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POWER MEASUREMENTS DURING MECHANICAL MILLING. AN EXPERIMENTAL WAY TO INVESTIGATE THE ENERGY TRANSFER PHENOMENA

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Abstract—Energy transfer and power consumption during milling have been theoretically evaluated through an already assessed model. Electrical and mechanical power consumptions during milling have been measured. The experimental results are perfectly in line with the prediction of the collision model and comparison between experimental and theoretical power absorptions is quite satisfactory. The dependence of the power consumption on the rotation speed of the planetary mill and the effect of the filling of the milling device on the energy transfer and power consumption have clearly been established. Practical consequences on the choice of the proper milling conditions have been illustrated by examining different kinds of reactions promoted under different milling conditions.

1. INTRODUCTION

Due to the general interest in the mechanical alloying process which has arisen over the last decade, several attempts at modelling have been made in order to establish predictive capabilities for this process. Such a capability should allow to infer which energetic parameters (rotation frequency, ball diameters, etc.) have to be properly considered in order to obtain one or another end product or, for a given reaction path, how long the milling time must be in order to achieve a given stage of that reaction.

A first comprehensive attempt has been performed assuming that collision is the primary event by which energy is transferred from the milling tools to the powder under processing. Kinematic equations of the ball movement, energy transferred per hit, collision frequency and total power transferred have been estimated by that approach [1]. Improvements of the collision model have been reported by McCormick *et al.* [2] and, recently, by Abdellaoui and Gaffet [3] who constructed a "dynamic phase diagram", on the basis of the calculated power injections, able to correlate results from different milling devices.

Modelizations apart, no experimental approach directed towards the quantification of the energies involved in the process has been carried out up to now. The only exception in this direction was attempted by Kimura and Kimura [4] who measured the torque applied on the rotating shaft of a suitable home made attritor. Therefore, until now, no systematic investigation has been undertaken to evaluate both the energies involved and the mechanisms underlying the energy transfer.

Recently, we indicated a way to measure, at a macroscopic level, the power involved in the process and to understand the laws governing the energy transfer [5, 6]. The main goal of the present work is to show such an approach in an exhaustive way and to show that previous evaluations are directly confirmed by the power measurements described in the present paper.

2. SUMMARY OF PREVIOUS INVESTIGATIONS

If we assume that collision is the primary energy transfer event during milling, then the energy involved in each collision event can be expressed by

$$\Delta E = K_a \tfrac{1}{2} m_b V_b^2 \tag{1}$$

m_b being the mass of the ball and V_b the *relative impact velocity*. K_a is a coefficient depending on the elasticity of the collision: for perfect elastic collision the energy release is null ($K_a = 0$) and is total for perfect inelastic collision ($K_a = 1$). The elasticity of collision has been dealt with in Ref. [7] to which one can refer for details.

From the kinematic equations of a *planetary mill*, derived in Ref. [1], it follows that the relative impact velocity of a ball is given by an equation of the type

$$V_b = K_b w_p R_p \tag{2}$$

where w_p and R_p are the rotation speed of the planetary mill disk and its radius, respectively. K_b is a constant that can be evaluated [6] and that primarily depends on the geometry of the planetary mill.

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The collision frequency of a ball can be expressed by [1]

$$v = K_v w_p \tag{3}$$

where K_v is a constant that, again, mainly depends on the geometry of the mill. Considering a suitable number of balls, N_b, for which reciprocal hindering of the balls is negligible (see [1] and below), the total collision frequency is given by

$$v_t = vN_b = K_v N_b w_p. \tag{4}$$

We want to draw the reader's attention to the fact that equations (2) and (3) have been derived in the original paper [1] under the assumption of inelastic collision ($K_a \rightarrow 1$) and are strictly valid for that condition. However, we assume, in a first approximation, that the equations still hold even for a regime different from the perfectly inelastic one, that is, for any K_a value provided that it can be assumed constant in a given series of milling experiments. We will see in the following that the assumption is realistic and is justified by the results.

The power involved in a milling process is given by the *intensive factor* of a single event, ΔE, multiplied by the number of events per unit of time (*extensive factor*), i.e.

$$P_{mod} = \Delta E v_t \tag{5}$$

where P_{mod} represents the power consumption during milling predicted on the basis of a collision model. Using the previous equations

$$P_{mod} = P^* \tfrac{1}{2} m_b w_p^3 R_p^2 n_b \tag{6}$$

where P^* includes all the previous K_a, K_b, K_v constants and $n_b = N_b N_v$ being N_v the number of vials used in a given experiment. Expressing all the variables in international units (m_b in K_g, w_p in radians, R_p in metres), P^* comes out as an adimensional factor. In a given series of milling experiments it is expected that P^*, for a given planetary mill, should only depend on the K_a value and should really be a constant factor as long as the reciprocal hindering of the balls [equation (4)] is negligible.

3. POWER MEASUREMENTS: EXPERIMENTAL SET-UP AND HANDLING OF DATA

3.1. Feasibility of electrical power absorption measurements

The energy *spent* during the milling process (due to collisions and other concomitant phenomena like attrition) derives from the movements of the milling tools and, ultimately, from the power coming from the electrical motor. In principle, therefore, it should be possible to reveal some absorption power difference when milling with the vials filled with a given charge or with the same empty vials. Further, as indicated by equation (1) (see Ref. [7] for details), the energy spent in each event is small when collision is elastic (typically bare balls without powder) and much greater in the presence of powder when the collision becomes inelastic ($K_a \rightarrow 1$). Therefore, again, carrying out milling experiments in exactly the same configuration by changing only the elasticity of collisions, differences of power absorbed should be revealed.

In order to verify the previous points, it is convenient to measure the power consumption during milling by a power meter as indicated in Fig. 1(a). By such a device the power absorption is measured with two empty stainless steel vials. Each vial has later been filled with 99 stainless steel balls. In a third experiment two vials having the inner walls coated with tungsten carbide were run with 99 tungsten carbide balls. The three sets of data are reported on Fig. 2(a) and they show that

(i) there is, indeed, an appreciable net electrical power difference, P_e, between empty, P_e°, and filled vials, P_e'

$$P_e = P_e' - P_e^\circ \tag{7}$$

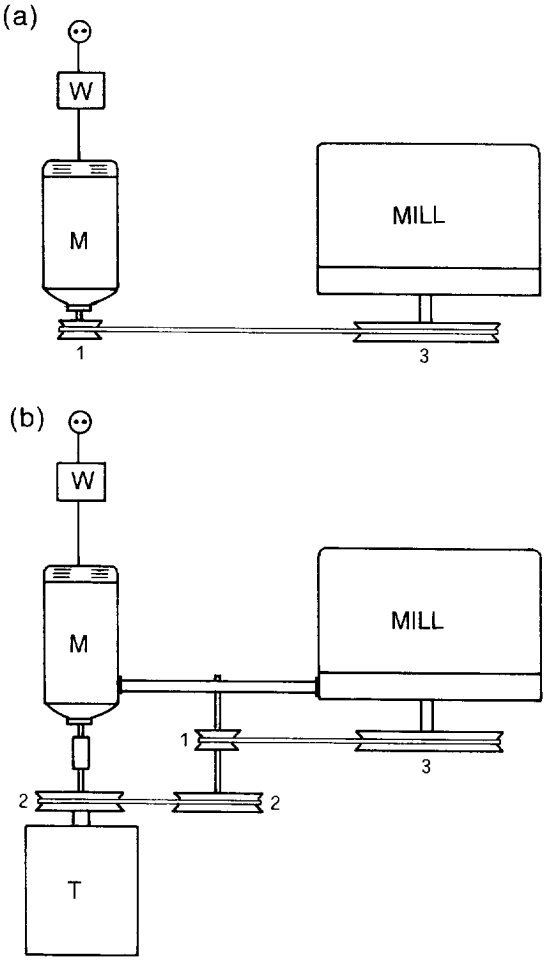

Fig. 1. (a) The power consumption of the mill is measured by the power meter indicated by *W* in the figure. *M* is the electric motor. (b) Scheme of the direct measurement of the torque applied on the motor shaft through the torque-meter *T*. 1, 2 and 3 on both (a) and (b) sketches indicate different dimensions of the various pulleys.

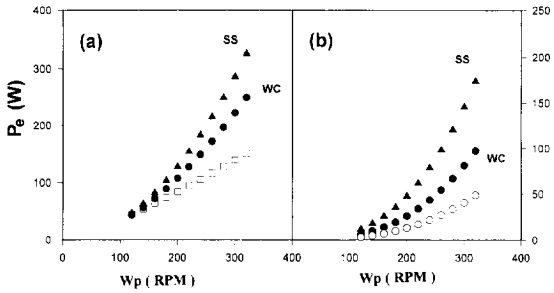

Fig. 2. Feasibility of electrical power measurements. (a) The gross electrical power absorptions measured with two 250 cm^3 empty vials made of stainless steel or having the inner walls coated with tungsten carbide (□). The same vials filled with 99 balls of 10×10^{-3} m stainless steel (SS ▲) and with 99 balls of tungsten carbide of the same diameter (WC ●). The powers are reported vs the rotation speed of the planetary mill disk plate expressed in RPM (rounds per minute). Note that the symbols thickness roughly include the average error in power measurements (y scale). (b) The net electrical powers for SS and WC balls (solid symbols). The open circles give the electrical power with WC balls recalculated for the same weight of the SS balls.

(ii) the power absorption registered with different materials is in line with the elasticity of their collisions.

The second point is better seen in Fig. 2(b) where the *net power differences* between filled and empty vials are reported. With tungsten carbide the collision is much more elastic than with stainless steel. Consequently the net power consumption is much greater in the latter case.

3.2. *Direct measurements of the applied torque*

The net power differences shown in Fig. 2(b) should actually represent the macroscopic answer to the sum of the microscopic events occurring during the milling process. According to the collision model these net values should obey an equation like (6). However, in doing the difference between filled and empty vials, we make the approximation of considering the yield of the motor constant in the different ranges of power consumptions that actually are somehow different with empty or filled vials. Therefore, in order to avoid errors due to the varying yield, we mounted a torque-meter on the shaft of the motor to directly measure the applied torque [see Fig. 1(b)]. From the measured torques the mechanical power absorptions are directly obtained. The net mechanical powers are obtained in the same way as the electrical ones by taking the difference between measurements from filled and empty vials

$$P_m = P'_m - P^{\circ}_m. \tag{8}$$

In a series of experiments both electric and mechanical powers were measured and the motor yield obtained by

$$\eta = \frac{P_m}{P_e} \tag{9}$$

over the whole range of measurable electrical power absorptions [i.e. in equation (9), the P values are from empty or variously filled vials]. The calibration curve is reported in Fig. 3. The continuous line represents the best fit through the experimental values covering the explorable range of electrical power absorptions.

We close this section by outlining that the direct measurements of the applied torque are much longer and delicate than the simple measurements of the electrical power absorption. Therefore, once the calibration curve of Fig. 3 had been obtained, most of the experiments were carried out by only measuring electric power and converting it to mechanical power using the calibration curve.†

3.3. *Milling experiments*

The planetary mill used was the model P5 from Fritsch. Two or four stainless steel vials of 250 cm^3 were mounted in each experiment. The walls of the vials were deliberately covered with the thin layer of powder that usually remains attached at the end of a prior milling process. In our case the vials were preliminary treated with Fe–Zr powder. In this way we were able to realize rather inelastic collisions. Bare stainless steel balls of different diameters (6, 15 and mostly 10×10^{-3} m) were used. The vials prepared in this way were charged with a given number of balls and then the power absorption recorded as a

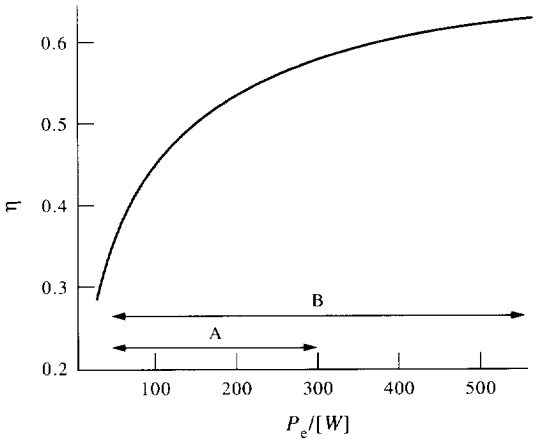

Fig. 3. Yield of the electrical motor $\eta = P_m/P_e$ (P_m and P_e mechanical and electrical powers respectively) plotted as a function of P_e. Region A indicates the range of the powers covered when working with empty vials. With filled vials, depending on the charge, any power included into B region, can be covered.

†It can be demonstrated, by a deeper analysis (not worth being exposed in the actual experimental paper), that the power really given to the process (to the powder and to the milling tools), let us say P_u, can be derived from the electrical (P_e) or mechanical (P_m) powers, considering suitable coefficients correlated to the dispersions occurring in the electrical motor or in the mechanical transmission, through simple relations of the type

$$P_u = P_m(1 - \varphi_m) = P_e(1 - \varphi_m)(1 - \varphi_e)$$

where $\varphi_m = 0.05$–0.1 and $\varphi_e = 0.25$–0.3.

Table 1. The α values of equation (11) (see text) from electrical (e) and mechanical (m) powers obtained as a function of the degree of filling of the vials n_v. d_b and m_b diameter and mass of the balls. The total number of balls used in each experiment n_b, is given by N_v (number of vials) multiplied by N_b (number of balls in each vial). The last columns give the power efficiency factor $\langle P^* \rangle$ evaluated by electrical and mechanical data (see text)

			n_b		α		$\langle P^* \rangle$	
n_v	d_b (mm)	m_b (g)	N_v	N_b	e	m	e	m
0.086	6.0	0.881	2	125	3.00	3.30	0.852	0.492
0.17	6.0	0.881	2	250	2.95	3.20	0.797	0.483
0.90	6.0	0.881	2	1315	3.00	3.20	0.128	0.079
0.12	10.0	4.074	2	35	2.70	2.90	0.832	0.500
0.16	10.0	4.074	2	50	2.75	3.00	0.856	0.510
0.23	10.0	4.074	2	70	2.85	3.15	0.821	0.486
0.32	10.0	4.074	2	99	2.78	3.05	0.826	0.472
0.32	10.0	7.760†	2	99	2.80	3.05	0.240	0.143
0.60	10.0	4.074	2	183	3.10	3.30	0.363	0.226
0.80	10.0	4.074	2	245	3.10	3.30	0.144	0.089
0.20	10.0	4.074	4	61	2.90	3.13	0.796	0.504
0.24	10.0	4.074	4	72	3.10	3.28	0.767	0.480
0.40	10.0	4.074	4	122	3.05	3.25	0.647	0.412
0.48	10.0	4.074	4	146	3.15	3.45	0.546	0.344
0.43	15.0	13.745	4	36	2.95	3.28	0.622	0.381
0.64	15.0	13.745	4	54	3.10	3.30	0.367	0.234
0.86	15.0	13.745	4	72	3.10	3.33	0.200	0.113
					2.96 (5)	3.20 (5)		

†WC balls.

function of the rotation speed of the planetary mill as shown in Fig. 2(a).

A given charge of balls was identified by the filling parameter

$$n_v = \frac{N_b}{N_{b,tot}} \tag{10}$$

where N_b is the number of balls (in each vial) used in the experiment and $N_{b,tot}$ is the total number of balls, of that diameter, necessary to completely fill up the vial so that no ball movement at all is possible. For comparison some experiments were carried out in completely different elasticity conditions; namely tungsten carbide lubricated inner walls and balls were used. At the beginning and at the end of a series of measurements, the power absorption from empty vials were recorded. Small variations from the empty vials are of importance and were properly considered in obtaining the net power differences.

3.4. *Data handling of the net power differences*

The sets of data obtained, that is (w_p, P_e) or (w_p, P_m) were least squares fitted to an equation derived from equation (6), i.e. of the type

$$P_{calc} = K w_p^{\alpha} \tag{11}$$

where K and α were parameters to be determined by least squares minimization of the error sum

$$U = \Sigma\, w_i (P_{calc} - P_{exp})^2 \tag{12}$$

with P_{exp} the measured power consumption (P_m or P_e) and w_i the weight given to each experimental point [note, see Fig. 2(a), that at low w_p, expressed in RPM—rounds per minute—, the powers from filled and empty vials approach to the same values so that their differences are affected by a much larger uncertainty than those at higher RPM. Hence the necessity of properly weighting each measurement].

The results are given in Table 1 where the α values found are given as a function of the filling parameter n_v. Figure 4 shows the power absorption, again as a function of the degree of filling, for two typically employed rotation speeds of the planetary mill.

Assuming that equation (6) is valid for the actual homogeneous series of experiments, we can use the P_{calc} values to obtain the normalised values P^*, namely

$$P_i^* = P_{calc,i} / [\tfrac{1}{2} n_b m_b w_{p,i}^3 R_p^2] \tag{13}$$

where the exponent of the rotation speed has been kept constant to the theoretical value of 3. The

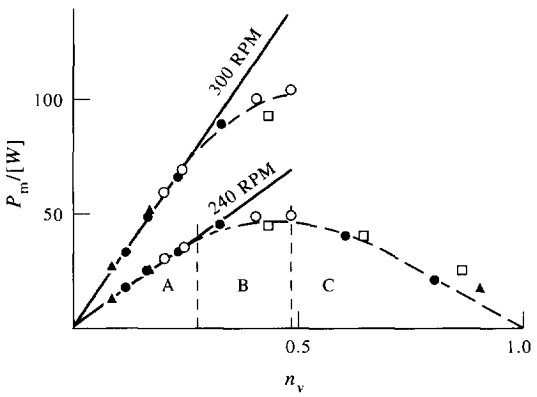

Fig. 4. Power absorption as a function of the degree of filling n_v for two typical rotation speeds of the planetary mill (240 and 300 RPM). The different ball diameters are indicated by triangles (6×10^{-3} m), circles (10×10^{-3} m) and squares (15×10^{-3} m). The solid symbols refer to values obtained with two vials. The open ones, refer to the values obtained with four vials normalized to two vials (i.e. divided by two). A, B and C indicate three different efficiency regions (see text).

average over the N experimental points of each milling experiment is given by

$$\langle P^* \rangle = \Sigma P_i^*/N \qquad (14)$$

where $\langle P^* \rangle$ can be regarded as a *power efficiency factor* (see Table 1, columns 8 and 9) and is plotted in Fig. 5 vs the degree of filling of the vials. We outline that $\langle P^* \rangle$ are experimental values since P_{calc} are nothing else than best fits through the experimental points.

4. ANALYSIS OF THE RESULTS AND DISCUSSION

4.1. About the law governing the energy transfer

In our first modelling approach, collision was assumed to be the main energy transfer event and equations of energy transfer properly derived [1]. McCormick *et al.* [2] have made some improvements of the original model introducing a "slip factor" that

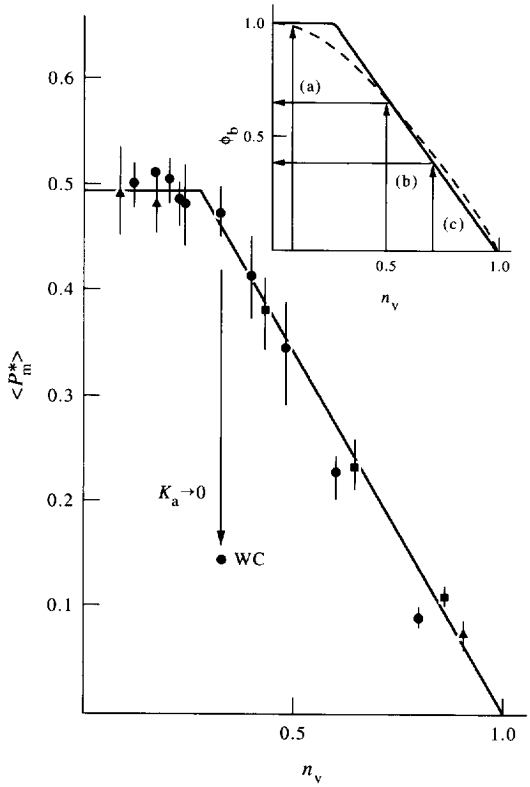

Fig. 5. Power efficiency factor, derived from mechanical powers, $\langle P_m^* \rangle$, as a function of the degree of filling n_v. Solid symbols identify the different diameters as in Fig. 4. The arrow in each symbol represents the maximum dispersion of the calculated P_i^* values using an α equal to three (see text). Region up to $n_v \leqslant 0.3$ identifies the *non-hindering* filling zone (see text). One experiment marked by WC (Tungsten carbide balls and vials) has been reported for comparison. The two solid lines are the best fits through the experimental points in the two different regions. The inset shows the hindering factor, ϕ_b, obtained through the actual experimental $\langle P_m^* \rangle$ values (solid rule) and by the semiempirical evaluation described in Ref. [1] (dashed rule). Three experiments with (a), (b) and (c) fillings have been carried out on the Pd–Si system (see text).

Table 2. α Values obtained from gross, empty or net powers (electrical, P_e, or mechanical P_m) for some milling experiments identified by n_v (see text). WC and FeZr labels indicate experiments with tungsten carbide balls and FeZr powder

n_v	$P_e P_m$	P' (gross) α	P° (empty) α	P (net) α
0.20	P_e	2.20	1.45	2.90
0.32	P_e	2.00	1.25	2.78
0.32 (WC)	P_e	1.80	1.25	2.80
0.32 (FeZr)	P_m	2.55	1.65	3.18

makes the predicted ball's trajectory very near to the one video recorded [2, 8]. Abdellaoui and Gaffet [3] improved the model still further by evaluating, other than the energy released per collision, the collision frequency and hence the shock power. In all cases collision was assumed to be the prevalent energy transfer event.

The actual results are in line with the previous assumptions. In fact, both electrical and mechanical power data (with a minor difference discussed below) can be fitted by equation (11) with an α exponent for the rotation speed very near to the value of 3 predicted by equation (6), that is, *predicted on the basis of a collision model.* This is true for any filling of the vials; that is the power dependence on the third power of the rotation speed is not substantially affected by the degree of filling as shown by the values reported on Table 1. This evidence supports the idea that even when the vials filling is high, still collision should be regarded as the main energy transfer event. McCormick showed by video registration [2] that even at low filling the balls, once detached from the wall, rather than *fly* along a given trajectory, follow that trajectory by tumbling over one another with a *cascade* of collision events. Therefore, what in the previous models [1, 3] was assumed to be a single collision event spending almost all the accumulated kinetic energy, should be rather regarded as a cascade of collision events through which the same kinetic energy is spent. Further, the approach of a collision cascade is certainly realistic at high levels of filling since surely balls hinder each other in that situation.

There is one significant objection that could be made to the previous arguments. In order to definitively prove that a "collision mechanism" is, in any case, the dominant energy transfer event, it should be verified that an "attrition mechanism" could be described by a set of equations different from equations (1)–(6) and expressing, at the end, a dependence from the rotation speed different from the third power. Just to mention, if we treat the "gross" or "empty" power data, completely different dependence on the rotation speed are obtained since *only the power differences* obey the third power law (see Table 2). Lacking such a description we can do, however, parallelisms with similar phenomena occurring in other fields. The power consumption registered when mixing a liquid with an agitator depends on the second power of the rotation speed until the flows created are laminar (equivalent of an attrition regime?). When the agita-

tor speed increases, vortices are created (turbulent flows) and the power consumption depends on the third power of the rotation speed [9] (equivalent of the collision regime?).

At present, therefore, we can state that the collision hypothesis as the dominant power absorbing process is strongly supported by the experimental findings either considering, for each launched ball, a single collision or a cascade of collisions. Concomitant attrition phenomena, likely occurring at high levels of filling, cannot be excluded, in principle, but they can be anyway included in the present approach based on collision as the experimental results indicate.

To conclude this section, a final remark about the values of α given in Table 1. The average α value obtained with electrical data is nearer to three and lower than the one obtained by mechanical data. Admitting that the small difference is significant, we trust more an α value greater than 3 rather than lower. In fact, the final equation (6) has been derived in the assumption (amongst others) that the elasticity coefficient (K_a) remains constant for a given series of data. However, it has been clearly verified by "free fall" experiments [7] that, at higher energies (i.e. highest rotation speeds), when all the other parameters remain constant, the energy spent in the collision increases, in percentage, slightly faster than expected (i.e. K_a increases a little) so that the powers measured at the highest rotation speeds can be relatively higher than those otherwise registered with a constant K_a value thus justifying an α value slightly greater than 3.

4.2. About the energy transfer itself

Figure 4 gives the power consumption due to the milling action at any level of filling. This power consumption represents the answer at macroscopic level to the sum of the microscopic events occurring during the milling process. In the present work the experiments have normally been carried out without powder (one of the exceptions is presented in a next section) but they are very close to the real milling process since the thin powder layer on the vial walls was enough to ensure an high K_a value. Therefore the information coming from the present results are of general interest and are of help to choose the right milling conditions in planning experimental works.

Figure 4 should really be a tridimensional pattern with w_p on the third axis. Two typical working rotation speeds (namely 240 and 300 RPM) are represented on the figure and several observations can be made. In a first region (A in the figure) the power consumption, at each w_p, *increases linearly* with the number of balls. Doubling the balls doubles the power consumption. The same effect is obtained by mounting four equally charged vials instead of two as can be seen by the perfect overlap of the open points whose original values were twice those represented in the figure. In this region we have really *no reciprocal hindering* of the balls up to about $n_v \leqslant 0.28$. Thus the prediction of equation (6) about a linear dependence of the power consumption with the number of balls is perfectly fulfilled. We want to emphasize that a n_v value very close to the one found here was derived for the non-hindering region in the original paper [1].

Above the non-hindering region, the power consumption increases less than expected. In other words the efficiency with respect to the charge decreases. This is true up to a plateau value around $n_v = 0.5$. Above the plateau, the power consumption decreases in absolute value and tends towards zero. We would like to point out that with completely filled up vials, the power data closely approach the values of empty vials further confirming that it is not the weight to determine the power consumption but exactly the sum of the occurring microscopic events.

Other information coming from Fig. 4 should be emphasized. For each filling, at the same w_p, we *do have* the same power consumption by combining a different number of balls and a different diameter. This fact directly confirms equation (5) that is, using larger balls we will have a greater intensive factor [ΔE governed by equation (1)] counterbalanced, however, by a lower extensive factor, [v_t, equation (4)] since a lower number of balls is needed to obtain the same filling of the vial. This observation and subsequent ones are of importance as we will discuss in the following.

The same and further information can be deduced from Fig. 5. In a first region, up to about the same n_v value of Fig. 4, the power efficiency factor $\langle P_m^* \rangle$ is really a constant. In equation (6), indeed, the P^* value (from which $\langle P_m^* \rangle$ derives) includes the parameters K_a, K_b, K_v that are constant for a given mill (K_a, K_v) and a given homogeneous series of experiments (K_a). Above the non-hindering region equation (4) is no more valid and the efficiency decreases dropping to zero as already described in Fig. 4. If, at the same filling, the elasticity of the collision (factor K_a) is drastically changed by using tungsten carbide the power factor drops towards much lower values as expected (see point marked "WC").

The inset of Fig. 5 represents the *hindering factor*, ϕ_b, obtained by normalizing to the value of one the asymptotic value of $\langle P_m^* \rangle$ (upper solid line of Fig. 5). The ϕ_b factor corresponds, ultimately, to the $\langle P_m^* \rangle$ (or $\langle P_e^* \rangle$) values, i.e. it represents *normalized power efficiency*. Now the power depends, following equation (4), on the product of ΔE (intensive factor) and v (extensive factor). The fact that the filling of the vial, beyond a certain value, decreases the power efficiency means that probably both ΔE and v are affected. In previous papers [1, 6, 10], in order to explain results coming from high filling experiments, the hypothesis has been done that the collision model *could be still assumed* just by introducing a suitable hindering factor so that the energy released per collision was given by

$$\Delta E^* = \phi_b \Delta E \tag{15}$$

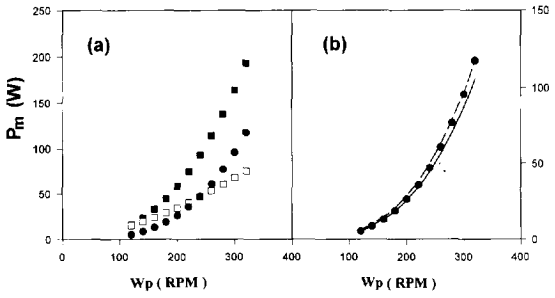

Fig. 6. (a) Power absorption, P_m, vs the rotation speed obtained with 99 balls (10×10^{-3} m) and 40 g of Fe–Zr (1:1, at.%). Gross, empty and net power differences are represented by solid squares, open squares and solid circles respectively. (b) Net experimental powers (solid circles) compared with theoretical ones using frequency factor from Gaffet [3] (solid line) and our modelization (dashed line) (see text).

where ΔE is still derived from equation (1) and the hindering factor was estimated in a semi-empirical way [1]. The original ϕ_b values are reported on the inset of Fig. 5 (dashed line) for comparison with the actual experimental results (solid line). As we will show in the next section, equation (15) has directly been confirmed by aimed milling experiments.

4.3. *Experiments with powder*

One of the main practical results drawn from the power measurements analysed in the previous sections is the experimental evaluation of the hindering factor ϕ_b. We stress the point that ϕ_b are experimental values since they come from the $\langle P^* \rangle$ values which have been, in turn, derived from the best fits through the experimental points.

In order to directly prove the validity of the power consumption predicted by equation (5) we have set up some milling experiments at low level of filling (in the non-hindering region with $\phi_b = 1$) and with powder [to ensure at most a K_a value, to be used in equation (1), very near to one].

Figure 6(a) shows the powers recorded in one of these experiments carried out using a Fe–Zr powder. The full symbols of Fig. 6(b) show the experimental net power differences compared with model values obtained using equations (1)–(5) and suitable values for K_a, K_b and K_v constants. The K_a value [equation (1)] has been set equal to one. The K_b value [equation (2)] is also near to one as can be derived from the planetary mill kinematic equations (1)–(6). The collision frequencies [equation (3)] have been evaluated by our model [1] and by the Abdellaoui and Gaffet [3] model (see Table 3). The final agreement between experimental and model powers is shown in Fig. 6(b) and is more than satisfactory. The agreement definitely proves that the collision model adequately describes the microscopic events occurring during milling and that, at macroscopic level, the final effect can be revealed by suitable power measurements like those reported in the present paper.

4.4. *Practical consequences for the choice of the right milling conditions*

In the previous sections we substantiated the main goal of the present investigation, that is the verification, by suitable power measurements, of the correctness of the collision model equations. The obtained results, however are not merely challenging speculations about the mechanisms of energy transfer. Rather, they do have practical consequences for the choice of the right milling conditions in order to obtain a given product or a given final processing stage. Some examples will be given in the following.

Any reaction is characterized by an activation energy. Normally, speaking of activation energies, one considers the thermally activated processes. In our case we are dealing with *mechanically* activated processes. However, the striking analogy existing between a solid state reaction activated by mechanical alloying and by thermal thin-films multilayers diffusion, has often been discussed (e.g. see Ref. [11]) and is nowadays accepted. We can just remember that formation of multilayers during the early milling stages [12]; subsequent reaction from the initial stage upon further milling [13]; temperature effects during mechanical alloying [14], have been observed and discussed. Coming to our approach, we can say that the energy released in a collision event can be thought of as the "equivalent" of a temperature rise (a review about temperature effects have been made by Koch [14]).

Having established this point, we can have a situation in which the activation energy of a given reaction is small compared to the range of input energies given by a mill (working at its lowest energetic conditions). We will refer to such a case as *cumulative* reaction in the sense that, practically, any milling condition can promote it. In this case, it is expected that the only parameter governing the end product should be the *integral work* done on the powder that is the product of the power consumption and the time of milling. Referring to Fig. 4 one would expect that milling at *constant power consumption*, for any different n_v and w_p, one should obtain the same end product for the same milling time. This argument will be dealt with in a forthcoming paper. On the other hand, if the activation energy of a reaction is high so that it can be reached or not by mechanical input, then we are in presence of a *threshold* reaction in the sense that only under given energetic conditions does the reaction occur. If these

Table 3. Collision frequencies (ν), as a function of the planetary rotation speeds, used for theoretical power absorption evaluations (see text)

W_p (RPM)	This work ν (s^{-1})	After Gaffet [3] ν (s^{-1})
100	5.6	5.1
200	11.3	10.1
300	16.9	15.2
400	22.5	20.3

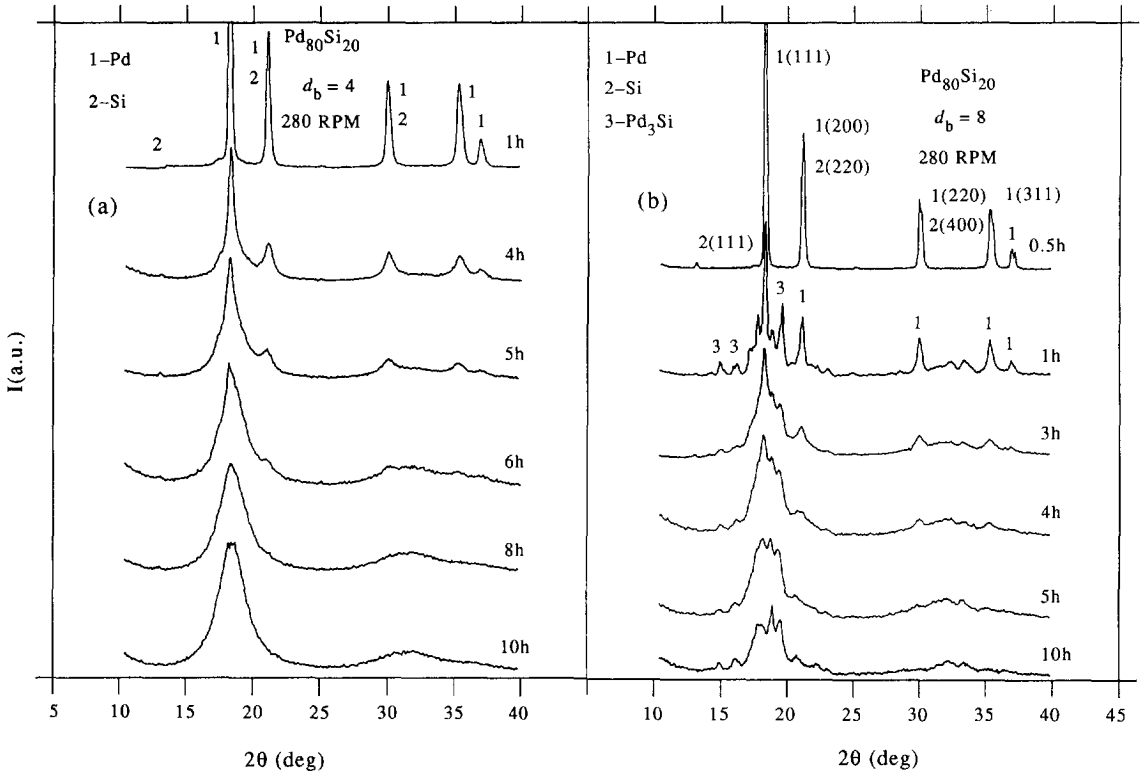

Fig. 7. X-ray patterns (MoK_α radiation) as a function of the milling times and the milling conditions (indicated by ball diameter d_b and rotation speed RPM) for the $Pd_{80}Si_{20}$ (at.%) composition. The filling of the vials for the two milling experiments is the same and equal to about $n_v = 0.04$. (a) Amorphous pathway. (b) Intermetallic pathway.

conditions are not reached, prolonging the milling time will be ineffective.

This second case has been examined by us, when dealing with the Pd–Si system [10, 15]. The threshold concerned the formation or not of an intermetallic compound depending on the milling conditions as reported in Fig. 7. The figure summarizes the behaviour observed in a large number of experiments [10]: below a given input value, Pd and Si react to form an amorphous phase [Fig. 7(a)]; above a threshold value an intermetallic compound is formed like in Fig. 7(b). With respect to this kind of reaction the parameter affecting the result is not the power consumption but rather the intensive factor ΔE. Indeed, referring to Fig. 4, the two examplificative experiments reported have been carried out at the same n_v value (in region A) and at the same w_p realizing thus the same power consumption P but different ΔE corresponding to the different ball diameters used. The consequence is that different end products, as a consequence of the different ΔE, were obtained.

The Pd–Si threshold reaction has also been used to verify the prediction of equation (15). According to that equation, the intensive factor ΔE is lowered according to the value of the hindering factor. Then for a threshold reaction, where ΔE governs the end product, it should be enough to vary the filling of the vials, keeping constant all the other milling

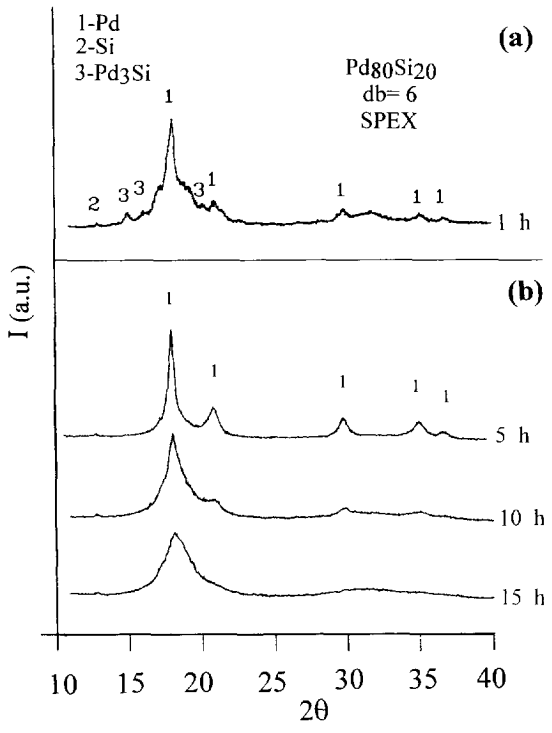

Fig. 8. Experiments carried out with a shaker device at the different levels of a filling n_v indicated in Fig. 5. Low ($n_v \leqslant 0.1$) and intermediate ($n_v \cong 0.5$) fillings follow the path indicated in (a) (intermetallic path). Large filling ($n_v \cong 0.7$) follows the path indicated in (b) (see text and Ref. [10]).

conditions, in order to vary the end product. This is exactly what has been verified with the Pd–Si system by the three filling factors indicated in the inset of Fig. 5. The results are given in Fig. 8 where it can be seen that at low and intermediate filling ($n_v \leqslant 0.5$ and $1 \leqslant \phi_b \leqslant 0.7$) the highest energy path is followed obtaining the intermetallic compound [Fig. 8(a)]. Filling up $n_v \sim 0.7$ ($\phi_b \sim 0.4$) reduces the effectiveness of each collision as predicted by equation (15) and the amorphous phase is formed [Fig. 8(b)]. The fact that the experiments of Fig. 8 have been carried out in a shaker device (Spex 8000 type) indicated that the hindering factor, obtained with experiments in planetary mill, has a general validity, as expected, for any milling device. On the other hand we have registered the same behaviour of the Pd–Si system by milling in a planetary mill the Fe–Zr system at different levels of filling [1].

5. CONCLUSIONS

The main conclusions that can be drawn from the present investigation can be summarized as follows.

1. The power consumptions due to the milling action can be revealed by suitable electric or mechanical power measurements. This has been done on a planetary mill but the principle can be extended to any milling device.

2. The experimental power measurements fit quite well with equations derived from a collision model. In particular the dependence of the power consumption on the third power of the rotation speed and the importance of the filling factor on the energy transfer have been established.

3. Some examples of the consequences of the obtained results on the choice of the right milling conditions to obtain wanted end products have been examined.

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