On the control problem in fluid energy milling and air classification processes: approaches for experimentation and modeling of particulate systems in an industrial scale plant.
Proceedings of European Congress of Chemical Engineering (ECCE-6)
Copenhagen, 16-20 September 2007

On the control problem in fluid energy milling and air classification processes: approaches for experimentation and modeling of particulate systems in an industrial scale plant.

L. Coutinho,[a] M. Embiruçu [a]

[a] *Universidade Federal da Bahia*
Escola Politécnica, PPGM - Programa de Pós Graduação em Mecatrônica – Mestrado. Rua Prof. Aristides Novis, 02 – Federação zip code: 40210-910 Salvador-BA. Phones: 55-71-3203-9701 / 55-71-9703 / 55-71-3203-9700
Fax: 55-71-3203-9712. Brazil

Abstract

The objective of this paper is to study the control problem in particulate processes, specifically the opposed air jet milling followed by particle size classification with a forced vortex air classifier. An overview of both processes is given and the control problem is defined, based on inputs, outputs and disturbance variables from a perspective of experimenting and modeling these processes on an industrial scale plant. The multivariable control problem and causality diagrams are presented and experiments to model these plants are described. The interactions existing between milling and classification operations are considered in order to identify a control strategy that enables increased energy efficiency and maintained or improved product quality. Difficulties in performing model identification of particulate processes without a pilot plant available and without on-line particle size measurement are discussed and results from preliminary experimentation are presented.

Keywords: Fluid energy milling, classification, modeling, particulate systems.

1. Introduction

Particulate systems and powder materials are used in many different industries such as pharmaceutical products, food processing, powder metallurgy, painting, mineral processing and chemicals in general. Particle size distribution and energy efficiency are important performance outputs for size reduction and classification processes. Particle size is important to provide quality characteristics for particulate products. For instance, it affects the most different things as the taste of the chocolate and the mechanical resistance of ceramic cutting tools. Particle size is significant for the quality of laser printing and can also modify the absorption of drugs by the human organism. In addition to the quality aspects, it is important to emphasize that size reduction processes are inherently poor in terms of energy efficiency. Many authors have reported that less than one percent of the total energy input is used in the breakage process. In fluid energy milling, where the comminution results principally from particle to particle collisions, most of the energy is consumed in placing particles in motion, and only around 0.1% of the total energy is involved in the breakage reaction (Gommeren, 1997). It is self evident that energy efficiency is probably the most important problem that the industry has faced in the recent past and industrial engineers will continue to face this problem now and for long time in the future.

A considerable effort has been invested by many scientists and powder processing researchers in order to obtain suitable models for this branch of industrial systems. Most of the works describing these systems are based on population balance models and breakage and selection functions (Epstein, 1948; Reid, 1965; Austin, 1971; Dodds, 1996; Dods, 1997; Berthiaux, 1999; Gommeren, 1999). More recently, a stochastic model based on Markov chains theory has been applied to study stirred bead mills and classifiers network (Berthiaux & Dods, 1999; Berthiaux, 2000). Some of the mentioned approaches are often deemed to be very time-consuming and testing intensive when defining and adjusting model parameters related to mechanical properties for the processing particles. It is not very common to find works in open-literature dealing with multiple operating input and outputs variables and very little research has been conducted based on black-box and nonlinear identification methods. In this paper, (1) fine grinding and air classification processes are described from a perspective of experimentation and modeling in an industrial scale plant. (2) Size reduction operation is defined as the combination of both air jet mill and air classification plants. (3) The multivariable control problem and main control objectives are discussed and (4) a detailed analysis on the input-output relationships is presented considering the real-world industrial environment. (5) Difficulties in performing process identification of dynamic particulate systems without a pilot plant available and without on-line particle size measurement are discussed. (6) Finally, the experimentation plan and preliminary results are presented.

2. Process Overview

Powder processing of bulk solids and particulate materials include operations such as size reduction and particle size classification. Size reduction, grinding, or comminution is the operation to reduce the particle size of solids. In most cases, size reduction is based on applying mechanical energy to reduce the particles via compression, collisions or friction to produce particle breakage. As an inherent result of the breakage phenomena, different sizes of particles are generated and it is not possible to define a unique particle size for the output material, but rather a particle size distribution. In the majority of cases of comminution, it is difficult, or even impossible, to control the shape of the size distribution by manipulating process variables. Thus, when size distribution is an imperative property for the particulate, subsequently particle size classification is necessary to separate the coarser particles from the fine fraction to adjust the shape of the size distribution. For the material in study, an excess of fine particles has negative influence on product and the classification process is used to reduce the fine fraction present in the product. The following sessions describe a size reduction plant (fluidized opposed jet mill) and posterior air classification process (forced vortex or rotor air classifier).

2.1 The fluidized bed opposed jet mill

The fluidized bed opposed jet mill is one of the most common fluid energy milling systems. The principle of operation of this mill is based on the conversion of high air pressure from air jet nozzles into kinetic energy for the grinding of particles. The material is accelerated during the expansion of the air and the breakage occurs mainly by mutual collision, friction and impact against internal wall or targets. This type of mill is especially suited for ultra-fine grinding of heat sensitive materials, with very narrow particle size distribution (Gommeren, 1997). Another important aspect of jet milling is the possibility of grinding without risk of contamination from other materials, which is possible because fragmentation is principally accomplished by autogenous comminution or mutual reduction of the particles that are being milled (Wellenkamp, 1999). Due to its high energy consumption it is recommended for high value added powder materials. The basic design configuration of the mill is composed by a chamber, a group of five convergent nozzles, and a set of wheel classifiers, assembled in the top of the chamber. For the milling operation, compressed, cooled and dry air is supplied from centrifugal air compressors, with flow rate from 100 to 300 Nm^3/h, and pressure from 6 to 10 Kgf/cm2. An exhaust system is used to balance internal pressure of the grinder chamber and remove particles from the mill.

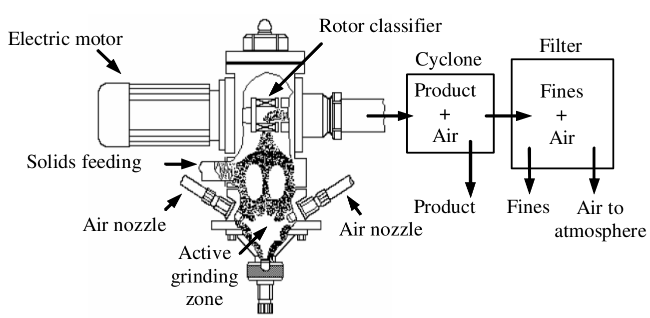


Figure 2.1 - The Fluidized Bed Opposed Jet Mill AFG-100.

Bulk solids to be milled are stored in a loss-in-weight feeder. A rotary valve and pneumatic conveyor are used to transport the material into the grinder. After entering the mill, the particles are entrained and accelerated by several high velocity air jet nozzles that are focused towards the center of the chamber, colliding with other particles, existing targets and internal walls. At the top of the chamber, a rotor classifier separates the particles with specified size that are ready to leave the mill or to return as coarse particles to the grinding zone. The particle size selection results from the force balance between the drag force, produced by the exhaust system, and the centrifugal force, caused by the rotor velocity. The particles of specified size leave the mill mixed on the air stream and pass to the air cyclone that separates the particulate material from the air flow. Jet mill product is collected from the bottom of the cyclone separator, while the air with residual fines particles is directed to the bag filters for final separation.

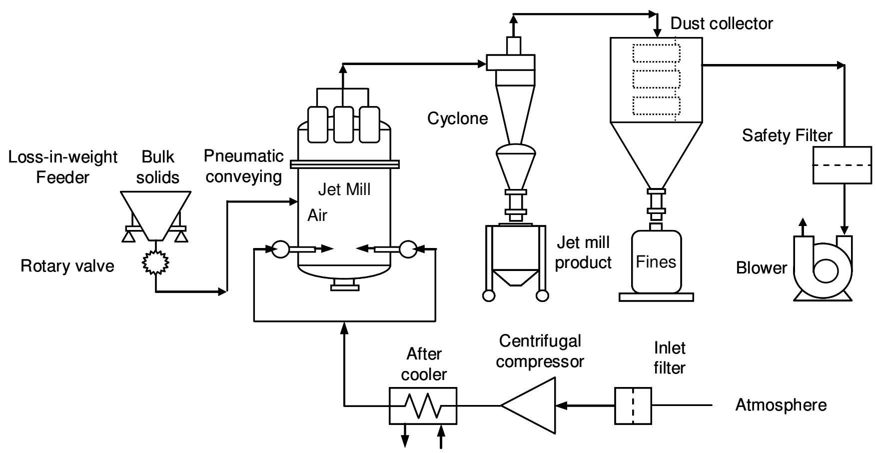


Fig 2.2 - General arrangement of the grinding plant.

2.1.1 Energy efficiency of Air Jet Milling

For the purpose of this work, energy efficiency of air jet milling can be expressed as the specific energy applied to produce a given amount of specified ground product. Jet milling efficiency is affected by many variables related to process parameters, equipment design and material properties. Some of these variables, if not correctly manipulated, may lead to undesirable collateral effects that can increase power consumption or introduce risks of product quality losses. The objective of proposed causality diagram showed in Figure 2.2.1.1 is to describe the complex cause-effect relationship which exists between jet milling energy efficiency and input variables related to process, design and material properties. This diagram takes into account the output of the jet mill plant as the total amount of the coarse product that is collected on the bottom of the air cyclone as ground product. The fines that are collected in the bag filter consist of out of specification material and can not be yielded as accepted product.

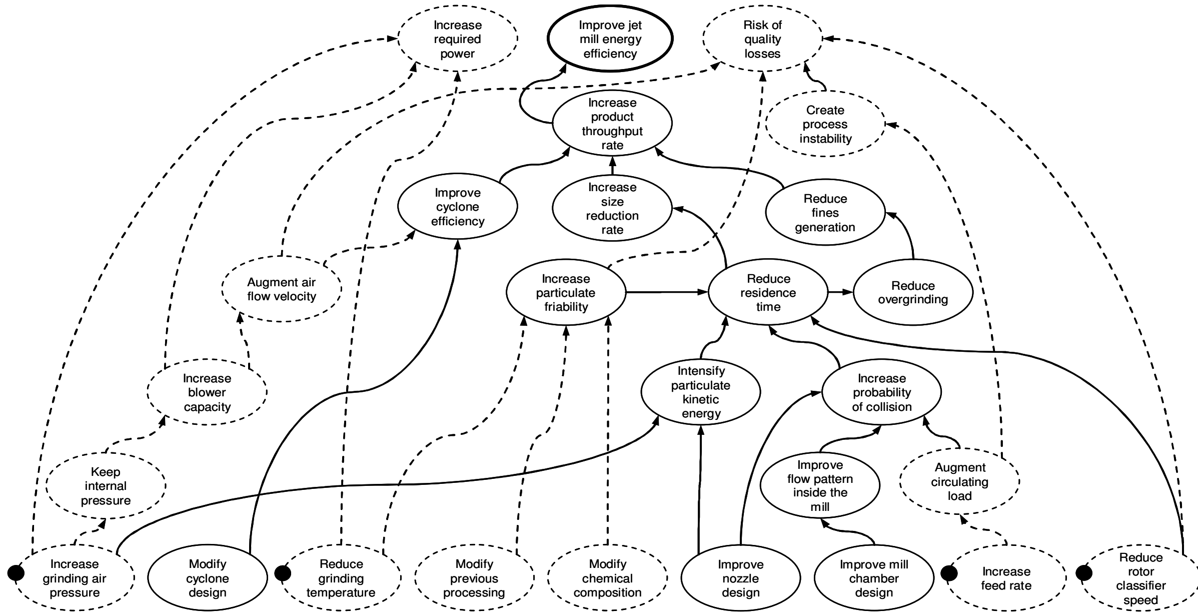


Fig 2.2.1.1 - Multivariable causality diagram for jet mill energy efficiency. (— No opposition between action and main objective; --- potential actions constrained by potential undesirable effects; • manipulated input process variable)

In fact particular, the efficiency of the breakage process is affected by two main drivers: the energy of particle to particle collisions and the probability of particle collision. Accordingly, higher grinding rates can be achieved by increasing collision energy and augmenting the quantity of particles entrained in the air jet streams. The collision energy is related to the velocity of the particles when the collision occurs. Once inside the mill chamber, the particles are entrained in the boundaries of the air jets and targeted by the nozzles to converge at the center of the mill, which is considered the active grinding zone. The majority of the collisions and the more energetic ones occur at the active mill zone. The initial particle velocity is proportional to the velocity of the air jet, which for a given nozzle geometry is increased with grinding air pressure. However, high pressure demands high power consumption and the problem is ultimately one of determining an operation point that delivers optimum energy efficiency, with maximum product throughput rate, and minimum power consumption. Probability of particle to particle collision can also be improved, for instance, by increasing of the circulating load inside the mill chamber. However, the effectiveness of this strategy is limited due to the process instability that can be generated with high feed rates. Increasing of grinding air pressure as well as augmenting of the circulating load are examples of process adjustments that can lead to potential undesirable effects as multivariable causality diagram is showed in the proposed causality diagram.

2.2 The Acucut® Air Classifier

Air classification is a solid-gas separation method used to classify particulates according to their size. The air classifier used in the plant is an Acucut® C-24 classifier, a type of high energy forced vortex air classifier, manufactured by Hosokawa Micron Powders Inc. An overview of several modern air classification devices, their operation principles, features, processing and performance parameters was given by Shapiro & Galperin, 2005, whom also refers to the Hosokawa classifiers as blade classifiers. The classifier and the classification plant are shown in Figures 2.2.1 and 2.2.2.

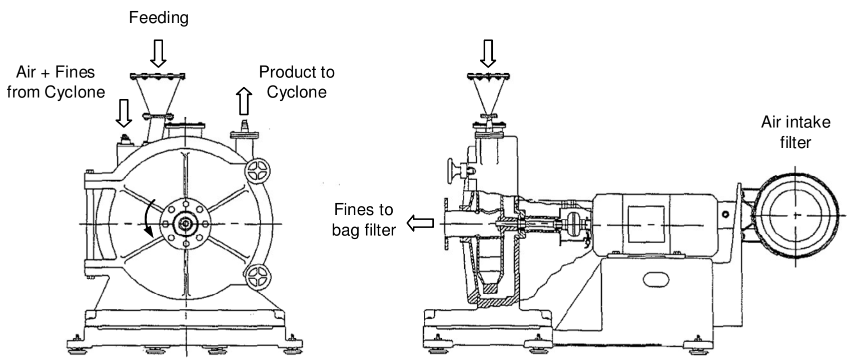


Figure 2.2.1 - Frontal and side view of a Donaldson Acucut® Air Classifier – Hosokawa Micron Inc.

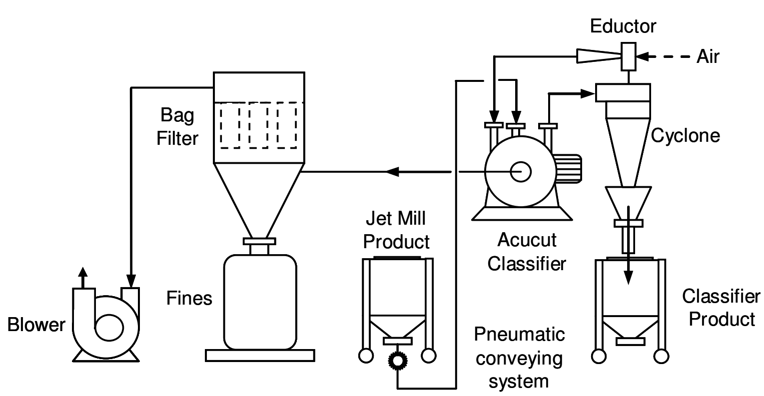


Figure 2.2.2 – Classification plant

Its principle was patented by Lapple in 1970 (Lapple, 1970; Lapple & Shaller, 1971). This type of classifier is also referred to as centrifugal countercurrent classifier. A model for performance of countercurrent classifiers and a phenomenological description of them was presented by Wang & others, 1998. Particle size classification plants are comprised of a pneumatic conveying system for feeding ground material, a cyclone to collect coarse product, a bag filter to collect the fine fraction, and an exhaust system as the source of the air flow for the plant.

As shown in Figure 2.2.3, the air enters the classification equipment from an inlet filter located on the rear of the classifier, passes through a flow meter and enters the classifier housing. Once inside the housing, the air flows into the rotor by a very narrow annular space between outside diameter of the rotor and inside diameter of the stator. This is the pre-classification area, also referred to as the high dispersion zone (Allen, 1990). This design provides the dense particle medium to be dispersed to enable posterior particle size classification. Inside the rotor, smaller particles are dragged by the air flow and moved radially inward to the central outlet. Larger particles and soft agglomerates are moved towards the stator until they reach the coarse outlet. Smaller particles, considered as fine fraction, are carried out with the air and to a bag filter, which is used to separate the air from the fines and collect the particulate material. Fines are further recycled as part of the raw material in the first step of the product processing The coarse fraction that was directed to the coarse outlet flows to a cyclone separator where the classified product is collected through the bottom of the cyclone. The vortex tube of the cyclone is the outlet for rejected fine fraction that returns to the classifier housing to be reclassified.

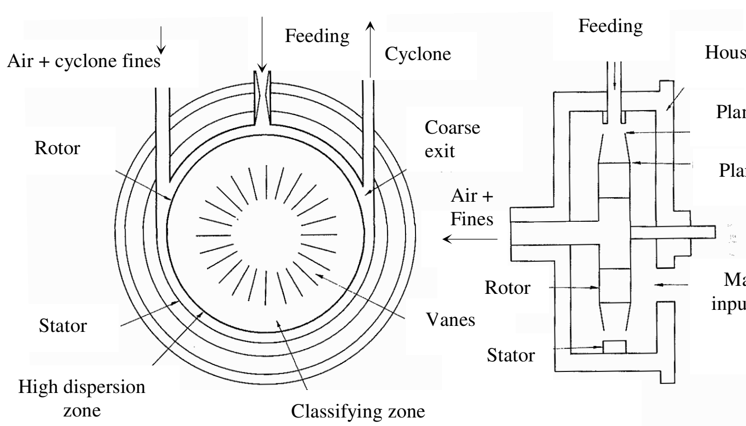
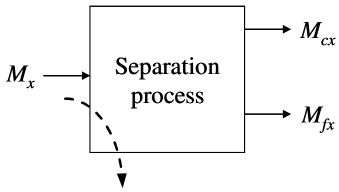


Figure 2.2.3 - Donaldson Acucut® Air Classifier

2.2.1 Classification process performance

The performance of a classification process can be expressed by different methods. Grade efficiency analysis is the most common approach to assess separation efficiency (Allen, 1990), (Gommeren, 1997). A summary of the grade efficiency method is given in Figure 2.2.1.1.



$$M = M_c + M_f \qquad (2.2.1.1)$$

$$M_x \cdot \frac{dF(x)}{dx} = M_c \cdot \frac{dF_c(x)}{dx} + M_f \cdot \frac{dF_f(x)}{dx} \qquad (2.2.1.2)$$

$$E_c = \frac{Mc}{M} \qquad (2.2.1.3)$$

$$G_c(x) = \frac{M_{cx}}{M_x} \qquad (2.2.1.4)$$

$$G_c(x) = \left[M_c \cdot \frac{dF_c(x)}{dx} \right] \Big/ \left[M_f \cdot \frac{dF_f(x)}{dx} \right] \qquad (2.2.1.5)$$

$$G_c(x) = E_c \cdot \frac{dF_c(x)}{dF(x)} \qquad (2.2.1.6)$$

Figure 2.2.1.1 Grade efficiency method for a single separation process

The mass balance and grade efficiency can be expressed by the equations 2.2.1.2 to 2.2.1.6, where:

- M is mass of feed material, M_c is the mass of coarse product and M_f is the mass of fine product.
- The subscript index x in M, M_c *and* M_f is used to designate theses quantities for particles with size x.
- $F(x)$ is the cumulative distribution for the particles with size x in feed material.
- $F_c(x)$ is the cumulative distribution for the particles with size x in the coarse product.
- $dF_c(x)/dF(x)$ is the relative fraction of particles with size x recovered in the coarse product.
- E_c is the total mass efficiency for the coarse product.
  - $G_c(x)$ is the grade efficiency which describes how efficient a given particle with size x is recovered from the feed material and collected in the coarse product.

Grade efficiency described in this way assumes perfectly dispersed particles in the air and no dead flux or by-pass, which is represented by a fraction of feed material that passes throughout the classifier without separation. In a theoretical case of total by pass, there is no separation and the product is collected after classification with virtually the same particle size distribution. Agglomeration and poorly dispersed particulate in classification zone are possible causes for increased dead flux.

3 The multivariable control problem for the extended size reduction operation.

For the objective of this work, the extended size reduction is defined as the operation that comprises both grinding and classification plants. The main control objective for the size reduction plant is not quite different from any other type of industrial process: improve or maintain product quality and reduce manufacturing cost. In spite of the physical arrangement of jet milling and classification equipments as stand alone operations in the size reduction plant, it is not possible to meet main control objectives unless considering them as an integrated plant. In fact, quality and cost of size reduction product depends not only on each plant as stand alone operations but on the interaction of these processes. This section describes the control objective and the control problem for each process and the reasons why they must be studied as an integral operation.

In fact, most of the open literature on the related control problem, as described by many authors, deals with pilot plants or stand alone equipment. Regardless of the quality of the proposed models, constructed upon the pilot plant control problem, many of the noise factors were not taken into account accordingly. A realistic discussion on the difficulties of implementing particle size control in particulate processes is found in Heiskanen, 1995. This paper, issued in the middle 90's, refers to certain reasons why particle size was not utilized as the control objective:

- Lack of proper models for the processes relating product size to input variables
- Lack of on-line particle size measuring methods
- Need for sophisticated control systems
- Need for cheap computing power
- Lack of "property functions"
- Lack of control actuators (input variables) to allow increased state space dimensions
- Increased system complexity
- Increased research effort involved.

Some of these reasons remain unsolved until today, and others, especially those referring to technology constraints were solved during the last decade. Not withstanding the advances in on-line particle size measurement and the current availability of computational power at decreasing costs, the physics of the milling and classification processes, certainly the kernel of the problem is still a complicated question to be solved, mainly because of the lack of proper phenomenological equations relating process input variables to product particle size. In this case, when the physics of the process is not sufficiently known, it is preferable to use system identification techniques to describe process behavior in terms of cause and effect relationships. Such models, also designated as black-box models, or empirical models, can be obtained from process plant experimentation, looking at the system as a set of input and output variables empirically correlated. In this case, previous knowledge about the identified system is required, but the phenomena occurring inside the black-box are less relevant than the effects that they produce. Nevertheless, it is not very common to find works in available literature dealing with multiple inputs and output variables (MIMO) in grinding and air classification operations, and only very little research has been published based on black-boxes and nonlinear identification methods.

3.1 The opposed jet mill problem

Comparing all industrial grinding processes, air jet milling is the most energy inefficient as virtually all energy supplied to a jet mill is being dissipated in the turbulent flow that leads to collisions needed for particle breakage (Gommeren, 1997). In a typical grinding plant with opposed jet mills most of the energy is applied to air compression necessary to place particles in motion inside the mill and the variable resultant cost of production is strongly influenced by the energy cost. The quality of the ground product depends principally on its particle size distribution (PSD) that is affected by process input variables, mainly the wheel classifiers speed, grinding air pressure and particulate feed rate. Suitable combination of process input variables must be pursued to improve energy efficiency and deliver ground products with the desired particle size distribution.

After describing the fundamental aspects of the fluidized grinder and rotor classifier, it is possible to initiate a description of the inputs-outputs variables. The following diagram in Figure 3.1.1 represents the real jet milling process as a black-box, with some of the multiple inputs and outputs that are measured.

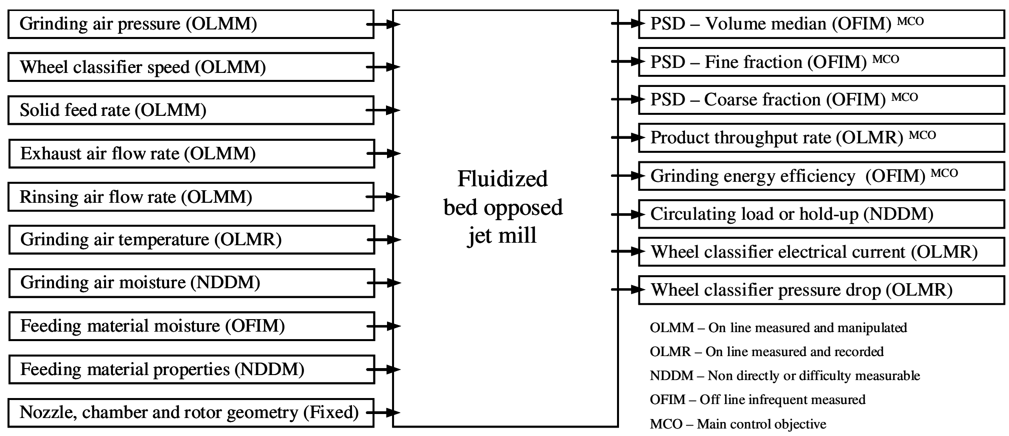


Figure 3.1.1 - The fluidized opposed jet mill as a black box: Inputs, outputs and available data acquisition.

Considering that identification problem was developed in a real industrial plant, it was necessary to consider possible constraints regarding process instrumentation availability. Each process variable was designated in terms of available instrumentation, including sensors and actuators, sampling or measurement interval, electronic data acquisition and recording. Accordingly, this work was instrumental in enabling further experimentation and visualizes instrumentation opportunities and constraints.

3.2 The forced vortex air classification problem

The principle of operation of the Donaldson Acucut® classifier was presented in the earlier section dedicated to the classification process overview. The phenomenon of fluid classification which occurs in this type of classifier is based on the force balance between the drag force, provided by the air exhaustion system, and centrifugal force that comes from the revolving classifier rotor. As mentioned before, the classification objective is to remove the excess of fine particles, or undersized particles, from the ground material. Hence, product collection efficiency is dependent on the particle size distribution that comes from the grinding process. For a given maximum allowable fine fraction, product collection will decrease with the amount of fines in the feed material. Additionally, due to several stochastic and operational factors, such as flow turbulences, air to particle concentration, particle to particle collisions and particulate agglomeration, some fines get into the coarse fraction and vice versa, which leads to a separation process that is not totally efficient. Therefore, product collection efficiency of particle classification is a combination of two factors:

i) An external factor, which is the size distribution of the ground material, and
ii) The separation efficiency of the classification process itself.

The energy efficiency for classification is also important, but not as critical as in air jet mill plant. The power usage of the classification plant is around 20% of the grinding plant power. Subsequently, the control problem for classifier system relies more on product quality aspects, which has direct influence on the product collection efficiency and consequently on the processing costs. Given the explanation above, improving the classification process can be achieved by increasing the coarse product collection rate, with a maximum allowable percentage of fines in the classified product, which can not be accomplished with grinding and classification process as stand alone operations. Because comminution inside the classifiers is fairly inexistent, the volume median x_{50} is determined mainly by the grinding operation, even though a slight increase in x_{50} can be observed with the reduction of the fines content. On the other hand, fines content for the final classified product is a function of the classification process only, since the extra amount of fines generated in the grinding process can be theoretically removed, regardless of the yield of the process. Thus, the higher the fines content in the product, the poorer is the classification process in terms of productivity and the higher is the processing cost, given the rejected fine fraction.

3.2.1 Input-output variables in classification process

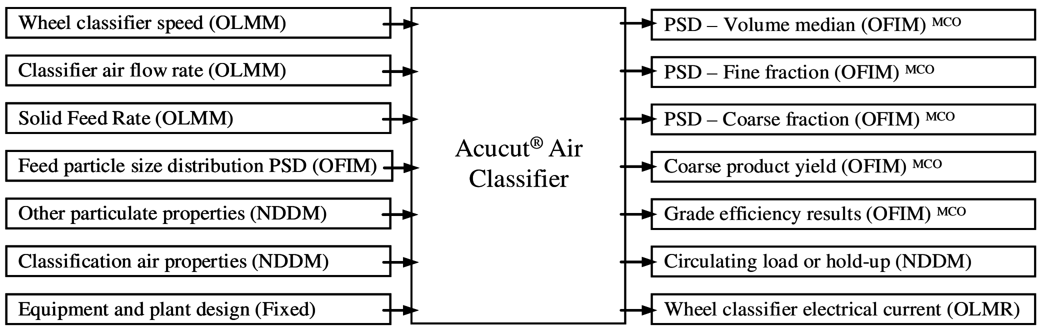
It is possible to define input variables for classification processes in many different ways, depending on the scale from which the particle-to-gas separation is observed. The table 3.2.1.1 provides a summary of different input variables to be concerned when analyzing particle size air classification, even for static or dynamic modeling. A similar structure applied for the analysis of the comminution problem was presented in Gommeren, 1997. Input variables for the particle level were defined based on the variables of influence for the resistive force against particle movement defined in Massarani, 2005.

Table 3.2.1.1 – Problem scales for the concerned input variables in the forced vortex air classification process.

Problem scale	**Concerned input conditions and variables**	**Variable type**
Plant	Particulate feed system: lay-out, mass flow regime, metering devices.	Design
	Feeding system operational parameters	Control
	Classification plant lay-out (number of classifiers, arrangement, etc)	Design
Classifier	Rotor and stator design (diameter, number of blades, blade profile, dispersion zone)	Design
	Inlets and outlets position and dimensions	Design
	Classifier air flow rate	Control
	Wheel speed	Control
	Particulate feed regime and mass flow rate	Control

Particle	Fluid to particle interaction	Material
	Particle size and shape	Material
	Velocity field without the influence of the particles	Design / Control
	Physical boundaries	Design
	Particle to particle interaction (surface properties, particle tribology)	Material
	Air properties (density, viscosity, thermodynamic properties)	Material / Control

Some of the variables in Table 3.1 are inputs or disturbances in relation to the control problem and others are fixed and dependent on the equipment or plant design. For the experimental work in the classification plant the following black-box diagram was considered.



OLMM – On line measured and manipulated
OLMR – On line measured and recorded
NDDM – Noise variable or not directly and difficulty measurable.
OFIM – Off line infrequent measured
MCO – Main control objective

Figure 7 - The Acucut Air Classifier as a black box: Inputs, outputs and available data acquisition

3.3 Control objectives

3.3.1 Product quality

A powder generally consists of a collection of particles with different sizes. Particle size distribution (PSD) describes the frequency distribution of the sizes that are present in a particulate sample. A complete discussion on particle size measurement is presented in (Allen, 1990). In order to represent PSD it is common to use density distributions and cumulative distributions. The distribution of density q(x), expresses the amount of particles in each size class, in relation to the total amount of particles – either by volume, mass or number. The cumulative distribution Q(x) describes the percentage of material in which size is smaller than the upper limit, or top size, of each class interval. Mathematically Q(x) is an integral of q(x), being represented by the S curve in the Figure 3.1.1.1. These curves are widely used in the industry because they enable a fast comparison of PSD, in both qualitative and quantitative perspectives (Gommeren, 1997). The graphs below show the PSD of a particulate material after grinding and after air classification. When visualized with the aid of a scanning electron microscope, samples of material that were measured are given in Figure 3.1.1.1. The visual appearance of the ground material confirms measurement results shown in the distribution graphs. Notice the large population of fine particles in the illustration at left and the narrow distribution exhibited at right. In fact, the classifier reduces the amount of fine particles represented by the fraction at the low end of the distribution.

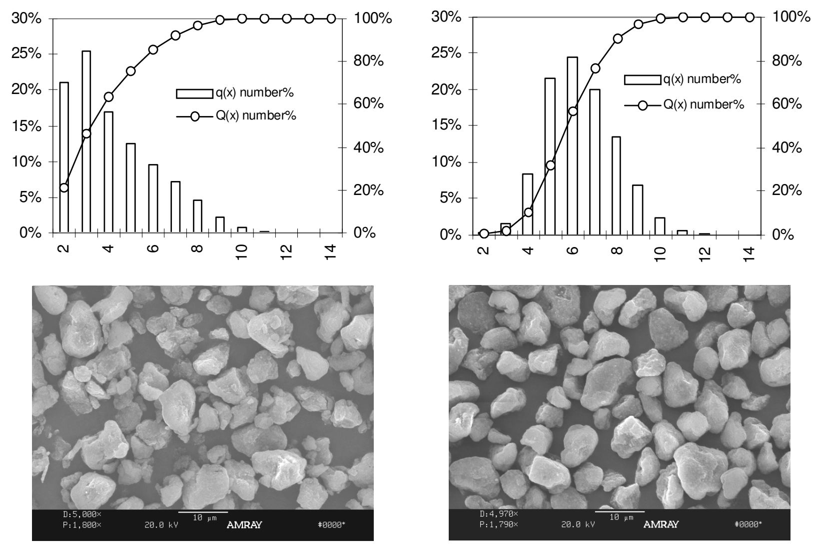


Figure 3.1.1.1 – Size distribution and of the material after the grinding process (left) and after air classification (right).
(Scanning electron microscope AMRAY®, magnification 5.000X)

In several instances, measures of central tendency and statistical dispersion are quite sufficient to concisely describe a typical PSD encountered after milling and classification processes. Hence, in spite of considering the entire distribution as one unique output variable, what is obviously more complicated, specific statistical metrics are utilized to represent PSD. The following metrics are used to characterize PSD, and thus product quality, for the particulate material in reference:

- **Median** (x_{50}) **-** It is a central tendency measure given by the particle size that divides the area under the distribution curve into equal parts. When the distribution is calculated by volume, 50% of the sample volume is composed by particles larger than the volume median and the other 50% contains particles smaller than the volume median. Considering all particles have the same density, the volume median is the same as the median by weight. In cases where the distribution is calculated by number, 50% of the counted particles are composed of particles larger than the number median and the other 50% contains particles smaller than the number median.
- **Maximum coarse fraction** (M_c) **–** The functional performance of a particulate material can be also influenced by the amount of particles larger than a reference size. Similar to the median, the coarse fraction can be expressed as the percentage of particulate within a specific size range (x_{uc} - x_{lc}), either by volume or number, which depends on the product requirements. Particles larger than an upper limit

level (x_{uc}) are not considered as coarse fraction but as individual large particles, which are measured in parts per million or individual counting.

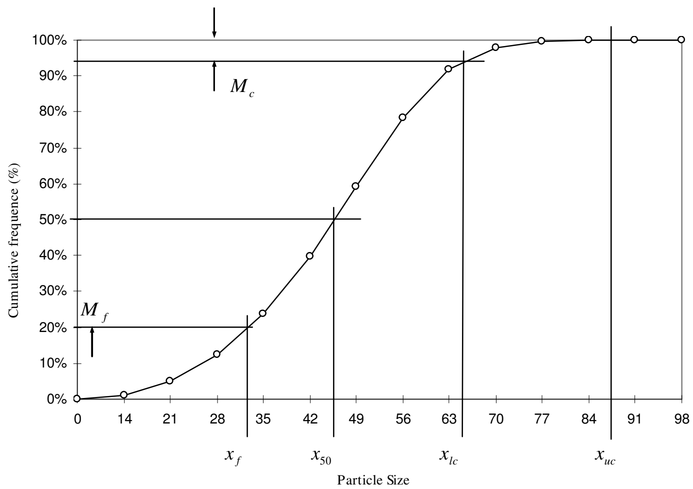


Figure 3.1.1.2 - Cumulative frequency distribution - PSD parameters used to express product quality.

- **Maximum fine fraction** (M_f) **–** Represents the excess of very fine particles in the product, smaller than a reference diameter, which may have a negative influence on product quality. As mentioned early in this paper, the fine fraction is an intrinsic output of the breakage process, and one of the objectives of this work is to explore certain possibilities to reduce the total amount of fines in the ground product without reducing material yield. An air classifier is used to reduce the amount of fines incoming from the grinding process, which results in material losses because of the excess of fines generated during the particles breakage. The Figure 3.1.1.2 illustrates PSD parameters taken as quality characteristics for the product in reference.

3.3.2 Production rate, energy consumption and cost of operation in the extended size reduction plant.

The cost of production in industrial activities comprises different factors such as energy, labor, taxation and the cost of added services. For the scope of this paper, the cost of operation in the extended size reduction plant is represented by the electrical energy consumed for the following equipments:

- Grinding air compressor and dryers, EGA.
- Jet mill exhaust and fines collection system, EGF.
- Classifier fines collection and exhaust system, ECF.
- Jet mill ATP classifier motor, EGC.
- Acucut® classifier motor, ECC.

Considering that the fine fraction can not be used as final product, due to quality requirements described in the previous section, size reduction product is only the coarse product collected at the end of the process, which is represented in Figure 2.4 as the classifier product. Accordingly, it is possible to define cost of the extended size reduction by means of the specific energy consumed in the plant to produce a certain amount of classifier product with a specified PSD. As explained in the section dedicated to the classifier efficiency, the amount of coarse particles recovered in the coarse product depends on the total mass efficiency of the separation process and also depends on the separation efficiency of particles with size x, as expressed by the equation 2.2.1.6. In fact, the extended size reduction plant comprises several classification processes, as represented in Figure 3.3.2.1. Therefore, the production rate is a function of the jet mill rate but is also affected by the grade efficiency of the ATP classifier, jet mill cyclone, Acucut® classifier and coarse product cyclone.

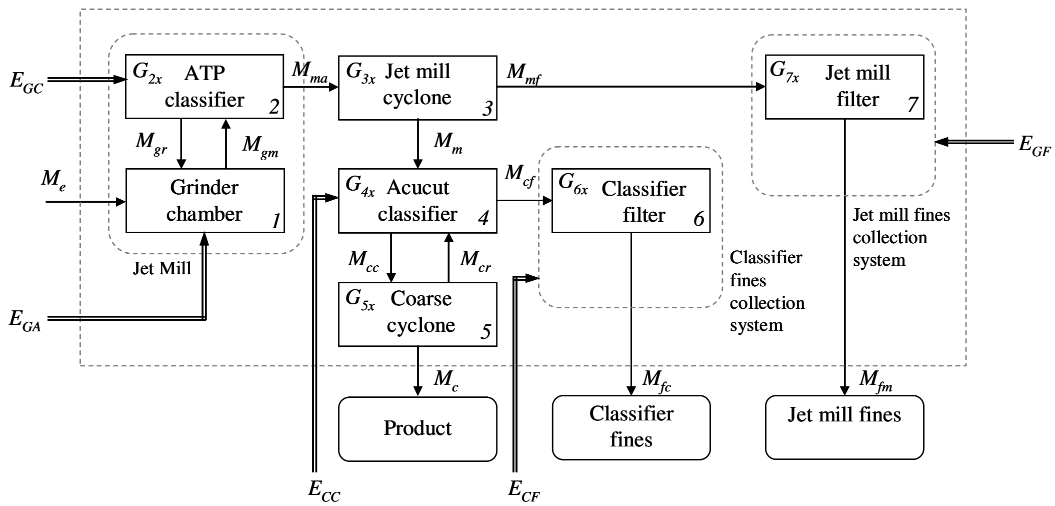


Figure 3.3.2.1 – Energy efficiency of the extended size reduction plant.

4 Experiments with an industrial size reduction plant

This topic describes the objectives of the proposed experiments and countermeasures that must be adopted to prevent equipment damage and incidents with the personnel involved with the experiments. A previous assessment of the possible behavior of the system was conducted based on the available practical knowledge, literature and preliminary experiments performed in the plant. The experiments were carried out in order to evaluate the dynamic response of the plant under manipulation of selected input variables. Only one input variable was selected to be manipulated each time, while keeping all the others fixed. Measurable disturbance variables were monitored and measurable fixed input variables were also evaluated depending on necessity.

4.1 Jet mill plant

The table 4.1.1 exhibits the general design of experiments and qualitative correlations that were considered a priori. Basically two aspects of the response were evaluated: the idealized intensity of the correlation and the response time of the output variable under evaluation. This previous assessment in addition to serving as a theoretical basis for the design of the experiments, aimed to enabling visualization of possible unsafe conditions and operational constraints given the existent interlocks with the automatic controller and supervisory system.

Table 4.1.1 – Jet Mill Plant - Experiment plan and qualitative correlations between input and output variables.

Experiment identification and manipulated input variable		Output variable #1. Electrical current in ATP classifier motor	Output variable #2. Pressure drop trough the rotor class	Output variable #3. PSD Coarse fraction	Output variable #4. PSD % Fines fraction	Output variable #5. PSD Volume median	Output variable #6. Product throughput rate	Output variable #7. Pressure drop throughout the cyclone	Output variable #8. Grinding energy efficiency
#1	ATP classifier speed	⤉⤉⤉	↑↑↑	⇟	⇞	↓↓↓	⤈	↑	⤈
#2	Grinding air pressure	⤉⤉	↑↑	↑	↑	⇟	⤉⤉	↑↑	↑↑↑
#3	Material feed rate	⇞⇞⇞	⤉⤉⤉	⇞⇞	⤉	⇟⇟	⇞⇞	⇞⇞	⇟⇟⇟
#4	Grinding air temperature	⇞	⇞	○	⇟⇟	⇞⇞	⇟⇟	○	⇟⇟⇟
#5	Nozzle diameter	↑↑↑	↑↑↑	↑↑↑	↑	↓↓↓	↑↑↑	↑↑↑	↓↓
#6	A/D rate	↓↓↓	↓↓↓	↓↓↓	↑↑	↓	↓	○	↓↓

* Legend: Arrows up: positive correlation, arrows down: negative correlation. The many arrows, the stronger the correlation: (○) fairly or not correlated; (↑) fairly positive; (↑↑) positive strong; (↑↑↑) positive very strong. The less crossed the arrow, the more rapid response: (⇞) Slow response, (⤉) fast response, (↑) very fast response.

The manipulated variables in the experiments #1 to #3 consist of the most common process adjustments made by the operators in order of decreasing frequency. The ATP classifier speed manipulated in experiment #1 is the most common process adjustment used by engineers and operators. This parameter is the first choice to control the product quality, given its response speed and the strong correlation with particle size distribution. Experiment #2 objectives to evaluate the energy efficiency as a function of grinding air pressure. Experiment #3 was planned to define the threshold values for the solid feed at which the milling process enters the instability zone discussed earlier in this paper. The feed rate threshold values were planned to be encountered by conservative increments in the feed rate set points while monitoring the power consumption of the electrical motor that drives the ATP classifiers and the pressure drop for the classifiers vanes. In the lack of an on-line PSD measurement, samples of milled material were collected in a fixed sample interval in order to evaluate particle size distribution response. Experiment #4 was planned to evaluate the influence of the grinding air temperature on the grinding energy efficiency and on the others output variables. Temperature range was limited by the current available air cooling capacity. Experiments #5 and #6 were planned to study the influence of nozzle design in the involved output variables. The main objectives of the proposed experiments were:

i) Evaluate the effect of input variables in the specific grinding energy.

ii) Identify possible correlations between product PSD, which is measured off-line, and other process variables measured on-line, such as pressure drop for the classifier vanes and ATP classifier power consumption.

4.2 Classification plant

Experiment plan for the classification plant is showed in Table 4.2.1. The same procedure used for qualitative analysis of the jet mill experimentation was developed. Given its physical construction, the response of the output variables in classification plant is in general much faster than the responses in jet mill plant. Experiment #1 was planned to evaluate the dynamic behavior of the output variables whit variation in classifier wheel speed. This variable, besides the classifying air flow rate manipulated in experiment#2, is the most common adjustment made by the operators to control fine fraction on classifier product. The solid feed rate was included as manipulated variable principally to investigate the response in grade efficiency. Increasing in solid feed rate is a choice to increase production rate but is limited because of the risk of equipment damage and quality losses due to generation of fused material in the classifier product or and must be used with caution. Grade efficiency is negatively influenced by the augmenting in solid feed rate because of the agglomeration and poor particle dispersion. Experiments #4, #5 and #6 were introduced to evaluate the influence of the jet milling in classification process.

Table 4.1.2 – Classification Plant - Experiment plan and qualitative correlations between input and output variables.

Experiment identification and manipulated input variable		Output variable #1. PSD – Fine fraction	Output variable #2. PSD – Volume median	Output variable #3. PSD Coarse Fraction	Output variable #4. Coarse product total mass efficiency	Output variable #5. Grade efficiency	Output variable #6. Classifier motor electrical current	Output variable #7 Production rate
#1	Classifier wheel speed	↓↓↓	↑	↑	↑↑	↓	↑↑	↑
#2	Classifying air flow rate	↑↑↑	↓↓↓	↓	↓↓	↓	↑↑	↓
#3	Solid feed rate	↓	↑	↑	↑↑	↓↓↓	↑↑↑	↑↑↑
#4	Feed material PSD – Volume median	○	↑↑↑	○	↑	↓↓	↓	○
#5	Feed material PSD – Fine fraction	↑↑↑	↓	○	↓↓↓	↑	↓	↓↓↓
#6	Feed material PSD – Coarse fraction	○	↑	↑↑↑	○	○	○	○

* Legend: Arrows up: positive correlation, arrows down: negative correlation. The many arrows, the stronger the correlation: (○) fairly or not correlated; (↑) fairly positive; (↑↑) positive strong; (↑↑↑) positive very strong. The less crossed the arrow, the more rapid response: (⇞) Slow response, (⤉) fast response, (↑) very fast response.

The main objectives of the proposed experiments were

i) Identify possible correlations between product PSD, which is measured off-line, and other process variables measured on-line, which could allow an indirect measure of the product PSD.

ii) Evaluate the effect of input variables in the grade efficiency and PSD.

5 Results from preliminary experiments

The experiments in tables 4.1.1 and 4.2.1 were conceived for realization in an industrial plant without put in risk equipments or people, production plan, product delivery or quality. This was the principal challenge in the experimentation activity. Part of the experiments were already realized and others are currently scheduled or waiting for a window in the production plan. The objective of the following trial was to evaluate the pressure drop for the classifier as an indirect measure of particle size. An indirect measure of the circulation load or hold-up using the power draw of the ATP classifier motor is showed in Figure 5.1 (Gommeren, 1997).

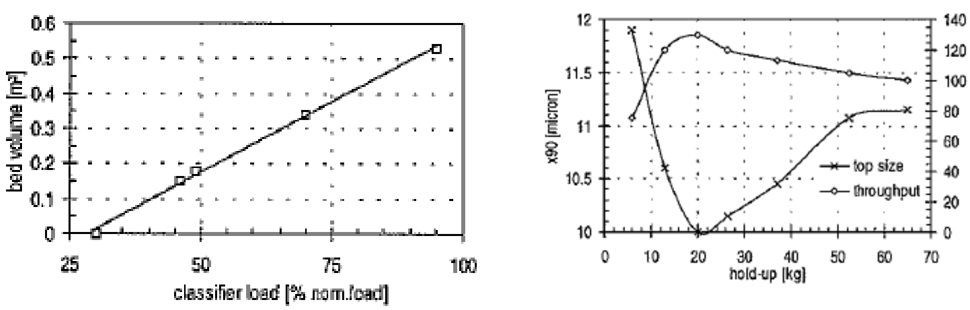


Figure 5.1 - Power draw of the ATP classifier motor as an indirect measure of circulation load (left) and correlation between hold-up and particle size (Gommeren, 1997)

The results obtained by Gommeren, 1997 shows that it is possible to use the power draw as an indirect measure of particle size. The following experiment was used to demonstrate that the pressure drop for the classifier wheel is a possible alternative to estimate ground product particle size (Figure 5.2).

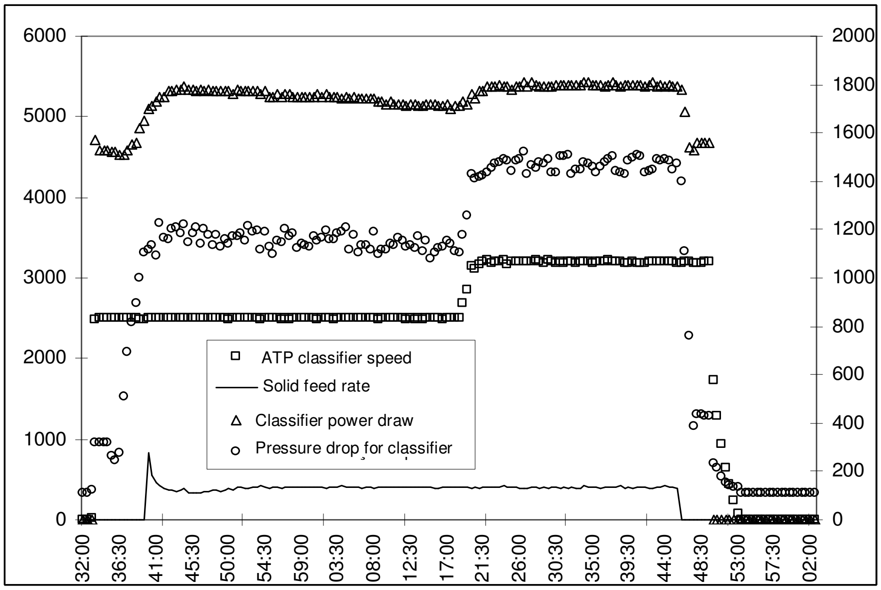
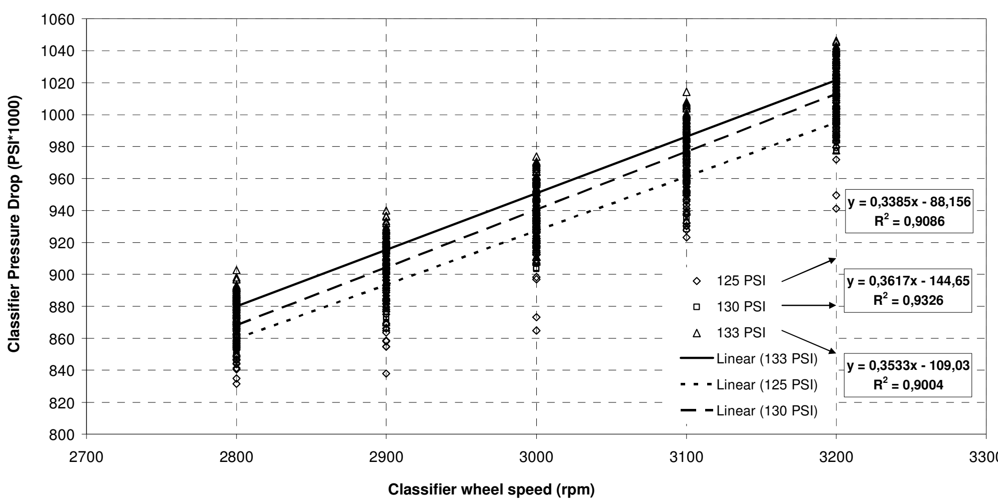


Figure 5.2 - Pressure drop for the classifier and power draw in the ATP classifier motor

Pressure drop for the classifiers was measured by the difference between pressure upstream and downstream the ATP classifiers. The power draw and the pressure drop are clearly correlated, which besides the results obtained by Gommeren, 1997



suggests the possible use of pressure transmitter readings as a virtual on line sensor for particle size. An additional experiment was used to evaluate the influence of rotor speed and

Figure 5.3 - Regression Analysis for pressure drop and wheel classifier speed

5 Conclusion and recommendations

A comprehensive study of the control problem in opposed jet mill and forced vortex air classier was made. It as possible to conclude that optimization in the extended size reduction plant is not possible considering both plants as stand alone equipments given the influence of the grade efficiency of the internal classification processes in the overall classification efficiency. The experimentation plan provided a suitable understanding of the required countermeasures necessary to safe experimentation in industrial plants in order to avoid compromising of production and quality. A new possible estimator for particles size was showed. Future investigation is necessary to complete planned experiments and confirm pressure drop for the classifiers as a indirect measure of particle size in opposed jet milling.

6 Acknowledgements

This work was possible because of the contribution of Xerox Corporation. Special thanks to my colleagues in Xerox for the revising the text and contributing in several aspects for the conclusion of the control problem analysis. Also thanks for my professors in Federal University of Bahia, specially for Professor Marcello Embiruçu, for the continued motivation and important opinions and orientation on the structure of this work.

7 References

B. Epstein, Logarithmico-normal distribution in breakage of solids, Ind. Eng. Chem. 40_1948.2289.

K.J. Reid, A solution to the batch grinding equation, Chemical Engineering Science. 20_1965.953.

L.G. Austin, Introduction to the mathematical description of grinding as a rate process, Powder Technology 5. 1971.

Dods, John. Berthiaux, Henri. Varinot, Christelle. Approximate calculation of breakage parameters from batch grinding tests. Chemical Engineering Science, Vol. 51, No. 19, pp. 4509 4516. 1996

Berthiaux, H; Dods, J. Modelling fine grinding in a fluidized bed opposed jet mill part 1: batch grinding kinetics. , Vol.106 no.1-2, Nov. 1999

Berthiaux, H; Dods, J. Modelling fine grinding in a fluidized bed opposed jet mill Part II. Continuous grinding. , Vol.106 no.1-2, Nov. 1999

Eskin, D. Voropayev, S.;VASILKOV, O. Simulation of jet milling. Powder Technology , v105, p. 257-265, 1999.

Berthiaux, Henri. Analysis of grinding processes by Markov chains. Ecole des Mines d'Albi-Carmaux, Campus Jarlard, Route de Teillet, 8113 ALBI, France. Chemical Engineering Science 55 (2000) 4117}4127.

Gommeren, Hericus J. C. Study of a closed circuit jet mill plant using on-line particle size measurements. PhD Thesis. Delft University Press**.** Delft, The Neterlands, 1997.

Welenkap, Franz-Josef. Moagem fina e ultrafina de minerais: uma revisão**.** Série Tecnologia Mineral, **75.** CETEM / MCT - Centro de Tecnologia Mineral / Ministério da Ciência e Tecnologia. Rio de Janeiro, 1999

Allen, Terence. Particle size measurement, 4th Edition. Powder Technology **Series.** Chapman and Hall London, 1990.

Heikanen, K. On the difficulties of implementing particle size control in particulate processes. Helsinki University of Technology. Espoo, Finland. Powder Technology 82 (1995) 13-19.

Shapiro, M. Galperin, V. Air classification of solid particles: a review. Chemical Engineering and Processing 44 (2005) 279–285.

Wang, Xiaoyan. Ge, Xiaoling. Zhao, Xuehua. Wang, Zhiwen. A model for performance of the centrifugal countercurrent air classifier. Powder Technology 98 (1998) 171-176.

Schönert, Klaus. Size Reduction. Ullmann's Encyclopedia of Industrial Chemistry. Wiley-VCH Verlag GmbH & Co. KGaA. Clausthal, Federal Republic of Germany, 2000.

O. Lecoq, P. Guigon, M.N. Pons. A grindability test to study the influence of material processing on impact behavior. Powder Technology 105 1999.21–29. France, 1999.

O. Lecoq,*, N. Chouteau, M. Mebtoul, J.-F. Large, P. Guigon. "Fragmentation by high velocity impact on a target: a material grindability test. Powder Technology 133 (2003) 113–124. France 2003.