# Simoloyer® CM100s, semi-continuously Mechanical Alloying in a production scale using Cycle Operation – Part II

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## abstract

The production of large quantities of powders for industrial applications e.g. in paints or soldering materials is an aim of this work. For these applications, Cu- and Ag-particles with a special geometry (flakes) are needed. Based on milling experiments resolved by the High Energy (ball) Mill, the Simoloyer CM01-½l, with a grinding unit capacity of 0.5 l for laboratory purpose, a new grinding device, the Simoloyer CM100s1 (capacity 100 liters), has been developed under the same principle. The new device is suitable for a semi-continuous production of *mechanically alloyed* and *mechanically particle deformed* powders [1-4], has been designed and already been described in part I of this work [5].

Part II focuses on testing for and on industrial application of the processing and the equipment as well as on principles regarding productivity.

The determination of the energy consumption (energy balance) by use of the special control-software will be discussed.

The batch operation procedure exhibited that the efficiency of the system is even much higher than expected. The well-known problems when processing CMB-materials (exhibiting a critical milling behavior due to ductility etc.) [6-8] were solved by using the Cycle Operation procedure controlled by the control-software [9]. A production capability of 600 kg/day with the testing plant has been achieved.

The testing regarding the semi-continuous operation was done and consequently these data is available in this paper.

If, what is expected, the semi-continuously procedure and the use of the corresponding equipment will be successful and the main processing parameters can be kept, the expected continuous capability is calculated to be about 4 tons of powder per day.

The already proved application in the batch process is a revolutionary step, for the process and for mechanical alloying. The now available data for one of two planned principles regarding semi-continuos operation are discussed.

## 1. Introduction – brief summary of Part I

The aim of the current work is to replace a common and cost-intensive powder production procedure by a modern and high efficient processing technology.

In Part I of this work [5], the initial testing, the possibility of reaching the product-standard of quality with the mechanical alloying technique [1, 2] using a laboratory-scale with a half liter milling volume has been proved with satisfying results regarding quality where at the same time the necessary processing time has been reduced to the surprising range of several minutes.

The following step is the scaling up of the laboratory-experience to the industrial application. Therefore the Simoloyer CM100s for batch and semi-continuous powder processing has been designed and manufactured. The testing of the batch operation procedure with this system was done to verify principle and function and in particular to prove the reproducibility of processing compared to the laboratory scale.

Due to a high demand for powder production in time, the first chosen principle for the testing of the continuous operation has been the *depression* method (based on suction and separation using cyclones) as the therefore necessary equipment had already been there.

The current paper describes the testing of different production principles. The results including energy consumption and total costs are discussed and compared.

## 2. Milling Experiments - Industrial Scale

The industrial scale milling machine in which the experiments were carried out is shown in *Fig. 1*.

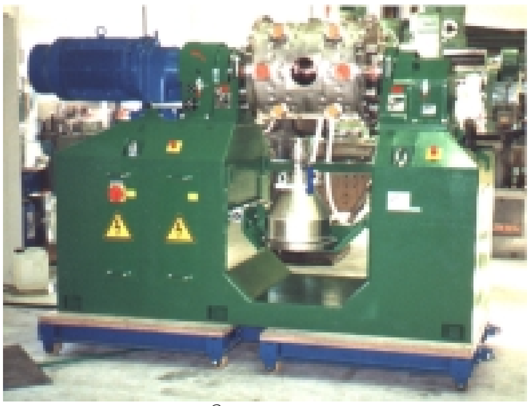

*Fig. 1: Simoloyer® CM100s, 100 l capacity for semi-continuous powder processing*

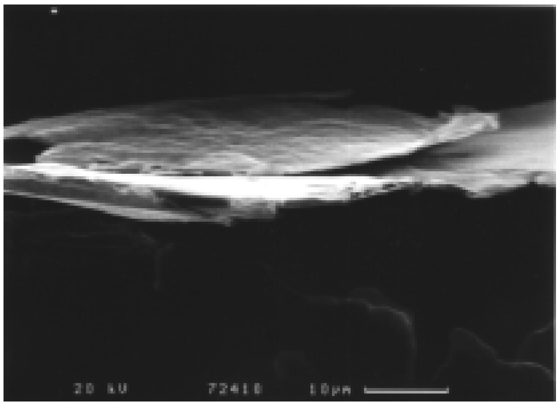

*Fig. 2: Lamellar thin Cu-particle, vertical oriented (SEM-micrograph)*

This system is based on the same principle as the laboratory-scale system that has been used for the initial testing [5]. Additionally, this mill has been designed to be used in different semi-continuous processing methods. The cycle operation procedure that is known to be very useful and partly necessary for the processing of ductile powders / CMB-materials [9] can be applied as well as an air-lock system with the through diameter of 100 mm (DN100).

The constant drive power of the milling device is pointed with 30 kW at a maximum rotational speed of 430 rpm. This refers to the maximum relative velocity [10] of grinding media in the system of 10 m/s approx. The maximum drive power is shifted to 45 kW by the frequency converter of the engine.
A part of the kinetic energy of the grinding media is transferred into the as atomized starting powder by the innumerable ball impacts [6, 7] and leads to a high deformation rate of the particles and the final product: a lamella particle shape (flake) with an approximately diameter-to-thickness proportion of 200:1 (see *Fig. 2*).
The specific energy of the system of 0.45 kW/l in maximum is lower than the value being applied for MA (0.55 kW/l) [11], but suitable for the here needed deformation-part of mechanical alloying that is correctly described as high energy milling (HEM).

In case of the production of Cu-flakes, the processing under inert gas or vacuum is not necessary and not wanted.
Only because of that, next to the well known batch operation, two different principles of a semi-continuous processing route are relevant:

One is the continuous transportation of the powder particles (product) through the impact zone (grinding chamber) using a high pressured carrier gas that is fed with powder particles outside the chamber. This carrier gas can be inert gas, but obviously not vacuum !
The second possibility is based on a vice versa principle: depression can be used to constantly discharge deformed particles due to their reduced apparent density and increased surface where the starting powder is constantly fed into the grinding chamber.

In both cases, the main criteria are the control of the transportation: in particular the remaining time of the particles in the process as well as the separation of processed, not processed and not ready processed particles. In case of batch operation these questions must not be answered because the process success can easily be checked by initial sampling and determination of the milling parameters.
In the following, these three different modes are discussed.

## 2.1 Batch Operation

For the batch operation experiments, the 100 liter grinding chamber of the milling device was filled with 200 kg of 4.76 mm chromium steel balls (100Cr6) which refers to a total filling ratio of 41 %. The Cu-powder charge was 20 kg which refers to a filling ratio of 268 % of the gap-space of grinding media between rotor-circle and chamber-circle and to a ratio of 12 % regarding the total grinding media volume [10].
Finally the powder-to-ball-mass ratio can be calculated to 1:10.

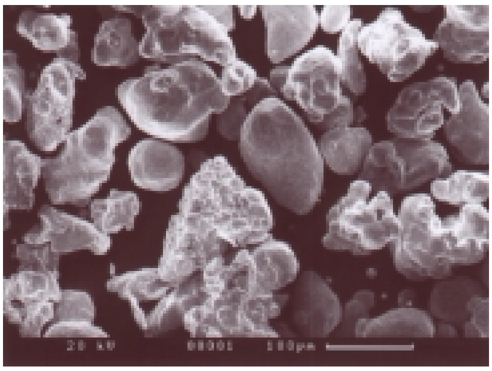

*Fig. 3.a.: Cu starting powder (SEM-micrograph)*

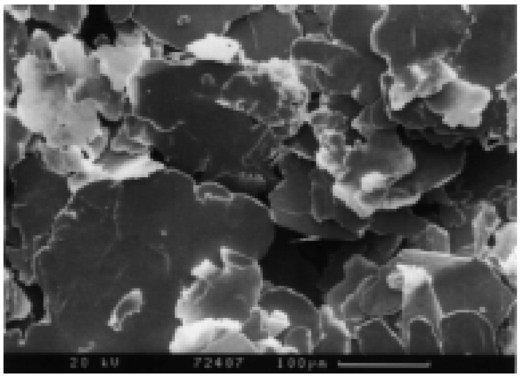

*Fig. 3.b:Deformed Cu-powder after 15 min of processing (SEM-micrograph)*

The Cu-starting powder (see *Fig. 3.a*) had a mean particle size distribution of 100 μm and different particle geometry. The particles exhibit spherical but also very fissured shapes which are due to the starting powder processing of water atomization. To minimize the cold-welding process between the Cu-particles and to enforce their plastic deformation by the high energy ball impacts, an addition of 0.5 wt.% of stearic acid was used. Furthermore, as a critical oxidation reaction of the Cu-powder is not expected, the high energy milling was carried out under air. The 20 kg-powder charge was processed for a duration of 3 minutes under a rotational speed of 430 rpm which refers to a total power consumption of 45 kW. The resulting Cu-flakes of a 15 min batch can be seen in *Fig. 3.b*. During processing the grinding unit, the rotor shaft, the pre-seal-units as well as the bearing-units were permanently water-cooled to avoid an over-heating of the powder and a damage of the machine parts. The temperature measurement is guaranteed by the compute control-software which allows a permanent and complete control of the milling device.

### 2.1.1 Discharging after Batch Operation

To discharge the product after batch operation under air, the blind-lid of the main port P01 is replaced by a draingrating that retains only the grinding media from passing but not the product.
The grinding unit is turned around 180º to get the main port P01 into the discharging position. Typically, without rotor motion almost no powder comes out due to the blocking effect of the grinding media and the powder itself (poor flowability of the powder).
Therefore the Simoloyer is usually set into (discharging-) operation, which is a constant rotation or in particular in case of ductile materials a discharging cycle [9].
In this case of lamella particles, even the standard discharging cycle procedure is not successful.
We found for discharging the whole powder stock, another special procedure being necessary. Several tests have shown, that during discharging most of the powder particles float on the surface of the grinding media level due to their large surface area and their lamella geometry. This is caused by the effect that the particles which have a specific low density are displaced by the milling balls with a higher density value. The result is that the flaky powder can not pass the gaps between the balls and therefore is forced to move into the opposite direction onto the top of the grinding media. The solution to get the powder out of the grinding unit was to apply a cyclic discharging procedure with extremely high peaks (violent-cycle). One discharging cycle here can be characterized by a time range of 3 sec at a rotational speed at 350 rpm followed by a duration of 13 sec at 65 rpm. Due to the inertia of the system and the initial ramp of the converter, the change of rotational speed can not be imagined rapidly.
The so called violent cycle had to be applied for 5 minutes to get the powder out.

The principle what happens with the powder particles during the violent cycle can be explained by *Fig. 4.* At 350 rpm the Cu-powder particles and the grinding media are completely distributed in the grinding chamber (“powder” and “grinding media gas”). In this state of the cycle, mainly the powder particles are concentrated close to the inner container wall. Now the rotational speed is reduced to 65 rpm and the gravity is getting active.

The result are pulsed high powder outputs for a short sequence each. However, the fact that the grinding media also drops under the influence of gravity and cover the discharging port, leads to the effect that the powder is blocked again. A second effect is that the low density flakes float on the surface of the grinding media so that again only a little powder comes out. As the rotational speed is increased to 350 rpm again, the grinding media starts cascading which leads to a limited higher powder output. After a short instant the powder output is decreasing again. Now the principle of the violent cycle is to get the short but very high powder output described at the beginning as often and as fast as possible. This works very effective and here discharges the 20 kg within 5 minutes.

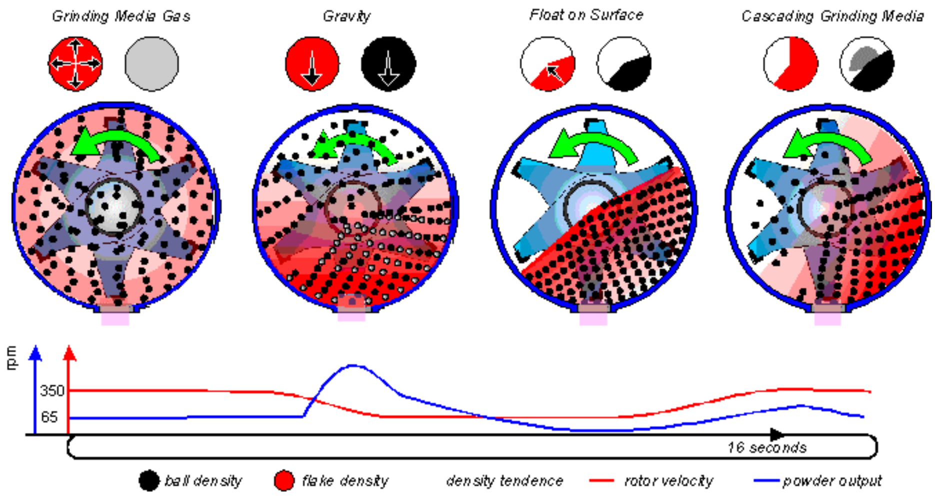


*Fig. 4: Principle of the violent discharging cycle*

If the position of the discharging port is inclined as shown in (*Fig. 5*), a higher powder output can be achieved, too. However the efficiency is much lower as in case of the here described batch of 20 kg, 15 minutes are needed for complete discharging.

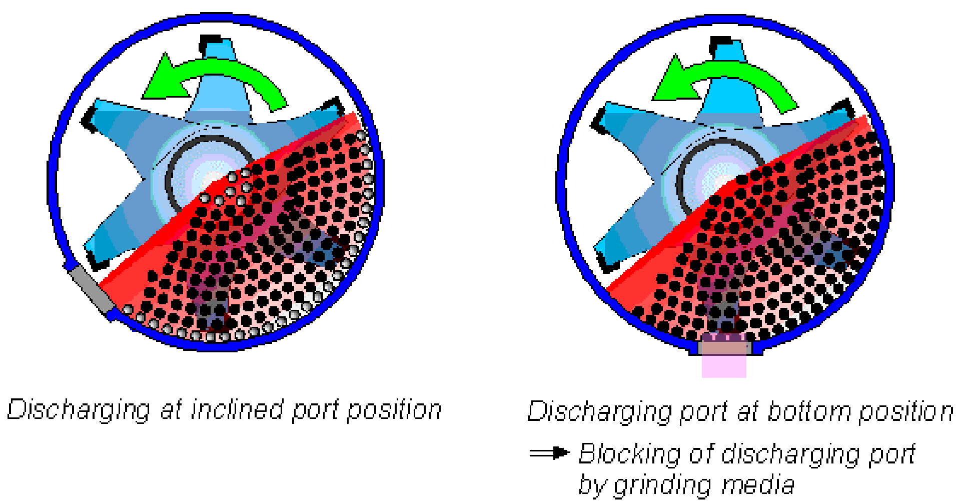


*Fig. 5: Scheme of the inclined grinding unit during discharging*

## 2.2 Semi-continuously Operation

One general goal of any industrial application or production is to receive the best possible product or often not more than the necessary quality but for the lowest possible price. As labor cost and time are very important cost-factors, a continuous processing method is in

general expected to be more efficient than a batch process at least regarding the production of large quantities.
The continuous mode saves expensive labor-times for charging and discharging if this can not be done automatically. The non-existence of interruptions in the process should safe time, too and should make the total process faster.
As far as we understand a continuous process being a process where 100 % starting material is transferred in a system to 100 % ready product and as it is not expected that the here discussed methods will reach this optimum stage, we describe the processing as a semi-continuous one. We do expect a continuous flow of material as well as a constant operation of the processing plant, but we do not believe that the here discussed production will be possible without any classification, separation and return of *not ready product* into the process.

In case of the production of Cu-flakes two different principles of a semi-continuous processing route are relevant (see 2.):
The compression-method where a high pressured carrier gas is needed and the depression-method where the principle of suction is used. In the following, these two different modes are explained:

### 2.2.1 Principle: using Compression

The scheme in *Fig. 6* shows the principle of semi-continuous operation inside the grinding chamber using *compression*. The additional ports M01-M07 designed for semi-continuously processing using *depression* (see 2.2.2) are not shown as they are not necessary here.

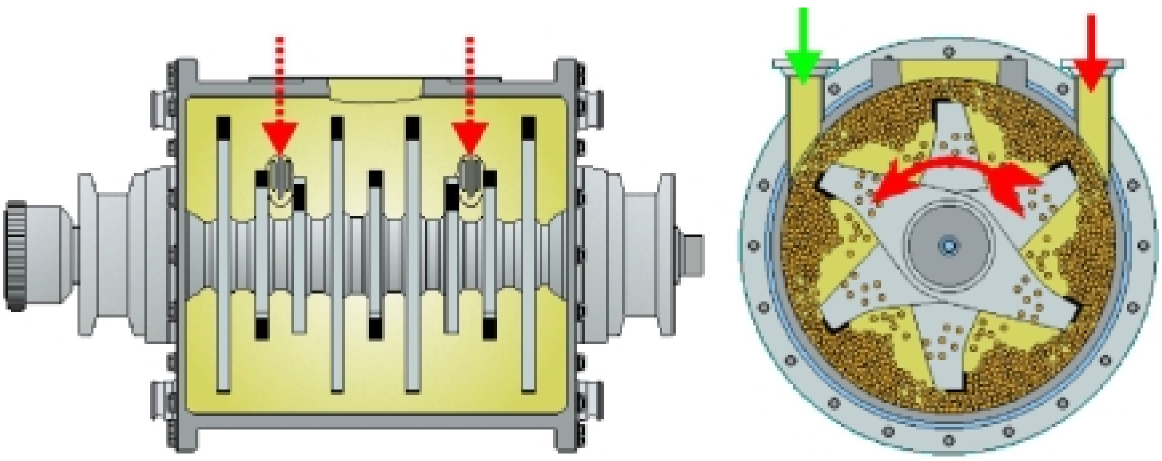

*Fig. 6: Principle of powder flow during processing*

The tangential ports Z01-Z04 (see figure 7) are intended for a powder transportation by means of a high pressured inert gas flow (carrier gas) into and out of the grinding chamber. For this configuration the main port P01 designed for batch operation is only necessary to get an easier access to inside of the grinding chamber e.g. for loading and unloading the grinding media. The tangential ports Z01 and Z02 respectively Z03 and Z04 (see figure 7) can be used in pairs either for powder and gas flow inlet or outlet. In this situation the powder is forced on a

tangential path into the grinding chamber and is injected either with the same direction or against the direction of the grinding media. The direction of the ball motion is important for the remaining time of the powder in the grinding chamber and the influence of the process on the powder particles. Due to the pressure gradient at those ports, the tangential accelerated powder particles are expected to be classified into light-weight/large-surface particles and heavy-weight/small-surface particles. This allows a first classification in the grinding chamber. The remaining time of the powder in the process is depending on the velocity of the rotor, the filling ratio of the grinding media, the powder content of the carrier gas, the carrier-gas pressure and the direction of gas/powder input.
Good results are expected to be obtained in case of a high rotational speed and or a high filling ratio when the input direction is chosen parallel to the rotation. In case of a low rotational speed and or a low filling ratio, the direction of input shall be in opposite direction to the rotation. A strong dependency to the accelerated mass of the balls is expected.
However, experiments concerning this configuration that is the most prospective possibility are not yet finished and are a main future task.

### 2.2.2 Principle: using Depression, testing

The second relevant possibility for the semi-continuous operation is the operation under depression. Concerning this configuration several experiments with the milling device have been carried out. The general idea is to charge and discharge the milling system continuously with powder in that way that always the same mass of powder is in the grinding unit. The transportation of the powder is done by depression.
Therefore this grinding unit is fitted with 7 additional ports in order to find out the optimum configuration by experience.
Regarding the filling-parameters, the same mass, size and quality of grinding media (200 kg of 4.76 mm chromium steel balls) as in case of the batch operation procedure was loaded into the grinding chamber. In several tests, the rotational speed has been varied from 250 to 340 rpm and the constant powder stock in the grinding unit has been varied from 5, 10 to 15 kg.

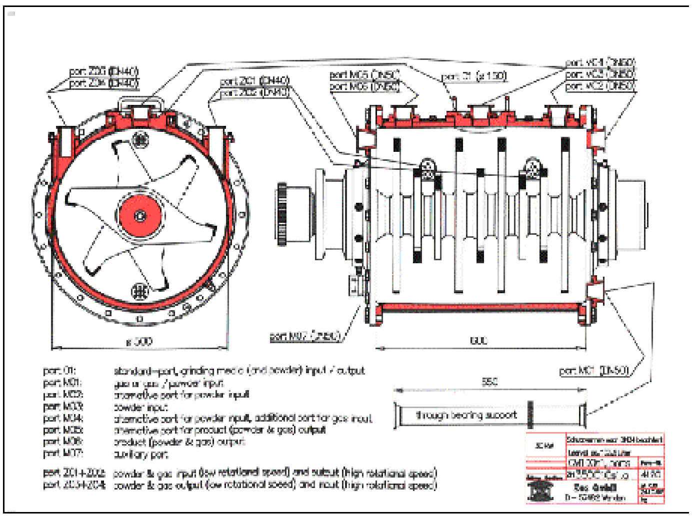

*Fig. 7: Scheme of the grinding unit, ports*

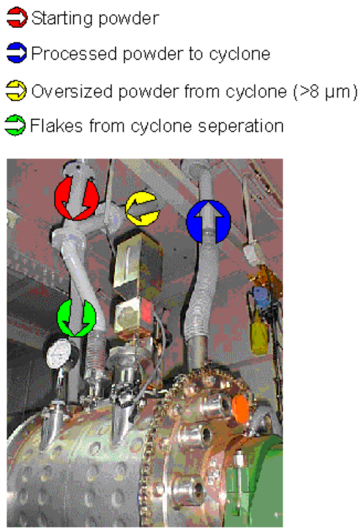


*Fig. 8: Piping-configuration (depression)*

The best results have been found with the following (piping) configuration:

The ports M03, M04 and M05 are connected to a first cyclone which manages the powder classification. The whole system including the grinding chamber is depressed by a fan installed outside of the system. The starting powder is fed into the grinding chamber by a cross-connection in the pipe between the port M04 and the first cyclone. A load cell measures the total weight of the machine and the amount of the powder in the process which is kept at a constant value of 10 kg by an electronic control system with an automatic feeder at the cross-connection. If the powder is varying from the constant value, the corresponding amount of starting powder is automatically reduced or added. Because of their reduced density and their increased surface area the powder particles are sucked out of the ports M03 and M05 and classified by the first cyclone. Heavy particles are returned through port M04, lighter ones are led through a pipe to a second cyclone were the particles are classified again. As a narrow distribution is needed, now to light particles are separated and the final product is deposited in a container which is positioned on a second load cell in order to measure the amount of produced powder. A defined amount of stearic acid (PCA) is added at a tangential port directly into the grinding chamber by another automatic feeder that is connected to the control-system. For the definition of the amount, the dependency to the power-consumption of the system is used. This is possible as this PCA has a lubricate-function and consequently influences the power-consumption where the filling parameters are constant. A necessity here are sensitive measurement systems that can not further be described here. Another tangential port is equipped with depression-measurement.

## 3. Energy Balance, measurement by the Maltoz-Software

The power measurement can be calculated by means of an implemented module of the control-software which is controlling the milling device. The idea is to receive the transmitted energy from the milling device into the powder. The stored energy of the powder after the milling process are mainly plastic deformation and surface energy. During milling a heat transfer is also taking place due to friction effects in the machine (bearing, grinding media, etc.). Furthermore, some unknown energy factors, e.g. noise and heat of powder due to powder reactions are present. By measuring the parameters of the process temperature, the drive shaft and their cooling (water flow, $T_{in}$ and $T_{out}$) as well as the temperature of the bearing units and the pre-seal units, an estimation of the energy transfer can be carried out if for an initial run without powder but the same other parameters the energy consumption of the engine is measured. Afterwards a second run with powder has to be carried out to receive the parameter for the energy under load. The difference of the two equations leads to the estimated energy which was necessary to achieve the deformation (s. *Fig. 9*).

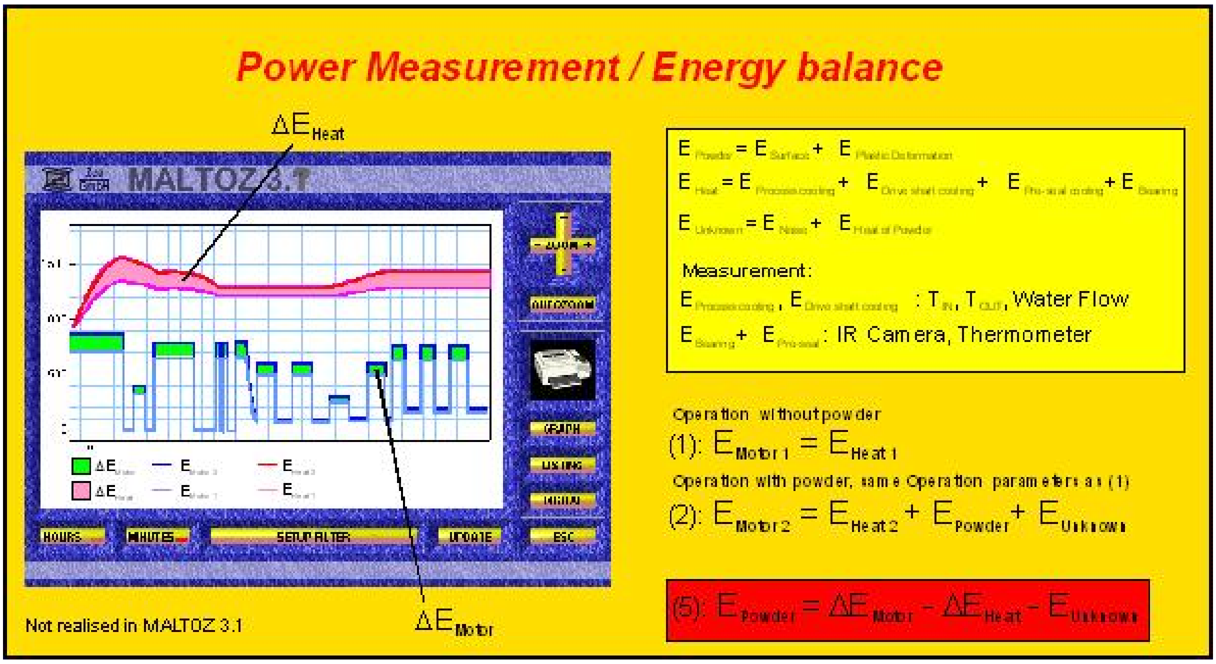


*Fig. 9: Power measurement and energy balance of the process*

# 4 Productivity

As for any industrial process the productivity - the relationship between input and output - is of major importance, several tests have been carried out in order to determine economical parameters regarding filling ratio (grinding media and powder), time and energy consumption.

## 4.1 Productivity of Batch Operation

By the described batch operation procedure (see 2.1, 2.1.1) it is possible to produce a powder batch of 20 kg in a satisfying quality within a processing time of 3 minutes. To discharge the powder, the described violent cycle (see 2.1.1) has to be applied for 5 minutes. Fixing the necessary time for charging the powder and handling the system to another 10 minutes, the total time for 1 trial is 20 minutes all together. This leads to a production-capability of 60 kg per hour (the theoretic process-capability refers to 400 kg/h). The needed total power had a value of 45 kW during processing and an average of 20 kW approximately during discharging by means of the violent cycle.

## 4.2 Productivity of Semi-continuous Operation using Depression

By the described semi-continuous operation procedure using *depression* (see 2.2.2), the most prospective results were achieved with 10 kg copper powder being constantly in the grinding unit.

Figure 10 describes the powder yield (received mass of ready product per hour), the measured apparent density of this product, the power consumption of the system as well as the calculated specific energy consumption per ton of powder yield for 5 tests with different rotational speeds (250 – 340 rpm). Additionally, on the right hand side of the graph, the corresponding data of the production-capability (not the process-capability !) of the described batch operation procedure is given.

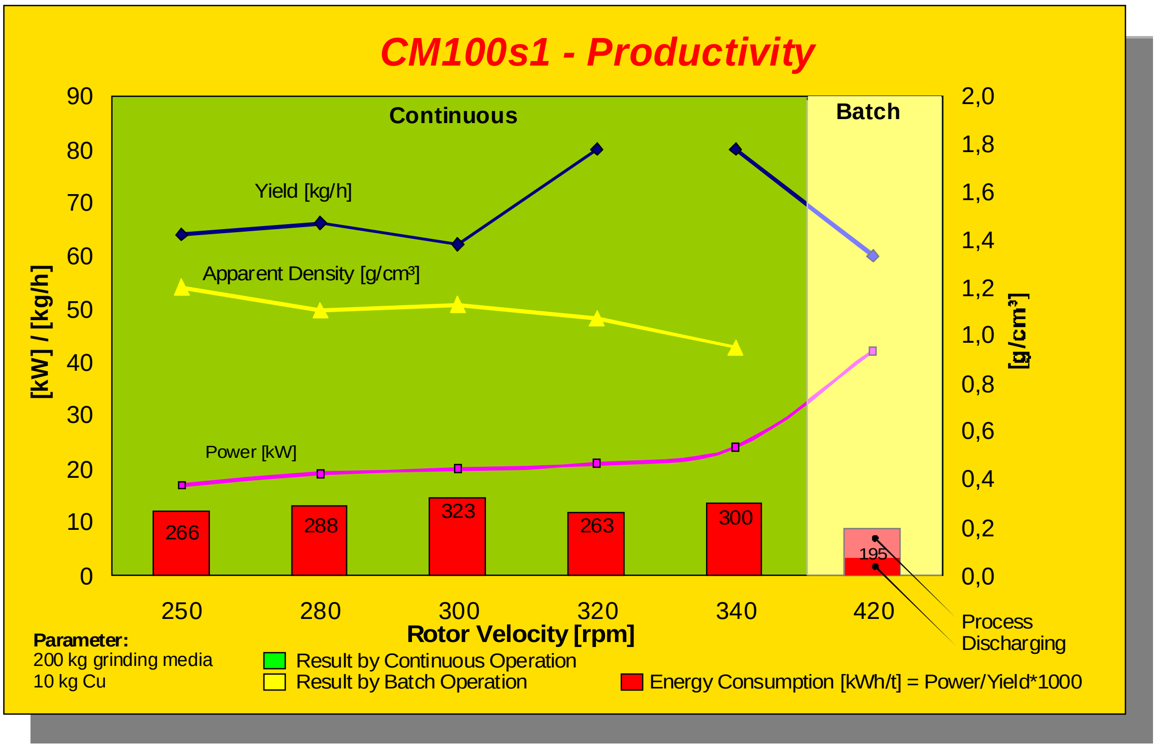


*Fig. 10: CM100s – Productivity*

The measured parameters show that at a rotational speed of 320 rpm a yield of 80 kg per hour with an apparent density of 1.07 g/cm$^3$ could be achieved at a minimum of an energy consumption of 263 kWh/ton.
Keeping in mind that the amount of powder and grinding media being in the process is constant, it can be explained that the apparent density is vice versa proportionate to the rotational speed of the rotor that is under these conditions proportionate to the maximum relative velocity of - and consequently proportionate to the kinetic energy transfer by - the ball impacts (collisions) in the chamber.

The comparison between batch operation and the depression-mode of semi-continuous operation regarding the production-capability shows that the result in case of the semi-continuous procedure is 25 % higher (60 kg/h : 80 kg/h). However also the total energy consumption is higher. This is explained by the much higher theoretic process-capability of 400 kg/h in case of the batch operation procedure.

## 5.1 Conclusions

*A very interesting and already existing industrial application of particle deformed powders has been described It has been explained that a part of Mechanical Alloying, the deformation-*

*part can be used with a much higher efficiency than any other common method to achieve the wanted goal (part I).*

It has been shown that the batch operation procedure leads to a high productivity of 60 kg/h. However, neither constant discharging nor the discharging cycle procedure could be successfully used to unload the powder (due to *float on surface*). Therefore a new discharging procedure (Violent Cycle) was described and introduced into the special control-software.

The two relevant principles (depression and compression) of semi-continuous processing were discussed.

The productivity of the depression method (80 kg/h) was found to be 25% higher than the productivity of batch operation. The total energy consumption was 26% higher (263 kWh/t : 195 kWh/t).
The even more prospective possibility, the semi-continuous processing using compression has not been tested yet!

## 5.2. References


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