

## ON THE ADAPTIVE DESIGN AND DEVELOPMENT OF A MINI VIBRATORY MILL APPLIED TO CERAMIC MATERIALS

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**Abstract.** *The development of a mini vibratory mill and adaptive design applied to the milling process of advanced ceramic powders is presented. There is a range of ceramic products that can be manufactured through forming processes based on powder compacting followed by sintering, likewise bioceramic medical devices, porous bearings and a variety of micro components. The milling process of small quantities of ceramic powders with small sizes (below 1  $\mu\text{m}$ ) is fundamental for sintering promotion by increase of the particle reactivity. The application of the methodology design techniques led to the adaptive design of a vibratory mill considering the necessary dimensions for the jar volume (10-300 ml). Dynamic analyses were carried out to determine the natural frequencies of the system and an experimental matrix was assembled based on the design of experiments. The investigated responses of the system were the vibration amplitudes and frequencies in each direction. Based on the obtained results, alumina powders with average initial size around 5.2  $\mu\text{m}$  were milled in the mini vibratory mill. The main objective of this work was the validation of the performance of the mini vibratory mill considering as reference a commercial vibratory mill. The powder was analyzed considering the grain size distribution.*

**Keywords:** *mini vibratory mill, design methodology, dynamic analysis, design of experiments, ceramic powders.*

### INTRODUCTION

New technological needs with increasingly multidisciplinary approach have levered up improvements and new developments to the manufacturing of a diversity of products including components obtained from advanced ceramics. Nowadays, many processes must produce ceramic powder in sub-micrometric scale in quantities in the range from 1 to 100g (Sheppard, 1994 and Smith, 1994). These reduced quantities imply, sometimes, in the reduction of the efficiency of mill process mainly in ball mill due to low force impact related to the reduction of the particle diameter. For this reason, the mill is a manufacturing process that can aggregate much value to the final product (Richerson, 1992). Certainly, obtaining the ultra-thin powder can allow the advanced ceramic sector to integrate competitive advantage in the ceramic sector of the National industry. The main reason to the development of this work is to obtain a ceramic powder in sub-micrometric scale to manufacturing of advanced ceramics which are relevant in several research areas within engineering, mainly to biomaterials and filtration areas. The conception, development and validation of a prototype of a mini vibratory mill are proposed using the concept of adaptive design. Design methodology techniques, design of experiments; dynamic analysis and optimization of the mill process were applied to this study. The prototype developed and analyzed allocated six jars with volumetric capacity of mill from 20 to 300ml for each jar. The mill process using alumina particles with initial average size on 5.2 $\mu\text{m}$  were analyzed by sedigraph. After this optimization process, a ceramic powder was obtained with average size of the particles around 0.7 $\mu\text{m}$  with 23% of the particles under 0.2  $\mu\text{m}$ .

### 2. REVIEW

#### 2.1 Advanced ceramics and milling process

Advanced ceramics can be applied basically in four areas: electronic ceramic, structural ceramic and ceramic covering <sup>[11]</sup>. In these areas, the size and granulometric distribution control have fundamental importance in the obtaining and determination of the performance functions in the product design. The mill process of raw material acts as a mechanism in which a reduction of the size particles occurs due to mechanical forces that actuate. During this process, the material is subjected to variations of stress and friction, simultaneously and repeatedly. This mechanism promotes several fractures in the particles, which allow the distribution, propagation and interactions among particles producing,

in the end of the process, the necessary reductions for different applications. To the processing of the thin powder and chemical process homogeneous, the mill process allows the improvement of the properties as well as the obtaining of new materials. Another important aspect related to size and granulometric distribution of the particles it is direct reliance to the forming process and final product. Wellenkamp (1999) classifies the ceramic thin particles as: thin (under 100 $\mu\text{m}$ ) and ultra-thin (under 10 $\mu\text{m}$ ). Small particles with size under 1  $\mu\text{m}$  are necessities in applications where advanced ceramic presents high reactivity contributing to the improvement of the sintering process that results in high mechanical strength of final product. During the mill process, the critical variables are: characteristics of raw material; choose of mill system; mill load; milling mode; covering mill; mill process (Fortulan and Purquerio, 1996). There are many kinds of mill that are employment to obtaining of the small ceramic particles, but no mill process amply applied is totally efficient to production of a high quantity of the particles in sub micrometrics rate. The mill process can be classified as two groups: dry mill and humid mill. The main kind of the equipments to mill include: ball mill, friction mill, vibratory mill and energy flux mill. The vibratory mill presents particular interest in this work.

## 2.2 Vibratory Milling System

The principle of operation of a vibratory mill involves the application of external vibration that promotes the effects of friction and impact between the mill elements and the material that will be milled. Normally, cylinders are used as milling elements due to better distribution of size of particles. This condition occurs as a consequence of the contact between the cylinders that occurs along a line resulting in a particle size distribution with narrow standard-deviation. The use of spheres as milling elements results in a more sparse distribution of particles than cylinders, because the contact between the spheres occurs punctually. The jars are frequently produced with polyurethane covering. Figure 1 presents a vibratory mill whose vibration source is an unbalance mass coupled to motor.

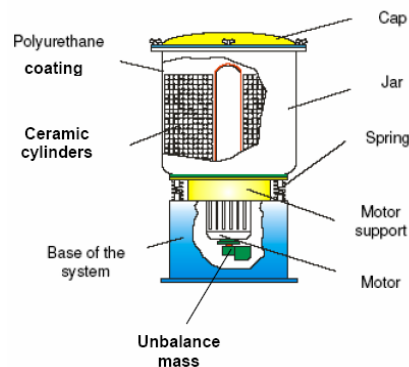


Figure 1. Vibratory mill system (Fortulan and Purquerio, 1996).

## 2.3 Design Methodology

The processes of design and development involve multidimensional activities that can be described considering different approach such as: labor psychology, methodological process and organizational view (Pahl *et al*, 2005). The design process acts as an intersection of cultural and technological activities through the knowledge in the areas of natural sciences, engineering, design, economy, marketing, psychology and politics (Pahl *et al*, 2005). According to Back (1983) "design" is a wide concept, but it must be integrated through all activities of product planning and design. These activities include, for example, market research, the product design, the manufacturing, use of the product, the maintenance and distribution plans and the defuse or disposal of the product. Pahl *et al*. (2005) emphasize the recent recognition of the importance of the design methodology or theory of design in the product conception, whether they are alternative, adaptive or innovative design have generated standards such as the VDI 2221 (1985) and guidelines suggest in ASME publications (1986). In these publications of the ASME in Mechanical Engineering recommendations and guidelines were presented to the teaching and research in the area. In the technical literature, there are some propositions to the systematic approaches to the design activities or strategies to find solutions (Back, 1983; Baxter, 2000; Pahl *et al*. 2005; Shabin, 1988). These propositions present, in a general form, small variations in face of techniques in the solution of the problems, as well as the approach, sometimes more technical (Back, 1983 and Pahl *et al*. 2005), sometimes more organizational . With regards to each activity in the design process, there is an analysis process and the subsequent synthesis process based on techniques and methods that lead the stages of work and decision taking. As the design activities evolve, the flux of information, which is initially conceptual, becomes gradually numerical results.

## 2.4 Concepts on design of experiments

The use of statistical techniques during the design process development is an important tool in the evaluation in the performance of the components and systems. This condition occurs as far as design improvement or innovations in the industries and researches center. Considering industrial products, the application of the design of experiments can reduce the time to market increasing the advantage competitive with quality and productivity (Montgomery and Runger, 2003; Silveira and Cavalca, 2004). Design of experiments is an alternative for traditional experiments that consider “one factor at a time”. This approach works simultaneously with design parameters or factors at different levels for system responses of interest (Box and Hunter, 1978; Montgomery and Runger, 2003). The experimental space should be understood as a cube for the factors exploration, in which each experiment represents a vertex, each side represents the level variation for each factor and each vertex represents the interaction between different factors. The factorial planning is represented by  $2^f$  or  $3^f$  where  $f$  represents the number of factors or design parameters and the base represents the numbers of levels. An experimental design formally represents a sequence of experiments, translated into a group of factors or design parameters adjusted in determined levels or pre-defined values (Box and Hunter, 1978). To factorial planning ( $2^f$ ) are obtained orthogonal contrasts for main effects and interactions, from Equation (1) and (2). The Equation (3) represents the Analysis of Variance to each effect (factors).

$$C = \sum_{i=1}^k c_i T_i, \text{ where} \quad (1)$$

$$\sum_{i=1}^k c_i^2 = \sum_{i=1}^k (\pm 1)^2 = k = 2^f \quad (2)$$

$$SS_c = \frac{C^2}{2^f \times n} \quad (3)$$

The design of experiments can generate empirical models to operation ranges for different applications of components and systems, exploring their behavior and characteristics at different levels of design parameters, showed by equation (4):

$$y(x) = f(x) + \varepsilon \quad (4)$$

Where  $\varepsilon$  represents random errors, considering they are normally distributed with a zero average and standard deviation ( $\sigma$ ). As the real  $f(x)$  function is unknown, the surface response  $g(x)$  is built to approximate  $f(x)$ . The equation coefficients are estimated using the approximation  $\hat{y} = g(x)$ . The empirical model generated by design of experiments can be represented using the polynomial function. Equation (5) establishes the relation of the second order between design parameters and system response by factorial planning, considering main effects and their interactions. The coefficients are adjusted using numerical methods, as least square method, and evaluated by Analysis of Variance (ANOVA).

$$\hat{y} = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i < j} \beta_{ij} x_i x_j \quad (5)$$

## 3. DEVELOPMENT OF THE MINI VIBRATORY MILL

The identification of the necessity is the activity that precedes any design. This necessity may arise from failure or improvement of the component or product, or it may be innovation originated from the scientific, technological and economic advances (Pahl *et al.*, 2005). The need of design and manufacturing of mini vibratory mill was identify mainly, due to the impossibility of Brazilian equipment to mill the sub micrometrics particles. The stages to the orientation of development and design of a mini vibratory mill are presented in the Flowchart represented by Figure 3.

### Stage 1 - Recognition of necessity

The necessity was recognized to obtain advanced ceramic powder in sub micrometrics scale because of the high demand in research areas such as nanotechnologies and biomaterials. Most national laboratories and research centers are not equipped to produce powders in this scale. This condition represents a lack in the design and manufacturing of the equipment to mill of ceramic powder and it is the motivation to the design and development of a mini vibratory mill.

### Stage 2 - Definition of the problem

The mini vibratory mill must be capable of coupling several jars with less volumetric capacity (from 1 to 100g) and useful volume from 10 to 300ml. The support of the mini mill can couple multiple jars (from 1 to 6) and the mill process can be made multiple and simultaneous. The design was classified as “adaptive design” (Pahl, *et al.*, 2005), once the principle solution is similar to the commercial vibratory mill. An estimative of the economical and physical viabilities was made to the design and manufacturing of a prototype of the system. The value calculated was US

3.300,00, including materials acquisition, use of machine tools, labor and experimental tests. This value was compatible with the financial support in high technology laboratories.

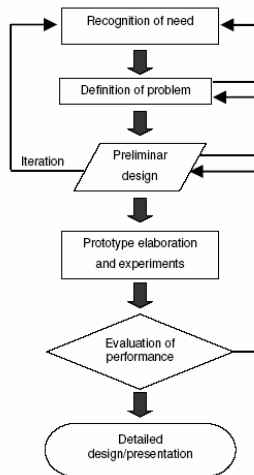


Figure 3. Methodology proposed to development of the mini vibratory mill.

### Stage 3 - Preliminary Design

The design of the mini vibratory mill had as reference the vibratory mill (Fortulan and Purquerio, 1996) is shown in Figure 1. Based on this first structure was maintained: the base, the motor and the springs. The components and corresponding actuating forces were re-calculated since the system was reduced. The unique jar was substituted by six small jars to multiple and simultaneous mill. The design of the jars considered the thickness of the wall as well as the covering, closure of cap and the fixation system on the vibratory mill. The useful volume to these small jars was set from 10 to 300ml. To obtain the vibration necessary to the mill process were set unbalance mass in the extremities of the motor shaft. The vibration amplitude can be changed with variation of the unbalance mass. During the preliminary design, systematic methods conducting the planning, search and solution of conceptual and technical problems are strongly recommended. In this phase, 90% of the technical information in the design is defined, corresponding to 10% of the total time of the design process.

Need of the users \ Technical Characteristics	Technical Characteristics									
	Reduced size (jars)	Motor power	Different sizes (jars)	Fixation of several jars	Fast engaged	Standard components	Vibrations in several axis	Materials	Small spaces	Closure system of jars
Reduced size	●	●						●	●	
Low consumption of power		●						●		
High efficiency of milling	●		●				●			
Simultaneous milling	●		●	●						
Easy assembly of system					●	●				●
Easy maintenance	●		●	●	●	●			●	●
Vibrations cycles		●					●			
Low weight		●						●	●	
Low contamination			●					●		●
Good closure of the jars					●			●		●

Subtitle:  
 Strong correlation: ●  
 Moderate correlation: ●  
 Weak correlation: —

Figure 4. Correlation matrix to the conceptual phase of the mini vibratory mill design.

Initially a correlation matrix was elaborated to the “mini vibratory mill system”. This matrix was the result of some research carried out with potential users and the design group where the main user’s characteristics and the possible technical characteristics are identified to each one. This result is presented in Figure 4. Based on the correlation matrix, intuitive and discursive methods are used to define the configuration, mechanisms and material to the jars and system.

A multidisciplinary group was formed to the brainstorming sessions classifying the resultant solutions as viable or potentially viable. After the brainstorming sessions, the viable results served as input to the morphological board. The use of discursive method allows exploring to exhaustion technical solutions to each design parameter. Figure 5 presents constructive alternatives to the design parameters from combinations of solutions. The dashed line was the solution chosen by the design group to jars design and manufacturing.

Design Parameter	Solutions					
External material of jars	Steel	Aluminum	Nylon	Ceramic	Epoxy	
Internal material of jars	Steel	Aluminum	Nylon	Ceramic	Polyurethane	HDPE
Fixation of the base support	Thread	Kind of liquidizer	Magnetic	Sucker	Kind of "marmita"	Belt
Fixation of the cap	Thread	Strap	Conical	Clip	Cork	Kind of "marmita"
Geometry	High cylindrical	Regular cylindrical	Prism			
Manufacturing of the jars (external)	Casting	Cutting	Injection	Foundry	Prototyping	
Manufacturing to the jars (internal)	Isostatic pressing	Slip casting	Extrusion	Machining		

Figure 5. Morphological method applied to jars.

According to the design information constructive solutions were proposed to the fixation system of jars. The advantage and disadvantage tables were elaborated to refine these solutions considering several design aspects such as: material, manufacturing, cost, physical limitation, weight, maintenance and usability. Some possible solutions were considered to effectuate the locking of the jars on the base. Figure 6 presents design alternatives found to the fixation of the jar in the base of the vibratory mill.

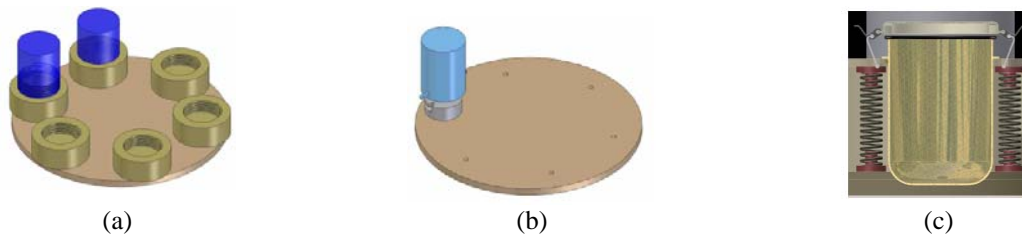


Figure 6. Fixation jars system; (a) screw; (b) fast clamp; (c) pressure closing.

#### Stage 4 - Prototype construction and experimental tests

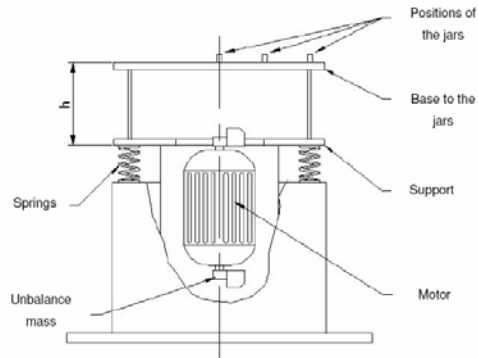
The objective of the prototype construction is to optimize unknown parameters, principally parameters related to dynamic behavior, which are fundamental to mill process. The prototype manufacturing, as well the simplified view, is shown in Figure 7 (a) and (b), respectively. The pre-defined design parameters are: diametric positions of the jar to mill process, rotating frequency of the motor, base height to jar related to vibratory base and number of spring's position in the vibratory base. To the dynamic analysis, a solid jar made of the aluminum was used with mass equivalent to the load of the jar with ceramic material. In the first place, the natural frequencies of the system as a rigid body are identified. After that, based on design of experiments, the design parameters are combined in three levels, obtaining the amplitudes of vibration of the jar at four different directions: ( $x$ ,  $y$ ,  $\theta_x$  and  $\theta_y$ ) to each combination. The objective of these experiments was to identify the variations in the responses due to variation of the pre-defined design parameters.

#### Stage 5 - Performance evaluation

The vibratory motor supplies 120W power with specification as follows: three-phase motor, from WEG (220/380V), 60Hz and 0.12kW, with 4 and 6 poles (shell 63) and 8 poles (shell 71). To obtain the necessary vibration to the mill process unbalance masses were set in the extremities of the motor shaft modified. The vibration amplitude can be changed with the variation of the unbalance mass.



(a)



(b)

Figure 7. Prototype of the mini vibratory mill: (a) physical system and (b) simplified view.

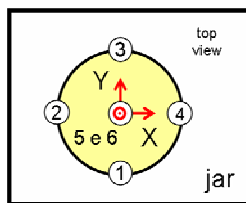
## A. Dynamic analysis

### A. 1 Modal analysis

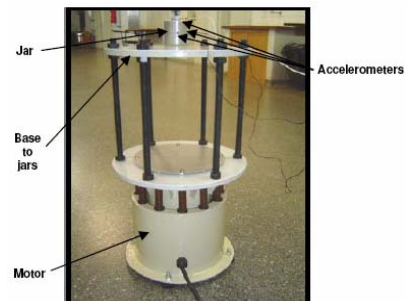
In the experimental assembly two translational accelerometers (PCB U353816) were positioned in the direction  $x$ ,  $y$  and an angular accelerometers (Kistler 8836M01) to measure at  $\theta_x$  and  $\theta_y$ . A data acquisition system was used with signal board of four channels (Tektronix – 2630) and two signal amplifier (ICP Sensor Power Unit 480C02). The external excitation was made using a shaker (MB Dynamics Modal 50A). The data acquisition was made using accelerometers, with the shaker positioned in four different points of the system: in the jar itself, in the superior base of the mini vibratory mill in the directions  $x$  and  $y$ ; in the inferior base in the directions  $x$  and  $y$ , as well. The first and second natural frequencies were found in the range of 0 to 20Hz, respectively to 3.16 Hz and 5.87Hz. These frequencies correspond to the two first modes of the rigid body of the superior base; the first mode of the translation and the second of rotation in the base related to  $x$  axis. A third frequency of resonance of the 107.5Hz was found to analysis range of 0 to 1000Hz. This frequency corresponds to combined modes of translation and rotation of the superior base.

### A. 2 Operational analysis

In this stage, the objective was to identify within the design parameters which ones have more influence on the response of the vibration amplitude. In this form, six points of measurement were chosen in the jar: four points divided by  $90^\circ$  placed along the external diameter of the jar and two other points located in the center of the jar. In the points along the diameter (listed of 1 to 4) the RMS amplitude of the translational vibration in direction  $z$  was measured in the center points (5 and 6) the amplitudes RMS of the rotation in the directions  $\theta_x$  and  $\theta_y$  was measured as well. Figures 8(a) and (b) present the reference coordinate system adopted and the instrumentation of the mini vibratory mill, respectively.



(a)



(b)

Figure 8 (a). Reference coordinates to dynamic analysis; (b) Instrumentation in the mini vibratory mill to data acquisition.

The analysis based in the vibration amplitudes of the prototype indicated that: i) the number of springs has less influence on the vibratory response in the jar; ii) the positioning of the jar in the extremity of the fixation base supplies high vibration amplitudes in all directions and, mainly: iii) an increased of the vibration amplitude occurs with the reduction of the base height where the jar is coupled. The graph of Figure 9 presents this tendency to three different base heights of vibratory mill to six points of measurement.

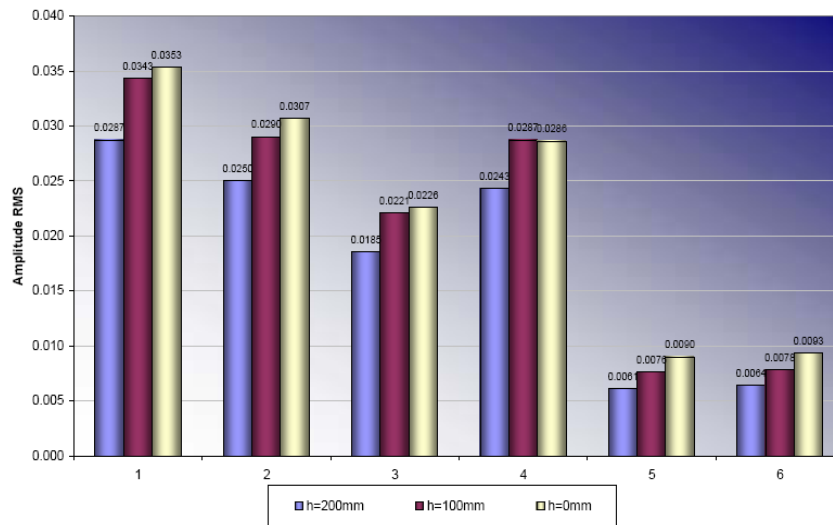


Figure 9. Variations of the vibration amplitudes.

**B. Design of experiments and data acquisition**

After the dynamic analysis following the assembly of experimental matrices based on factorial planning (2<sup>4</sup>) with repetition in the center point. The initial objective of this planning was an exploratory study of parametric sensitivity of the design parameters on the response to identify the best dynamic condition to mill ceramic powders. The design parameters or factors were: base height to fixation of the jars (100, 200 e 300mm), number of poles (motor) (4, 6 e 8 polos), spring number (6, 9 and 12) and diametrical position of the jars (1, 2 and 3 positions). The experimental matrix obtained from the factorial planning to the amplitude vibrations at directions x and y [g], respectively and angular  $\theta_x$  [rad/s<sup>2</sup>] is presented in Table 1. The response was treated individually to each direction.

Table 1. Experimental matrix to evaluate the dynamic responses of the prototype of the mini vibratory mill.

Exp.	Base height [mm]	Spring number [.]	Polo Number [.]	Jar Position [.]	-x [g]	y [g]	$\theta_x$ [rad/s <sup>2</sup> ]
6	300	12	8	1	0.00307700	0.00313810	0.015993
8	300	12	4	1	0.01065100	0.01050200	0.015993
4	300	6	4	1	0.01024800	0.01007900	0.016913
2	300	6	8	1	0.00262450	0.00262570	0.016064
12	300	6	4	3	0.01012300	0.01047600	0.016595
10	300	6	8	3	0.00266660	0.00270600	0.015844
17	200	9	6	2	0.00396230	2.90080000	0.014824
18	200	9	6	2	0.00396230	2.90080000	0.014824
19	200	9	6	2	0.00396230	2.90080000	0.014824
9	100	6	8	3	0.00099426	0.00076065	0.014832
11	100	6	4	3	0.0010910	0.00073913	0.016697
3	100	6	4	1	0.00142430	0.00063793	0.016498
1	100	6	8	1	0.00123010	0.00056989	0.014645
15	100	12	8	3	0.00186630	0.00059265	0.014636
13	100	12	4	3	0.00172690	0.00069738	0.016424
5	100	12	8	1	0.00247100	0.00292090	0.014376
7	100	12	4	1	0.00252400	0.00305150	0.016085
14	300	12	4	3	0.01047300	0.00258680	0.015366
16	300	12	8	3	0.00377550	0.00320510	0.014433

**C. Results and discussion**

The Analysis of Variance (ANOVA) from the calculation of the contrast of the main effect and interactions as well as the confidence level adopted indicate that the parameters with more influence on the interest response. Consequently, to the responses of the vibration amplitude [g] at direction x, the base height of the jars varied the response on  $5.037 \times 10^{-3}$  [g]; the number of springs varies  $3.6 \times 10^{-3}$ . In this condition, the base height of jars is either the design parameters with more influence on the vibration amplitude or it is the sensitivity factor in the experimental matrix. The factorial planning's were made and analyzed with Optima Program (Silveira and Cavalca, 2004). The linear model of 1<sup>st</sup> Order was fitting from the factorial planning by Equation (6) and Table 2 present the ANOVA of the experiments. The

percentage of variation explained by the regression method (ratio between the square sum due to regression and the total square sum) was 74.56%. The ratio value between the square medium and square medium residual is bigger than the tabled value ( $F_{4,14} = 3.11$ , with confidence level of 95%) this value indicates there was a regression significant. However, the value obtained by ratio of the square medium of lack of fitting on square medium of pure error is bigger than the value tabled  $F_{12,9} = 2.21$  to the same confidence level of 95%.

$$y = 0.00415 + 0.00252x_1 \quad (6)$$

Table 2. Analysis of variance to fitting of first order model to response in direction x.

Variation source	Square Sum	Degree of freedom	Square Mean
Regression	$1.59 \times 10^{-4}$	4	$3.97 \times 10^{-5}$
Residue	$5.41 \times 10^{-5}$	14	$3.87 \times 10^{-6}$
Lack of fitting	$2.52 \times 10^{-4}$	12	$2.10 \times 10^{-5}$
Pure error	$6.89 \times 10^{-10}$	2	$3.45 \times 10^{-10}$
Total	$2.13 \times 10^{-4}$	18	$0 \times 10^0$

This result indicates that there is lack of fitting to the data fitted by least minimum method to first order model. These lacks of fitting were repeated to responses to the direction y (63.14%) and direction  $\theta x$  (78.98%). Originally, the lack of fitting is related to the choice of polynomial order of the function; in this case, the order must be increased to second order which implies in changing the type of design of experiments. If the pure and random errors are higher, then new values to levels of design parameters must be defined and new designs of experiments are assembled. These procedures are called response surface method that use design of expandable experiments. However, the objective of the design of experiments in the dynamic analysis of the mini vibratory mill was an exploratory study of the design parameters, identifying which design parameters are more important for vibration amplitude. Thereby, the results of the effect contrast and analysis of variance indicated that the vibration amplitude at direction y is affected just by the number of poles varying the response at  $1.34 \times 10^{-3}$  [g]. To the response at direction  $\theta x$ , the parameter with more influence on the vibration amplitude, as well as direction y is the number of poles (motor) where the response is varied on  $7.14 \times 10^{-6}$  [g]. The previous behavior by dynamic analysis and the results obtained through the design of experiments indicated that the base height of the jars is the parameter with most influence on the vibration amplitude of a prototype of vibratory mill, following by number of poles of the motor. It is expected that these amplitude variations have decisive influence over the mill process of ceramic powder.

#### D. Tests and milling process results

To each mill process, using Alumina (Alcan 5 SG) with initial diameter of 5.2  $\mu\text{m}$ , the material was analyzed and the distribution of the medium spherical diameter [ $\mu\text{m}$ ] was obtained and it is shown in Figure 10. The subtitles used were: MB: Ball Mill; MV: Vibratory Mill; Mill time [h] and  $N^\circ\text{mm}$ , base height of the jars [mm]. The results obtained with the mill at position 38 from de motor base resulted in ceramic powder with particles of medium diameter ( $\varnothing_{ee}$ ) equal to 2.05  $\mu\text{m}$ , to position 100 mm; and particles of medium diameter ( $\varnothing_{ee}$ ) equal to 2.30  $\mu\text{m}$ , to position and  $\varnothing_{ee}$  equal to 2.5  $\mu\text{m}$ . The position 38 indicated the best condition of the mill process to obtain sub micrometric powder, according to the results obtained from the design of experiments to dynamic analysis. By the way, a comparison shows that the medium diameter ( $\varnothing_{ee}$ ) obtained of 2.05  $\mu\text{m}$ , from the vibratory mill without optimization is superior to the values obtained from ball mill (3.8 $\mu\text{m}$ ) at mill time of 96 hours, as presented in Figure 10. This condition implied the vibratory mill is more efficient than the ball mill considering small jars.

#### E. Optimization of milling process

From the results obtained with the design of experiments to dynamic analysis, following experiments to fit the diameter equivalent of the particles of the ceramic powder. The objective was to obtain powders in sub micrometric scale. To the first experiment (01), the mill elements of 130g with zirconium dioxide spheres of diameter 6.7mm were substituted by 170g with zirconia's spheres of diameter 3 mm and the mill time was increased to 120 hours. The base height of jars was set on 12mm from the inferior base of the motor following the new mill. The substitution of mill elements increases the superficial area around 160%. The medium diameter of ceramic particles was 1.6  $\mu\text{m}$ , with 100% of the particles under 3.5  $\mu\text{m}$  and above 0.22  $\mu\text{m}$ , besides 22% of the particles presented values under 1  $\mu\text{m}$  (Figure 11 – MV12-20H-12). To the second experiment (02), the conditions in 01 were repeated without the superior base of the jars. The jars were coupled in the inferior base and 12 kg of the total system were eliminated. This modification resulted in a medium diameter of ceramic particles equal to 1.15 $\mu\text{m}$  with 100% of the particles under 2.8  $\mu\text{m}$  and above 0.2  $\mu\text{m}$ , besides 40% of the particles present values less than 1 $\mu\text{m}$ . It is observed that the reduction of mass in the system improved the milling process (Figure 11 – MV12-120h).



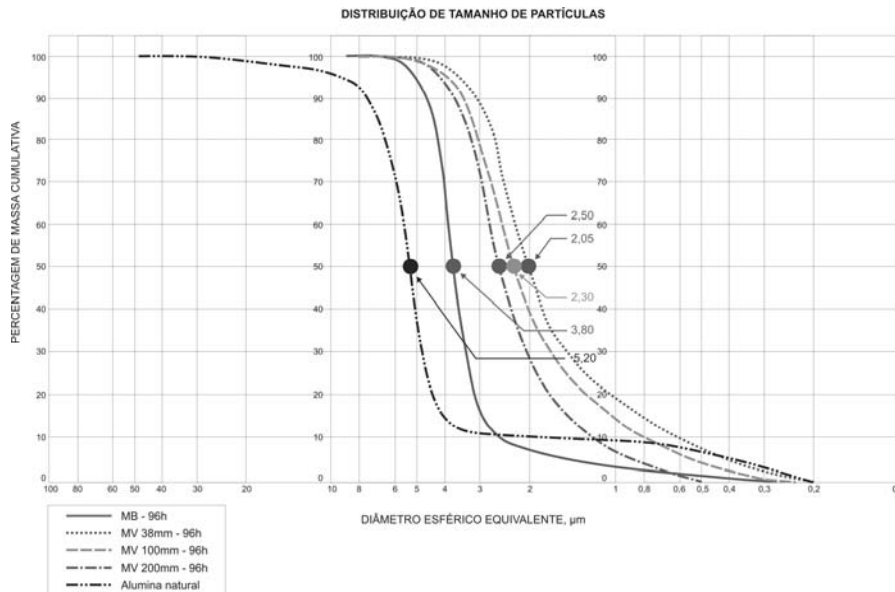


Figure 10. Mill curve to the mini vibratory mill: Mass (%) x diameter equivalent spherical ( $\mu\text{m}$ ). MB

In the last experiment (03), the unbalance mass in the motor was increased in 230g each. This modification generated an increase in the centripetal force of 43% (from 747N to 1070N) to each extremity of the motor shaft. Figure 11 (MV12-120-01KN) presents the result of the modification. It is observed that the medium diameter of ceramic particles was  $0.7 \mu\text{m}$ , a value that satisfies the initial objective of this work, to obtain ceramic powder with scale sub micrometric. All of the particles were less than  $2.2 \mu\text{m}$ , and notably 23% of the medium diameter ceramic particles were less than  $0.2 \mu\text{m}$  indicating a considerable fraction of the powder in nanometric scale.

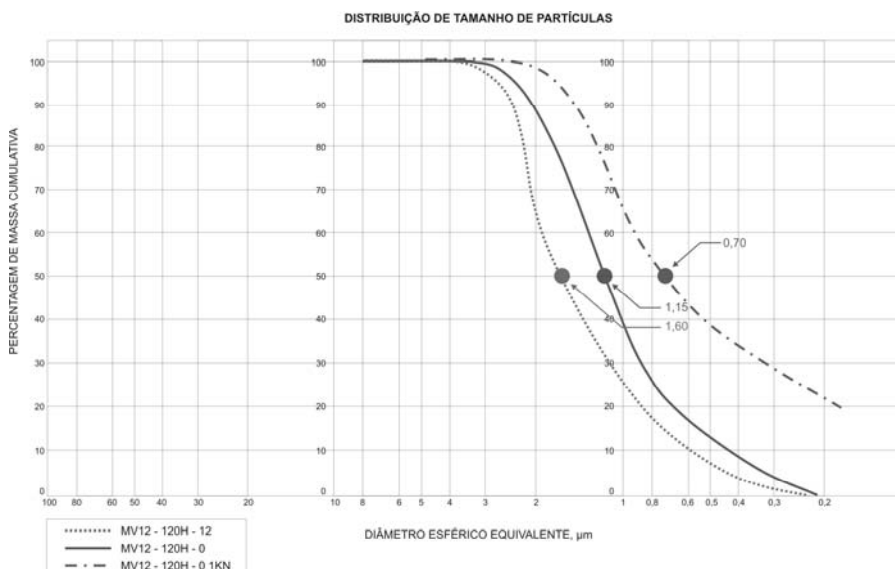


Figure 11. Mill curve to the mini vibratory mill to 120 hours with jars set on the inferior base.

## Stage 6 - Detailed Design

According to the information and results obtained from Flowchart presented by Figure 3 it was proposed feasible designs. This solution considers mini vibratory mill with capacity to couple to maximum six jars manufacturing externally by aluminum and internally covered with ceramic of the high alumina. The jars are fixed to mill through fixation clamps joining the own jar and the cap, simultaneously. The base that supports the jars was set to inferior position next to springs. The motor of 2 polos of the 120W is coupled to mobile structure supported by 12 springs with rectangular profile. The unbalance mass located on the motor shaft apply the centripetal force of the 1070N each extremity of the shaft. The assembly of this solution is presented by Figure 12.

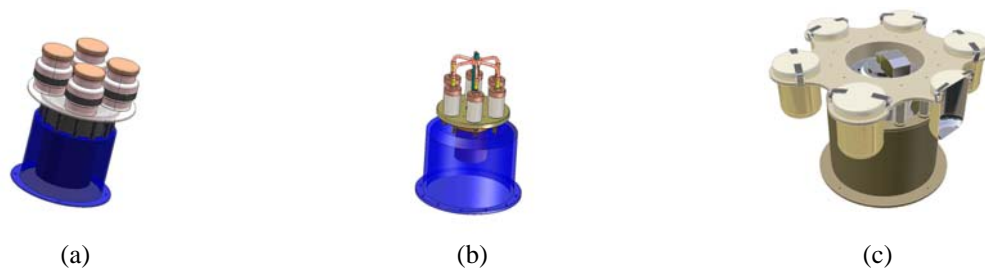


Figure 12. Viable options of the design for mini vibratory mill system.

#### 4. CONCLUSIONS

The methodology proposed was capable of supplying the necessary information and to direct the development, design and construction of a prototype of mini vibratory mill. The results indicated that powder in sub micrometric scale was obtained through mini vibratory mill of easy manufacturing, easy maintenance, satisfactory performance and accessible cost. The optimization process to mill ceramic powder based on the results from design of experiments of dynamic analysis lead to sub micrometric powder with average size of particles of  $0.7 \mu\text{m}$ . The performance of the vibratory mill to small jars to milling time of 96 hours exceeded the expectation, resulting in a medium value of the particles diameter equal to  $2.05 \mu\text{m}$  without optimization against the medium value of the particles diameter equal to  $3.8 \mu\text{m}$  obtained by ball mill. This condition is related to low height of the particles drop in jars of little dimensions in ball mill which do not generate sufficient power to break these particles. On the other hand, it was possible to prove that in a mini vibratory mill, the impact force supplied by vibratory motor maintains the efficiency of the mill process to small volume of the ceramic powder.

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