

DEVELOPMENT OF PARTICLE BREAKDOWN AND ALUMINA STRENGTH DURING CALCINATION

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Abstract

Since the replacement of rotary kilns with stationary calciners, the impact from hydrate properties and calcination technology on the quality of Smelter Grade Alumina (SGA) have been studied frequently. F.L. Smith is studying the complex interaction between calcining conditions, particle breakdown and development of alumina particle strength in both Pilot-scale and industrial calcination units using conventional analytical techniques. The pilot-scale units is simulating the pre-calcination step at 320-380°C, common to all stationary calciners and the final calcination stage at 1070°C in Gas Suspension Calciners without Holding Vessel. The calcining capacity of the full scale units are ranging from 2200-4500 TPD of SGA and cover stationary calciners with and without Holding Vessel.

Representative hydrate samples from more than eight (8) different alumina refineries are calcined and compared.

The first results of the above work will be presented with focus on how the calcining conditions impacts the properties of calcined industrial hydrates with respect to alumina strength and particle breakdown.

Introduction

Since the development of the F.L.Smith Gas Suspension Calciner (GSC) for alumina was initiated back in 1976, the complex interaction between calcining conditions, particle breakdown and development of alumina particle strength in both pilot-scale and later industrial calcination units have been studied. The results of the first pilot-scale study was reported as early as 1982 [1,2] before the first 1000 TPD GSC unit was commissioned at Hindalco, India, in 1986 [3].

At that point in time relatively few stationary calciners were in operation and the major share of world alumina was still produced in rotary kilns. Consequently, the precipitation circuit in very few refineries was designed for producing a hydrate with sufficient strength to withstand the change in calcining conditions from rotary kilns to stationary calciners with respect to particle breakdown.

As time has passed, more and more rotary kilns have been replaced with stationary calciners [4], while new refineries was equipped with stationary calciners, with a few exceptions only [5]. Simultaneously, more [6] and more hydrate precipitation circuits were modified and optimized for producing a hydrate suitable for stationary calciners.

In view of the above development, and the fact that there is still a lot to learn and understand FLS and Alcoa decided to initiate a new hydrate calcination study in 2005 involving eight different alumina refineries equipped with FLS or Alcoa stationary calciners. In doing so, the major objectives were to study and understand the factors responsible for particle breakdown and development of alumina particle strength.

Gas Suspension Calciner Process Flow Sheet

FLS Gas Suspension Calciner comprises four main sections:

- Drying and Pre-heating/Pre-Calcination (PO1,PO2);
- Calcination Furnace (P04) and Furnace Cyclone (P03);
- Heat Recovery from alumina by direct cooling with Air;
- Indirect alumina cooling with water in a Fluidized Bed Cooler.

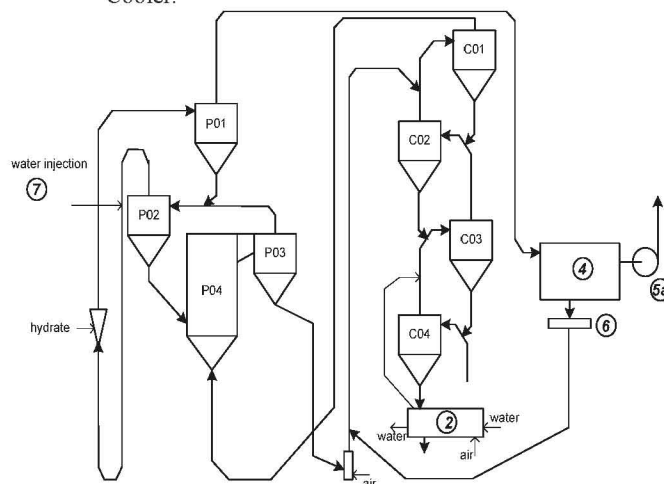


Figure 1. FLS Gas Suspension Calciner (GSC) flow sheet.

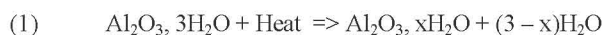
The Drying and Pre-Heating/Pre-Calcination (Hydrate, PO1 & PO2) comprise two stages that are very similar in all calcination flow sheets and dictated by the drying requirement of hydrate with typically 6 – 9% moisture and the thermo chemistry of the calcination process. In the FLS and Alcoa calciner flowsheets, dust from cyclone PO1 enters an ESP (4) or Bag House [4], from where it can be either fully or partially recycled back (dust management) to the calcination process. The dust collected from cyclone PO1 contains both gibbsite and a relatively high fraction of α -alumina phase as pointed out elsewhere [7, 8].

Thermo Chemical Reactions

The overall basic and simplified thermo chemical reactions from alumina hydrate to smelter grade alumina for a GSC is:

Precalcination (P01-P02):

250 – 380°C



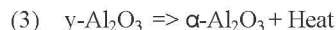
Calcination (P04-P03):

380 – 1075°C

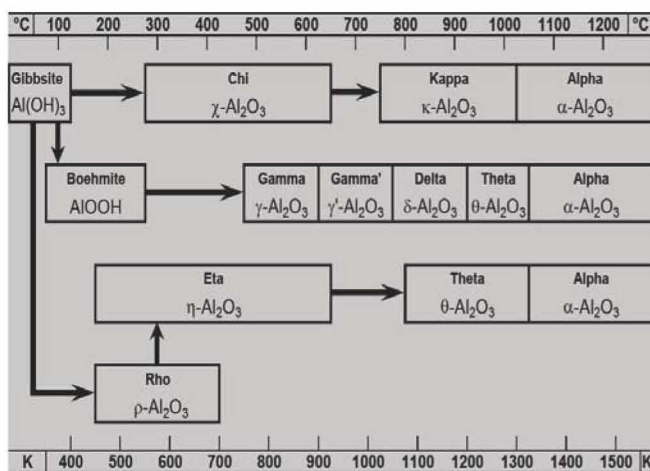


Alpha alumina formation (P04-P03)

1075°C



In the above reactions (1) and (2) the calcination reactions remove water by the rate of heat-transfer to the reaction front of the solid particles. The final reaction (3) is the formation of α -alumina from “ γ -alumina”, which takes place through several intermediate phase changes as illustrated in the below Figure 2.



[L. Perander, et al., AQW paper 2008. Adapted from Wefers and Misra, Alcoa 1987]

Figure 2. Gibbsite Dehydration Pathways to α-alumina.

The physical properties of the alumina changes with the dehydration as indicated in Figure 3 below.

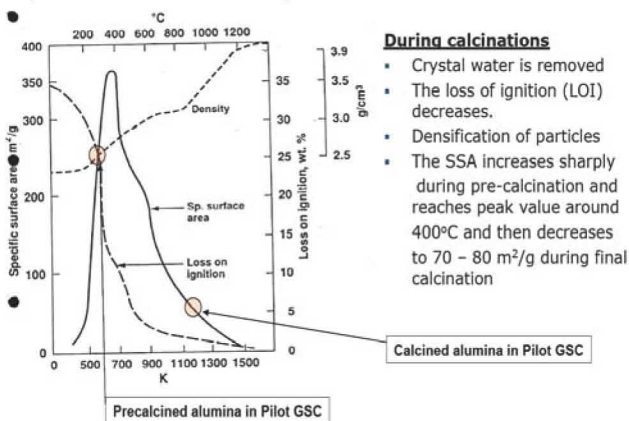


Figure 3. Physical Changes with Gibbsite Dehydration [Alcoa]

The alumina quality obtained from GSC units is typically:

Specific Surface Area (BET):	72–80 m ² /gram
LOI (300–1000°C):	0.69–0.95 Wt. %
Alpha Alumina:	2 - 9% by XRD
Gibbsite:	< 0.1 Wt. %
Attrition Index (±45 μm):	7 - 21 Wt. %
Alumina < 45 μm*:	6 - 10 Wt. %
Alumina < 20 μm*:	0.7 - 2.3 %

*The amount of fines (< 45 micron) and super-fines (<20 microns) depends on the hydrate quality, calciner throughput versus rated capacity and dust management (open or closed loop).

With the availability of more and more advanced solid state analytical methods [7,8] the dehydration pathway from gibbsite to α -alumina phase as well as the development of SGA microstructure and properties have received considerable interest lately [7, 8, 9, 10]. Unfortunately, with one exception [10], all of these investigations use laboratory scale calcining techniques which do not adequately simulate the calcining conditions in industrial calciners with respect to fluid dynamic conditions, heating rates and retention times as closely as to be reported in the present study.

Hydrate and Alumina Test Materials

Representative alumina and hydrate samples from more than eight (8) different alumina refineries are included and the hydrate is calcined and compared.

Thin section microscopy has been used to determine the internal particle hydrate structure as Mosaic, Radial, and Pseudo-Radial structures [1,2].

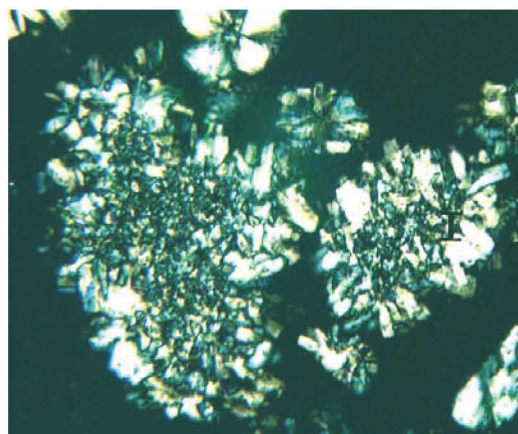


Figure 4. Mosaic Hydrate Structure

The Mosaic particle structure is obtained by agglomeration of small seed hydrate particles to produce a particle with a high concentration of grain boundaries.

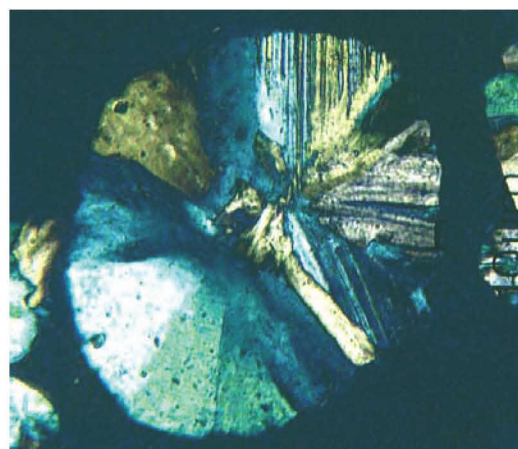


Figure 5. Radial Hydrate Structure

The Radial particle structure is obtained by agglomeration of a few small seed hydrate particles followed by crystal growth to

reach the final particle size with a low concentration of grain boundaries.

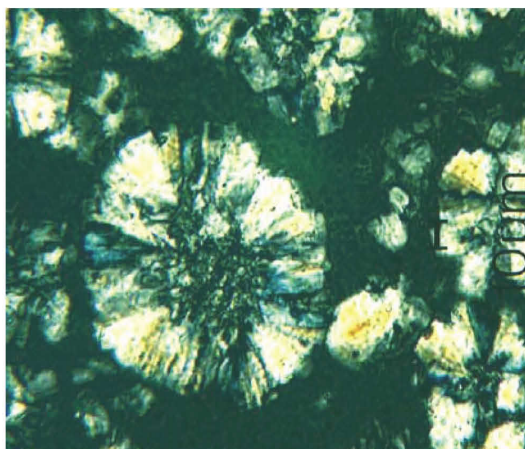


Figure 6. Pseudo-Radial Hydrate

The Pseudo-Radial particle structure is obtained by agglomeration of small seed hydrate particles followed by crystal growth to cement the agglomerated particles together to obtain a medium concentration of grain boundaries.

In quantitative terms, Radial + Pseudo-Radial structure is referred to as Non-Mosaic Structure (NMS).

Simulation of Industrial Size Calciner

The Pilot-scale GSC unit shown in Figure 3 below, simulates the pre-calcination processes between cyclone PO1 discharge and inlet to cyclone PO2 (see Figure 1) and the full calcination process: P01-P04 (see Figure 1).



Figure 7. Test installation of Pilot-scale GSC test.

The test installation comprises an inverted U-shape Gas Suspension Calciner with multiple gas burners along the flow path to control the temperature profile, collecting cyclone, gas fired heat generator, bag house and ID fan.

The typical operating condition for the **pre-calcination** test run was as follows:

- Hydrate feed rate: 5 kg/h
- Average calcining temperature: 360°C
- Gas velocity into cyclone: 10, 15, 24 m/sec
- Average gas velocity in riser duct at 18 m/s: 5 m/sec
- Gas retention time in riser duct at 18 m/s: 1 sec
- Degree of pre-calcination: LOI ≈ 12%, corresponding to outlet of cyclone PO2 (see Figure 1).

The specific surface area (SSA) obtained at pre-calcination in the Pilot-GSC, is seen to have a peak value (240-245 m²/g) like the SSA indicated in Figure 3. In full scale calciners, the SSA in the cyclone PO2 underflow material is somewhat lower (150-180 m²/g) owing to internal fines circulation caused by cyclones having less than 100% collection efficiency. The pre-calcined samples contained Gibbsite and about 5% Boehmite plus a large fraction of X-ray amorphous material, all in line with expectations and previous findings in pilot plant and industrial calciners [2, 11].

The typical operating condition for each **full calcination** test run was as follows:

- Hydrate feed rate: 5 kg/h
- Average calcining temperature: 1075°C
- Gas velocity into cyclone: 12, 15, 18 and 22 m/sec
- Average gas velocity in riser duct at 18 m/s: 5 m/sec
- Gas retention time in riser pipe at 18 m/s: 1 sec
- Degree of calcination: LOI < 0.1%, corresponding to outlet of cyclone PO3-CO4 (see Figure 1).

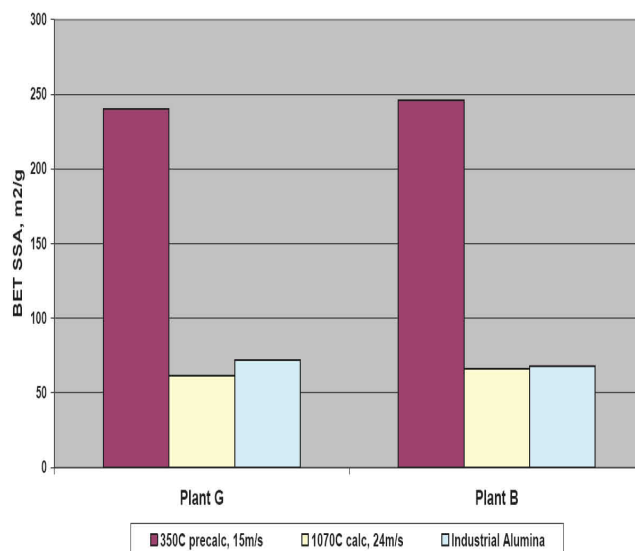


Figure 8. Specific Surface Area versus Calcining Conditions.

The SSA of the fully calcined hydrate is seen to correspond to the SSA in full scale calciners as shown for hydrates from Plant G (72-73 m²/g) and Plant B (68-69 m²/g) respectively. Neither Gibbsite nor Boehmite phase was detected in these samples. With

respect to the α -phase formation, this was about 9 – 20% owing to the distributed gas burner heating needed to maintain the temperature profile in the Pilot-scale GSC.

In general, the same alumina phases are detected by XRD in the pilot and industrial scale calcined samples.

It is therefore concluded that the Pilot-scale GSC is able to simulate industrial scale calciners with respect to degree of calcination (LOI & SSA). However, the heating rates in the Pilot-scale GSC was on purpose somewhat higher than in industrial calciners, because the full calcination is performed in one single stage only.

Development of Alumina Particle Strength

A modified Forsythe-Hertwig Attrition Index (AI) test has been used to measure the particle strength of hydrates and calcined alumina.

The higher the AI value the weaker is the hydrate and alumina particles tested.

The attrition index test is a very rough test with respect to the fluid dynamic conditions when compared to industrial calciners and focuses on one cut point (45 μ m) only of the entire particle size distribution.

However, as the modified Forsythe-Hertwig attrition index test today is a standard in the alumina industry, it has been included in the evaluation. In addition, FLS has found that the test gives highly valuable information about the particle breakdown as shown below.

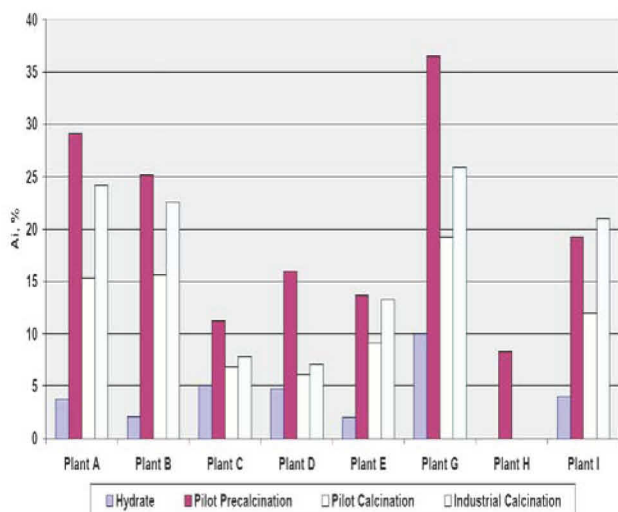


Figure 9. Attrition Index versus Degree of Calcination

As seen for all hydrates tested, the pre-calcined alumina phase formed as per reaction (1) above, is much weaker (higher AI) than its corresponding hydrate. The alumina regains some of its particle strength (lower AI than pre-calcined alumina during final calcination in accordance with reaction (2 & 3) above.

The alumina from industrial calcination plants are seen to weaker (higher AI) than alumina from the Pilot Calcination unit, but this is mainly due to the higher particle breakdown taken place in the Pilot-scale GSC for the samples compared in Figure 9 as discussed below.

Since the pre-calcined material is the feed material to the final calcination furnace in any stationary calciner flowsheet, it must be

handled as gentle as possible until it is fully calcined in order to minimize particle breakdown while gaining its final strength.

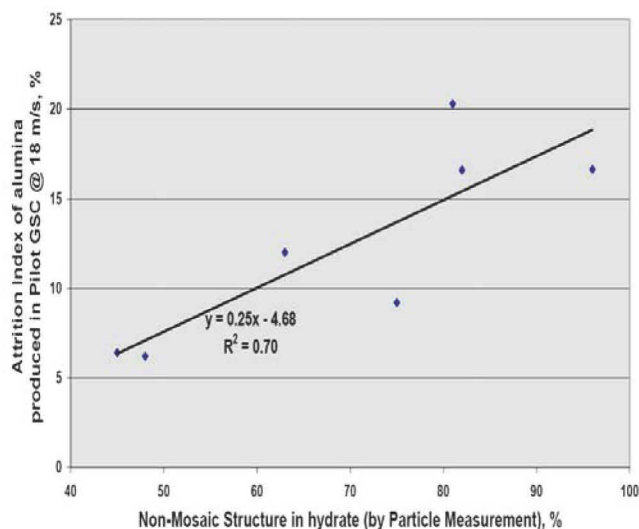


Figure 10. Alumina Attrition Index versus Particle Structure

The weakness of the alumina particle as measure by the AI is seen to increase with increasing content of Non-Mosaic particle structure. With squared correlation coefficient of only 0.70 as seen in Figure 10 above, other factors than the hydrate structure is responsible for obtaining a strong alumina particle after calcination. However, earlier bench-scale investigations have concluded that a high heating rate during calcination [12] produces a relatively stronger particle with lower AI.

Particle Size Distribution (PSD) during Calcination

When hydrate is calcined in the Pilot-scale GSC at different gas velocities, the PSD of the hydrate changes as a result of particle breakdown, taken place during calcination.

In the below Figure 11 it can be seen that if Pilot-scale GSC is operated at a gas velocity between 15-18 m/sec, then the same amount of alumina below the 45 micron particle size will be generated as from a industrial scale Calciner, the column to the very right in Figure 11.

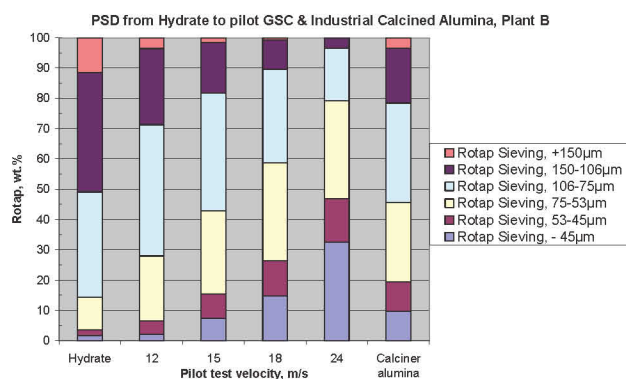


Figure 11. Hydrate and Alumina from Plant B

It is therefore reasonable to conclude that the Pilot-scale GSC is representative with respect to simulating the fluid dynamic condition in industrial calciners.

Comparing the change of PSD of Plant B during calcination with the PSD of Plant E hydrate (see figure 12 below), it can be deduced that Plant E hydrate is more resistant to particle breakdown than Plant B hydrate, because Plant E hydrate can be calcined at 18-24 m/sec in order to breakdown the same amount as Plant B hydrate.

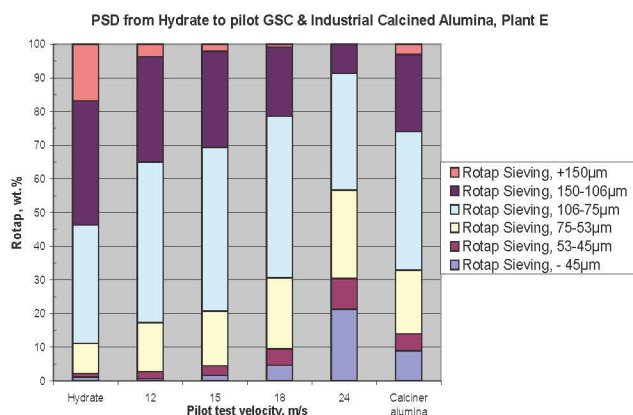


Figure 12. Hydrate and Alumina from Plant E

In general considering the changes in PSD from hydrate to alumina, it is possible to identify whether particle size has an influence on the particle breakdown. It can be seen and concluded that a main contributor of particle breakdown is related to the fraction of coarse hydrate particles (+106µm). The coarse particles are exposed to relative higher centrifugal force on the wall (and have higher kinetic energy) compared to smaller particles of the same velocity which lead to higher particle breakdown.

Particle Breakdown Models

The first generation particle breakdown model developed by FLS was solely based on pilot plant testing in 1980 – 81 [2] and included the below parameters:

- a) Solid Sodium Oxalate occluded in the hydrate particles
- b) Quantity of Non – Mosaic Particle Structure
- c) Attrition Index of the Hydrate and Calcined Alumina
- d) Hydrate particle size distribution

However, recent particle breakdown data from industrial GSC units on different industrial hydrates showed that the first generation model was not fully adequate any more and consequently a revised model is to be developed.

Centrifugal Acceleration Model

When a particle passes the curved riser duct and cyclones (see Figure 13) in the calcination plant, the centrifugal acceleration acting on the particles caused by the swirling gas flow, forces the particles against the wall of the riser ducts and cyclones. The centrifugal forces acting on the particles when they pass a riser duct or cyclone with a characteristic radius of curvature of R at a tangential velocity of U_t is:

$$(4) \text{ PB} \propto \text{Centrifugal Force on particle} = m_p \times U_t^2 / R \propto U_t^2$$

Where:

- m_p = Particle mass $\propto \rho_{\text{particle}} * dp^3$
- ρ_p = Particle density
- dp = Particle diameter
- U_t = Tangential velocity (eg cyclone inlet gas velocity)
- R = Radius of curvature (eg cyclone radius)

Coloured lines indicate gas velocity direction and magnitude in cyclone

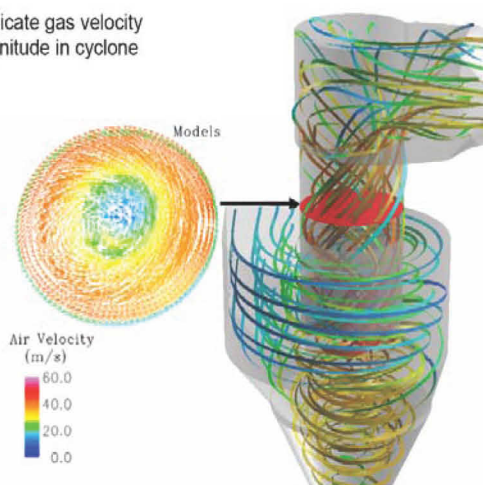


Figure 13. CFD Model of Cyclone

From the above relation one would expect that particle breakdown will increase with:

- > Increasing particle size due to the increased kinetic energy of the particle as observed below; and
- > The gas velocity into the riser ducts and the cyclones raised to the 2nd power;

This suggests a simple 2nd Order Power Law Model with respect to gas velocity. However, the particle breakdown in an industrial GSC is typical about 30-40% of the particle breakdown in a Pilot-scale GSC, when operating at the same gas velocity, which is due to the different size and geometry of the cyclones.

Momentum Model

When the underflow material from cyclone PO2 enters the PO4 vessel by gravity, it is suspended into the incoming gas stream from RPO4 and transported through the PO4 vessel suspended in the upwards flowing gas stream. The necessary force for accelerating the incoming solids almost to the gas velocity is provided by the momentum of the incoming gas stream, which is equal to:

$$(5) \text{ Momentum of gas flow} = M_g \times U_{gi} = A_i \times \rho_g \times U_{gi} \times U_{gi} \propto U_{gi}^2$$

Where:

- M_g = Mass flow of gas
- A_i = Inlet area of cross section
- ρ_g = Gas density
- U_{gi} = Inlet gas velocity

Again, the theoretical forces acting on the particles causing particle breakdown is seen to dependent on the gas velocity squared suggesting a similar 2nd Order Power Law Model as found above.

Agglomerate Breakage Model

Particle breakage studies of agglomerates have correlated the observed attrition with the Weber number (We) defined as [9]:

$$(6) \quad We = \rho_p \times dp \times (U^2/\Gamma) \propto U^2$$

,Where:

We = Weber number – relative attrition rate for agglomerates;

U = Particle velocity;

Γ = The surface energy of the alumina particle – assumed constant for specific hydrate with given particle size distribution;

From eq. (6), it is again observed that increasing the particle size increases the Weber number and thus the attrition of the agglomerate. The reason is that the larger particle has a higher kinetic energy which has to be dispersed upon collision by which attrition takes place. The Weber number model is an empirical 2nd Order Power Law Model similar to the theoretical models found above.

Summarizing, the above three particle breakdown models the particle breakdown (PB) can be described by the following power law equation:

$$(7) \quad PB(U) = \beta + \alpha U^2$$

Where:

U = Characteristic gas velocity – cyclone gas inlet velocity used in this evaluation.

α = Velocity rate factor for particle breakdown for specific hydrate.

β = Characteristic constant, specific for hydrate used to model pilot test results and including other factors than gas velocity.

Particle Breakdown Calculation

The amount of fines (<45 micron on Rotap Sieve and <20 micron on Malvern laser diffractometry) before and after calcination are calculated from the sampling and analysis.

It is well known that there is a difference in measured particle breakdown between the Rotap sieving, and the Malvern Laser PSD Analyzer. The difference has been found to be related to the specific hydrate.

The procedure of the Malvern Laser Analyzer is considered to be very sensitive to a lot of factors including the particle size distribution of hydrate and alumina. Rotap sieving is thus used for evaluating the -45µm particle breakdown.

However, Malvern Laser PSD has been included in this study as information about super fine formation (-20µm) can be estimated using Malvern Laser PSD, which is not possible with Rotap.

To reduce impact on uncertainties on the mass balances during the tests, a simple but stable method is used by comparing the fine fraction sizes of hydrate feed and calcined hydrate:

$$(8) \quad PB(45 \mu m) = X_{Hydrate} \div X_{Alumina}$$

$$(9) \quad PB(20 \mu m) = Y_{Hydrate} \div Y_{Alumina}$$

Where,

X_i = +45µm of hydrate or alumina measured by Rotap Sieve or Malvern Laser PSD; and
 Y_i = +20µm of hydrate or alumina measured by Malvern Laser PSD;

This calculation neglects the amount of dust overflowing from the cyclone, because the cut-size (particle size of which 50% is collected) of the cyclone was in the range 2 – 5 micron, and therefore only a vey small error is introduced.

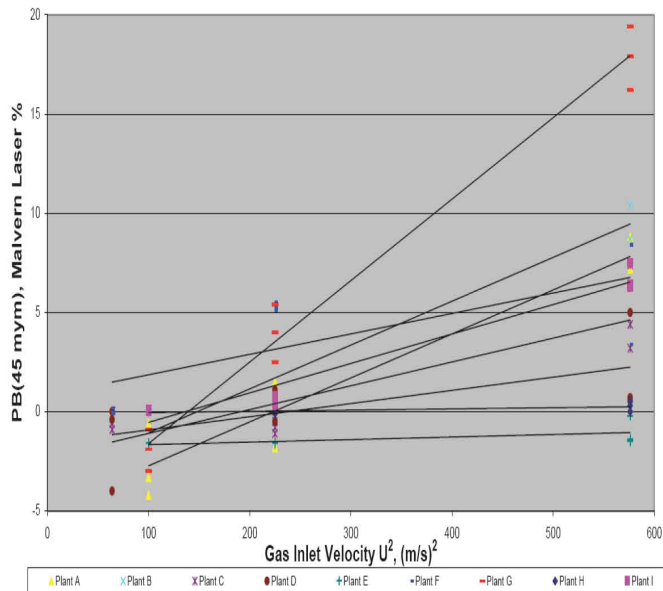


Figure 14. Pre-Calcination Particle Breakdown - 360°C

Figure 14 shows the linear increase of particle breakdown for individual hydrates as a function of the cyclone inlet velocity raised to the 2nd power (U²) at pre-calcination conditions. The linear regression coefficient squared ranges from 0.36 – 0.98 with only one hydrate out of eight having a β value > 0.

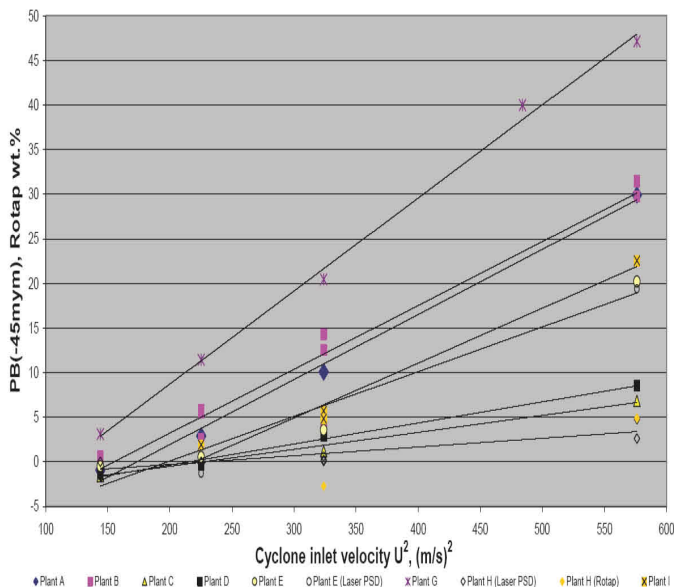


Figure 15. Full Calcination Particle Breakdown - 1075°C

The 1075°C calcination test, shows that the particle breakdown for each individual hydrate tested is a linear function with respect to the cyclone inlet gas velocity raised to the 2nd power (U^2) in the gas velocity range studied. The linear regression coefficient squared was ranging from 0.77 – 1.00, with all β values < 0. It is seen that the particle breakdown is 0 wt.% at about $U^2=150\text{m}^2/\text{s}^2$, i.e., at a velocity of about 12.2 m/s.

In Figure 16 below, the particle breakdown data from the 1075°C calcination test of all the hydrates is plotted versus the attrition index of all the alumina products collected from the test.

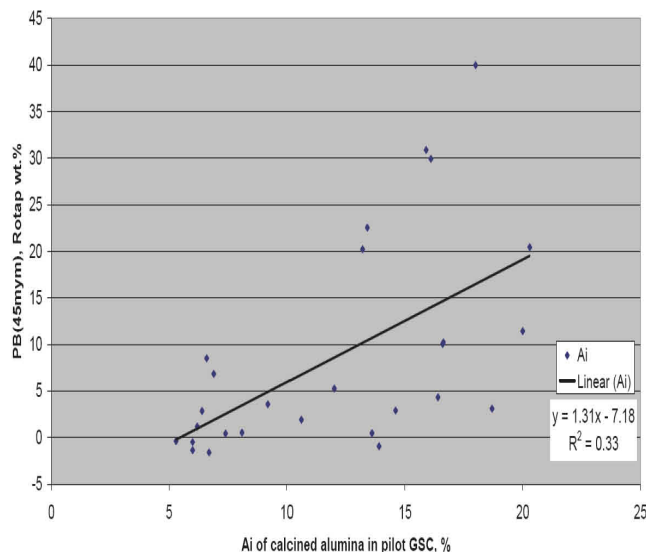


Figure 16. Particle Breakdown vs Alumina Attrition Index

Though a certain trends can be seen, there is no simple correlation between the two parameters [13].

Now, defining a new independent parameter $X = AI_{Alumina} \times U^2$ we find the below empirical curve fit with a squared correlation coefficient of 0.9837:

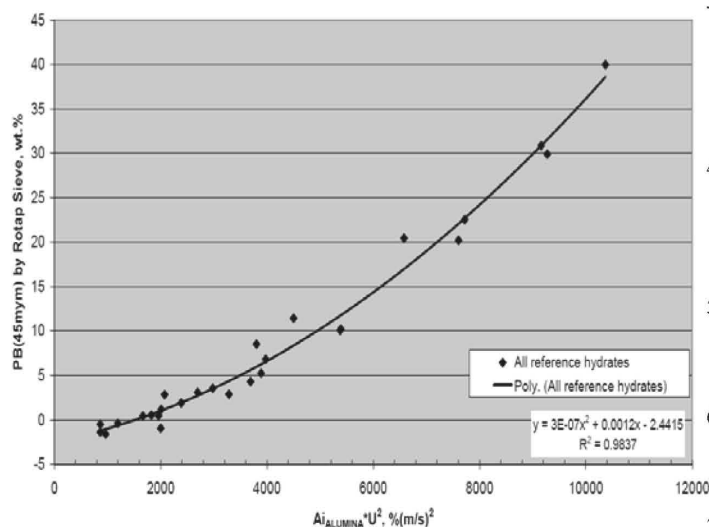


Figure 17. Generic Particle Breakdown Model

Based on the alumina hydrate strength R&D project reported here FLS and Alcoa have discovered a new Generic Particle

Breakdown Model for the Pilot-scale GSC calcination test unit used by FLS. The Generic Particle Breakdown Model has been established on 8 reference hydrates from different alumina refineries and precipitation technologies. The Generic Particle Breakdown Model is based on a material property parameter (attrition index of calcined alumina) and a GSC design parameter (cyclone gas inlet velocity):

$$(10) PB(45\mu\text{m}) = 2.7 \times 10^{-7} \times X^2 + 1.2 \times 10^{-3} \times X - 2.4 \text{ wt.}\%$$

Where,

$$X = AI_{Alumina} \times U^2$$

$AI_{Alumina}$ = Attrition index of calcined alumina, %

U = Pilot cyclone inlet gas velocity, m/s

The reason for the 2nd order equation of the Generic Particle Breakdown Model (eq. (10)) has not yet been identified. However, since it has also been demonstrated that the Pilot scale GSC is representative for the fluid dynamic conditions in an industrial calciner plant, there are strong indications that the Generic Particle Breakdown Model also applies to industrial calciners. A similar model have been obtained for the pre-calcination test conditions with a squared correlation coefficient of 0.9318 and with no residual particle breakdown for $X = 0 \Leftrightarrow U = 0$ m/sec.

Conclusions

The above reported work suggest the following conclusions:

- 1) The Pilot-scale GSC operation is representative for industrial calciner operation with respect to degree of calcination and fluid-dynamic conditions.
- 2) During pre-calcination some boehmite phase is formed, corresponding to the amount found in the underflow of cyclone PO2 in industrial calciners, confirming the gibbsite dehydration pathways already reported many years ago [11].
- 3) The observed particle breakdown at pre-calcination (360°C) and full calcination (1075°C) can be explained by a single parameter $X = AI_{Alumina} \times U^2$, including the Attrition Index of Alumina (particle weakness) and the gas velocity into the cyclone, squared.
- 4) The observed particle breakdown is reduced to ZERO, when the gas velocity into the Pilot-scale cyclone is reduced to below about 12 m/sec, indicating that no measurable particle shrinkage takes place.
- 5) Coarse particles (> 100 micron) are the main source of particles breaking down to less than 45 micron. This has been verified in industrial calciners as well.
- 6) Hydrate over-coarsening is the wrong way to make alumina with maximum 10% < 45 micron because weaker alumina particle results, as previously reported [14].
- 7) Despite higher heating rates in the Pilot-scale GSC compared to industrial calciners, NO explosive shattering of the grains or particles takes place which is of practical significance. This confirms previous experimental findings that such a

mechanism is not causing particle breakdown in industrial calciners either.

In view of the above, calciners can be designed with lower gas velocities to minimize particle breakdown, but this may not result in a strong alumina particle.

Consequently, the real challenge is for the Alumina refiner to produce a hydrate which upon calcination becomes alumina with a low Attrition Index, so that a strong alumina particle is produced. This will minimize attrition or particle breakdown during calcination and transport from the calciner to the smelter, and subsequently when the alumina is used in the dry gas-scrubbing of the smelter off gases [15].

Needles to say this challenge points to the hydrate precipitation stage in the refinery, and the requirement for proper balancing of the many factors at play in precipitation of the hydrate. However, since a very coarse particle size distribution is not the answer a gain in yield may be achieved at the same time.

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