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Abstract

The article presents a novel design for a distribution plate. The solution is suitable for a reactor vessel where a reactant gas needs to be maintained at a different temperature from the reaction chamber in order to avoid unwanted occurrences, such as clogging of the distribution plate. A normal procedure involves cooling of the distribution plate which is reported to either increase heat loss substantially or yield insufficient temperature in parts of the reaction chamber. The problem is especially important for reactors where the difference in reactant inlet temperature and desired reaction temperature is large. The investigated design utilized materials of very different thermal conductivity to only cool specific parts of the distribution arrangement and thereby minimize heat loss. Our system is a distribution plate for use in a fluidized bed reactor for silane pyrolysis. However, the solution is general and may be utilized in many types of vessels and chemical reactors.

KEYWORDS: polysilicon, fluidized bed, distribution plate, heat loss

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1 Introduction

1.1 Background

The worlds energy demand is ever increasing and there is a growing interest in finding alternative renewable energy sources. The concept of photovoltaic (PV) cells is over a century old. But the increasing energy prices in combination with more cost effective production methods and higher efficiencies already makes PV competitive in several countries and regions today. Over 95% of todays PV cells contain silicon. Being one of the most abundant elements on earth, the accessibility of the raw material is in most aspects unlimited. The processing is however complicated and energy consuming as the most efficient cell concepts require a contamination of selected impurity species as low as ppb level (Jenny Nelson 2009). These purity levels are today only obtainable by going through gas and chemical vapour deposition (CVD).

Silicon based PV cells are expected to play a major role in the utilization of solar energy and the market for polysilicon is expected to continue to grow rapidly in the next five years. The projected worldwide capacity in 2012 is predicted to be about 200,000 Mt./year and individual plant capacity is expected to be of the order of 10,000 Mt./year, if not larger (Gerald Parkinson 2008), (M. Javidi & P. Ramachandran 2009). Correspondingly, the share of power generation from PV is expected to increase and is already of the order of 3.8 GW in Germany. The target cost of silicon production is set around $20-25 per kg (M. Javidi et al. 2009).

1.2 Application

Fluidized Bed Reactors (FBR) have been widely used for many different applications. The fundamental function of a FBR is to fluidize solid particles by an ascending gas flow of high enough velocity. In a silicon CVD FBR, a thermally decomposable silicon compound is introduced and decomposed through heating. The two commonly used reactants are silane and trichlorosilane. Silane is utilized by Rec silicon and MEMC while trichlorosilane is utilized by Wäcker chemie. For many other FBR applications, one tend to insert the reaction gas diluted in the fluidization gas and inserted both through a common distribution plate at the bottom of the reactor volume. This layout has been explored for silicon CVD to some extent. Both Hsu. et al of Caltech (G. Hsu, N. Rohatgi , J. Houseman

1987), Iya of Union Carbide Corporation (Sirdhar K. Iya 1987) and Yoon et al. of KRICT (P. Yoon and Y. Song 1988) have all explored introducing a premixed fluidization gas and tried to avoid clogging through cooling of the distribution plate. However, the layouts being used commercially today is mostly based on different principles.

1.3 Fundamental problem

Silane decomposes at 420 °C (S. P. Walch & C. E. Dateo 2001), even though clogging is reported to be a problem at even lower temperatures (Hsu, et. al 1987), (S. K. Iya 1987), (P. Yoon et al. 1988). To assure a crystalline silicon structure, the beads need to be exposed to temperatures above 710 °C (S. M. Lord & R. J. Milligan 1995). This temperature may either be applied at the time of deposition or alternatively later through annealing. The fundamental problem is therefore that one needs to keep the temperature low at the point of reactant gas insertion while keeping the temperature high in other areas of the reactor in order to assure correct crystal structure

If one tries to solve this problem directly by intense cooling and heating at subsequent locations this leads to severe thermal losses. This is especially the case if the bottom of the reactor is kept cold while the top is heated since the heat is then continuously removed.

1.4 Earlier solutions

A number of different reactor layouts have been proposed for FBR silicon production. The interested reader is referred to the work of Filtvedt et al. (Filtvedt et al. 2010). The first FBR reactors for silicon production were all based on premixed fluidization and reactant gas (Harry W. Ling 1961), (H.S.N Setty et al. 1974), (S. K. Iya 1987), (Robert H. Allen 1988). However, because of the challenges with either clogging or severe heat-loss in the bottom of the reactor, the new designs of the 90s and 00 where mostly based on different types of separate reactant and fluidization gas inlets. New challenges using undiluted reactant gas involved increased fines production and impuriy incapsulations.

One promising two inlet design is heating and fluidizing the bottom and introducing the reactant gas to the upper part of the reactor like Kim et al. (Kim et al 1994). Alternatively running the reactor semi continuously like S. M. Lord (Stephen M. Lord 2002) where high temperature and reactant gas are applied alternately in time. Other solutions include inserting the reactant gas through a central nozzle while fluidizing the bed through a peripheral distribution plate as proposed by Kulkarni et al. of MEMC (Kulkarni et al. 2009).

A modern premix design is applying the heat within the reactor by an infrared light source through a spout jet like Lord et al. (Lord et al. 1995) this solution is the one being utilized for the largest production volumes today. The solution includes depositing at lower temperatures, and then anneal the deposited layer to form a crystalline structure. A number of publications have been made on the design over the last years including recrystallization of beads of Dahl et al. (Dahl et al. 2009) and modelling of bed behavior Piña et al. (Pina et al. 2006). The publications don't claim to be the actual process being utilized for commercial production. A challenge with the solution is the bubble size which is bigger within a spout than a bubbelig bed and it is found that an increase in bubble size is correlated to an increase in fines formation.

The earlier bubbeling bed designs based on premixed reactant and fluidization gas have utilized fluidizing through a homogeneous perforated distribution plate. The problem is therefore quite straight forward; the reactant gas decomposes at a certain temperature and the reactor needs a certain temperature to assure correct crystallization. Both these temperatures needs to be maintained. The thermal losses are then only depending on the internal heat transport mechanisms as described by Gunn and Hilal (D. J. Gunn & N. Hilal 1996) among others.

The silicon CVD FBR reactor is somewhat unusual in the sense that the required inlet conditions of the reactant gas is so far from the required reactor operational temperatures. Several researchers has acknowledged this difference and called out for unique designs to help aid the problem.

Other research groups to investigate the distribution plate clogging problem include Lackey et al. (Walter J. Lackey, Jr. & John D. Sease 1975) who proposed a thick distribution plate with thinner regions for letting the distribution gas through. The solution is not especially made for silicon production, but the goal is nevertheless to avoid depositions from decomposition of the fluidization gas.

2 Grid design

Many researchers on silicon FBRs report high quality beads when keeping the bed in bubbling regime (Hsu, et. al 1987), (S. K. Iya 1987), (P. Yoon et al. 1988), (Lord et al. 1995). In order to keep the bed in this regime, the process needs to be continuously monitored and controlled. The beads continuously grow in the reactor and since the bubbling regime offers limited mixing of the bed, there is need to have control of the average bead size as well as the bead size distribution through the bed. The largest beads tends to move downwards to where the deposition is largest (Lord et al. 1995). This mechanism might spiral down and eventually kill the bed. Several methods may be utilized to aid the problem, but continuous bead removal from the bottom of the bed and reintroduction of the smallest beads at the top is a well known solution.

In order to achieve a good gas distribution over the whole bottom of the bed, several different distributor designs are possible. This involves using a distributor plate or cone alternatively to utilize a tube construction at the bottom of the bed, often referred to as a sparger grid. Sparger grids may be especially desirable in large beds where the preferred injection direction might be horizontal or downward.

Due to contamination problems, sparger grids are not especially desirable in silicon FBRs. Most designs either utilizes a distributor plate of some sort, alternatively a spout layout. Established design equations proposes this relationship when designing a distributor plate; the pitch L_h in Fig 1 depends on the hole density N_d according to equation 4. The hole density is given in number of holes per unit area. (Karri et al 2001)

PARAMETERS

C_d	0.746	= discharge coefficient
d_h	$1\text{x}10^{-3}$	= grid hole diameter, m
g	9.81	= gravitational acceleration, m/s^2
K	0.3	= grid pressure-drop coefficient, upward gas entry
L_B	0.15	= operating bed depth, m
N	66	= number of grid holes
N_d	3499	= number of hole density, holes/m^2
ρ_B	1190	= operating bed density, kg/m^3
$\rho_{g,b}$	0.14729	= density of gas at bed operating conditions kg/m^3

$$\Delta P_{grid} = Kg\rho_B L_B \tag{1}$$

$$U_h = C_d\sqrt{\frac{2\Delta P_{grid}}{\rho_{g,h}}} \tag{2}$$

$$Q = N\frac{\pi d_h^2}{4}U_h \tag{3}$$

$$L_h = \frac{1}{\sqrt{N_d}} \tag{4}$$

RESULTS

L_h 0.0169 = grid hole pitch, m
U_h 62.9 = velocity of the gas through the grid hole, m/s
Q 3.26 x $10^{-}3$ = total volume flux through grid, m^3/s
ΔP_{grid} 525 = pressure drop across grid, Pascal

Based on these equations one realizes that for normal size reactors, the pitch between the distributor holes may be several orders higher than the hole diameter. This simple concept yields the possibility of building up a composite distribution plate. One may cool certain areas and insulate certain other parts of the plate. Starting from this standpoint one may already reduce the total thermal losses by only cooling a certain area around each hole and not the entire distributor. By exploring different geometry options of different materials, we where able to reduce the theoretical thermal losses even further.

Our reactor had ID 155mm and possible bed height of 500mm. A number of different operation conditions is possible in the bed but to meet the range of possibilities the grid consisted of 66 holes with a diameter of 1mm. Square pitch was chosen for the pattern see Fig 1. As one may see the viritual velocity through the bed at the outlet of the distribution plate is for this setup 0.185 m/s.

3 New design

The design was based on the fundamental textbook equations reported by Karri et al. (Karri et al 2001) among others. Based on the number and length of the holes, the design was taken further in the commercial CAD

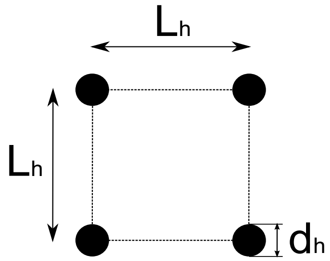

Figure 1: Hole pattern in distribution plate, square pitch

program Solid Works. The basis for the layout was how to assure low enough temperature in the thermal boundary layers of the individual holes in combination with minimizing the heat loss from the reactor volume. The chosen design was a highly conductive plate at the base with a number of pins enclosing each hole through the insulation see Fig 2. The pins finished in a conical shape in order to reduce the amount of heat transfer into the pins. A practical problem was the sum of contributions from each pin from the center of the plate to the wall. Because of this effect the temperature in the center of the plate would be significantly higher than by the wall as shown in Fig 2.

Another obvious challenge with the design is the length and diameter of the individual pin. This problem is also dependent on the type of material in the baseplate and the pins. One desirable solution is to use an anisotropic material with different k-value in each direction for the pins and for the baseplate. Different types of composite carbon materials may be desirable for such applications and especially combinations of different composite carbon materials.

However, for the chosen design, isotropic materials were used. The base plate and the pins were made from one piece of aluminium. Because the plate was inserted between two cooled flanges, the aluminium had to sustain several installations. Because of this, the chosen aluminium alloy was Al 6082. For insulation a commercial available high temperature compact wool was used. The k-value for this material was 0.08 W/mK.

To predict the temperature distribution in the different regions of the baseplate a solid CFD model was tested in Fluent. Since the plate has two symmetry planes only one quarter of the plate needed to be modelled. The model was quite simple. The top of the plate was subjected to a constant temperature of 600 °C while the side of the base plate was main-

Figure 2: Distribusjonsplate design

tained at 100 °C. The bottom of the baseplate is facing the windbox and the entering gas. Because the windbox is kept at a temperature close to the bottom baseplate temperature and since the entering gas has limited heat capacity the chosen boundary condition is anisotropic. The chosen mesh was T-grid will average cell length of 0.5mm. From these simulations we found that the temperature rise from the side to the center of the baseplate was around 25 °C as can bee seen in Fig 3, the scale has 25 °C steps.

The silicon FBR through silane pyrolysis is most commonly performed in bubbling regime. For the one familiar with the art of FBR engineering the bed height, hole pattern and minimum fluidization velocities are easily obtainable through textbook examples. However, the problem is somewhat out of the ordinary because of the large volume expansion given from the raise in temperature in the first few centimeters of the reactor (Lord et al. 1995). The problem arises because of the large difference between decomposition and ideal crystallization temperature. This problem is usually avoided by reference in the conditions at the inlet of the reactor and sufficient fluidization conditions here makes sufficient fluidization of the whole bed (Lord et al. 1995), (Kim et al. 1994). This means that one takes reference in the properties of the incoming gas and use the inlet velocity as the virtual velocity through the whole bed although the large volume expansion increases the virtual velocity substantially during the first few cm of the bed.

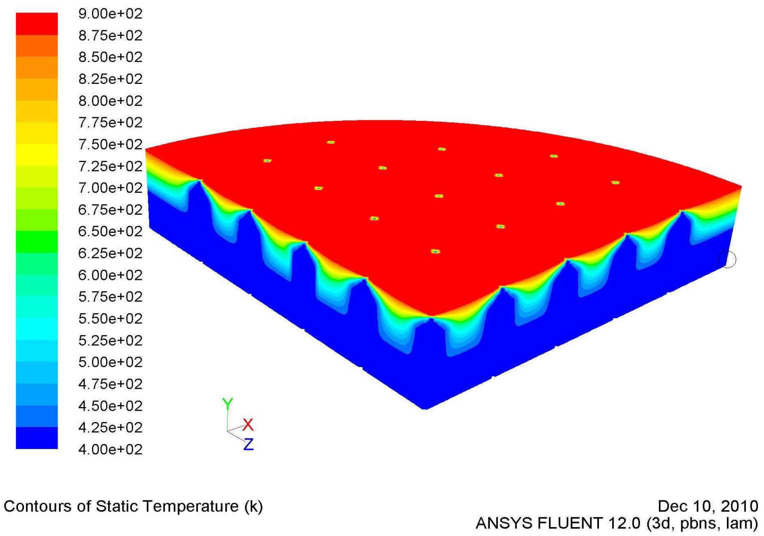

Figure 3: Temperature analysis distribution plate

4 Experimental testing

4.1 Experimental setup

The distribution plate was installed between two cooled flanges at the bottom of a fluidized bed. The two flanges had individual cooling systems and where cooled by boiling water at about 95 °C, see Fig 4. The instrumentation involved temperature probes at each side of the plate. For these initial runs the plate was tested without gas flow and beads, this made it possible to gain access to both the windbox and the reactor internals.

One could therefore do thermography imaging of the distribution plate from both sides. There were also Termo couple probes 10 mm outside each side of the plate to monitor the surface temperature realtime. All four zones had individual temperature sensors see Fig 4. The heating of the bed was done by irradiative heating elements from outside the reactor wall.

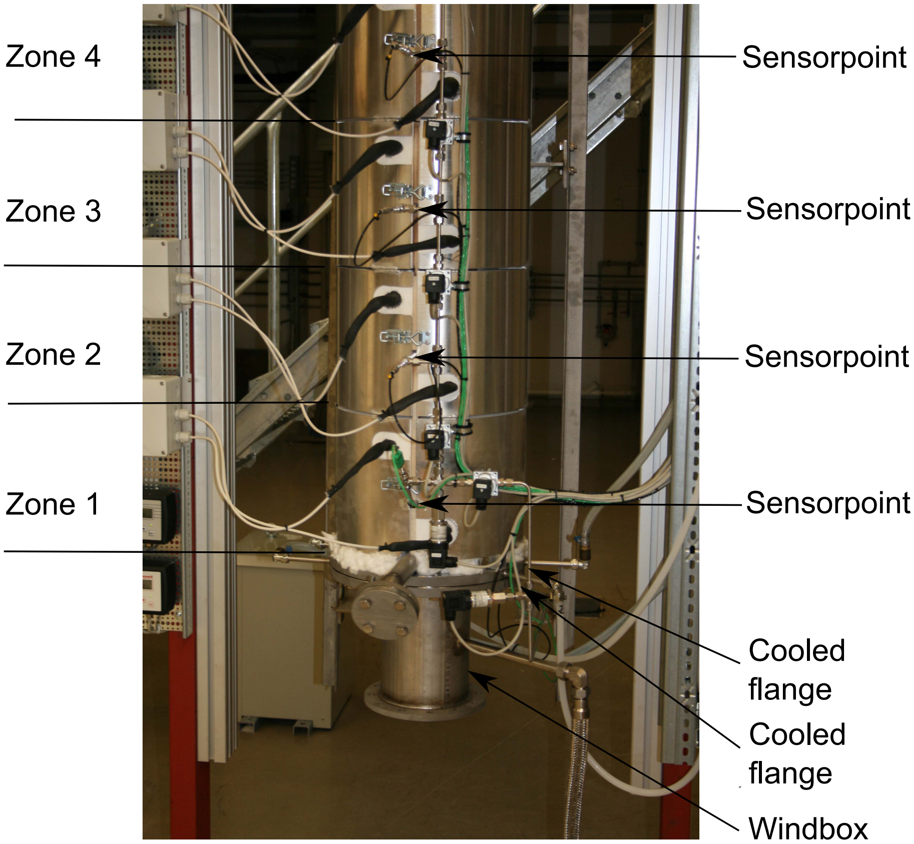

Figure 4: Experimental setup

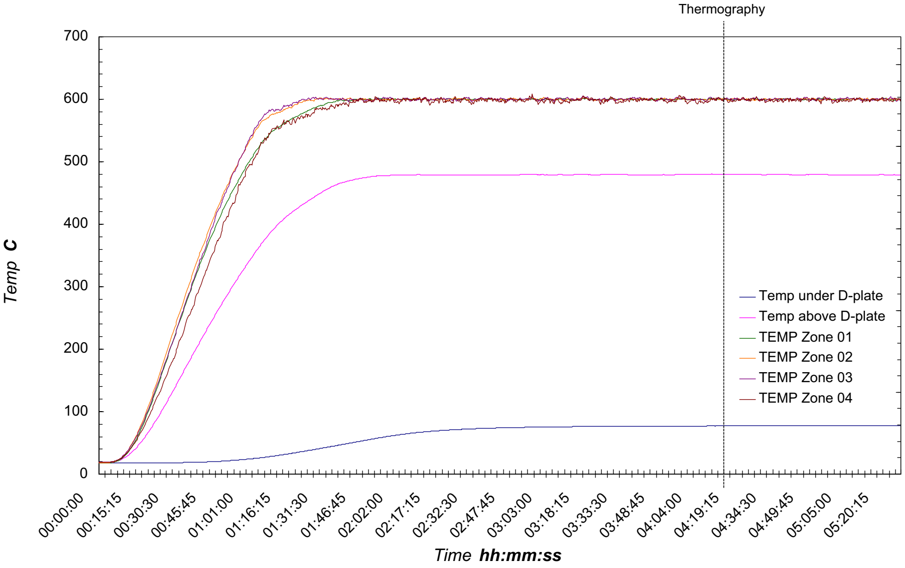

Figure 5: Temperature development

4.2 Results

4.2.1 The individual setups

The heating and cooling system was tested under a number of different configurations to test different heating and cooling loads over the plate. For the initial run the plate was intensively cooled and kept below 15 °C while the temperature above the plate was risen to 178 °C. This initial run was problematic since the thermal losses around the plate was quite severe in addition to insufficient temperatures in the lower regions of the reactor volume.

To aid the problem the flange cooling was reduced for the second run, the inlet temperature of the cooling media was 10 °C while the outlet temperature was 44 °C. This setup resulted in a temperature of 278 °C while the temperature below the plate was about 35 °C. The success in this run was the quite substantial temperature difference on each side of the distribution plate. The problem was however the still insufficient temperatures in the reactor volume in addition to the large temperature difference around the cooling flange.

The third next subsequent and last run was done with water at boiling temperature. The inletflow was reduced to a bare minimum such that the whole cooling flange reservoir reached boiling. At the outlet, the first section was made from a 1,5m vertical section which overflow when the water level got too high. This setup yielded a close to constant temperature around the flange and the extracted heat was removed through evaporation. By using this setup, the temperature just below the baseplate hold a constant temperature of about 78 °C as can be seen in figure 5 while the temperature 10 mm above the distribution plate reached 423 °C. The temperature in zone 1 rose to 600 °C sensor placement may be seen in Fig 4.

4.2.2 Heat distribution

The reactor was heated from room temperature to operation temperature of 600 °C. When all zones reached operating temperature and the temperature over and under the distribution plate reached steady state, the temperature distributions were investigated by thermography measurements as shown in Fig 6.

The CFD model had predicted the heat temperature distribution over the bottom of the baseplate to be close to uniform from the center to the border see Fig 3. The temperature difference through the plate from the bottom to the top was challenging to measure during the tests, but from the CFD analysis this was predicted to be close to constant.

When analysing the bottom of the distribution plate a thermography picture was taken from underneath the wind box see Fig 6. The black shadow in the picture is the gas feed line which is below and colder than the plate. As one may clearly see from the picture, the temperature distribution is close to constant over the plate thus making the results consistent with the CFD findings.

The reactor is equipped with an inspection glass at the top of the reactor, making it possible to investigate with optical instruments. To map the temperature distribution over the top of the plate, a thermography picture was taken from the top of the reactor as seen in Fig 7.

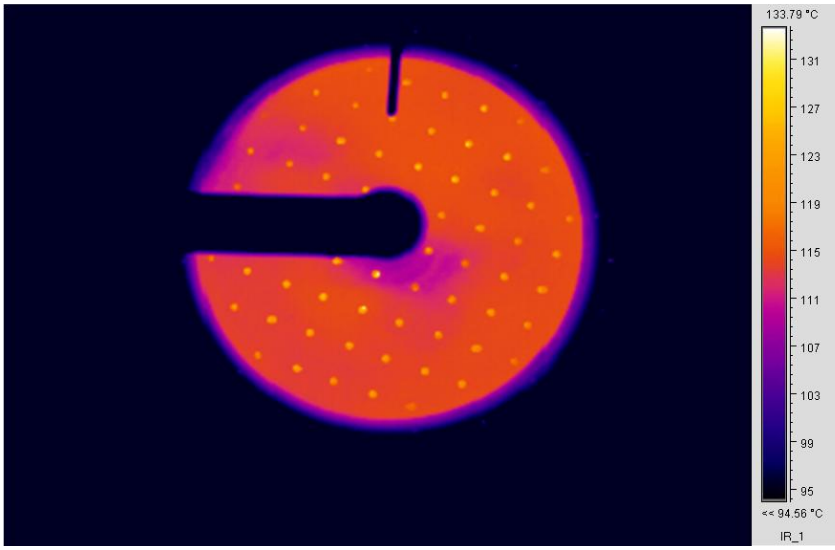

Figure 6: Thermography picture from the bottom of the plate

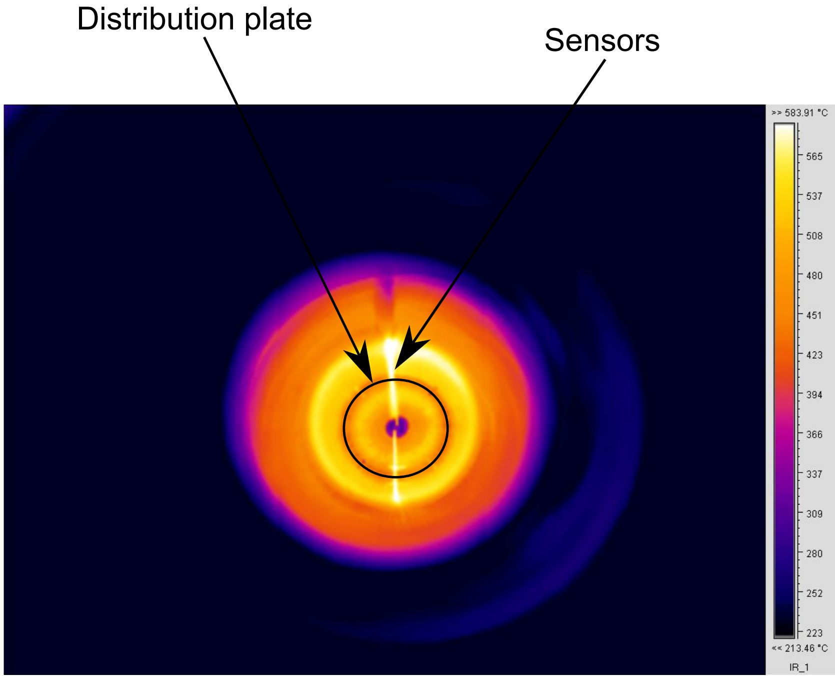

Figure 7: Thermography picture from the top of the plate

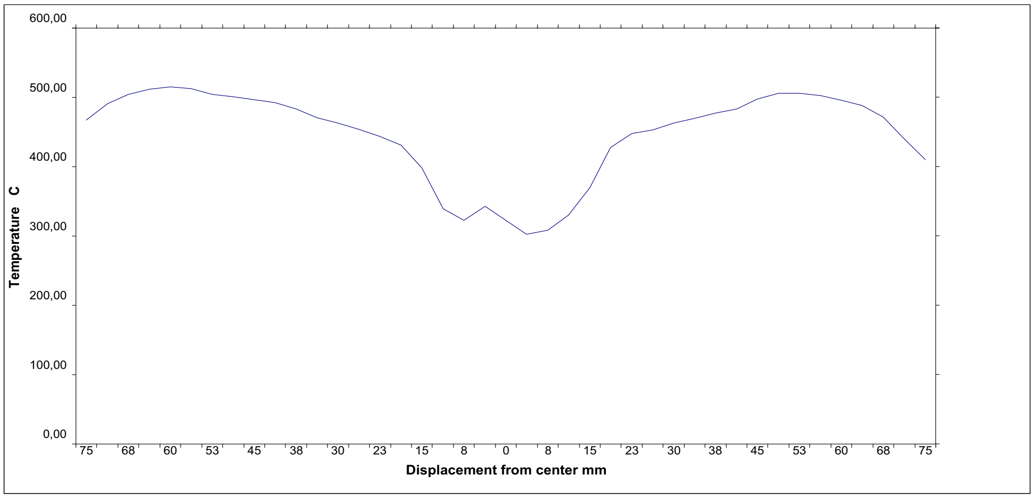

Figure 8: Temperature distribution over the top of the plate, extracted from the thermography raw data

Since the reactor is over 2.5 m tall, the distance to the plate in combinations with the limitations of the camera yields a picture low on details compared to the picture taken from the bottom. Nevertheless, it is clear that the temperature of the distribution plate is lower than the measuring probe just above the distribution plate see Fig 5. This is possible because the gas flow is shut off and thus that the heat transport mechanisms from the plate to the overlying gas is limited. It should in this regards also be noted that the heat transport from the wall to the inside of the reactor is limited since the reactor is lacking particles. When both these mechanisms are operational, one may expect the heat transport to be more efficient and thus a more even temperature distribution than when running the reactor empty. Another feature is that the temperature is low by the wall then going up to a maximum about 30mm from the wall and gradually decreasing until the center of the plate. There seems to be a step in temperature in a 15mm radius around the center of the plate, see Fig 8.

This effect is not fully investigated. However, it might not be physical, but a result of reflection or other optical effects. When doing the initial runs, the bottom of the windbox was untreated and thus shiny. This resulted in a much too low observed temperature as well as the hole pattern from the distribution plate was reflected onto the inner wall of the box, showing a periodic pattern of light coloured spots, thus indicating higher temperature at these spots. The inside of the box was thus coated with a high temperature, black paint in order to control these effects. This resulted in Fig 6 which was in good coherence with probe measurements.

The head-load over the distribution plate is expected to be more uniform when the reactor is filled with beads.

5 CFD model

All runs were done without particles and fluidization gas. There was therefore interest in finding how the temperature would be transferred through the insulation, into the aluminium and further into the flow. The motivation for the exercise was not to model the complete picture, but merely to investigate the heating of the flow.

The commercial software Fluent was used to model the heat transfer. The problem was defined by a finite temperature on each side of the plate and the thermal properties of the materials involved. The fluid mechanical and thermal properties of the fluid was calculated by the commercial software Ergun. The grid was produced in Gambit with a triangular mesh in the solid region and a quadric mesh in the fluid region. The motivation for this difference was the sharp corners of the solid regions which yielded a triangular mesh while the fluid region could be meshed with a quadric mesh for better precision. The interfaces separating the regions were however meshed first in order to define boundary regions and assure a good cell to cell transport of properties. A typical cell size of 2.8 x 10^{-5} mm^2 was used for both solid and fluid regions see Fig 10.

The Reynold number of the problem was in the order of 1.5 x 10^3 and the CFD analysis was thus performed without a turbulence model. To assure no loss of precision and since the case was in the transitional region, some runs were done with a k- ϵ model. There was minimal difference with or without the turbulence model and the presented run was thus made without a turbulence model.

Physicalproperties, gasmixture100 °C

Density = 0.244 kg/m^3
Thermal Conductivity = 0.17571 W/mK
Specific heat = 11962.98 J/Kg K
Molecular weight = 8.03048 g/mol
Viscosity = 1.231 x 10^{-5} Pa S

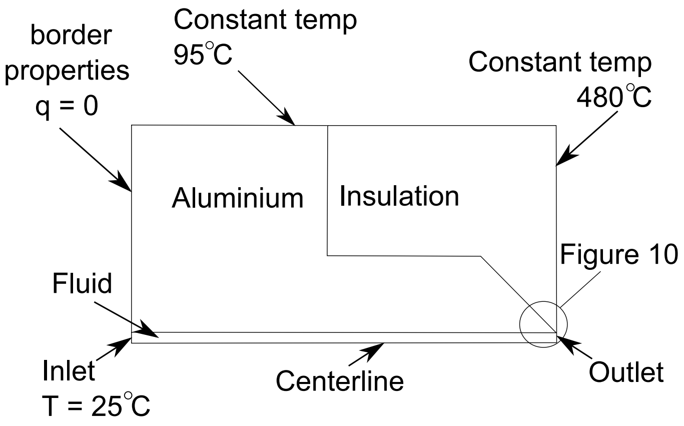

Figure 9: The case

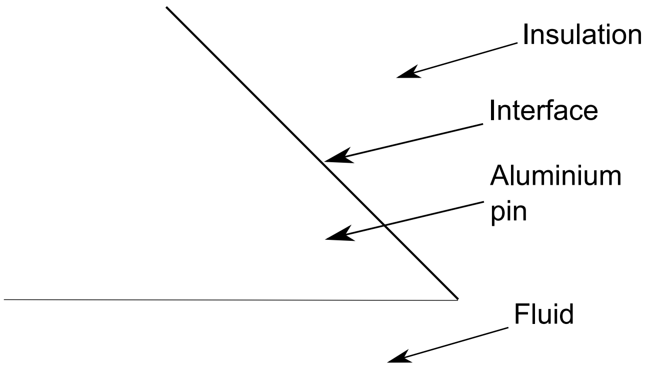

Figure 10: Part of the mesh, section from Fig. 9

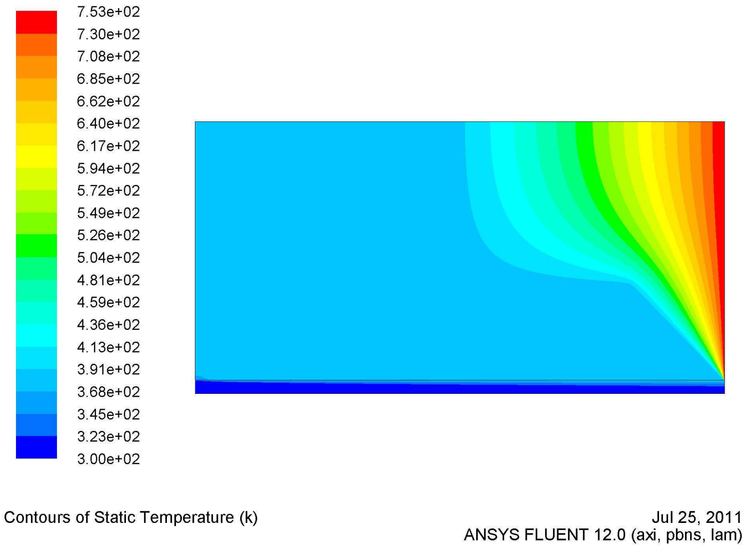

Figure 11: CFD analysis on the distribution plate

The presented design was based on the layout described in Fig 2. This layout minimized the thermal losses and it was possible to keep the reaction gas heating to a minimum. If one took reference in keeping the edge of the baseplate at 95 °C, it was quite easy to keep the entire base sufficiently cold if the design had a sufficient transverse crossection.

As one would expect, there was a thermal boundary layer by the tube wall which grew slowly depending on the velocity. At 80 m/s through a 1mm dia tube the thermal boundary layer would occupy about 30% of the tube radius at the exit, see Fig 11. From these results it is quite clear that the baseplate needs to be maintained below the decomposition temperature in order to avoid decomposition and possible clogging. Another conclusion one might draw from this is that the gas entering the reactor will have quite low temperature if one choose not to heat either the wind box or the supply line. There is therefore important to keep a good mixing of the bottom bed, to keep an efficient heat transfer from the wall to the reactant gas.

6 Conclusion

Using a composite distribution plate consisting of materials with different heat conduction properties was very successful. Earlier researchers have reported problems with too high temperatures in the distribution plate and insufficient cooling when utilizing only flange cooling. Although the experiments done in this article have been executed in a limited manner, there is still sufficient results to draw a conclusion on the applicability of the method. The distribution plate was capable of sustaining a large temperature difference on each side. This gives that the thermal losses over the distribution plate was limited although the wall temperature of the individual holes in the plate was kept sufficiently low.

More empirical results are needed in order to find a design suitable for full scale use. Especially the execution of the individual pins and perhaps a varying pin design over the plate should be investigated. Other designs might include several materials or even gradually varying materials to improve performance.

Another desirable execution would be to utilize materials of anisotropic thermal properties. Such layouts would have a better chance of succeeding when the design process is aided by extensive use of CFD analysis.

One should note that the exercises in this articles have limitations. There are several mechanisms relating to fines generation that needs to be investigated further in order to map out other challenges that might occur. Being able to introduce the reactant gas cold and heating it to decomposition temperature rapidly after insertion, might not be desirable because of other mechanisms that these tests have not been able to discover. The team wishes to continue the research with several tests with different reactant gas concentrations, flow rates and particle sizes in order to study the applicability further. These tests will be aided by CFD modelling in order to increase the cost effectiveness of the research.

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