S8_oxy". They increased in the oxyfuel clinker samples taken on 28.03.2017 with increasing duration of the trial. The phase content of the layer could not be identified. The layer was not observed in the three samples cooled with air ("S9_air", "S10_air", "ØS3_air") and in sample "S4_oxy". Since the layer does not occur in all samples cooled with CO_2, its formation cannot be correlated with the high CO_2 concentrations in the cooling medium alone. Two measurements revealed very high moisture contents in the cooling medium (Fehler! erweisquelle konnte nicht gefunden werden.). Moisture content measurement was not repeated in the trial phase on 28.03.2017. Possibly the high moisture content together with the high CO_2 content in the cooling medium were responsible for the formation of the layer. The layer on alite crystals in contact with pores probably consisted of very fine grained belite and free lime crystals, resulting from alite decomposition. Possibly the free lime has, at least partly, reacted with CO_2 to form calcite, which was found in all samples exhibiting the layers on alite in contact with pores in significant amounts (0.6 to 1.6 mass %). Calcite was found only in traces in all other samples. An estimate based on plausible inner surfaces of clinker (0.02 m^2/g), an alite content of 65 volume %, an intermediate thickness of the observed layer of 0.5 µm and the density of alite (3.02 g/cm^3) result in up to 2.0 mass % of decomposed alite in the samples. A further estimate, based on stoichiometric calculations and the assumption that the calcite (up to 1.6 mass %) in the clinker samples has formed from free lime, which itself formed due to alite decomposition, leads to up to 3.7 mass % of decomposed alite in the samples. Both estimates are in the same order of magnitude and correspond with the slightly lower alite contents in the samples "S7_oxy" and "S8_oxy" compared to the other samples. These two samples showed the highest calcite contents and the highest occurrence of layers on alite surfaces. The

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decomposition of alite might have been accelerated by the presence of the cooling medium rich in CO_2 and water in the clinker pores at temperatures that were high but already below the stability limit of alite (~ 1250 °C). The absence of the layer in one sample cooled with CO_2 might than be explained by lower water contents in the cooling medium. However, there are currently not enough data to support this theory. Earlier investigations on the effects of the Oxyfuel process on clinker characteristics did not show any comparable effects in laboratory clinker burned and cooled with CO_2 ([2], see Appendix A – Bibliography and references).

Table 15 Microstructural features of the clinker samples

Sample short name	**S9_air**	**S10_air**	**ØS3_air**	**S4_oxy**
Component	**HC clinker 02.02.2017 11:15**	**HC clinker 28.03.2017 16:35**	**Ø Pilot plant clinker 16./17.01. 2017 (air)**	**Pilot plant clinker 02.02.2017 11:20 (oxy)**
average crystal size alite	13 µm	11 µm	10 µm	10 µm
average crystal size belite	10 µm	10 µm	9 µm	9 µm
max. size of belite clusters	1000 µm	850 µm	730 µm	1000 µm
max. size of free lime clusters	250 µm	400 µm	230 µm	230 µm
cooling (content of domains with different ground mass crystal sizes in %: very fine / fine / intermediately fine / coarse / very coarse)	intermediately fast (0/16/50/ 24/10)	intermediately fast (0/18/62/ 18/2)	fast (10/60/ 30/0/0)	fast (3/28/ 17/2/0)
length of the precooling zone (indicators for short length: corroded alite surfaces, secondary belite forms rim; indicators for high length: recrystallised alite surfaces, secondary belite forms single crystals)	intermediately long	short to intermediately long	short	short
content of clinker grains with high burning degree in %	96 %	87 %	100 %	90 %
content of clinker grains with intermediately high burning degree in %	4 %	13 %	0 %	10 %
evidence for reducing burning conditions	no	no	no	no
layer around alite in contact with pores (probably belite and free lime / calcite)	no	no	no	no

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Table 16 Microstructural features of the clinker samples

Sample short name	**S5_oxy**	**S6_oxy**	**S7_oxy**	**S8_oxy**
Component	**Pilot plant clinker 02.02.2017 15:15 (oxy)**	**Pilot plant clinker 28.03.2017 16:35 (oxy)**	**Pilot plant clinker 28.03.2017 16:55 (oxy)**	**Pilot plant clinker 28.03.2017 17:20 (oxy)**
average crystal size alite	10 µm	11 µm	10 µm	10 µm
average crystal size belite	11 µm	9 µm	10 µm	10 µm
max. size of belite clusters	1000 µm	1450 µm	870 µm	1470 µm
max. size of free lime clusters	240 µm	240 µm	220 µm	240 µm
cooling (content of domains with different ground mass crystal sizes in %: very fine / fine / intermediately fine / coarse / very coarse)	fast (6/68/22/ 4/0)	fast (6/46/44/ 4/0)	fast (4/56/36/ 4/0)	fast (8/66/22/ 4/0)
length of the precooling zone (indicators for short length: corroded alite surfaces, secondary belite forms rim; indicators for high length: recrystallized alite surfaces, secondary belite forms single crystals)	short	short	short	short
content of clinker grains with high burning degree	92 %	85 %	87 %	86 %
content of clinker grains with intermediately high burning degree	8 %	15 %	13 %	14 %
evidence for reducing burning conditions	no	no	no	no
layer around alite in contact with pores (probably belite and free lime / calcite)	yes	yes	yes	yes

5.2.8 Summary and conclusions regarding clinker quality

The comparison of clinker samples from the plant cooler and from the pilot cooler (operated with air or CO_2) showed systematic differences only with respect to the cooling conditions. Systematic differences in the chemical composition of the samples were not found. The mineralogical analysis with XRD showed significant contents of α'-belite besides β-belite in the samples cooled in the pilot cooler, whereas the samples from the plant cooler contain mainly the β-modification of belite. The α'-modification is mostly preserved by fast cooling conditions. The clinker microstructure of the clinker samples from the pilot cooler also showed indicators for fast cooling (mostly fine ground mass crystals) and a short precooling zone (alite in contact with ground mass corroded and surrounded by rim of secondary belite). In the samples from the HC cooler indicators for slower cooling (coarser ground mass crystals) and a longer precooling zone (alite surface in contact with ground mass and secondary belite partly recrystallized). The signs for a shorter precooling zone and faster cooling in samples from the pilot cooler compared to the plant cooler could result from the experimental setup. The clinker led into the pilot cooler was systematically enriched in finer granules compared to the usual plant clinker. Finer granules can be cooled faster due to the higher surface-volume-ratio. The comparison of air-cooled clinker samples (plant and pilot cooler) with CO_2-cooled clinker samples (pilot cooler) showed that four of the five analyzed CO_2-cooled clinker samples contained relatively high amounts of CO_2, most probably bound in calcite, which was also found in unusually high contents in the same samples. Under the microscopes these samples also showed unusual layers of up to 2 µm thickness around alite crystals in contact with pores. The layer was very fine grained and the contained

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phases could not be identified. However, the observations indicate that it probably consisted of very fine grained belite and free lime crystals, resulting from alite decomposition. Possibly the free lime has, at least partly, reacted with CO_2 to form the calcite found in the samples. Different estimates suggest that the alite decomposition resulting in the formation of the layer in contact with pores led to a loss of up to about 4 mass % of alite in the investigated samples. If this affects the product properties remain unclear and should be investigated. The layer did not occur in the three samples cooled with air and in one sample cooled with CO_2. Therefore its formation cannot be correlated with the high CO_2 concentrations in the cooling medium alone. Some trial data indicate that the cooling medium had high water content. The decomposition of alite might have been caused by the presence of the cooling medium rich in CO_2 and water in the clinker pores at temperatures that were high but already below the stability limit of alite (~ 1250 °C). The absence of the layer in one sample cooled with CO_2 might than be explained by lower water contents in the cooling medium. However, there are currently not enough data to support this theory. Earlier investigations on the effects of the Oxyfuel process on clinker characteristics did not show any comparable effects in laboratory clinker burned and cooled with CO_2 [2]. Further investigations into the phenomenon are recommended. If high water contents in the cooling medium prove to be responsible for alite decomposition, and if this affects the product quality, future designs of process technology for Oxyfuel could prevent this effect by incorporating a condenser, resulting in low water contents in the cooling medium.

5.3 Experimental results - false air ingress and sealing efficiency

False air ingress was the unexpected experimental challenge that demanded most time and effort from the team to overcome. The experiment could not be performed at all without solving this problem. In the beginning the intended relatively high and stable CO_2 concentration in the cooling gas could not be achieved. False air ingress reduced considerably after HC has changed kiln hood under pressure set point, but nevertheless, it was still considerably high. The team concluded that a single false air stream pulled through the screw conveyors would be divided in two separate streams inside the pilot cooler (Figure 5.2). One of the false air streams would be mixed with CO_2-rich gas inside the pilot cooler and then recirculated. The other false air stream would also be mixed with CO_2-rich gas inside the pilot cooler but would flow through the hot clinker extraction tube into the kiln hood. VDZ could only measure the chemical composition of the recirculated gases at the measuring point where the gas probe was installed. The proportion of recirculated false air and escaping false air into the kiln hood could not be estimated. Filling the pilot plant with 100% CO_2 and measuring CO_2 concentration decay to calculate false air ingress was not possible anymore (cooling gas composition setting b - Table 3) and the team decided to abandon this procedure.

Different clinker filling degrees of the screw conveyors had been already tested during the experiment in order to assess the importance of this variable on false air ingress. High filling degrees of the screw conveyors could theoretically improve tightness. However, the tests proved that the filling degree of the screw conveyors did not play a very relevant role with respect to false air ingress. Nevertheless, the team was aware of the importance to estimate false air ingress for WP6 and decided to use an anemometer to measure false air velocity at the clinker sampling point (chute located between two consecutive screw conveyors - Figure 5.2). The measurements with the anemometer could only be performed when clinker was not being fed to the pilot plant. The diameter of the chute was known and thus the total false air volume flow could be calculated. Several tests were performed in order to evaluate the effect of the cooling fans load on false air ingress volume flow (Table 17).

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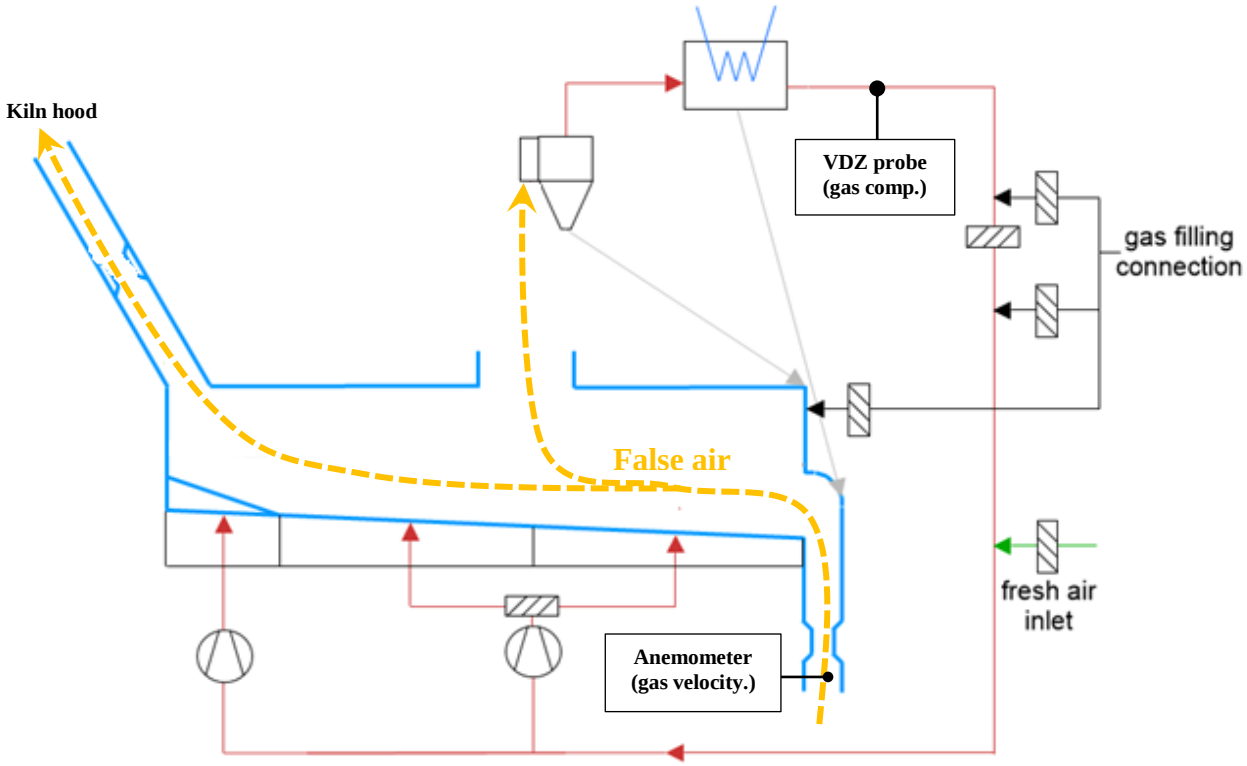

Figure 5.2: False air streams

Table 17 False air measurements with an anemometer

Fans load	air speed (m/s)	Volume flow (Am^3/h)	Volume flow (Nm^3/h)
Fans off	2.7	305	297
Fans @ partial load (33-45% rpm)	2.7	305	297
Fans @ full capacity (100% rpm)	3.3	373	363

*** Note: $T_{amb.}$ = 8 °C; Kiln hood pressure = -0.9 mbar; Chute inner diameter = 200 mm**

The measurements confirmed that the impact of the load of the cooling fans on total false air ingress was very limited. This means that kiln hood under pressure level was playing a key-role regarding false air ingress. A maximum false air volume flow of about 360 Nm^3/h was measured in the worst-case scenario (highest kiln hood under pressure level and cooling fans running at full capacity). Nevertheless, false air ingress would be expectably lower during the trials (up to about 300 Nm^3/h). The false air intake into a future oxyfuel cement plant will be of lower effect since the ratio of grate cooler surface to clinker outlet will be different and the discharge system will consist of a clinker crusher and pan conveyor rather than screw conveyors. Furthermore the pressure conditions as driving force for the false air intake can be controlled in a better way. Therefore, the false air volume flow measured in the pilot plant can only be used to estimate up-scaling effects very roughly (comparison basis). False air intake in the clinker cooler was underestimated and will need special attention in the design phase of a full scale oxyfuel cement plant. The sealings of the pressurized areas of the pilot cooler performed well. Neither the CO_2 warning system installed at ground level nor the individual mobile CO_2 detectors have detected

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any CO_2–rich gas leakage during the trials. Similar performance of the sealings is expected in a future oxyfuel cement plant. Therefore, one may presume that most of the false air ingress in an industrial scale oxyfuel clinker cooler will be pulled through the clinker crusher. Nowadays, false air in industrial clinker coolers is irrelevant for the clinker manufacturing process and is not measured at all. The lack of data gives no basis of comparison for future oxyfuel clinker coolers. In order to overcome this problem and deliver the necessary data to WP6, the team made a very rough estimate of false air ingress through an industrial scale clinker crusher based on Bernoulli equation (Table 18). False air ingress of about 1000 Nm^3/h was estimated for a 3000 t/d clinker crusher.

Table 18 False air volume flow through an industrial clinker crusher (3000 t/d)

Number of rollers	Length of rollers (m)	Gap between rollers (m)	Total gap area (m^2)	% total gap area covered with clinker	Pressure difference in clinker crusher (mbar)	False air volume flow (Nm^3/h)
4	5	0.02	0.5	90%	0.2	~ 1000

5.4 Experimental results - cooling medium moisture content

The absolute moisture content of the clinker cooling air is normally below 1 Vol.% (taking European climate as reference). Therefore, no higher moisture content was expected in the cooling gas of the pilot plant. Instead, it was expected that the cooling gas would be recirculated and, without any relevant sources of water in the recirculation circuit (e.g. injection of water), the absolute moisture content of the clinker cooling gas (air or CO_2–rich gas) would not change over time. Nevertheless, one measurement of moisture content for each cooling medium was performed. The measurements indicated a surprisingly high cooling gas moisture content well above 1 Vol.% (Table 19).

Table 19 Cooling medium moisture content

Cooling medium	CO_2 (Vol.% - dry)	Moisture (Vol.%)
Air	0%	8%
CO_2-rich gas	74%	13%

The reason for such high moisture content in the cooling medium remains unclear. The accuracy of the measurements and the indicated absolute values remain questionable. The moisture measurement was performed by controlled cooler gas extraction and measurement of the weight difference in silica gel, which absorbs the gas moisture. One hypothesis for misleading moisture measurements is referred to the presence of submicron particles in the clinker cooler gas that could not be collected by the filters installed upstream from the silica gel. However, this hypothesis does not seem to be plausible. Firstly, an unrealistic mass of those particles would have to be transported through the filters. And secondly, it would then have to effectively settle on the silica gel during the time of the measurements (only 30 minutes) in order to accumulate any significant mass.

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Finally, it seems unquestionable that the moisture content in the recirculating gas was high. The blockage of the grate plates due to the presence of fine dust particles combined with moisture in the recirculating gas has been observed during the experiment (Figure 6.2 and Figure 6.3).

Moisture concentration in the recirculated gas above 1 Vol.% could only be reached if the system would have any source of water (moisture enrichment). No water was used in the pilot cooler to cool down the clinker. For that reason, a different source of moisture is to be found. The thesis of moisture enrichment due to false air ingress and recirculation of the cooling gas seems to be hardly plausible. The same conclusion is valid for moisture enrichment with origin in the CO_2 gas bottles. False air ingress, as well as CO_2 gas injected continuously during the trials may hold only very small amounts of moisture. The purity of the CO_2 gas contained in the gas bottles is very high and water amounts only some PPM. The moisture measurements were performed during very cold winter days. Low ambient air temperatures considerably limited the maximum moisture content that air can hold. Therefore, an absolute moisture content much lower than 1 Vol.% would be expected. Furthermore, moisture would have to be somehow separated from the other recirculated gas components inside the pilot plant in order to promote its enrichment and reach the concentration levels shown in Table 19. In conclusion, this thesis is not plausible and other water source must be found.

Refractory material can hold a considerable amount of water. If not properly dried, the newly installed refractory material may have released water to the system during the operation of the pilot plant and thus increased moisture concentration of the recirculated gas during the measurements. The operation of the pilot plant was performed discontinuously and sometimes with long shutdown periods between consecutive trials. This fact combined with pilot cooler exhaust gas temperatures well below 555 °C (Table 8) may have had an impact on the drying speed of the refractory lining. The refractory material can have continued to release moisture into the system during the trials, even if the hot commissioning tests and some trials already have taken place before. Moisture contained in the refractory lining seems to be, at the present moment, the most plausible source of moisture in the recirculated gas. Despite its general plausibility, it is impossible to prove this thesis with the available experimental data.

The ECRA CCS study as well as the CEMCAP framework for an oxyfuel process already foresees the construction of a condenser to extract moisture from the recirculated gases. The origin of moisture in the case of the complete oxyfuel process is (i) the raw materials and (ii) the combustion of fuels (Figure 5.2).

Based on the blockage of the grate plates observed during the CEMCAP trials with the pilot cooler, it is evident that the moisture content in the recirculating gas was high. Any effect of moisture on clinker quality as e.g. the observed effects in the clinker samples from the pilot cooler reinforce the importance of a condenser in future industrial scale oxyfuel cement plant. Condenser layout and dimensioning will be important for preventing effects of increased moisture on clinker quality.

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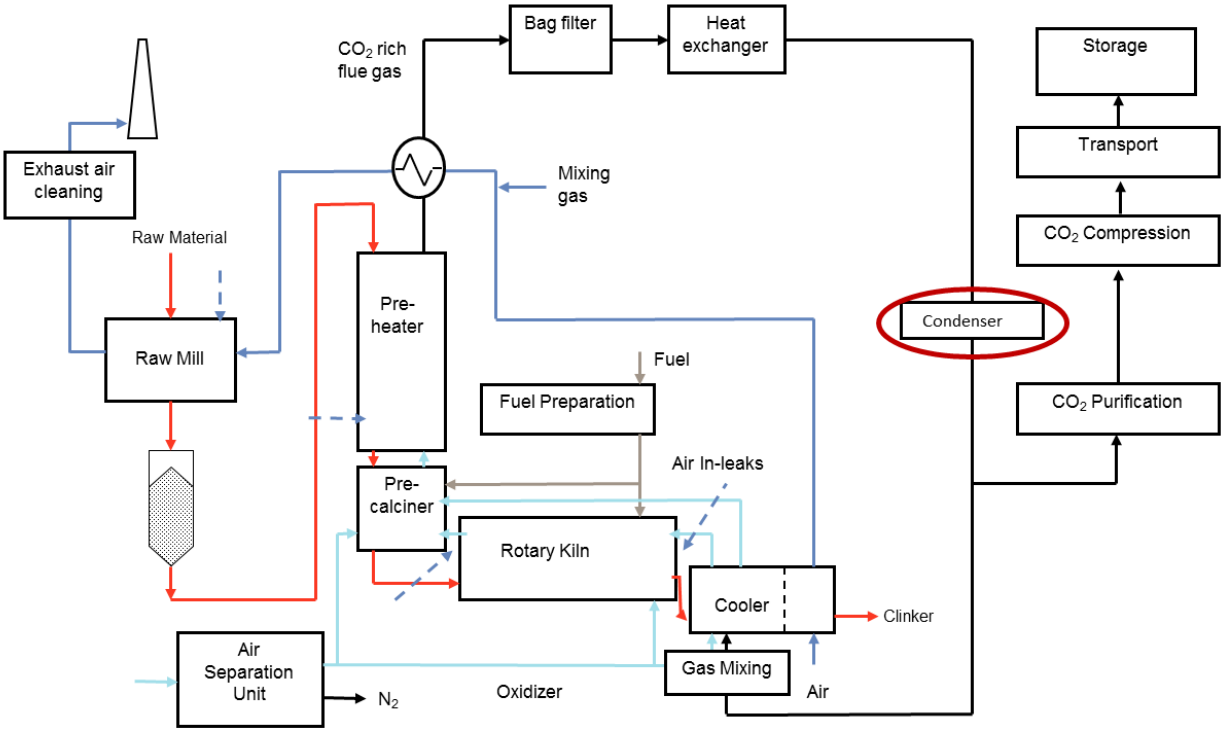

Figure 5.3: ECRA CCS Project – Oxyfuel cement plant flow sheet with condenser

5.5 Experimental results - cooling curves

The quantity and quality of the experimental data was limited by the already extensively reported experimental challenges. Therefore, the team had to expend a substantial amount of time and effort in assessing the available experimental data in order to extract as much information and knowledge as possible from it. The objective to calculate the cooling curves with air and CO_2–rich gas could in this way be partially achieved.

In order to relate all pilot plant control variables with each other, VDZ developed a mathematical model (VDZ model – see 10 Appendix C) based on an energy and mass balance of the pilot cooler (Figure 10.1). This model comprises the following information:

- Clinker properties at pilot cooler inlet and outlet (m; T and E)
- Exhaust gas properties (chemical composition; T; V; and E)
- Cooling medium properties (chemical composition; T; V and E)
- CO_2 injection properties (T;V and E)
- False air ingress properties (T;V and E)
- CO_2–rich gas escape through the hot clinker extraction tube properties (chemical composition; T;V and E)

Note: E = enthalpy; T = temperature; V = volume flow; m = mass flow

Experimental data collected during the trials were used as model inputs (temperatures, volume and mass flows). However, some process parameters relevant for the heat and mass balances were not measured. Those parameters only became relevant due to the considerably high false air ingress, which in turn was totally unexpected during the concept development phase of the pilot plant. Some of them could be very roughly estimated based on existent process data. Some

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others, like the hot clinker temperature at pilot cooler inlet, could not even be estimated without high uncertainty. The relevant missing process parameters, which were not measured during the experiment, are:

- False air volume flow through the cold clinker extraction system
- CO_2 injection volume flow
- CO_2–rich gas escape
- Hot clinker temperature at pilot cooler inlet

Maximum false air volume flow estimate was based on some measurements performed with an anemometer (Table 17). Those measurements could only take place when clinker was not being fed to the pilot cooler. Therefore, on-line measurements of false air ingress during the trials were unfeasible. As already explained in chapter 5.3, false air ingress strongly depended on kiln hood under pressure level and on cooling fans load as well. Assuming the maximum false air ingress estimate as input data for the VDZ model would be unreasonable, once both parameters varied during the trials. CO_2 injection volume flow is closely related to false air ingress. In order to keep constant a certain CO_2 concentration level in the cooling medium, the higher the false air volume flow, the higher the CO_2 injection volume flow has to be. Following this line of reasoning and some basic assumptions, false air volume flow could be roughly estimated based on CO_2 injection volume flow.

CO_2–rich gas volume flow was measured upstream and downstream from the CO_2 injection point. Since gas composition upstream from the CO_2 injection point was known (measured continuously by VDZ), it was possible to estimate the CO_2 injection volume flow and the CO_2–rich gas composition downstream from the injection point as well (cooling medium).

CO_2–rich gas volume flow escaping through the hot clinker extraction tube was not measured. However, it could be estimated based on the principle of mass conservation. Knowing the estimated values of CO_2 injection volume flow and false air ingress, one could estimate CO_2–rich gas volume flow escaped through the hot clinker extraction tube. The team assumed that the CO_2-rich gas escape volume flow would have exactly the same volume concentration as the recirculated gas before CO_2 injection takes place (measured by VDZ). Moreover, it was assumed that CO_2-rich gas escape temperature would be the same as the cooler exhaust gas temperature measured upstream from the dedusting cyclone. Assuming two false air streams inside the cooler (as explained in chapter 5.3), the real temperature would be expectably lower.

Hot clinker temperature cannot be measured. Depending on the clinker cooler technology installed in the cement plant (grate cooler, satellite cooler or rotary cooler), a hot clinker temperature range at the cooler inlet is usually estimated. In the case of a kiln with a grate cooler, a hot clinker temperature in the range of 1400 to 1450 °C is widely accepted in the industry. However, the hot clinker temperature at pilot cooler inlet was lower than 1400 °C. CO_2–rich gas volume flow escaping through the hot clinker extraction tube promoted a fast cooling of the hot clinker inside the extraction tube before reaching the pilot cooler. The analysis of the clinker samples confirmed the same phenomenon (chapter 5.2.8). Hot clinker cooling rate inside the extraction tube strongly depends on gas composition, temperature and volume flow of the escaped CO_2–rich gas. Those variables could only be very roughly estimated and the uncertainty range was not possible to determine. Therefore, hot clinker temperature at pilot cooler inlet could not be estimated. Hot clinker temperature can be related to cold clinker temperature to a certain extent after performing an energy and mass balance to the pilot cooler.

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The cold clinker sampling point was at a chute between two consecutive screw conveyors (cold clinker extraction system) located about 10 meters downstream from the pilot cooler outlet. A cold clinker temperature lower than the cooling medium temperature was observed in many trials. That would be physically impossible, unless another cooling stage would take place between the pilot cooler outlet and the clinker sampling point. False air ingress cooled the clinker inside the screw conveyors even further and misled the clinker temperature measurements. Therefore, real cold clinker temperature at pilot cooler outlet could not be determined accurately. Despite all data uncertainties and constraints the team focused on calculating the cooling curves as best estimate with the available data. The calculation of the clinker cooling curves in grate coolers is based on a mathematical formula widely found in the technical literature. The mathematical formula represents a simplified model of a cross-flow heat exchanger.

$$\frac{Tcli - Tmedium}{Tcli\ in - Tmedium} = \exp(-\frac{Vair}{Efactor})$$

with:

T cli = clinker temperature at cooler outlet (°C)
T medium = cooling medium temperature (°C)
T cli in = clinker temperature at cooler inlet (°C)
V air = specific cooling medium volume flow (Nm^3/ kg clinker)
E factor = dimensional factor that depends on the cooling medium and cooler design.

The *E factor* is unknown, since it depends on the cooling medium and cooler design. Therefore, all the other variables of the equation have to be known in order to calculate the *E factor*, which is considered streamlined constant for the same cooling medium and clinker cooler.

As explained before, hot clinker temperature and cold clinker temperature at pilot cooler outlet could not be determined exactly due to lack of data. In order to overcome this issue and to be able to estimate the cooling curves, a cold clinker temperature 5 °C higher than the cooling medium temperature was assumed. This is in fact unrealistic but it was useful to determine the boundary of the best case cooling scenario. Assuming a cold clinker temperature, it was possible to calculate the hot clinker temperature at the pilot cooler inlet by closing the energy and mass balance. All variables became known and the cooling curve for the best-case cooling scenario could be calculated. During the trials the team noted that the clinker from the pilot cooler was finer than the clinker from HC plant (Figure 5.4). Fine clinker cools down faster than coarser clinker for the same cooling conditions, since the specific surface of the first is higher, which promotes heat transfer.

For the same cooler, an *E factor* is expectably lower for CO_2–rich gas as cooling medium than for air, due to higher specific heat capacity (assuming the same cooling medium temperature). The faster the cooling, the lower the *E factor* is expected to be. Following this line of reasoning, the *E factor* of the pilot cooler is expectably lower than the *E factor* from an industrial cooler. Taking the data presented in the CEMCAP framework as reference, an industrial cooler has an *E factor* of 0.69 (with air as cooling medium). Therefore, in the worst case cooling scenario the pilot cooler would have the same *E factor*, since an *E factor* lower than 0.69 would be expected. With that the cooling curve for the worst-case cooling scenario in the pilot cooler could be calculated and the upper boundary cooling curve determined (Figure 5.5).

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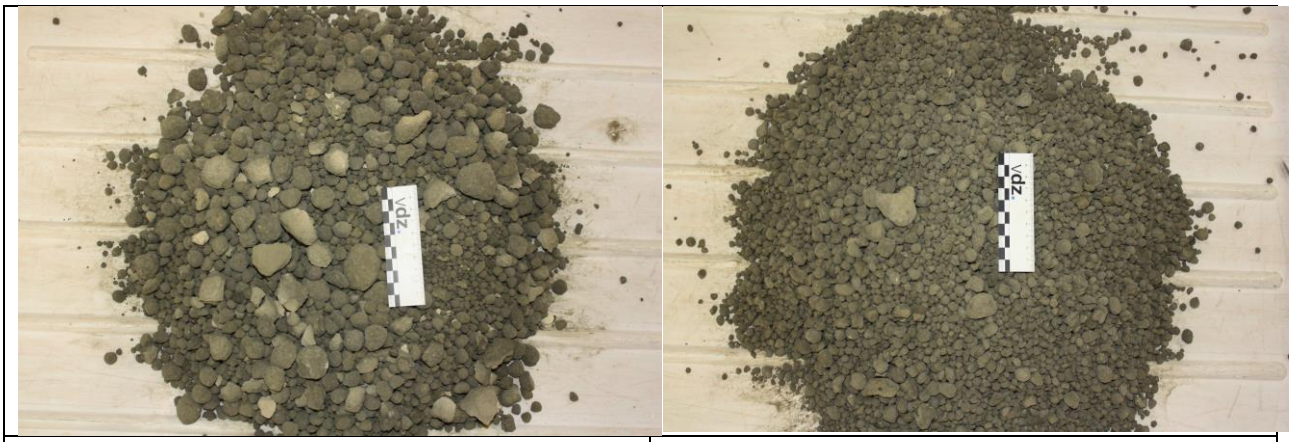

HC plant clinker	Pilot cooler clinker

Figure 5.4: Clinker particle size comparison

The calculation of the boundary cooling curves was based on scarce and very imprecise data. The uncertainty region located between the upper and lower boundaries of the cooling curves is significant for both cooling mediums. The boundary cooling curves presented in Figure 5.5 do not take into account most of the uncertainties with origin in the many assumptions and simplifications undertaken in the VDZ model (energy and mass balance of the pilot cooler), which are very hard to quantify. Therefore, only very cautious conclusions can be drawn. Nevertheless, WP6 simulated clinker cooling curves are located in the estimated uncertainty region (Figure 5.5). Due to lack of better information, WP6 simulated clinker cooling curves shall be used to go on with the simulations of an oxyfuel cement plant. Finally, the objective to determine the clinker cooling curves with CO_2–rich gas was only partially achieved.

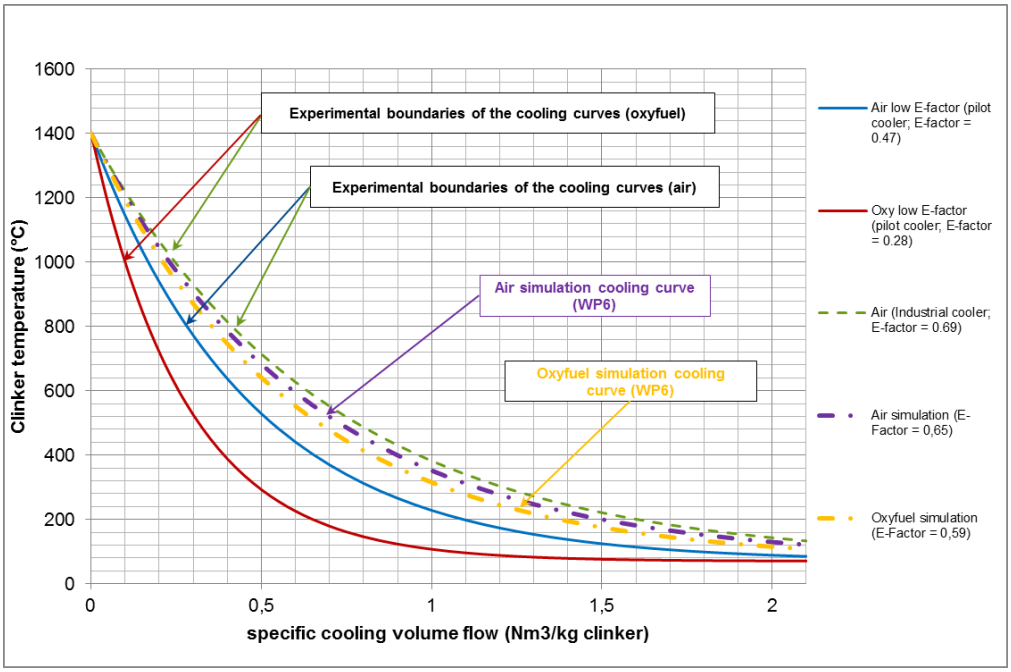

Figure 5.5: Experimental boundaries of the cooling curves with air and oxyfuel cooling medium

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6 LESSONS LEARNED AND UPSCALING

The experiment, the several operational challenges led to a comprehensive list of lessons learned that will help to give advice for future design of an industrial scale oxyfuel clinker cooler and introduces particular points that have to be taken into account for up-scaling the oxyfuel pilot cooler design. The following chapter lists and discusses the challenges, which were experienced during the oxyfuel pilot cooler operation and important lessons learned are pointed out. Required improvements are addressed including potential changes in design for a future oxyfuel (pilot) cooler. A distinction is drawn between the oxyfuel pilot cooler itself and the oxyfuel pilot cooler system (incl. peripheral equipment). Furthermore, design improvements as well as lessons learned are classified as either relevant in pilot scale only or relevant for industrial scale, too (Table 20).

Table 20 Design improvements and lessons learned short list

Section	Design improvement and lessons learned	Pilot oxyfuel cooler	Industrial oxyfuel cooler
6.1	Holes shall be open in the hot clinker extraction tube for removing blockages easily.	x	
6.1	Design the hot clinker extraction tube's tip for easy removal.	x	
6.2	Choose the dimensioning and type of equipment very carefully (e.g. heat exchanger and/or dedusting system). The design and dimensioning of the equipment shall be based on the expected operating points but shall have enough margin to handle unexpected operation conditions.	x	x
6.2	Dust in combination with moisture from the recirculated cooling gases can block the ventilation slots of the cooler grate plates. The installation of a condenser seems to be necessary. Moreover, the design and operation of the dedusting system for the recirculated gases may play a relevant role in the availability of the cooler.	x	x
6.3	For oxyfuel operation it is recommended to reduce static pressure differences to the surroundings as far as possible in order to reduce the driving force for false air intake.	x	x
6.4	For a higher flexibility and for better fan operation in regard to controllability and engine smoothness it is recommend to introduce an additional fan in series to the intrinsic cooling fans.	x	
6.5	Boundary zones such as the cold clinker discharge system demand special attention regarding minimisation of false air ingress.	x	x
6.6	It is recommended to integrate flow meter for the injected gas in future oxyfuel pilot systems that demand the continuous injection of CO_2.	x	
6.7	Clinker weighing is recommended to determine the actual clinker production rate and check the accuracy of the installed mass flow sensors.	x	
6.8 / 6.9	Cooler downscaling is very challenging and several design parameters shall be taken into account during design.	x	

6.1 Hot clinker extraction tube

One of the main challenges not only during the experimental operation but also during engineering was the hot clinker extraction tube. To operate a pilot-scale oxyfuel clinker cooler in parallel to an existing industrial scale clinker cooler of an existing kiln line it was essential to route part of the hot clinker falling from the kiln to the pilot cooler system. As the clinker from the kiln has a temperature up to 1450 °C and because the pilot cooler system is located more than 10 m straight-line distance from the kiln outlet this was considered to be a challenge from

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the beginning on. Several designs of a hot clinker extraction device were considered. Finally a design was chosen, which was finally chosen and which is described in detail in section 2.3.

Immersion of a refractory lined steel tube into a kiln hood and, more specifically into a curtain of falling hot clinker holds many pitfalls. It already happened during oxyfuel pilot cooler system equipment testing that the hot clinker extraction tube was hit by a large lump coming out of the kiln, which completely destroyed the extraction tube's refractory tip (Figure 6.1). Hot clinker was not able to enter the extraction tube anymore, which required for repair works during kiln stoppage. The tip of the hot clinker extraction tube could be repaired while the risk of a further fail led to a modified tip design, which allows for rapid replacement.

Due to the high mechanical impact of big lumps falling from the kiln in combination with the high thermal stress the extraction tube slightly bent over time, which led to poor operating smoothness and sporadic failures over time making it very hard for the team to establish a steady-state oxyfuel pilot cooler operation. These difficult conditions and the permanent risk of destruction led to the conclusion of not running in 24/7 operation, already at an early stage. This decision was supported by the fact that the oxyfuel pilot cooler system could reach a steady-state just approx. 2 hours after start-up. Despite of cautious experimental operation the damage of the hot clinker extraction tube tip occurred 3 times. The last time led the team to end the experiment.

It is important to underline that the hot clinker extraction device was only needed to provide the oxyfuel pilot cooler system with hot clinker. As the oxyfuel clinker cooler will become an integral part of the overall kiln line in industrial scale, described challenges are relevant for the oxyfuel pilot cooler operation only. Nevertheless, in case of redesign it is recommended to also consider opening holes over the complete length allowing for easy removal of blockages. Furthermore, a design should be chosen which allows for easy replacement of the tube's tip in case of damage, at best during kiln operation. A design permanently resisting the high mechanical and thermal impact of the hot clinker is considered unfeasible.

Figure 6.1: Hot clinker extraction tube during repair works after 2nd damage

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6.2 Partial-load operation

Although the hot clinker extraction tube worked fine in some time it was never possible to reach clinker flows greater than 46 t/d. As the overall oxyfuel pilot cooler system was designed and built for a nominal clinker capacity of 80 t/d this partial-load operation has led to a whole range of side-effects. With lower clinker capacity, the overall recirculation gas flow was reduced, too. The reduced gas flow has led to a decreased separation efficiency of the de-dusting cyclone allowing the dust of the cooler vent gas passing the de-dusting cyclone and recirculate with the main gas flow. In combination with the high moisture content of the recirculated gas in the pilot plant dust clogging appeared at different locations. The most visible impact was found on the cooler grate plates, where dust clogging slowly narrowed the ventilation slots. After several weeks the grate plates from the static inlet were found completely blocked (Figure 6.2) not allowing aeration of the static inlet section anymore and also the linear units of the grate suspension were clogged by dust (Figure 6.3) leading to grate drive failures from time to time. Sporadically arising raw meal flashes from the kiln line introduced very fine raw meal into the oxyfuel pilot cooler system, which could not be removed from the recirculation gas stream sufficiently. Furthermore, the gas-to-air heat exchanger was negatively affected by the partial-load operation as controllability of the heat exchanger gas outlet temperature was worsened by the low gas flow amounts. Consequently, keeping the cooling gas temperature (heat exchanger outlet temperature) at a constant level was challenging. During winter times these low gas flows in combination with low ambient air temperatures made the two axial fans of the heat exchanger almost unnecessary. Cooling in the heat exchanger was already efficient without operation of the fans. The separation efficiency of a de-dusting cyclone is very dependent on the overall gas volume flow. The efficiency of a bag filter or electrostatic precipitator is less dependent on the same, while having a much higher de-dusting efficiency at the same time. The biggest drawback is the much higher false air intake in those systems and especially for the bag filter the need for a purging gas. For the industrial scale oxyfuel plant it is therefore recommended to:

(i) choose the dimensioning and type of equipment very carefully

(ii) base those choices on the expected operating points, but with enough margin to handle unexpected operation conditions.

Figure 6.2: Raw meal and clinker dust clogging the lamellas of the cooler grate plates

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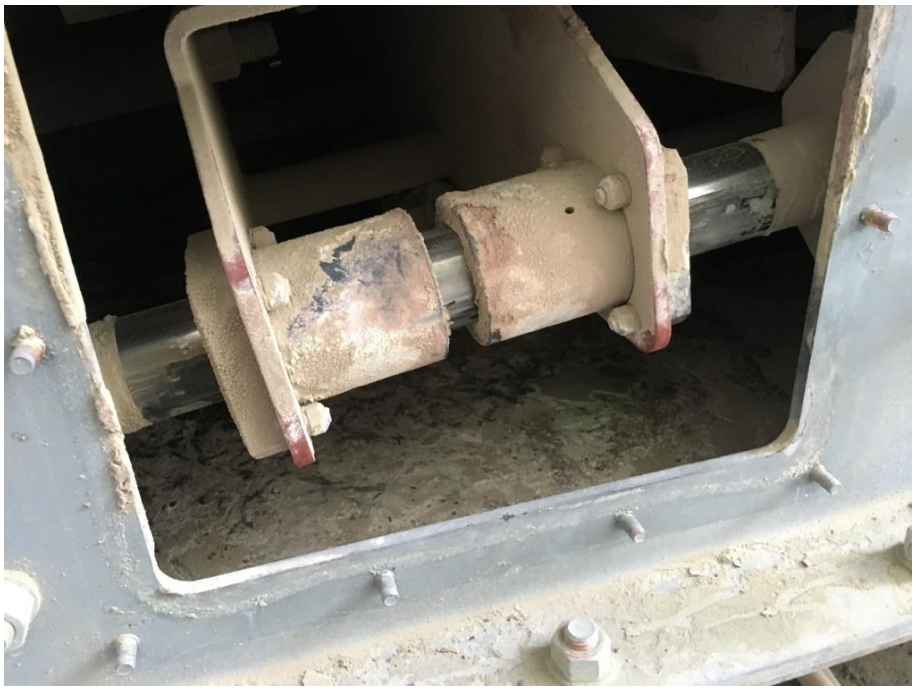

Figure 6.3: Raw meal and clinker dust clogging the linear unit of the grate suspension

6.3 Kiln hood pressure

In most cement plants the kiln hood pressure is controlled by the cooler vent gas fan to a set-point ranging between 0.1-0.3 mbar under ambient pressure. Within the HeidelbergCement plant in Hannover this value is set to 0.9 mbar under pressure for higher process reliability. It could be observed that the amount of false air intake into the oxyfuel pilot system is strongly dependent on the plant's kiln hood pressure. By increasing the kiln hood pressure during time of pilot plant operation to a set-point of 0.2 mbar under pressure the actual static pressure difference between kiln hood and pilot cooler could be reduced, while the false air amount decreased significantly. For later oxyfuel operation it is recommended to reduce static pressure differences to the surroundings as far as possible in order to reduce the driving force for false air intake.

6.4 Recirculation/cooling fans

A recirculation design was chosen for the oxyfuel pilot cooler system which allows for easy reuse of the cooling gases. Thereby the number of CO_2-bottles needed for such trials can be kept low. For this type of layout, a de-dusting cyclone as well as a gas-to-air heat exchanger for reprocessing of the cooler vent air gas becomes necessary (removal of dust and excess heat) to be used again as cooling gas for the clinker cooling process in the oxyfuel pilot cooler. Two fans in parallel were used to recirculate the cooling gas and by routing it back to the static inlet section (fan #1) and under grate compartments (fan #2) of the pilot cooler (Figure 2.1). These two fans were operated in mixed operation. This means on the one hand these two fans had to overcome the pressure drop from the de-dusting cyclone, the gas-to-air heat exchanger as well as the ducting on the suction-side. On the other hand, they had to provide required overpressure to supply the static and moving grates with sufficient gas flow at pressure-side. The mixed operation made it hard to control the static pressure at certain locations in the pilot system. Due to high difference in static pressure between kiln hood and oxyfuel pilot cooler, and therefore high gas losses via the hot clinker extraction tube, it would have been desirable to shift the static

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pressure in the oxyfuel pilot cooler. By adjusting the fans, the static pressure level could only be shifted in the whole pilot system at once. For a higher flexibility and for better fan operation in regard to controllability and engine smoothness it is recommend to introduce an additional fan in series to the intrinsic cooling fans. Acting as a booster fan with inlet operation, the pressure at pressure port of this fan would be close to ambient pressure allowing the cooling fans to work in discharge operation only. A 3rd fan was eliminated from the concept during detail engineering due to space constrains and underestimation of the possible negative influence. The pressure above the grate would have been better controllable and false air intake would have been much lower with the installation of a 3rd fan. For future oxyfuel cement plants, as well as for current cement plants with a grate cooler installed, an additional fan in series to cooler cooling fans is already in place (cooler vent air filter ID fan).

6.5 False air ingress

One of the biggest challenges during basic and detail engineering was to design a gas-tight oxyfuel pilot cooler system that certainly avoids any CO_2-leakage. This was achieved very well as the tightness-tests before commissioning phase of the pilot plant have shown. However, the sealing efficiency of the pilot system's boundaries, i.e. the material inlet and outlet, was not tested. The material inlet was implemented via the so-called hot clinker extraction tube, which allowed for the extraction of a small percentage of hot clinker falling from the kiln into the oxyfuel pilot cooler. Length of the extraction tube, permanent material flow through the tube as well as similar static pressure in the plant's kiln hood and in the oxyfuel pilot cooler led to the assumption during engineering that gas exchange between both systems would be very small and negligible. Moreover, in a closed recirculation oxyfuel pilot cooler system, gas can only leave the system when the same amount of gas enters the pilot system at another location at the same time (based on the principle of mass conservation and assuming a constant pressure level on the pilot system). In practice this gas entry position could quickly be identified as being the cold clinker discharge screw conveyors. These screw conveyors route the cooled-down clinker from the pilot cooler grate's rear end back to the plant's main clinker pan conveyor (Figure 6.4). It was assumed that due to the high material filling level of the screw conveyors the material outlet would work as a self-tightening system. The filling level of the screw conveyors was even additionally raised during trial operation to improve sealing against air ingress. But during the experiments it was found out that the natural sealing effect of the material inside the screw conveyors was greatly overestimated. Experiments to investigate the sealing efficiency of the cold clinker discharge screw conveyors showed that even with filling degrees close to 100% a false air intake could not be avoided and made up a substantial part in the overall gas balance. It is therefore recommended to not only rely on the sealing effect of the clinker material itself but to also consider additional measures like e.g. double pendulum flaps or sealing gas ports.

The hot clinker extraction tube, as well as the cold clinker discharge screw conveyors, where only necessary to attach the oxyfuel pilot cooler system to the plant's kiln line. In an industrial oxyfuel environment the oxyfuel clinker cooler will be highly integrated into the overall kiln line. A hot clinker extraction tube will no longer be necessary as hot clinker falls directly from the kiln into the clinker cooler. The cold clinker discharge system comprising of several screw conveyors will be replaced by a clinker pan conveyor following the clinker crusher system at the rear part of the movable grate. Although the clinker discharge system in an oxyfuel cement plant will look different the clinker extraction from the system will remain a critical point for false air intake as at this part the pressure in the clinker cooler lies below ambient pressure sucking false

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air from ambient via the crusher's openings. Sealing the oxyfuel kiln line system at this location remains a challenge and should be further investigated.

Figure 6.4: Cold clinker discharge onto the HC clinker pan conveyor

6.6 Filling the oxyfuel clinker cooler pilot plant with CO_2

For the quick and easy gas filling of the oxyfuel pilot cooler system three different positions were chosen (see also section 3.3.4 and Figure 2.2). Before starting the oxyfuel experiments the shut-off gate between the gas-to-air heat exchanger and the recirculation/cooling fans was closed. With the first gas injection location just after the shut-off gate next to the fans it was originally planned to fill the ducting, fans and cooler compartments with CO_2. Additionally, it was originally planned to inject CO_2 on the other side of the gate just between the shut-off gate and the gas-to-air heat exchanger. It was expected that by this measure the heat exchanger would slowly be filled with CO_2 from the bottom to the top, followed by the de-dusting cyclone right

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behind. After a while the third injection port at the rear part of the oxyfuel pilot cooler should be used to push the remaining air inside oxyfuel pilot cooler. Initial gas would be vented through the hot clinker extraction tube into the plant's kiln hood.

It turned out that the difference in gas density between air and CO_2 and hence the expected separation between both gases was not relevant. It was not possible to flood the oxyfuel pilot cooler system slowly with CO_2 as originally planned. In fact both gases mixed-up and even the occurrence of the so-called air-pockets could be observed. It was therefore decided to inject CO_2 with the shut-off gate in open position while recirculation fans already running. This procedure requires for a lot of CO_2 from the CO_2-bottles to reach high CO_2 Vol.% of the gas in the oxyfuel pilot cooler system with CO_2. Nevertheless it was the only effective way for filling the oxyfuel pilot cooler system. Filling the oxyfuel pilot cooler system with air on the contrary was quite easy as it only required opening of a set of inspection ports. It then took only a few minutes to completely change the cooling gas in the system back to ambient air. During CO_2-filling it sometimes happened that a pressure reducer froze up and locked due to the cooling effect of gas relaxation, as shown in Figure 6.5. This could be counteracted by usage of electric heating blanket. CO_2 injection volume flow became an important variable for the performance assessment of the pilot cooler. Therefore, it is recommended to integrate an injection gas flow meter in future oxyfuel pilot systems that demand the continuous injection of CO_2.

There are no further recommendations to be given for a future oxyfuel pilot plant. In industrial scale filling/draining of the CO_2-rich gas will not be necessary as prevalent gas composition will be integral part of the oxyfuel process and recirculated gas will continuously enrich with CO_2 from the process.

Figure 6.5: Frozen up pressure reducer of one of the CO_2-bundles

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6.7 Determination of clinker mass flow

For measurement of the clinker mass flow through the oxyfuel pilot cooler a contactless solid flow sensor was chosen, as shown in Figure 6.6. This sensor is developed for dusts, powders and granules in the experiment it showed high measurement uncertainty of up to 50% divergence of the measurement value. This measurement inaccuracy complicates establishing of a consistent mass and energy balance of the oxyfuel pilot cooler system.

In order to enhance the accuracy it was then decided to determine the clinker mass flow by extracting clinker from the pilot plant and weighing. Therefore, cold clinker was extracted from the withdrawal opening at a chute between two screw conveyors (Figure 6.6). The clinker material was then collected in a big-bag while noting down the time of filling. Total weight divided by the filling time results in the clinker mass flow. As this procedure is very time-consuming and laborious it was not performed for all experimental settings. During the experiments it could be observed that even with the same settings for the hot clinker extraction tube the clinker mass flow could not be replicated on a daily basis. Kiln production, fuel mix and overall kiln line stability changes the clinker dropping point and therefore has an impact on the amount of hot clinker extracted from the kiln hood. Repeated measurements by clinker weighing were required, which is the same approach followed in industrial scale to determine the actual clinker production rate and raw meal and/or kiln feed to clinker factor.

Figure 6.6: Contactless solid flow sensor and cold clinker extraction for further weighing

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6.8 Local constraints

The overall design of the oxyfuel pilot cooler system was strongly affected by the local structural conditions. In the first place a solution to provide the pilot cooler with hot clinker by overcoming several meters in height and length had to be designed. On the rear part of the pilot cooler a discharge system had to be established to return the cooled clinker to the plant's clinker conveying system and storage site. The former was realized by the hot clinker extraction tube while the latter was realized by several screw conveyors routing the cold clinker back to the main clinker pan conveyor. The location and orientation of individual equipment had to be decided under several aspects. Due to proximity to the cooler building and kiln pier walls, orientation of the heat exchanger was already pre-defined. Also, cable ducts and the plants sewage system had an impact on the oxyfuel pilot cooler systems footprint. Therefore, the oxyfuel pilot cooler itself had to be positioned in parallel to the cooler buildings front wall and not in center line to the hot clinker extraction tube. Thereby the hot clinker did not fall centrally onto the static inlet section of the pilot cooler as planned, as shown in Figure 6.7. In addition, the extensive ducting between the several pilot system components made it impossible to meet all flow measurement requirements regarding the length of the run-in and run-out distances in front of and behind the measuring point. This complicated the measurements and increased the flow measurement uncertainty.

Figure 6.7: Hot clinker partially missing the static inlet section of the oxyfuel pilot cooler

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6.9 Limits of downscaling for the pilot system

The extensive ducting between the several components already mentioned in section 6.8 is one of many consequences of the significant down-scaling. With the oxyfuel pilot cooler being the smallest clinker cooler ever build (only ~2 m² aerated surface) several effects of down-scaling became evident:

(i) Grate width of the pilot cooler is 0.6 m only. With such a narrow grate the influence of side wall friction in combination with the operating principle of a moving grate became strongly visible in the form of a wavy clinker bed surface inside of the oxyfuel pilot cooler (Figure 6.8).

(ii) The surface to volume ratio of such small equipment is different to the industrial scale leading e.g. to different specific surface heat losses.

(iii) The small and compact design requires for a lot of elbows in ducting leading to turbulent, hard to determine and non-optimal gas flows.

Downscaling of the overall clinker grate cooler technology incl. grate suspension, grate drive system, refractory mounting, etc. to the pilot scale was a big challenge during engineering but was achieved very successfully. In an industrial scale oxyfuel clinker cooler mentioned limits will be overcome again and proven technology (e.g. pendulum grate suspension, hydraulic grate drive system, etc.) can be used.

Figure 6.8: View onto the wavy clinker bed in the oxyfuel pilot cooler

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7 CONCLUSION AND OUTLOOK

VDZ, IKN and HC were able to design, construct and operate the world's first oxyfuel clinker cooler prototype ever tested in an industrial environment. Several samples of clinker cooled down in an oxyfuel environment were taken during the trials for further analysis in VDZ's laboratories. Facing the experimental challenges and overcoming difficulties the team gained valuable know-how during the operation of the pilot oxyfuel clinker cooler. An important outcome of this experiment was a comprehensive list of lessons learned during the trials, which will help to give guidance in future design of an industrial scale oxyfuel clinker cooler. Several unexpected experimental challenges limited the experiment to a certain extent and the envisaged experimental plan had to be adapted to the new circumstances. Despite all experimental challenges reported, the 3 measuring campaigns performed by the team during the operation of the oxyfuel clinker cooler prototype were considered a success. Three of the four main objectives of the experiment have been totally achieved and one partially achieved (Table 21).

Table 21 List of objectives

Objective's description	Status	Section
Determine the influence of cooling gas composition on clinker chemistry	Achieved	5.2
Determine the gas leaking rate to ambient	Achieved	5.3
Determine false air in-leakage	Achieved	5.3
Determine the clinker cooling curves and efficiency with CO_2 rich gas	Partially achieved	5.5

CO_2 detection systems (static and mobile) did not detect any leakages of CO_2–rich gas during the experiment. This proves that the sealings installed in the pressurized zones of the pilot plant (from cooling fans to clinker cooler undergrate area) performed well. However, the use of similar sealing technology in industrial scale shall be regarded with some caution, as the operation conditions are more severe in industrial scale. Moreover, sealing's performance over time was not assessed during the limited time of the experiment.

The operation of the oxyfuel clinker cooler prototype revealed that boundary zones such as the cold clinker discharge system demand special attention regarding minimisation of false air ingress also in industrial scale projects.

The absolute moisture content of the recirculated cooling gas measured during the experiment was much higher than expected (up to 13 Vol.%). The reason for such high values remains unclear. Several hypotheses have been presented and discussed in the report. The most plausible explanation is that moisture had its origin in the refractory lining of the test facility. Despite its general plausibility, it is impossible to prove this explanation with the available experimental data. More important than the values themselves, whose accuracy is somewhat questionable, it is unquestionable that the moisture content in the recirculating gas was relatively high. The blockage of the grate plates due to the presence of fine dust particles combined with moisture in the recirculating gas has been observed during the experiment. More precise measurements regarding the moisture content in the recirculating gas are recommended in future experiments.

The calculation of the cooling curves with oxyfuel operation was based on scarce and very imprecise data. The uncertainty range located between the upper and lower boundaries of the cooling curves is significant for both cooling mediums, which narrows the validity of the conclusions. Further investigations are recommended.

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The assessment of the impact of CO_2–rich gas on clinker quality was performed by VDZ in its laboratories. A total of 10 clinker samples (clinker samples from HC plant main cooler as well as from the oxyfuel pilot cooler) were selected for further analysis and comparison. The comparison of clinker samples from the HC plant cooler and from the oxyfuel pilot cooler (operated with air or CO_2) only showed systematic differences only with respect to the cooling conditions. The clinker microstructure of the clinker samples from the pilot cooler indicates very fast cooling and a short precooling zone. On the other hand, in the samples from the HC plant cooler indicators for slower cooling and a longer precooling zone have been observed. The signs for a shorter precooling zone and faster cooling in samples from the oxyfuel pilot cooler compared to the HC plant cooler likely resulted from the experimental setup. A fast cooling of clinker started already inside the hot clinker extraction tube. No systematic differences in the chemical composition between HC and pilot cooler samples when operated in air mode were found.

The comparison of air-cooled clinker samples (HC plant and oxyfuel pilot cooler) with CO_2-rich gas cooled clinker samples (oxyfuel pilot cooler) showed that four of the five analyzed CO_2-rich gas cooled clinker samples contained relatively high amounts of CO_2, most probably bound in calcite, which was also found in unusually high, but nevertheless limited amounts, in the oxyfuel mode samples. Under the microscope these samples also showed unusual layers of up to 2 µm thickness around alite crystals in contact with pores. The layer was very fine grained and the contained phases could not be identified. However, the observations indicate that it probably consisted of very fine grained belite and free lime crystals, resulting from alite decomposition. Different estimates suggest that the alite decomposition resulting in the formation of the layer in contact with pores led to a loss of up to about 4 mass % of alite in the investigated samples. Such layers did not occur in the three samples cooled with air, as well as in one sample cooled with CO_2 in the oxyfuel pilot cooler. Therefore its formation cannot be correlated with the high CO_2 concentrations in the cooling medium alone. The decomposition of alite might have been caused by the presence of the cooling medium rich in CO_2 and water in the clinker pores at temperatures that were high but already below the stability limit of alite (ca. 1250 °C). However, there are currently not enough data to confirm this hypothesis. Earlier investigations on the effects of the Oxyfuel process on clinker characteristics did not show any comparable effects in laboratory clinker burned and cooled with CO_2 [2]. Further investigations are recommended. If high water contents in the cooling medium prove to be responsible for alite decomposition, and if this affects the cement quality, future designs of oxyfuel process technology for cement plants shall incorporate a condenser, resulting in low water contents in the cooling medium. In that hypothetic scenario, not only the design and dimensioning of the condenser (minimum moisture level of the recirculated gases after condensation), but also its control and operation would play a key-role in the performance of an oxyfuel cement plant with regard to oxyfuel clinker product quality.

Two new research questions arose from CEMCAP WP9 experiment:

(i) how the layer detected in some clinker samples may affect cement properties

(ii) and how alite decomposition and layer formation are related to the presence of water in conjunction with CO_2 in the cooling medium.

In order to find an answer to those two critical and new research questions, VDZ designed two more lab experiments. Both were not in the original scope of CEMCAP WP9. Despite this, and knowing in advance the importance of answering both questions for the development of future oxyfuel projects, the new experiments will be conducted and possible conclusions will be amended in 2018.

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Some improvements can be proposed for the design and operation of future oxyfuel clinker cooler prototypes. Moisture and dust from the recirculated gas in combination increases the risk of narrowing the ventilation slots and blocking the grate plates, which compromises the availability of the pilot plant. Therefore, the installation of a condenser to control the moisture content of the recirculated cooling gas seems to be necessary. A cyclone is an effective dedusting system but it is not reliable for operation settings much different from design capacity. The installation of a different type of dedusting system shall be evaluated as well. False air ingress through the cold clinker discharge system shall be minimized and a tighter control of the CO_2 injection volume flow shall be considered for the design. CO_2–rich gas leakage through the pilot cooler feeding point shall be minimized as well. The need to extract cold clinker from the pilot plant and weighing during the trials to determine the actual clinker production rate shall be taken into account in the design of future oxyfuel clinker cooler prototypes. Clinker samples shall be taken as close as possible from the pilot cooler outlet. This procedure will improve the measurement accuracy of the cold clinker temperature.

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8 APPENDIX A – BIBLIOGRAPHY AND REFERENCES

[1] ECRA CCS study project phase III (https://ecra-online.org/research/ccs/)
[2] Koring, Kristina: CO_2-Emissionsminderungspotential und technologische Auswirkungen der Oxyfuel-Technologie im Klinkerbrennprozess. Schriftenreihe der Zementindustrie, Heft 79 (2013)

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9 APPENDIX B – PHOTOS FROM CLINKER QUALITY ASSESSMENT

Table 9.1: Identification-key of the clinker samples

Setting	Original name shown in the sample	Description	Short name used in the report
1	Klinker Pilotanlage 16.1.17 14.40	Pilot plant clinker 16.1.17 14.40 (air)	S1_air
2	Klinker Pilotanlage 17.1.17 15.50	Pilot plant clinker 17.1.17 15.50 (air)	S2_air
1-2	Ø Klinker Pilotanlage 16./17.01.2017 Luft	Ø Pilot plant clinker 16./17.01.2017 (air)	ØS3_air
3	Oxyfuel-Klinker 02.02.2017 11:20 >80% CO_2	Pilot plant clinker 02.02.2017 11:20 (oxy)	S4_oxy
4	Oxyfuel-Klinker 02.02.2017 15:15	Pilot plant clinker 02.02.2017 15:15 (oxy)	S5_oxy
5	Pilot-Klinker 28.03.2017 16:35	Pilot plant clinker 28.03.2017 16:35 (oxy)	S6_oxy
6	Pilot-Klinker 28.03.2017 16:55	Pilot plant clinker 28.03.2017 16:55 (oxy)	S7_oxy
7	Pilot-Klinker 28.03.2017 17:20	Pilot plant clinker 28.03.2017 17:20 (oxy)	S8_oxy
3-4	Werksklinker 02.02.2017 11:15	HC clinker 02.02.2017 11:15	S9_air
5-7	Werksklinker 28.03.2017 16:35	HC clinker 28.03.2017 16:35	S10_air

Figure 9.1: Sample "S9_air"

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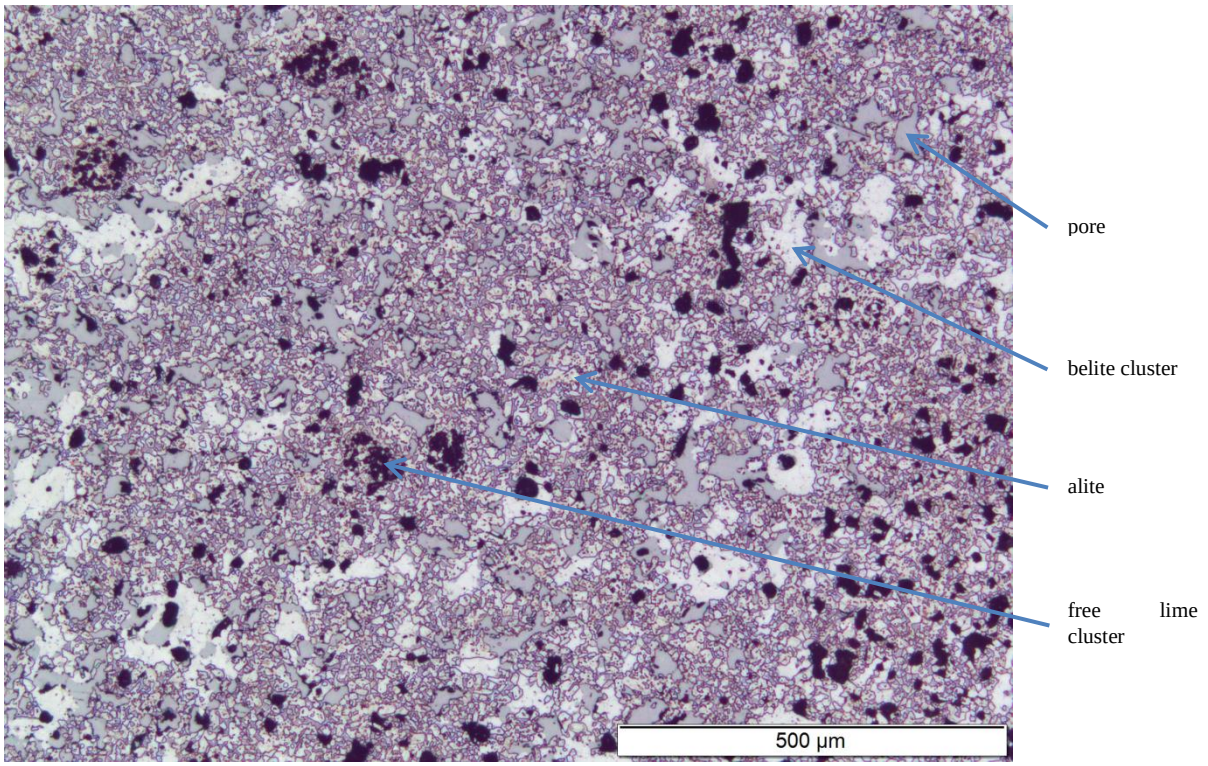

Figure 9.2: Polished section of sample "S9_air"; domain with high burning degree

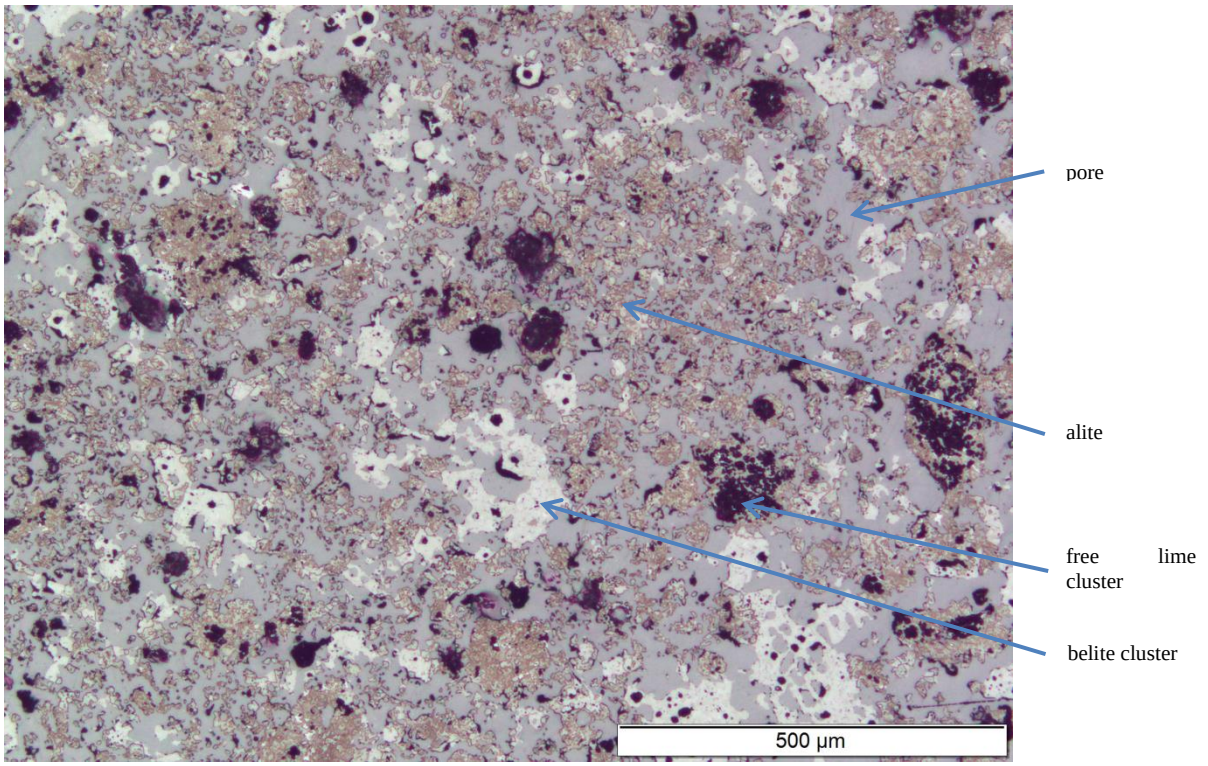

Figure 9.3: Polished section of sample "S9_air"; domain with intermediately high burning degree

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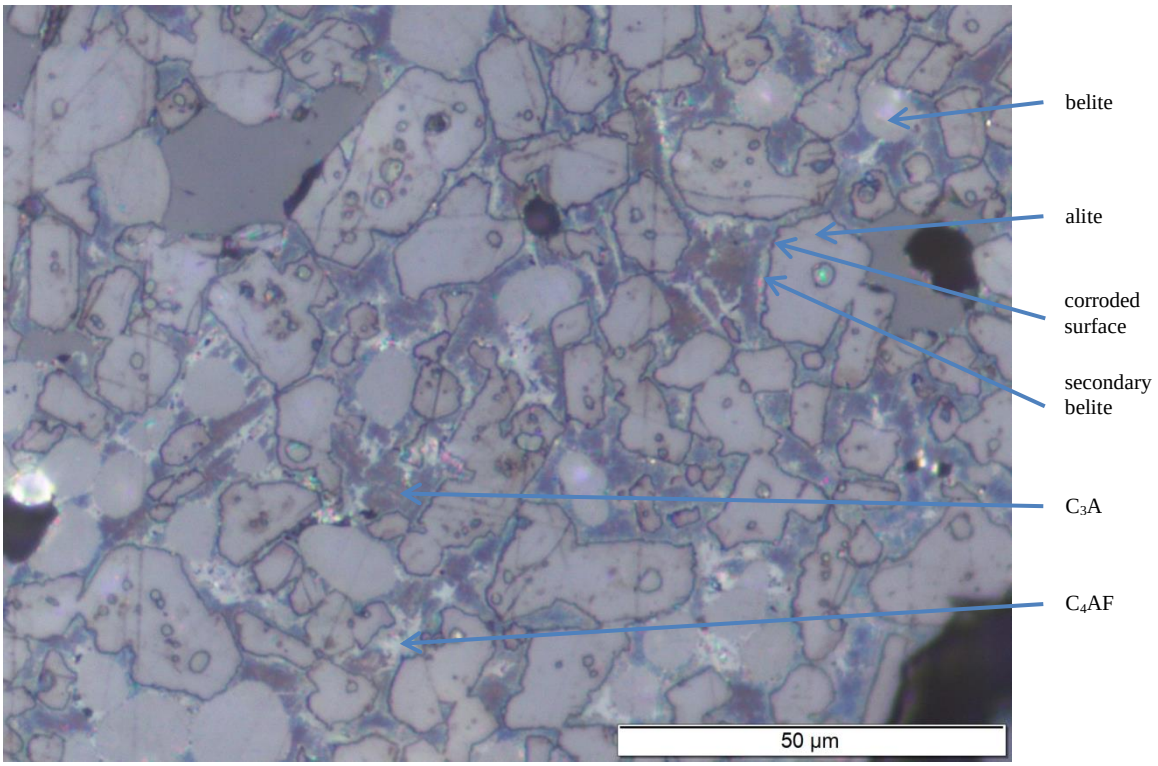

Figure 9.4: Polished section of sample "S9_air"; domain with intermediately fine ground mass crystals, corroded alite surfaces and rims of secondary belite

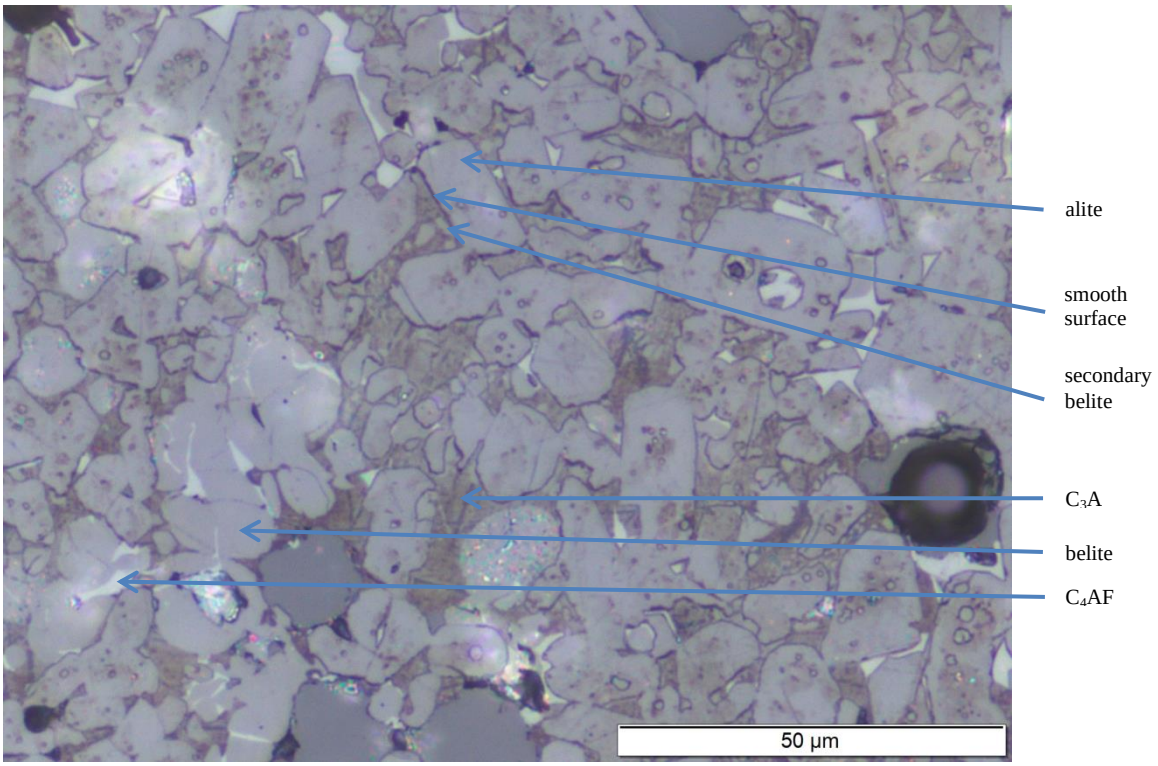

Figure 9.5: Polished section of sample "S9_air"; domain with coarse ground mass crystals, recrystallised alite surfaces and secondary belite recrystallised to single crystals

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Figure 9.6: Sample "S10_air"

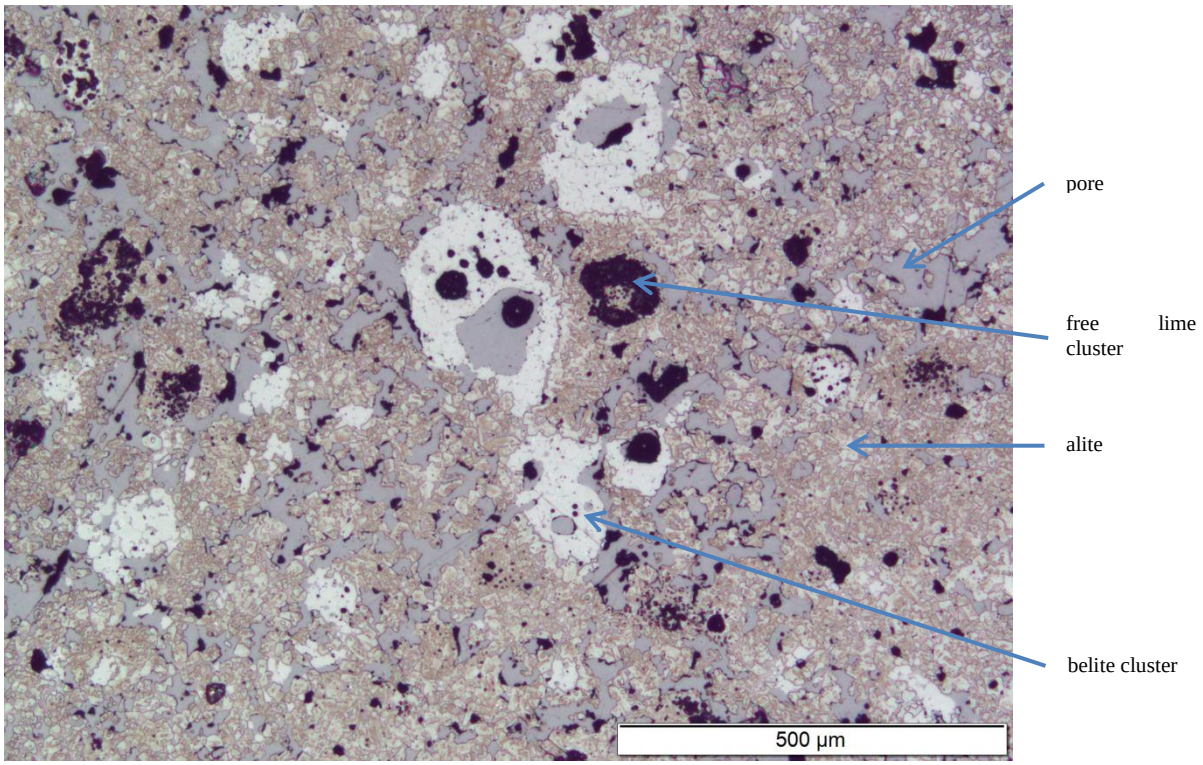

Figure 9.7: Polished section of sample "S10_air"; domain with high burning degree

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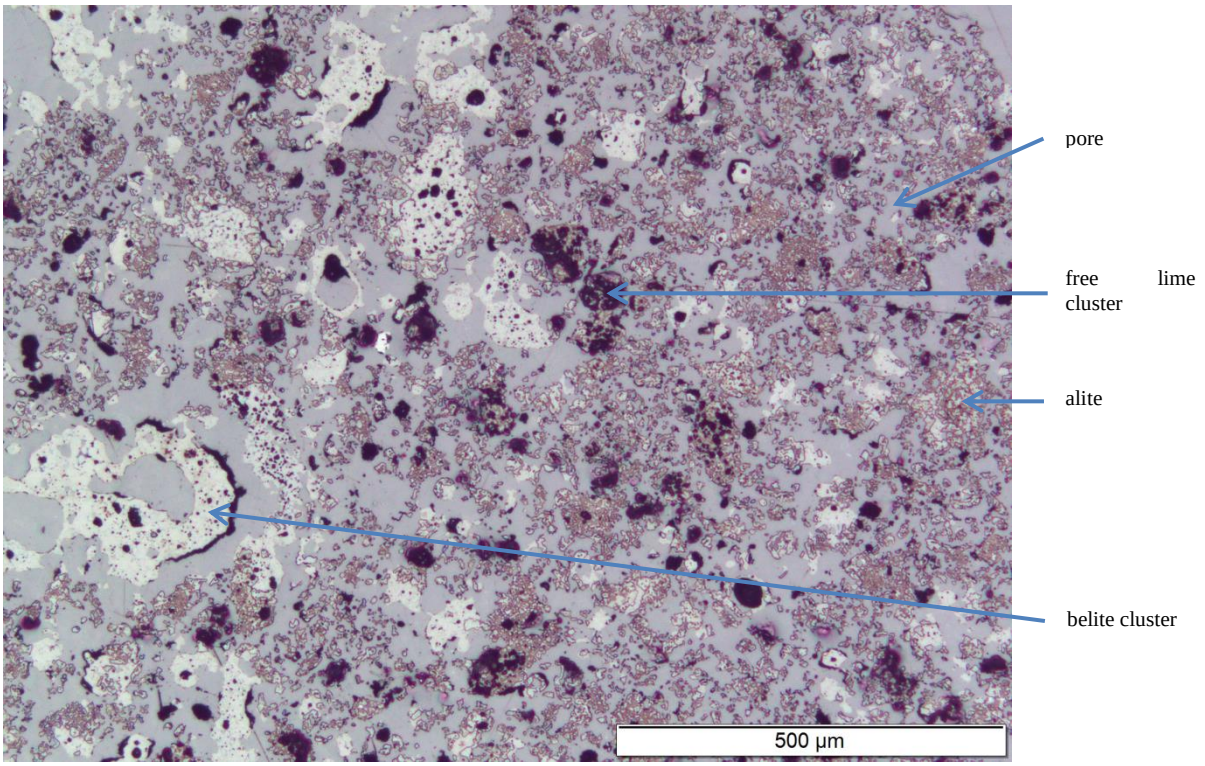

Figure 9.8: Polished section of sample "S10_air"; domain with intermediately high burning degree

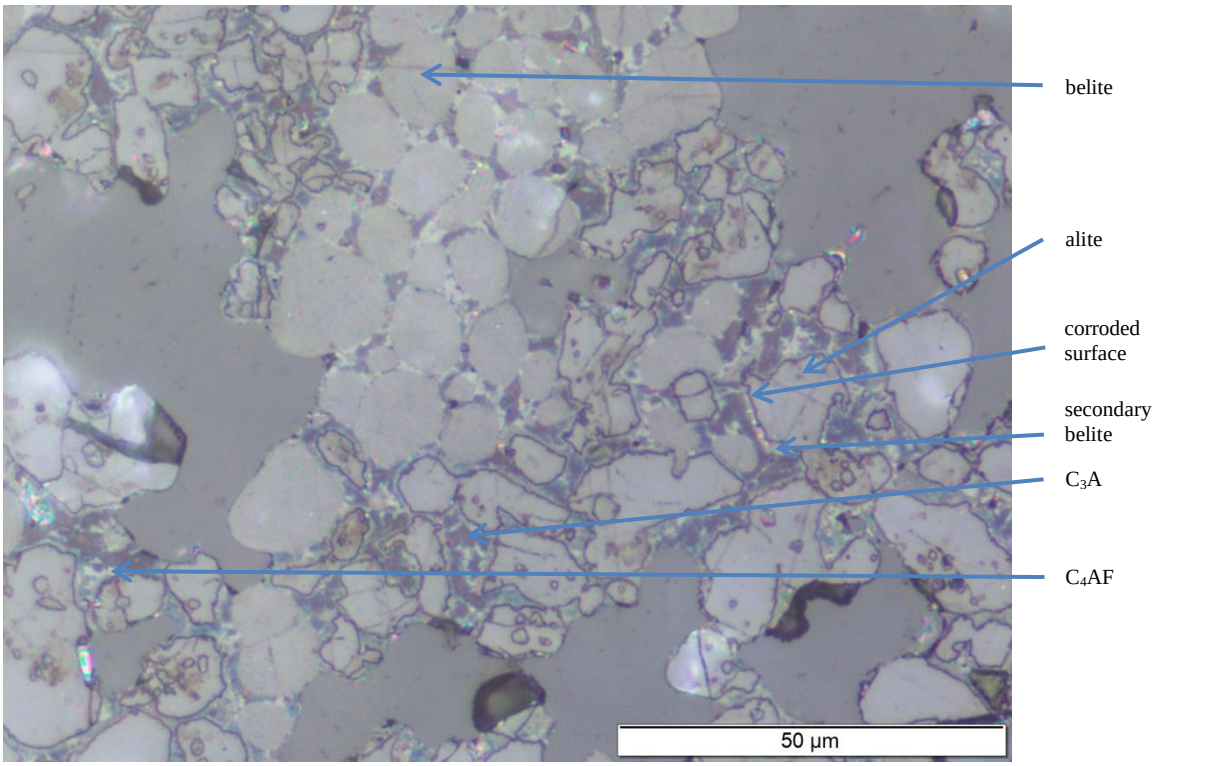

Figure 9.9: Polished section of sample "S9_air"; domain with intermediately fine ground mass crystals, corroded alite surfaces and rims of secondary belite

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Figure 9.10: Sample "S1_air", part of sample "ØS3_air"

Figure 9.11: Sample "S2_air", part of sample "ØS3_air"

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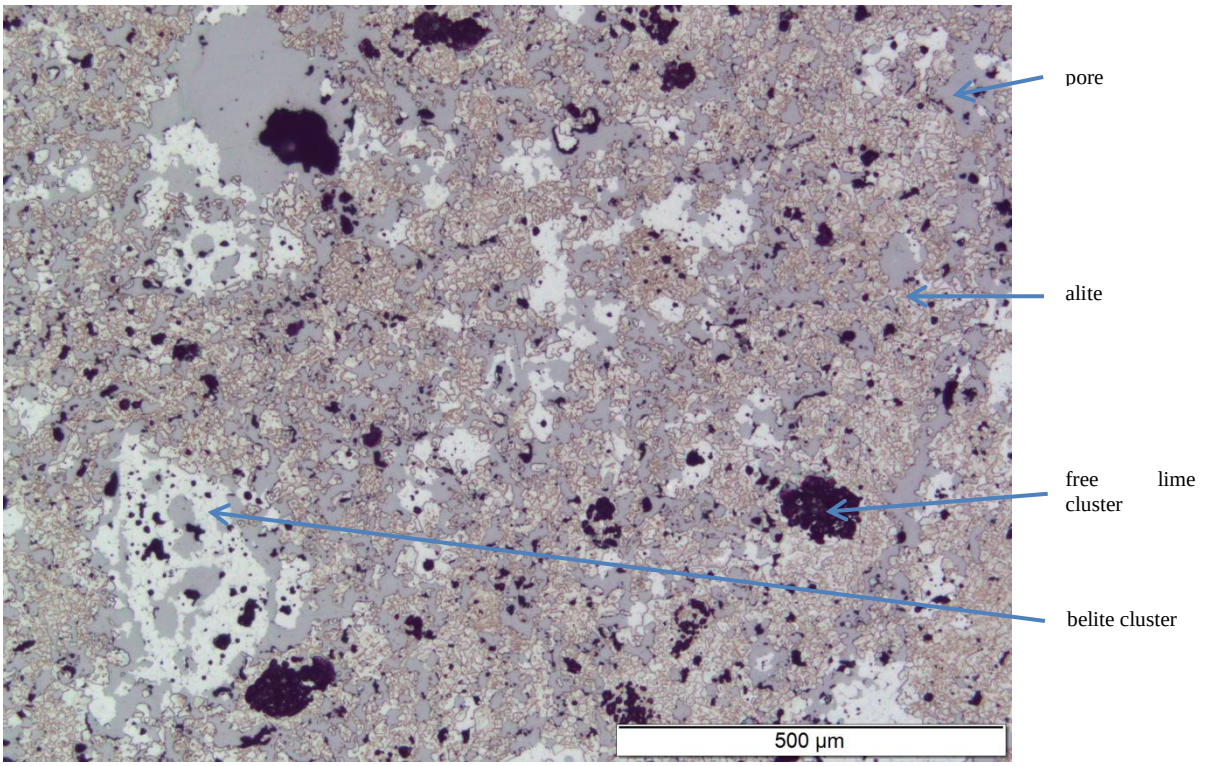

Figure 9.12: Polished section of sample "ØS3_air"; domain with high burning degree

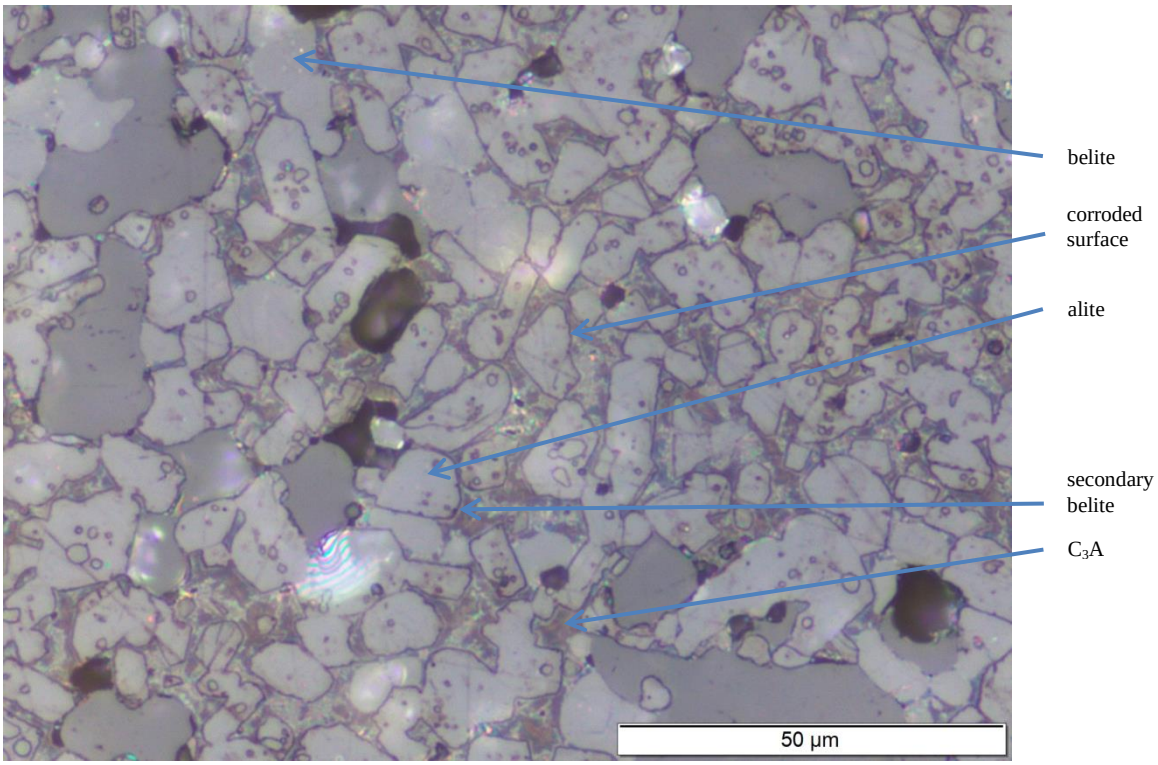

Figure 9.13: Polished section of sample "ØS3_air"; domain with fine ground mass crystals, corroded alite surfaces and rims of secondary belite

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Figure 9.14: Sample "S4_oxy"

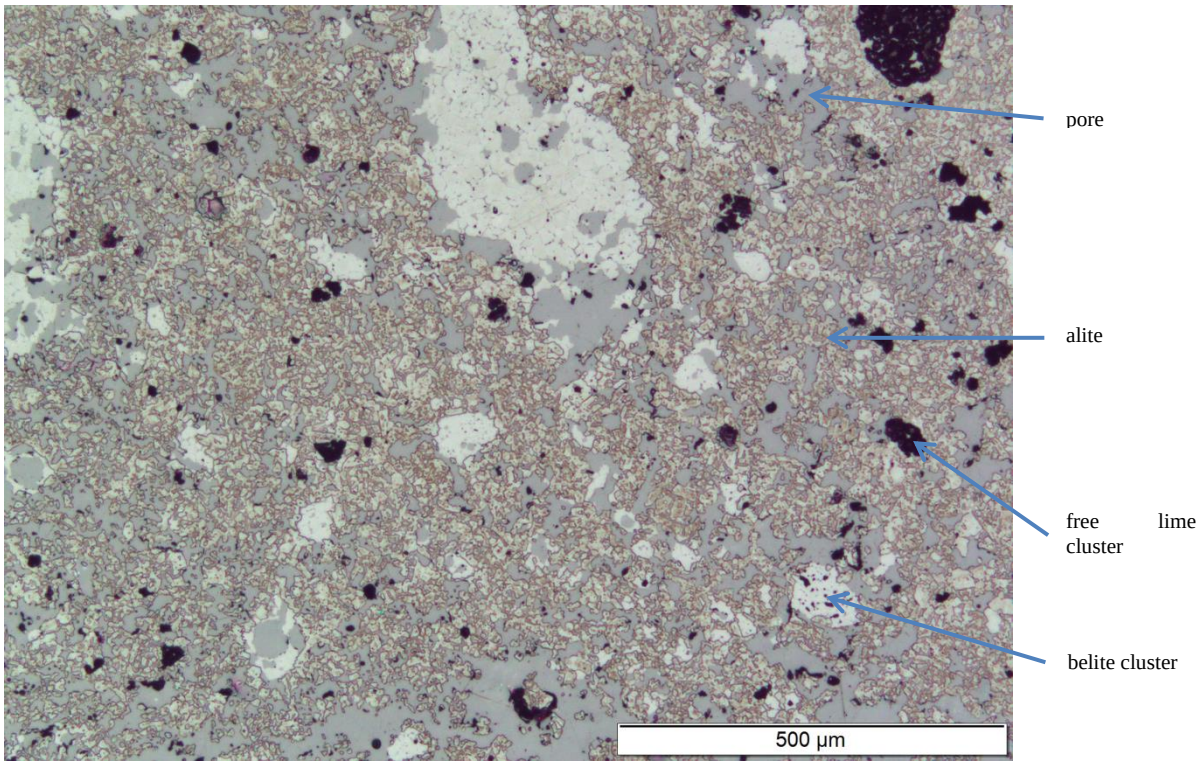

Figure 9.15: Polished section of sample "S4_oxy"; domain with high burning degree

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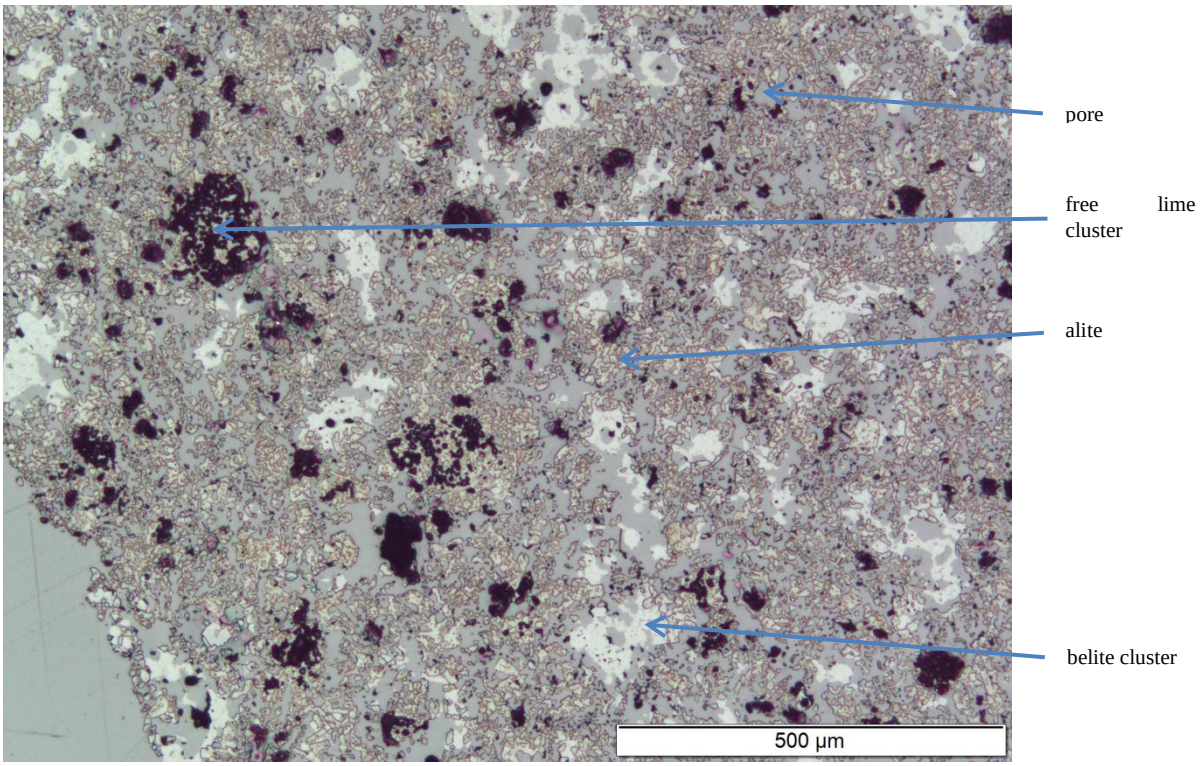

Figure 9.16: Polished section of sample "S4_oxy"; domain with intermediately high burning degree

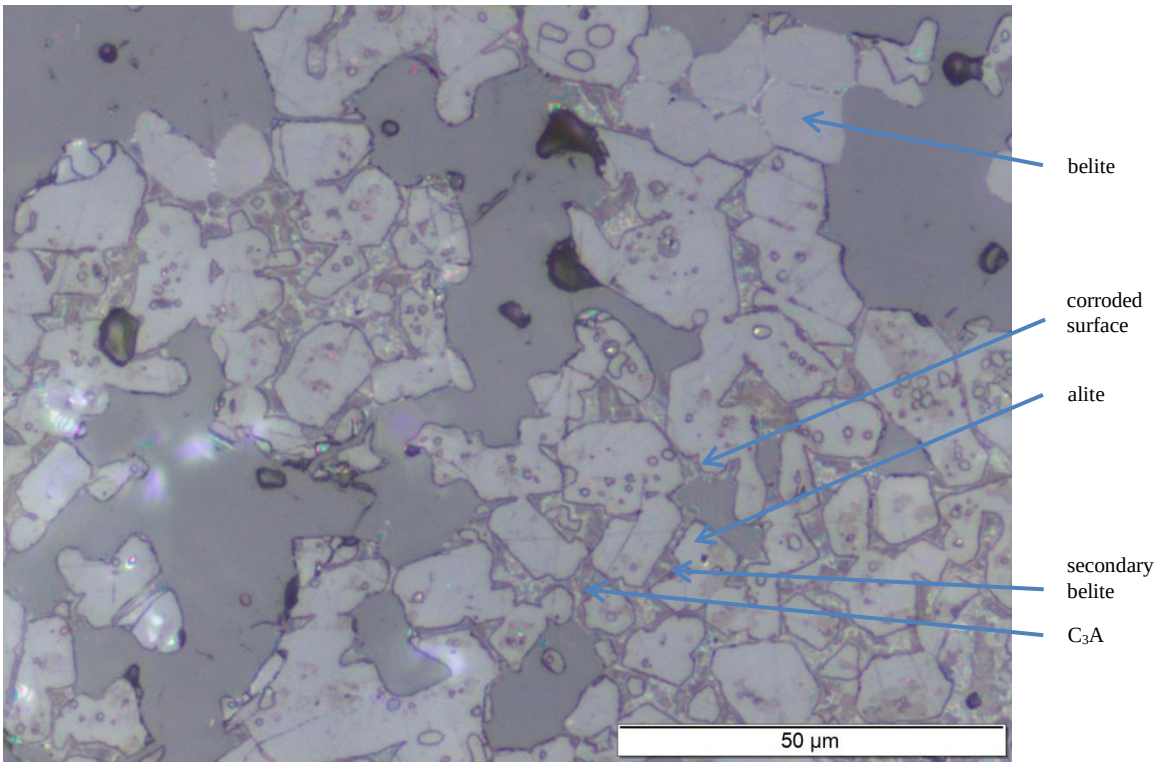

Figure 9.17: Polished section of sample "S4_oxy"; domain with fine ground mass crystals, corroded alite surfaces and rims of secondary belite

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Figure 9.18: Sample "S5_oxy"

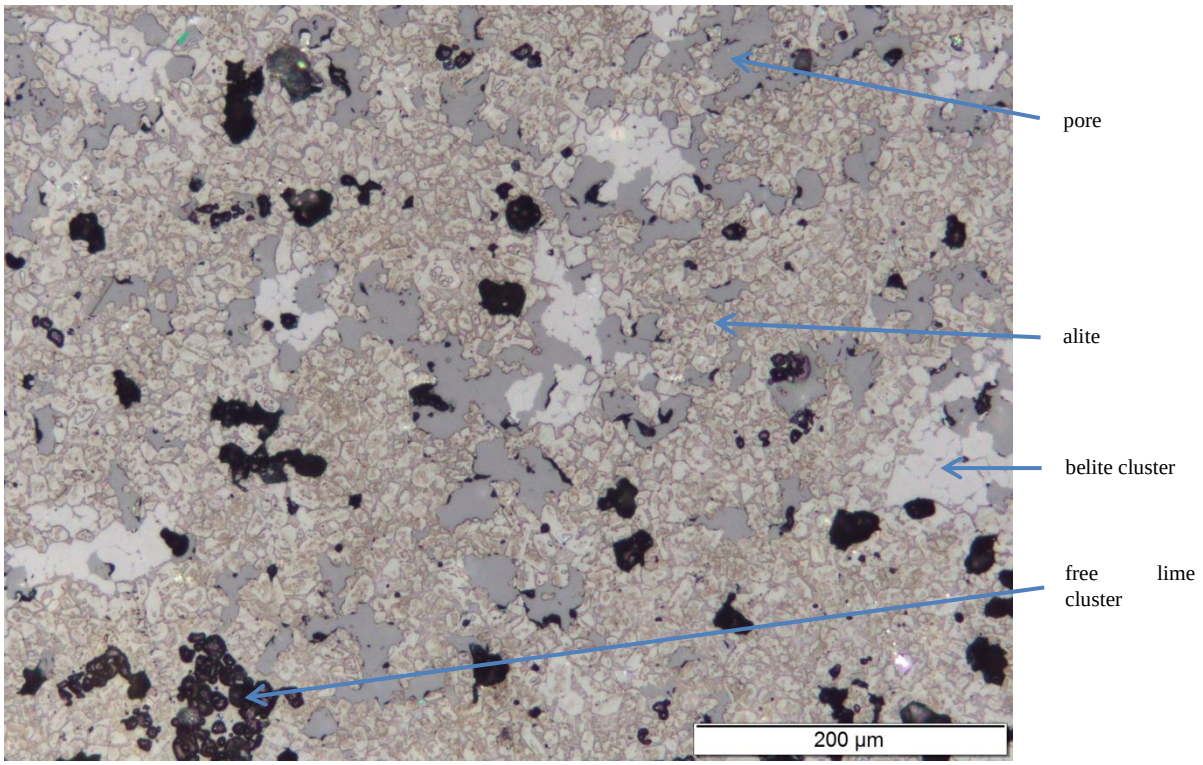

Figure 9.19: Polished section of sample "S5_oxy"; domain with high burning degree

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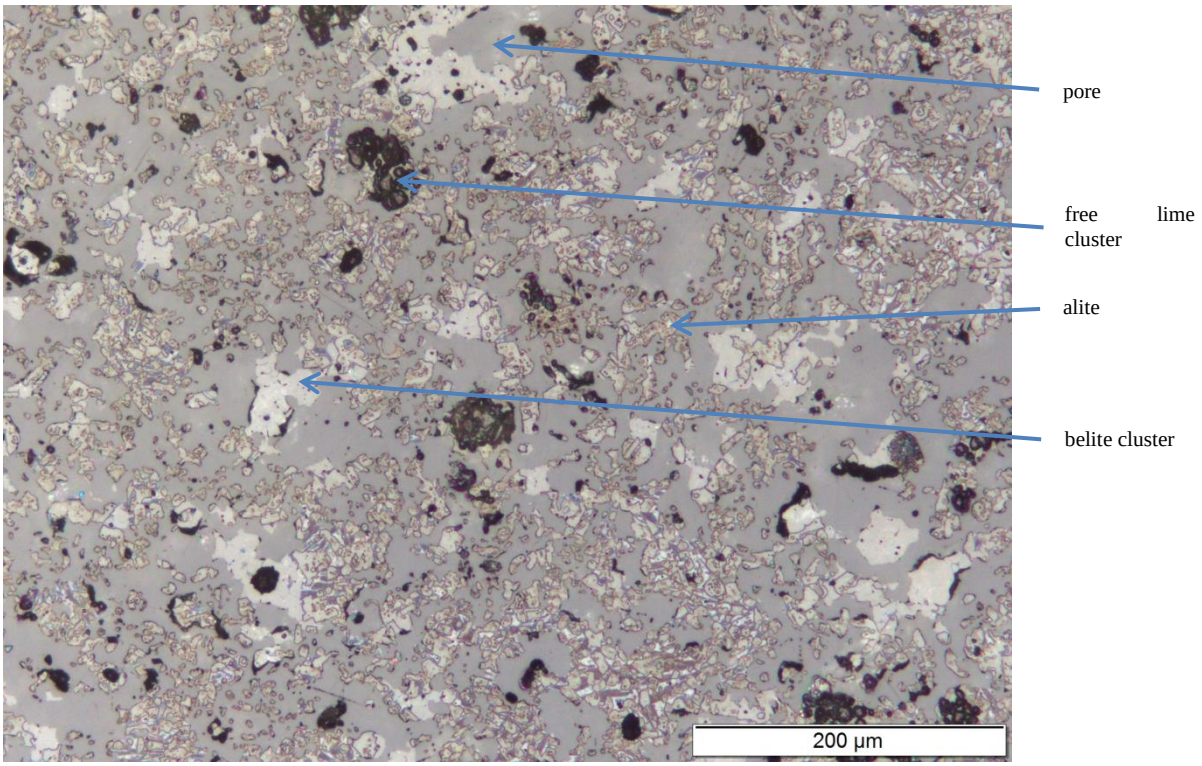

Figure 9.20: Polished section of sample "S5_oxy"; domain with intermediately high burning degree

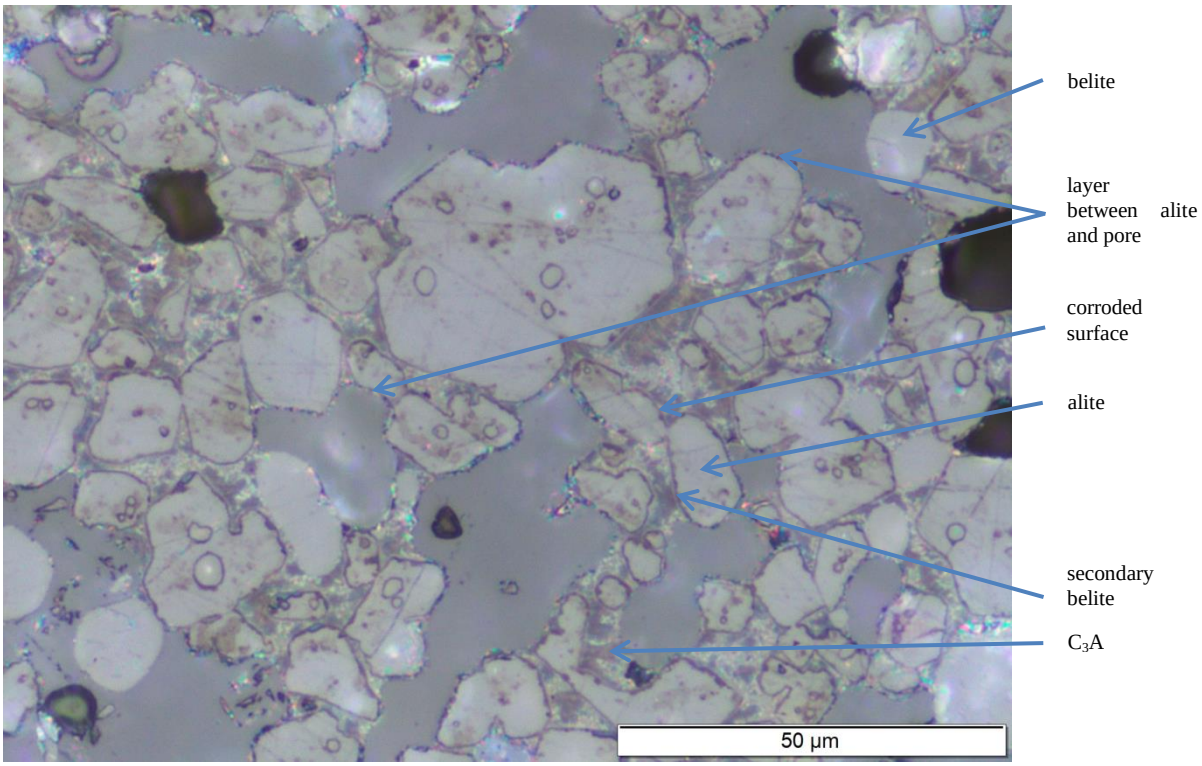

Figure 9.21: Polished section of sample "S5_oxy"; domain with fine ground mass crystals, corroded alite surfaces and rims of secondary belite; very finely crystalline rim on alite surfaces in contact with pores

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Figure 9.22: Sample "S6_oxy"

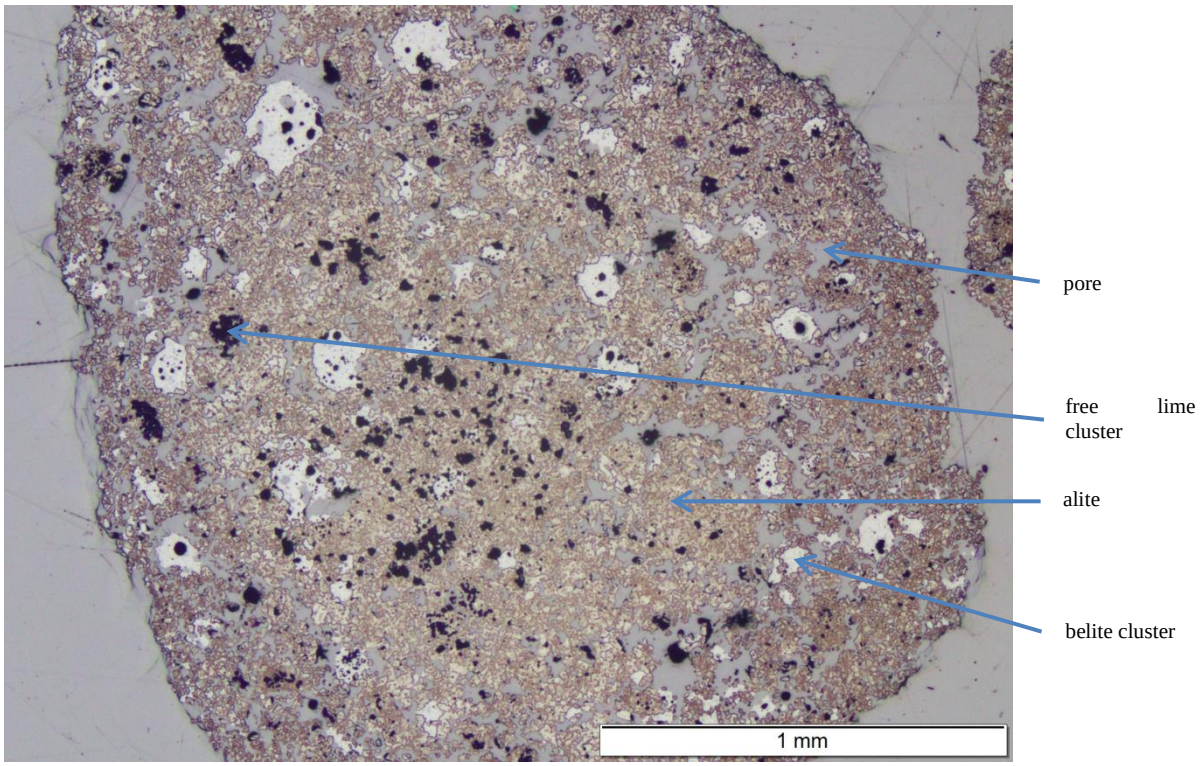

Figure 9.23: Polished section of sample "S6_oxy"; domain with high burning degree

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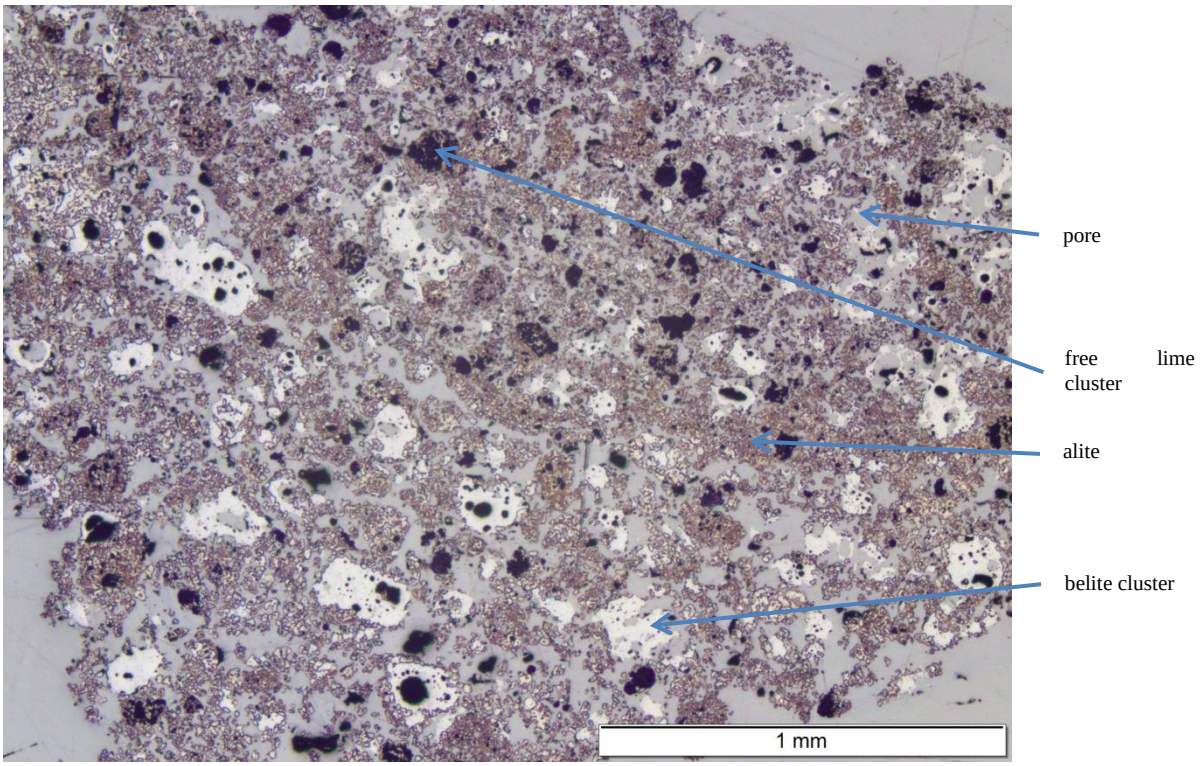

Figure 9.24: Polished section of sample "S6_oxy"; domain with intermediately high burning degree

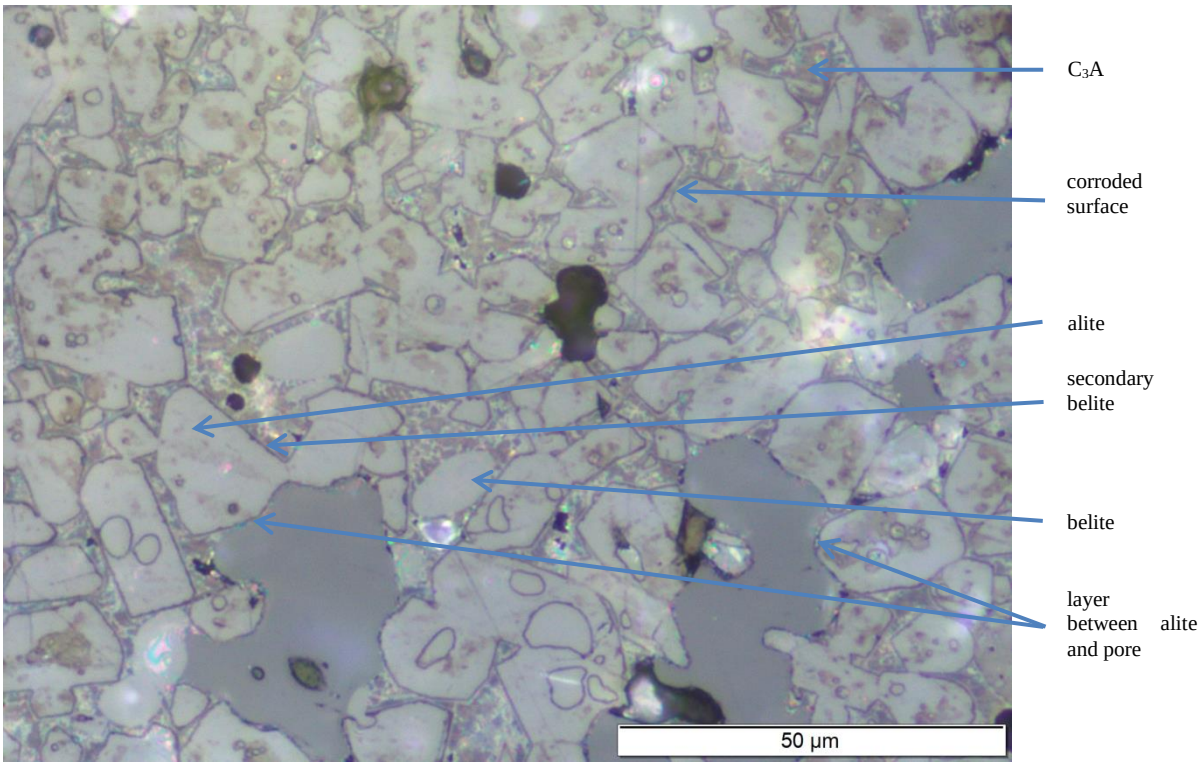

Figure 9.25: Polished section of sample "S6_oxy"; domain with fine ground mass crystals, corroded alite surfaces and rims of secondary belite

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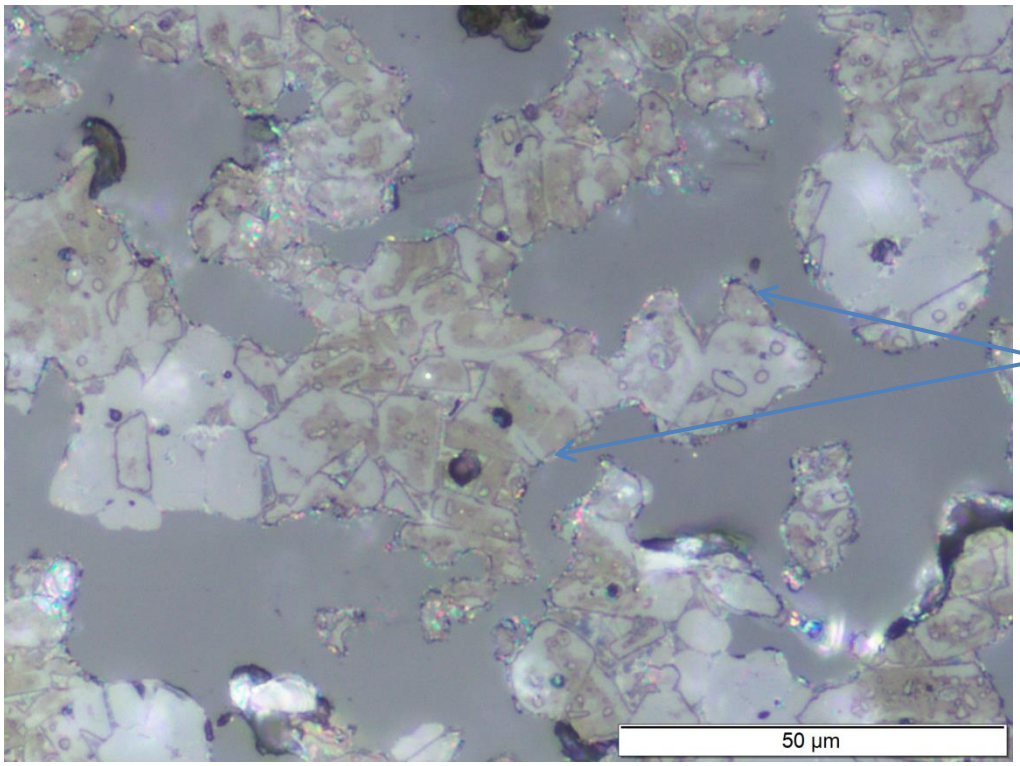

Figure 9.26: Polished section of sample "S6_oxy"; very finely crystalline rim on alite surfaces in contact with pores

Figure 9.27: Sample "S7_oxy"

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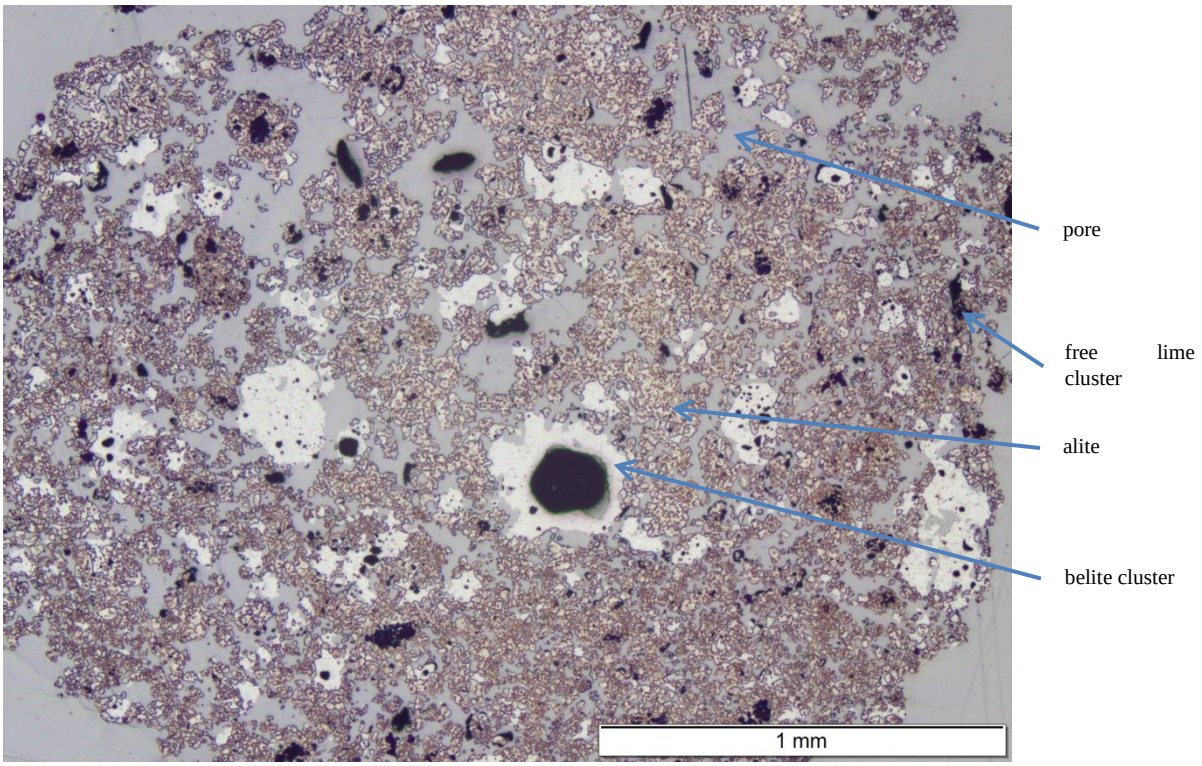

Figure 9.28: Polished section of sample “S7_oxy”; domain with high burning degree

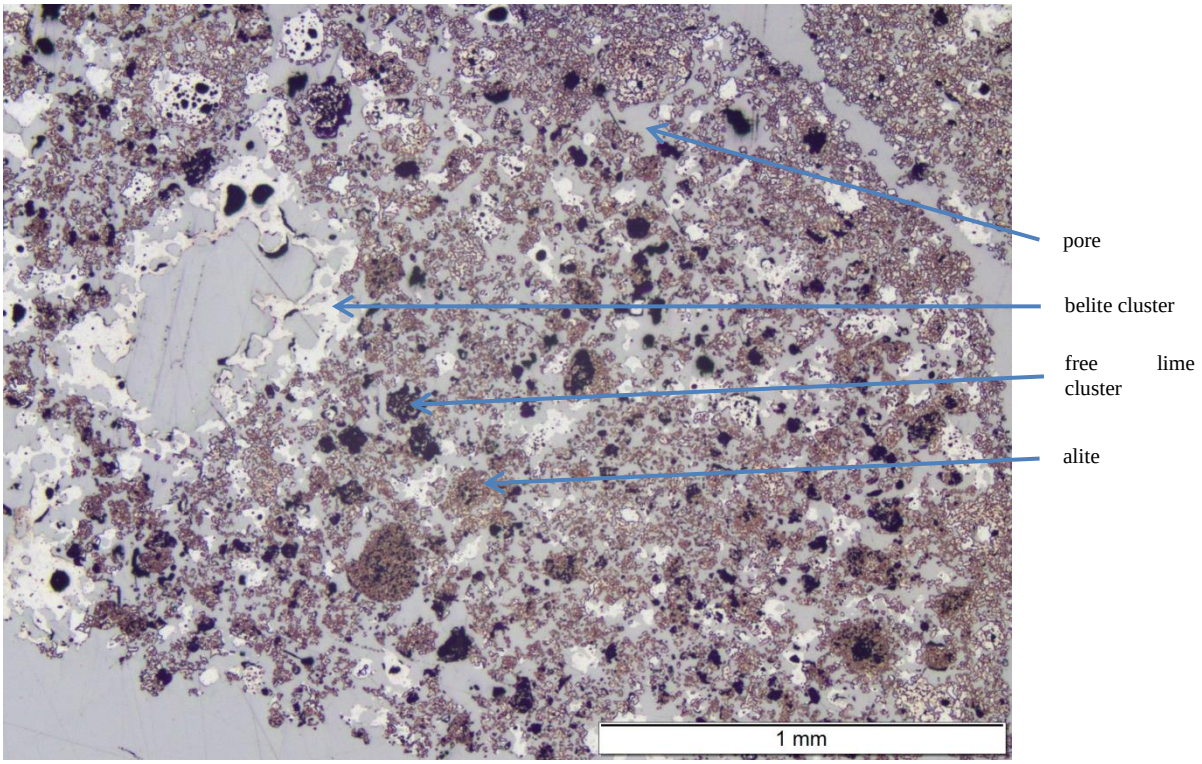

Figure 9.29: Polished section of sample “S7_oxy”; domain with intermediately high burning degree

This project has received funding from the European Union's Horizon2020 Research and Innovation Program under Grant Agreement No 641185

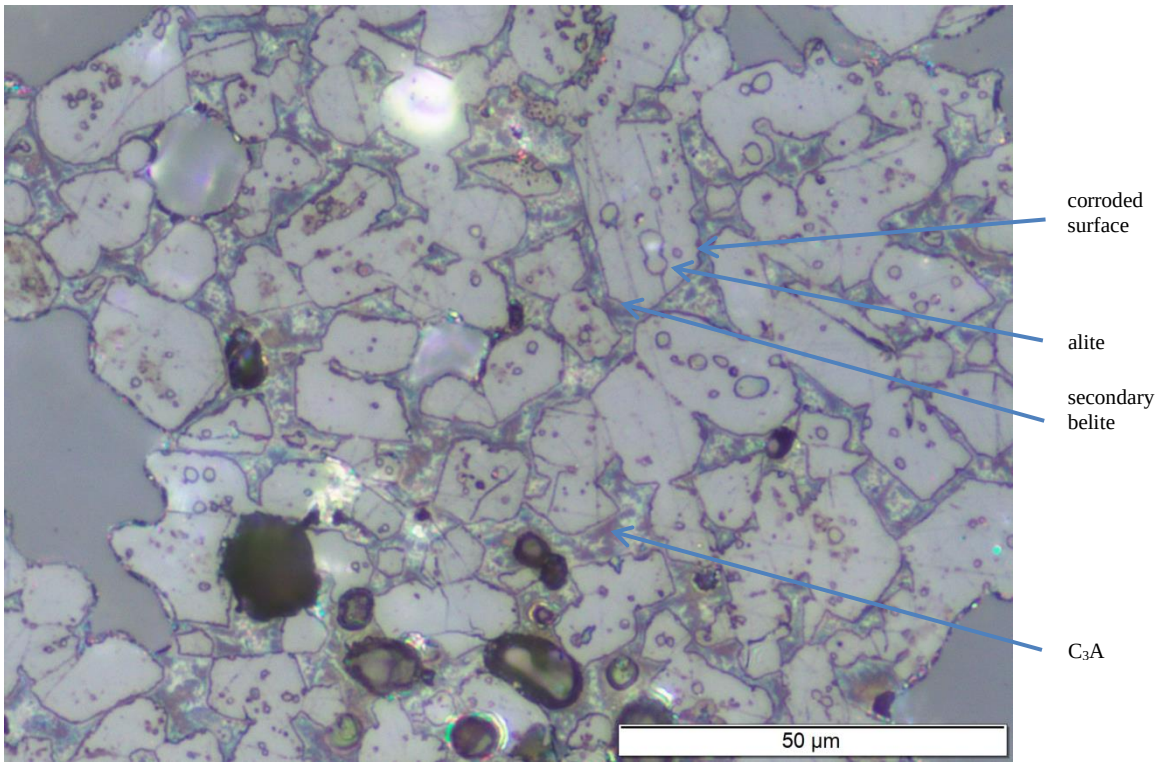

Figure 9.30: Polished section of sample "S7_oxy"; domain with fine ground mass crystals, corroded alite surfaces and rims of secondary belite

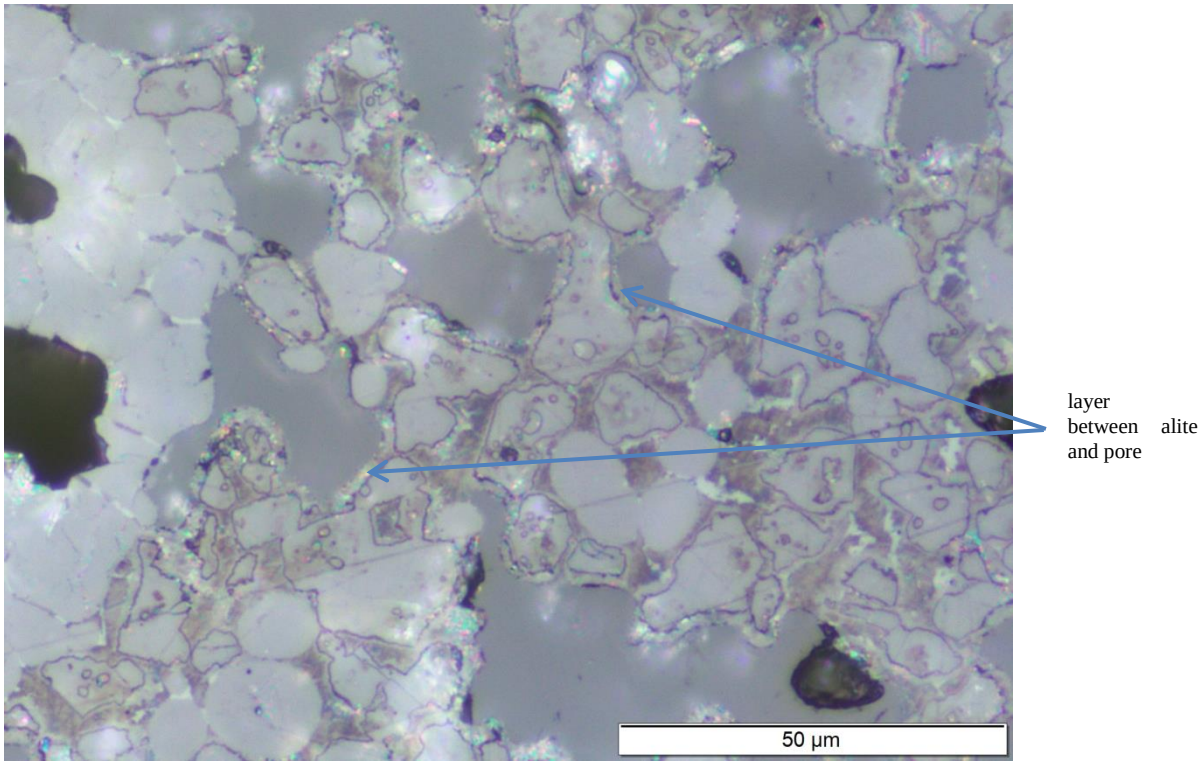

Figure 9.31: Polished section of sample "S7_oxy"; very finely crystalline rim on alite surfaces in contact with pores

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Figure 9.32: Sample “S8_oxy”

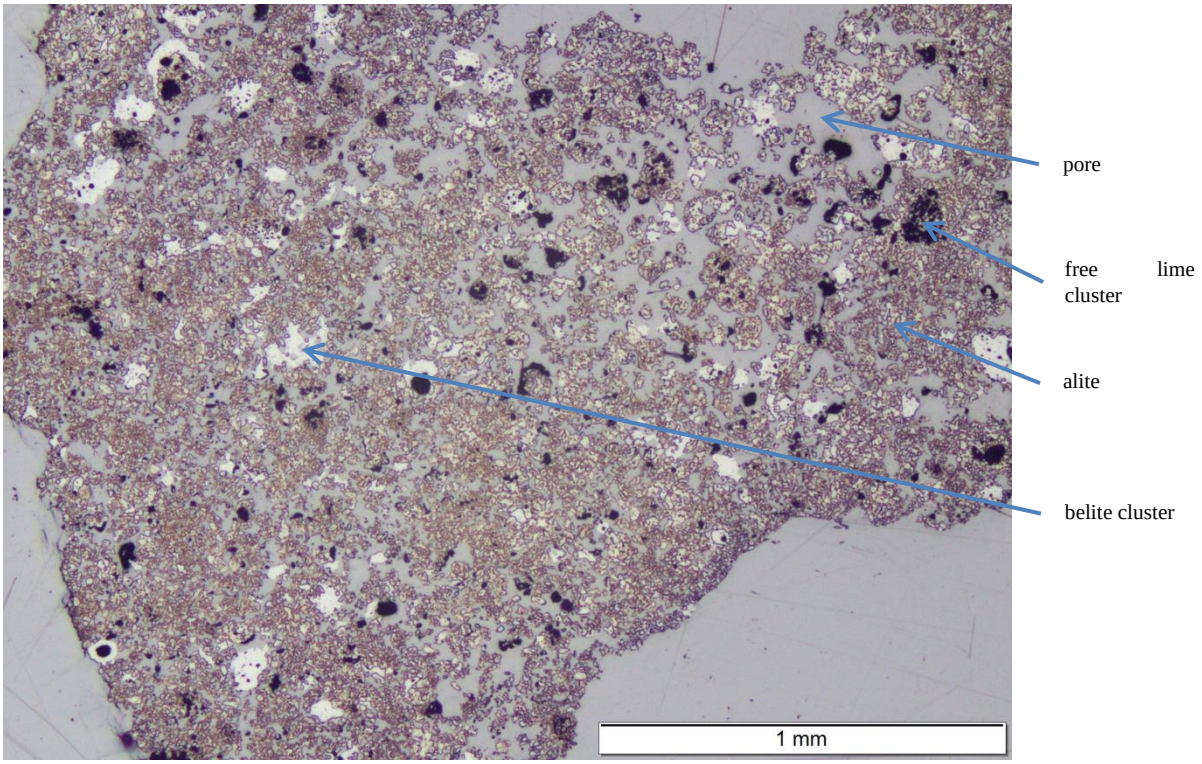

Figure 9.33: Polished section of sample “S8_oxy”; domain with high burning degree

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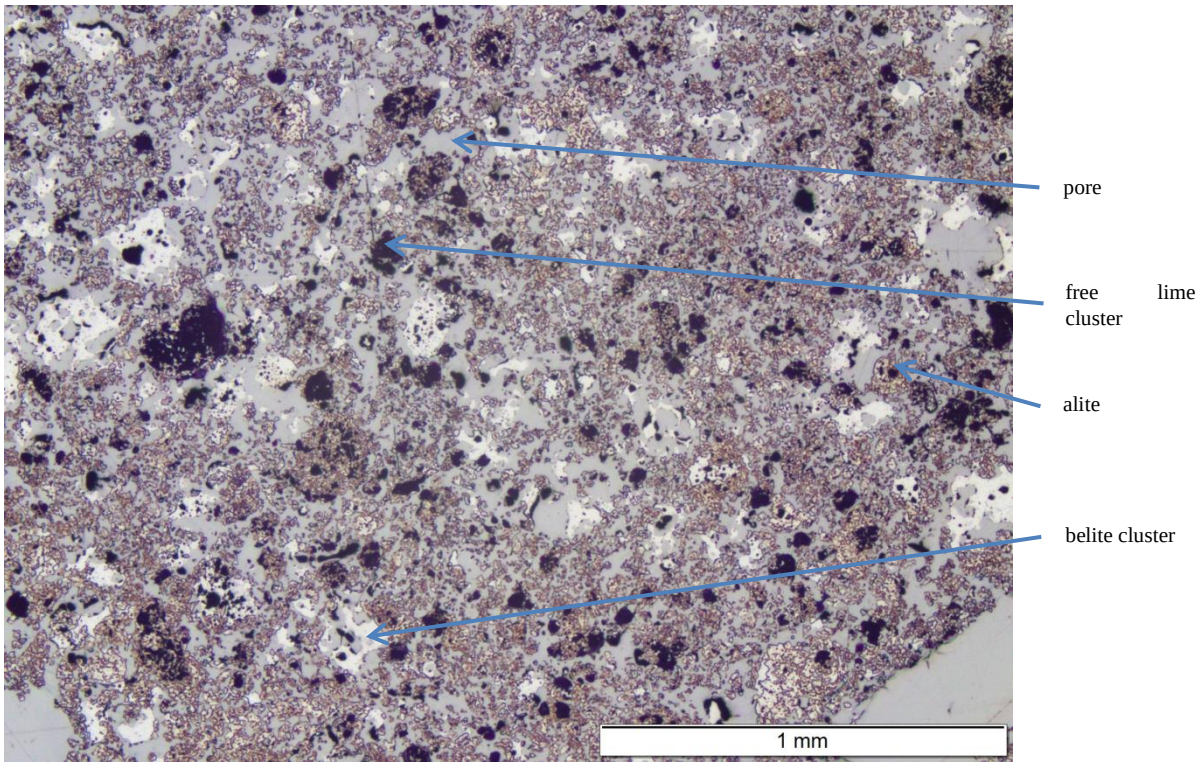

Figure 9.34: Polished section of sample "S8_oxy"; domain with intermediately high burning degree

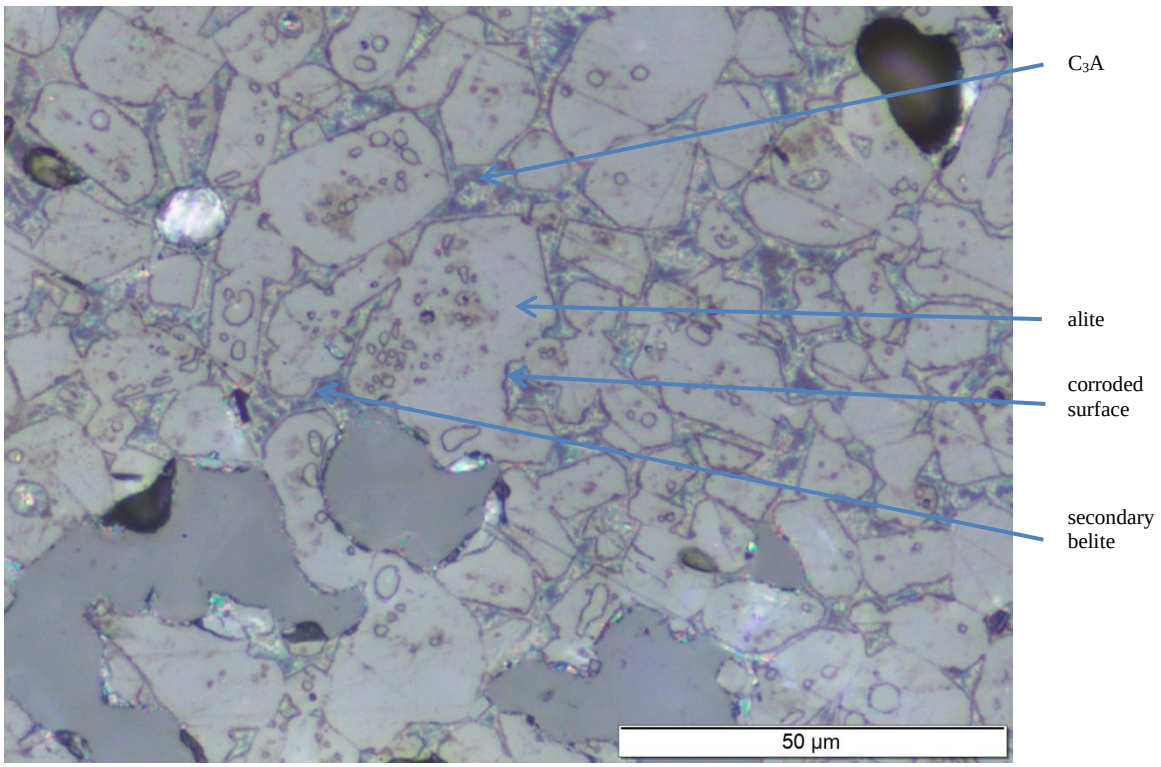

Figure 9.35: Polished section of sample "S8_oxy"; domain with fine ground mass crystals, corroded alite surfaces and rims of secondary belite

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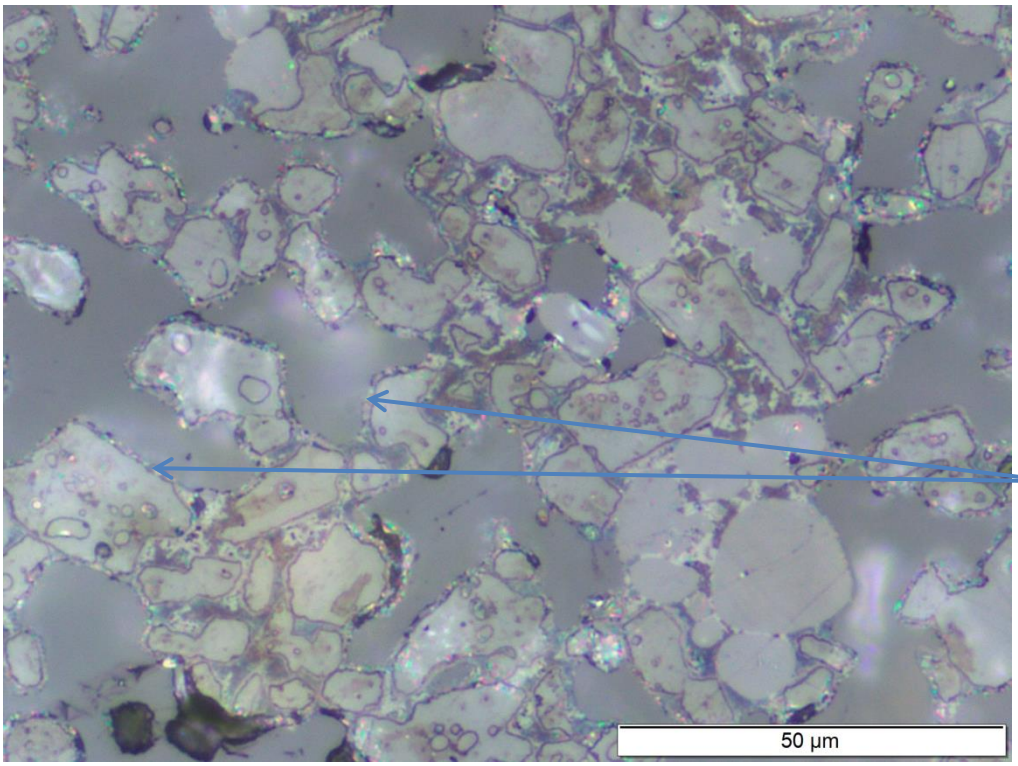

Figure 9.36: Polished section of sample “S8_oxy”; very finely crystalline rim on alite surfaces in contact with pores

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10 APPENDIX C – PILOT PLANT HEAT AND MASS BALANCE MODEL

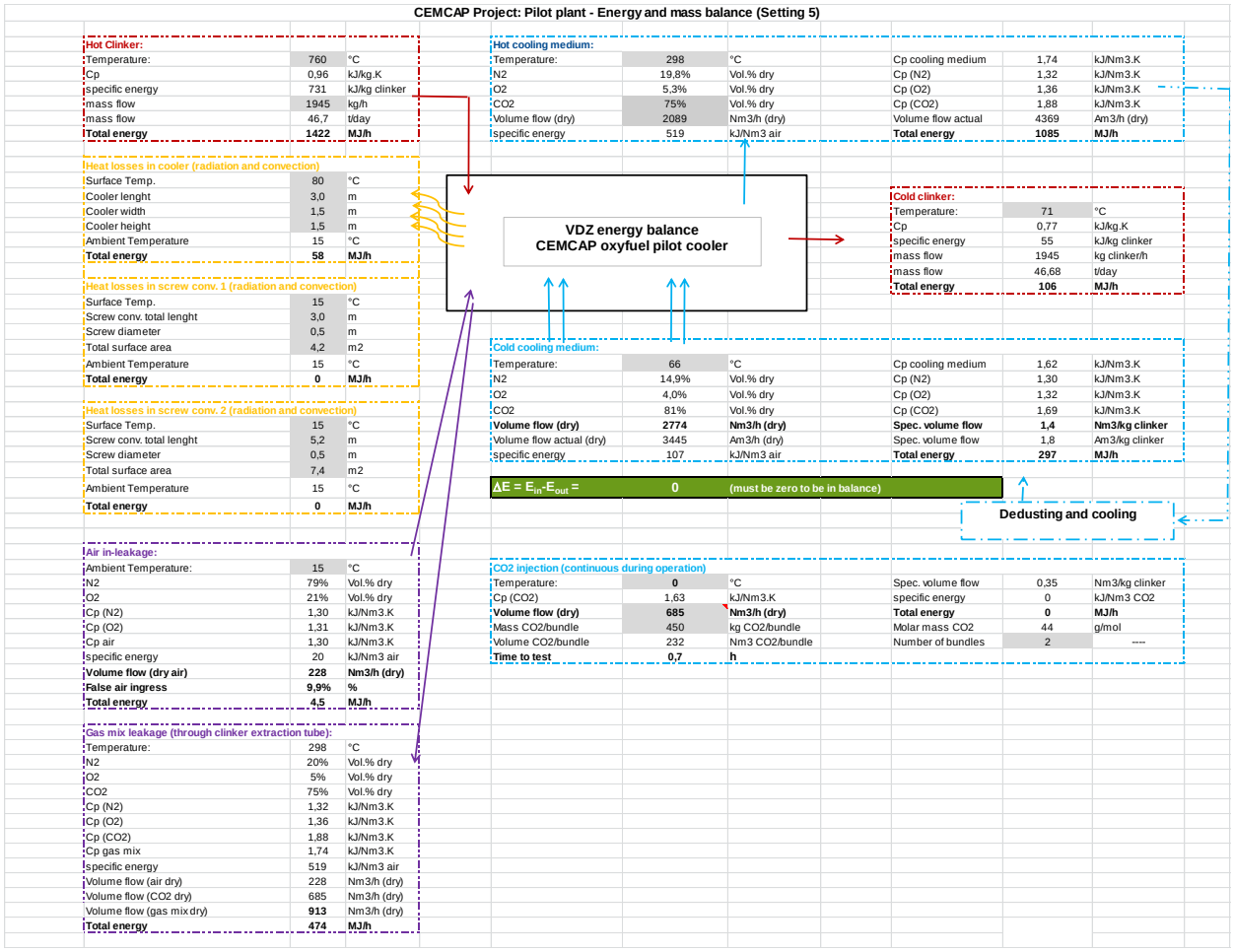

Figure 10.1 Heat and mass balance example – setting 5

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