HKP using Carrier-gas Assisted Discharging

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Abstract

The Simoloyer-mill is well known as a horizontal-rotary-ballmill that allows high-kinetic energy impact at relative velocity of the grinding media up to 14 ms^{-1}.
This often leads to very short processing times in the range of some seconds to some minutes for particle-size-reduction, particle-shape-deformation and reactive-milling.
If the device is applied for a batch-process, these short processing times result in a major barrier for the scaling up res. the commercialization of the process. The reason is the needed discharging procedure that often needs similar or longer time sometimes even at similar velocity. Thus the material that is discharged e.g. after 1 minute is expected to be different from the material discharged after 2, 3, 4 etc. minutes.
To solve this problematic, a carrier-gas-discharging unit TGD20 has been developed to rapidly unload processed material out of the vessel of a CM20-Simoloyer (20 liter chamber volume) at low rotational speed in a closed gas circuit under severe oxygen measurement and control.
The present paper explains the new device and describes the initial testing after particle size reduction of a glass-system under air as well as soft-MA of a metal-system under inert gas.
Results will be given in terms of powder yield/time relation, different gas-flow-velocities and rotational speeds. The materials were characterized by SEM and laser diffraction.

1. Introduction

Zoz GmbH mainly based in Germany is manufacturing equipment for High Kinetic Processing like Mechanical Alloying, High Energy Milling and Reactive Milling and also using this equipment for the production of advanced materials e.g. in the range of MMC's, MCC's and CCC's and finally producing a limited number of parts with some of these materials by HIP and LPS/HIP. Significant efforts are spend in R&D in materials science and some in process engineering. Toyota Central Labs, in co-operation with the Toyota Group and research organizations, based in Nagakute, Japan is one of Zoz's highly innovative customers covering the automotive field.
In this fruitful relationship, several steps ahead have already been made e.g. in order to bring a still exotic processing technique, which HKP in terms of industrial application still is, closer to commercial relevance.
We report here on one of these quasi joint-developments where Zoz is currently working on the development of carrier-gas assisted HKP. In the present work, the main goals are an easier, faster and less energy applying discharging process, which is very important in mass-production, after the batch operation step in HKP since the discharging procedure in the base-work took relatively long time at relatively high velocity and additionally the yield was not close to 100 % as well.
Therefore it had been considered, how the ongoing but not at all finished development at Zoz related to the carrier-gas assisted HKP could be of support for mass-production in early stage.
The present paper is a summary of a part of this process, to be more precise, the discharging part.
For the carrier-gas assisted discharging (CAD), a special device (TGD20a) had been designed, produced and tested which is content of this paper.

2. High Kinetic Processing, technique and application

Mechanical alloying (MA) has been described as a process where powder particles are treated by repeated deformation, fracture and cold welding by highly energetic collisions of grinding media in a milling process [1-3]. By this technique it is possible to synthesize new materials with new properties that cannot be created by conventional route e.g. due to a not present thermal equilibrium or immiscibility of their components. By structural design, important materials properties can be influenced (e.g. nanocrystalline, amorphous).

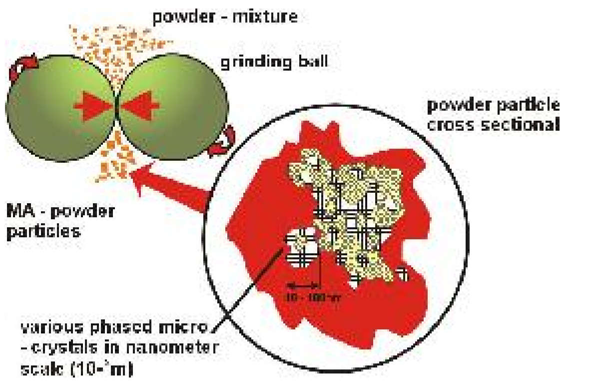


Figure 1: schematic of the collision as the main event of energy transfer

High energy milling (HEM) and reactive milling (RM) are performed by the same processing principle where the variation is in general based on the target of the processing, the transformation effect by the kinetic energy and the starting materials.

The different procedures can be described as High Kinetic Processing (HKP) where the collision of the grinding media is the main event of kinetic energy transfer from the milling tools into the powder [1-3]. Fig. 1 shows the schematic of the collision. The basic equation (1) describes the relation between the kinetic energy (E_{kin}) and the mass m and the velocity v of a single ball:

$$E_{kin} = \frac{1}{2} m v^2 \qquad (1)$$

It is clearly seen that the maximum relative velocity of the grinding media is the most determining factor contributing to the kinetic energy.
A typical example of practical importance that demonstrates the effects that are inherent in Figure 1 has been described in detail [4] in the system:

$$24\ Ti + Al_{11}Nb \rightarrow Ti_{24}Al_{11}Nb$$

In this work the influence of the milling parameters have been evaluated and basically the processing route of Cycle Operation for CMB-materials [5] had been invented. By this technique, the problematic agglomeration and sticking behavior of the in this case Ti-Al-based material in a dry milling process could be significantly reduced which did lead to an increase of the powder yield from < 4 % to 80 %. Later on similar effects were found in the Ti-Ni-system [6] and in the processing of a number of ductile metal flakes [7-8]. In 1999, Kim etal. investigated the difference of Cycle Operation and Constant Operation in the Fe-Co-system and found tremendous effects on particle- and in particular crystallite-size reduction [9].
If the here described processing method is applied for particle and /or crystallite size reduction or particle deformation (e.g. flakes) in single systems, this route is described as HEM. The goal can be to receive a special particle geometry e.g. for rapid and large-scale production of ductile metal-flakes in dry process without solvents and less energy e.g. for paint-pigments, conductive pastes and anti-corrosives [10-11]. On the contrary rapid particle size reduction of brittle solids like Enamels or Glass Fluxes has recently been introduced as a new application field [12-13].
The definition of RM is suitable if during milling a chemical reaction is wanted and observed.
By this route, a dispersion of ultra-fine (nano-scaled) crystals and/or a homogeneous dispersion of transformed phases in a matrix can be achieved [14].
A typical example of practical importance here has been demonstrated in the Ag-Sn-system [15]:

$$Ag_3Sn + 2\ Ag_2O \rightarrow 7\ Ag + SnO_2$$

Due to the CMB-behavior of this ductile system, again the processing has to be performed by applying Cycle Operation. With respect to the starting materials (e.g. Ag_2O instead of Ag), RM in this kind of principle can show economical advantage. Often these processes are environmentally benign as they avoid wastes e.g. in solid state synthesis where organic solid-solid reactions and others can rapidly proceed without any wastes and in the absence of solvents [16-17].

3. Batch processing, auto-batch and semi-continuously processing

3.1 Batch processing

The processing in batch operation is the most common and most simple procedure in HKP. The principle is to load a process chamber with starting material, to process the material and finally to unload the processed material. For charging, gravity is used which means the main-port of the grinding unit is arranged in top-position. For discharging, the grinding unit is turned for 180° (main-port in bottom position) and gravity and centrifugal force of the rotating rotor are used.
The main criteria and issues in batch operation are:

- Safe and complete charging (loading) of the starting material, e.g. under controlled condition like inert gas or vacuum.
- Safe processing of the starting material, e.g. under controlled condition like inert gas or vacuum including temperature control.
- Complete processing of the starting material, e.g. like no dead-zones or dead-layers during processing.
- Safe discharging of the processed material, e.g. under controlled condition like inert gas or vacuum.
- Complete discharging with a powder yield close to 100 %
- Acceptable relation between processing and discharging time res. kinetic impact during the same

One of the major issues here is safety which means both, protection for human and hardware and also safety for a successful process. To make it shorter, only the most critical processing shall be considered which would be e.g. a Ti- or Al-base material. In order to avoid e.g. oxygen pick up and a high kinetic oxidation reaction after processing which usually would end in an explosion, the starting material would have to be handled in a glove-box where the charging container is loaded. This container is then connected with the air-lock at the Simoloyer, the entire system is evacuated and the material finally charged into the process-chamber. Then this is either flooded with inert gas or further evacuated. After processing, the material must be discharged in safe condition again which is in so far the most critical part. Air-lock and (charging) container can easily be disconnected and cleaned or exchanged, however, the processing chamber can not be opened to outside at any time. This means the entire process can only be executed with a draingrating of Ask-type [18] which is shown in figure 2. And here it is already difficult to guarantee that during charging, first, the powder can pass the screen-grating in the first ball-valve and second, that no starting powder remains inside the draingrating before this is locked since if not, then later during discharging, the processed powder would be polluted with non-processed powder.

Figure 2: draingrating Askv-08-20-50 (G11/2v-G11/2-DN50

During processing it is important, not to have any dead-zones inside the chamber and not to create dead-layers during res. by the processing since then the process would not be successful and the powder product would not be treated unique. For the here regarded CMB-material [5], Cycle Operation [4-5] must be applied in order to counter sticking and agglomeration tendency where in some cases even an addition of a PCA, e.g. a lubricant like stearic acid can not be avoided.
For discharging, it is additionally important, to completely unload the material which means a close to 100 % yield is important. This is first because the target of the process is to receive the material and second because remaining material in the chamber will cause severe problems in terms of opening the same.
For the entire process, a proper temperature control is necessary which is usually cooling but sometimes also heating or cooling to extremely deep temperature. The oxygen-content must be controlled either by vacuum- or oxygen measurement and the temperature by a number of sensors connected to the operating software of the device.

Figure 3: Simoloyer CM01-2l (a), CM20-20l (b), CM100s2 (c)

Figure 3a shows a laboratory-scale Simoloyer CM01-2l that can be placed on a table next to the process controlling computer and is operated with water cooling or heating at rotation frequencies up to 1800 rpm. The grinding unit is connected with an Ask-draingrating to the DN40-air-lock and DN16-sampling-unit and is in charging/operation position and the air-lock is connected to gas- and vacuum supply. Figure 3b shows a semi-production Simoloyer CM20-20l with a DN50-air-lock in discharging position. Figure 3c shows a production Simoloyer CM100s2 with a special draingrating for metal-hydrides connected to a DN40-sampling-unit and just turned from operation- to discharging/sampling position.

3.2 Auto-batch processing

Auto-batch processing is in general a batch process with adapted and automatic charging and discharging procedure. The Simoloyer is then operated only in operation/charging position where for discharging not gravity but a carrier-gas flow [10-13] is used. By this, also the before needed effect of centrifugal force by rotor rotation can be tremendously reduced which can be of important advantage (see chapter 4).

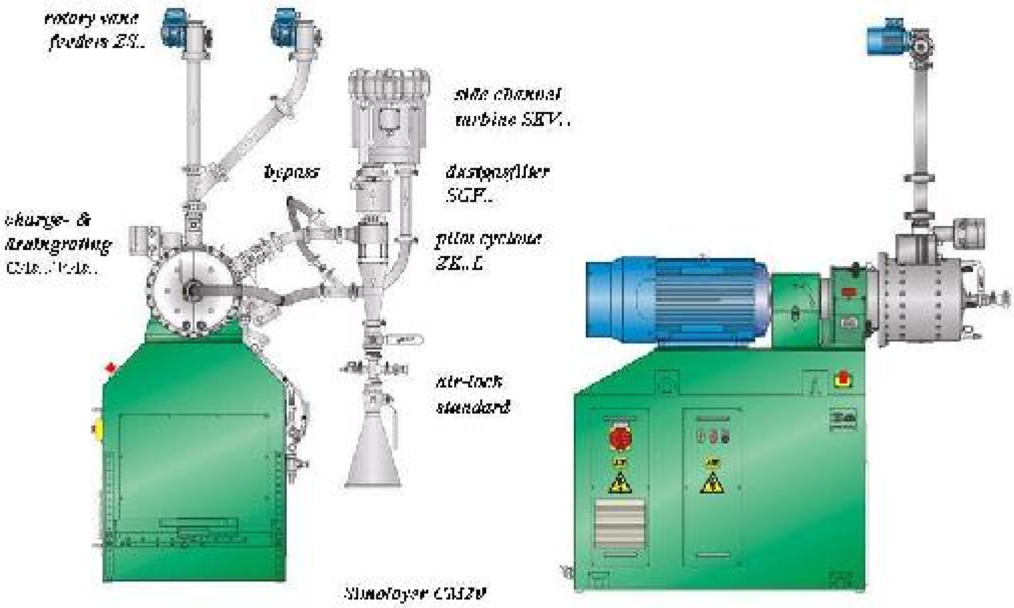


Figure 4: Simoloyer CM20-20ls2 in auto-batch configuration

Figure shows the configuration of a Simoloyer CM20 in auto-batch configuration. The grinding unit is equipped with a second main-port in order to adapt charging and discharging devices at the same time. The draingratings still must be Ask-type but equipped with automatic valves. The charging unit is

equipped with air-locks (not shown) following in line the rotary vane feeders. The discharging unit is based on a closed carrier-gas circuit where the gas is entered at the side-port of the grinding unit, then inside the chamber a multiphase-flow (gas and powder) is formed which is separated outside the chamber in the cyclone (see chapter 5). The operating software of the Simoloyer is extended and controls also the 4 valves at the 2 draingratings as well as the side channel turbine (gas drive) and one or more rotary vane feeders at the charging unit.
By this configuration and in case of exchanging the charging- and discharging containers into much bigger ones, it is possible to use this device for a constantly repeated batch process automatically. But then a main requirement is that complete discharging can be guaranteed (see chapter 3.1).

3.3 semi-continuously processing

The semi-continuous operation procedure can be divided into two principles, the one using depression and the one using compression. The difference describes a different use of carrier-gas where in depression mode the gas system is open and suction is used to carry powder particles out of the grinding chamber into a cyclone. In compression mode, the carrier-gas is cycled in a closed gas-circuit which may also be divided into a primary and secondary circuit. In both cases, the Simoloyer is continuously fed with starting material and continuously processed material is discharged. Important issues here are the control of the amount of powder in the grinding chamber and the separation res. classification of the product out of the gas-flow.

Figure 5a shows a graphic of the semi-continuous configuration of a CM100 Simoloyer in depression mode. The picture in Figure 5b shows an image of this unit where the cyclones are located in the second floor and can therefore not be seen on this picture.

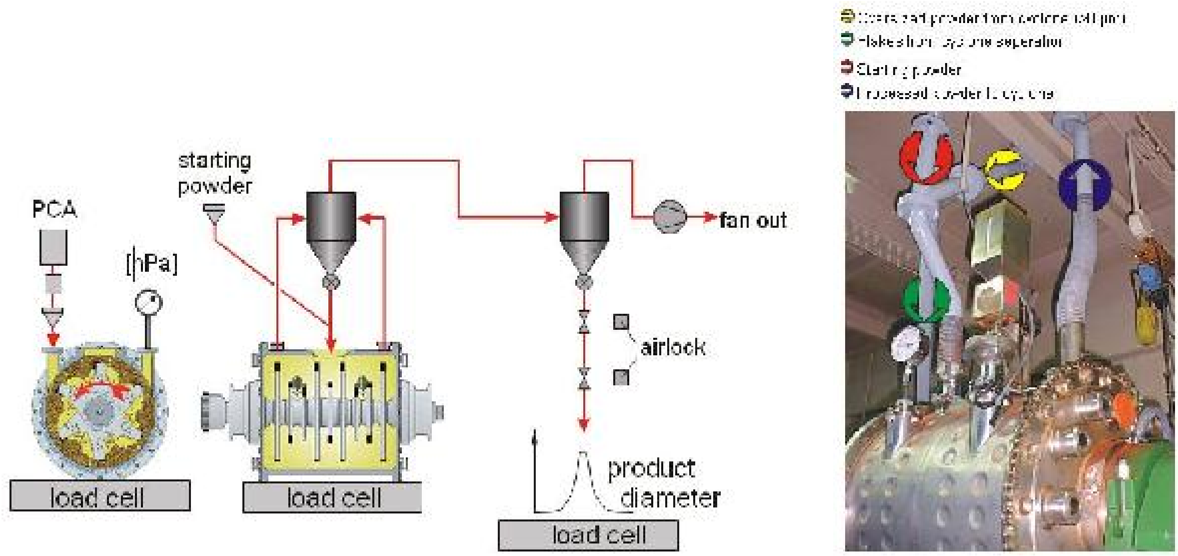


Figure 5: Simoloyer CM100-s1 in semi-continuous configuration (depression mode)

The entire unit is based on a load cell which is connected to a control unit that also controls the automatic feeder for the starting powder and in this case another one for a PCA. A fan is initiating depression through 2 cyclones that are connected with the grinding chamber where the first one separates to heavy particles and returns the separated fraction into the Simoloyer, the second one then separates product and gas.

Figure 6 shows a graphic of the semi-continuous configuration of a CM100 Simoloyer in compression mode.

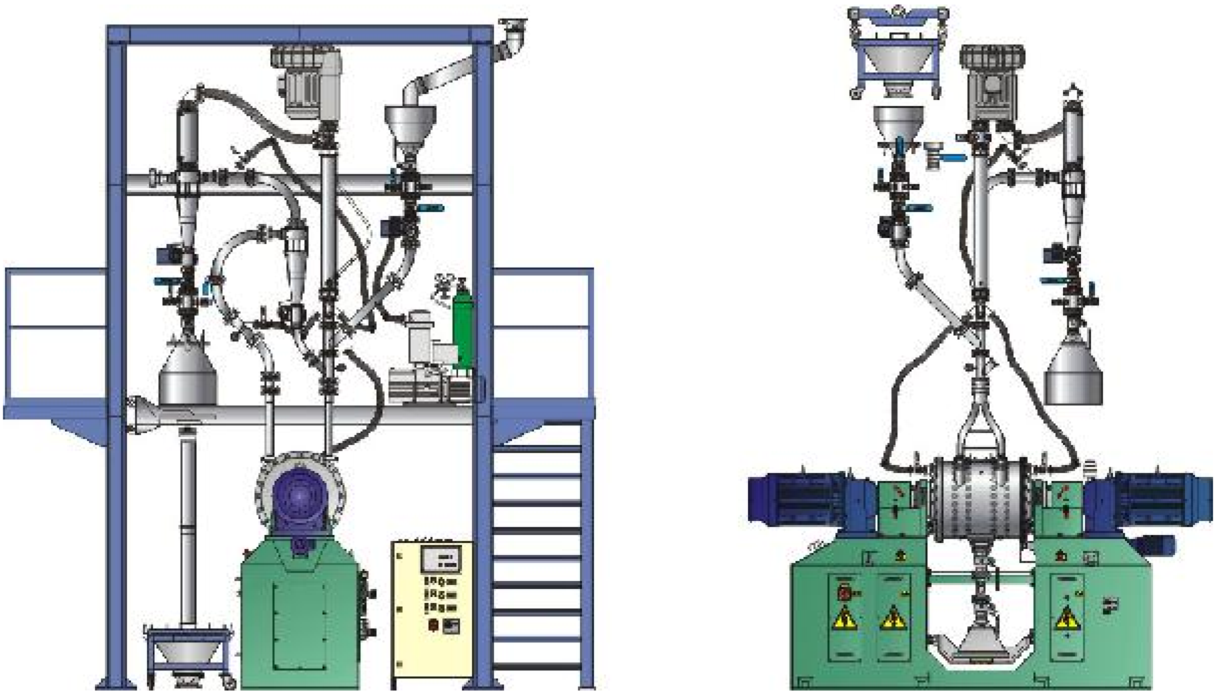


Figure 6: Simoloyer CM100-s1 in semi-continuous configuration (compression mode)

This unit that is currently under design to be used for the continuously refinement of OPC (ordinary Portland cement) in large volume is based on the experience of the earlier pilot-plants VS01a and VS20a based on CM01 and CM20 scale [12-13]. In general, the Simoloyer is equipped with a closed gas circuit where the side channel turbine on top blows the gas at high velocity through a heat-exchanger, then the carrier-gas picks up the starting material at the injection unit and goes into the grinding chamber at 2 tangentional ports. On the other side of the unit, the multiphase flow leaves the chamber via 2 more tangentional ports and goes into the first cyclone which has a return path into the chamber. In line after the first cyclone the classified multiphase-flow is separated in the second cyclone and the final material is collected in a container that is adapted with and automatic air-lock. Before the carrier-gas is returned into the turbine, it has to pass a filter-unit that automatically returns filtered dust into the second cyclone. In order to classify the multiphase-flow at the first cyclone, the gas system is equipped with various by-pass units which allow to adjust different velocities in each of the cyclones, the grinding chamber and the piping.
With respect to application, the main criteria is the remaining time. Up to now, this principle can only be realized for those processes that require in batch operation a processing time in the range of several minutes e.g. rapid particle size reduction of enamels [12-13]. Due to the effect, that the fine fraction of particles is continuously, which means here also immediately discharged out of the vessel, the dumping effect of this fraction (like liquid) is terminated and consequently the kinetic of the system is increased tremendously. Additionally it is important to understand, that in case of treating large brittle particles in up to several mm-scale by collisions of grinding media at high kinetic, these particles will collapse not just into two pieces but into a number of pieces of different size and finally with a size distribution. This means that also a very fine fraction is present right away. For the today's understanding, this all together leads then to the found extremely short processing times in continuously mode in the range of several seconds for these materials-systems which is also valid for the OPC [12-13].
Since in mid 2002, the carrier-gas technique was still in early stage development, but since it could already be expected, that this can be of significant support to discharge materials in particular when it is necessary to eliminate or at least significantly reduce the impact of the process during discharging, it was decided to separately bring ahead just the discharging part by carrier-gas and the idea of carrier-gas assisted discharging (CAD) and the idea for the TGD20a (see chapter 5) was born.

4. Discharging procedures, problematic

In case of any Mechanical Milling (MM) process and so in HKP, the goal is to receive a processed material which means quality and quantity is expected. Quantity here means a good powder yield which is a yield close to 100 % and this means a complete discharging is necessary.
The Simoloyer is know to supply easily complete powder yield in case of non-CMB materials which would be materials that are easy to process and would here mean that show no significant sticking and agglomeration tendency during processing and discharging. Ameyama etal. compared the Simoloyer and the planetary ball mill with respect to MM efficiency in refinement of HS-steel [19] and confirmed the Simoloyer at a yield around 100 % and the PBM below 50 %.

But even in case of non-CMB material, just the complete powder yield is not always enough. Very important is also the relation of processing time and discharging time which is an expression for the kinetic impact during both processing steps. Discharging shall not supply kinetic impact at all but of course does because the centrifugal force on the powder by rotor rotation is needed for discharging (in batch operation). Additionally, since during discharging, the relation between grinding media and powder inside the chamber is drastically increased (powder goes out, grinding media stays in), the kinetic is dramatically increased. In case of short discharging times after long processing, e.g. 2 hours processing and 10 minutes discharging, this is usually not problematic. But if this relation is completely different, like 3 minutes processing and 5 minutes discharging, then this cannot lead to a unique product since the material being discharged after 3+1 minutes must be expected to be different from the material being discharged e.g. after 3+5 minutes. Exactly this problematic with the given numbers was met in 1997 at a Cu- flake production with a CM100 Simoloyer [10] and this was the reason that this unit had to be operated in semi-continuous route [11] as given in figure 5.
This means sometimes it is a strict requirement to achieve a short discharging time at lowest kinetic impact and always this is of course a challenge for a more economical process.

In case of CMB-materials additionally we have the problematic, that the material tends to stick to the milling tools which in standard procedure would end in extremely long discharging times like more than 1 hour to receive a full powder yield, if this is possible at all. This must in particular countered already during processing by Cycle Operation and if necessary and acceptable, also by the addition of a PCA in order to avoid the formation of dead-layers [4, 6] and strong agglomeration. Only "free" powder in the chamber is processed successfully and only "free" powder can be discharged. And of course it is very non-opportunistic to accept the formation of dead-layers and then later during discharging to try to loose them by grinding since this will end in non-unique powder as described before. Cycle Operation then shall also be applied for the discharging step.

If we conclude the topic here into groups, then the third group might be that in case of any commercial production, first a complete powder yield is favorable and second a quick processing time (including discharging) is wanted. Additionally, since production usually means ongoing processing, a kind of automatic procedure is wanted and this means there can be no increased residues after every processing acceptable because this would end in a kind of overloaded grinding chamber. Next to this, and also in case if only batch processing can be applied, any interruptions shall be kept as short as possible and labor intensity of the processing shall be reduced to the minimum.
And this last paragraph contains the motivation to use the carrier-gas flow as described in chapter 3.3 for mass-production.

Table 1 tries to summarize the 3 groups as above, gives some hints for solution attempt and figures 7-9 in this table give a materials example for each of the groups:

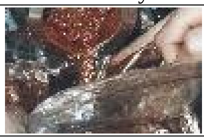
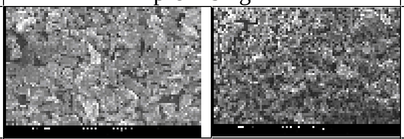
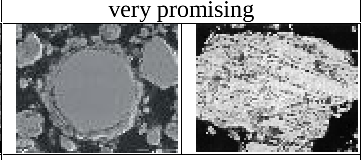
	group 1	group 2	group 3
why HKP ?	much faster process	only potential solution	optimization in general
related to	process engineering	materials science	production / pilot plants
problematic	discharging takes long time after extremely short processing time	material can not or almost not be discharged	discharging takes to long after medium processing time
main reason	short processing times reflect in must for short discharging	material is very ductile, tends to agglomerate and stick (CMB)	no definite reason
typical for application	rapid particle size reduction rapid flake processing (HEM)	MA, RM	MA, HEM, RM
typical for material	enamel, glass fluxes Cu, Ag…	Ti or Al- base material under MA / Al-flakes without PCA	metals and composites (MMC, CMC, CCC)
solution Cycle Operation	a must for ductile metal flakes	often a must	often a must
solution PCA	often helps, sometimes a must	often helps	often helps, sometimes a must
solution Cryo Milling	no need	little promising, scale !!	not realistic
potential CAD	necessary	promising	very promising
Figures 7-9			
	Cu-fakes after 3 min processing time, discharging by cycle operation	Ti-24Al-11Nb with and without cycle operation	discharging, no industrial solution yet
today's solution	semi-continuously, depression & compression mode	batch-process auto-batch	auto-batch semi-continuously

Table 1: attempt of listing discharging problematic in 3 groups

5. Carrier-gas Assisted Discharging (CAD)

The general idea of CAD is to use a carrier-gas to transfer processed powder-material out of the grinding chamber of a Simoloyer or any other MM-device. Here the main expected advantages are a decrease of discharging time, a decrease of kinetic impact during discharging (Simoloyer must only operate very slowly) and easier adaptation of any automatic process.

Figure 10 shows a picture of the carrier-gas discharging unit TGD20a (adapted to a CM20-Simoloyer) that has been developed in a co-operation inside a public funded project of the State of NRW in Germany. In general, TGD20a blows the carrier-gas (usually argon) at high velocity into the side-port of the grinding unit. The gas picks up the powder inside the grinding chamber where the rotor must only be operated very slowly in order just to mix the powder into the gas-flow. The multi-phase flow is exiting the grinding chamber at the main-port through the Ask-type draingrating and then arrives into the TGD20a again. Additionally there is a by-pass short-cutting the grinding unit which allows to adjust a different gas-flow velocity inside the chamber and TGD20a.

Figure 10: TGD20a adapted to SimoloyerCM20

Figure 11 shows the graphic of the main assembly sheet of TGD20a. The side channel turbine is driving the gas-flow which first passes the gas-control-module where the oxygen-content is recorded and controlled. Then the gas enters the heat-exchanger in order to cool down the gas temperature that is mostly heated up inside the turbine by compression and expansion but also inside the grinding unit of course. Then the gas-flow is guided by some connecting pipes into the side-port of the grinding chamber res. into the by-pass short-cutting the same. After the gas-flow is loaded with powder particles inside the chamber and therefore is now called multiphase-flow, it returns to the TGD20a in the completely closed circuit and goes into the pilot-cyclone. In there, solids and gas is separated. The powder (solids) is than transferred into an adapted standard air-lock with a powder container (not seen on the graphic but on the picture). The gas is sent from the cyclone into a dust-gas filter which protects the turbine from extremely fine particles in submicron scale that my pass the cyclone without being separated. Then finally the gas enters into the turbine again and the circuit is closed.
Additionally TGD20a is equipped with a number of valves and flow-control units that are used for initial evacuation before TGD20a is flooded with inert gas (Argon) res. that are used for some process control e.g. in terms of by-pass effect etc. (see chapter 7).

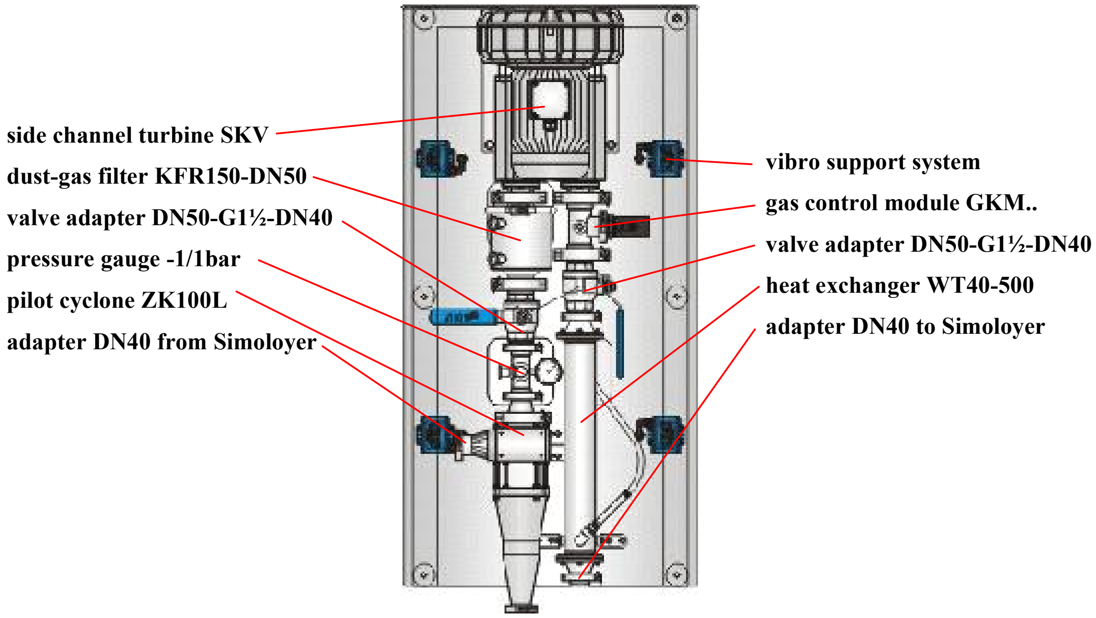


Figure 11: main assembly sheet of TGD20a

All before described units are fixed on the main assembly sheet (excluding some flow-control units) and this sheet is fixed in a kind of flexible connection to the base frame of TGD20a. Then the main assembly sheet carries 4 exocentric motors that initiate a vibration to the entire main assembly sheet with all the single units. Since all the flow-directions inside TGD20a are arranged vertically, this converter driven and in so far adjustable vibration supports the powder flow and avoids sedimentation inside the piping. In particular it is important for the function of the dust-gas filter since this unit (KFR1-DN50) is designed that the vibration will return filtered material trough the cyclone into the collection-container as soon as the turbine is stopped. Additionally this filter unit is equipped with 2 cleaning-gas ports that can be used for blowing argon-gas through the filter but in vice versa direction than the turbine does.
Figure 12 shows the electrical control unit of TGD20a as well as the control unit of the gas-control-module (GKM15a) for the oxygen control. The electrical control allows to adjust the turbine, the vibration module of the main assembly sheet (vibro-support system) and is already prepared to control a rotary vane feeder that is needed for later upgrade to auto-batch modus. All these units are converter driven and shall later be controlled by an extended Maltoz-software that today only operates the Simoloyer.
Additionally, on figure 12, the vacuum-pump and the gas-supply-bottle can be seen, that supply the inert carrier gas in the TGD20a and are assembled on the same.

Figure 12: electrical control unit of TGD20a and GKM15a

Figure 13: conductive flexible pipes and replaced flex-metal-tubes

Since in the closed gas-circuit of TGD20a, a gas-flow loaded with powder particles is moved at high velocity, careful attention must be given to the problematic of electrostatic charge of the unit. Therefore it was very important, that no electrically isolated units are present in the entire gas-circuit. On the other hand the first used flex-metal-tubes for the connection of TGD20a to the Simoloyer turned out to be not suitable since they have no smooth inner surface at all. Therefore we finally had to replace them with self-made conductive flexible tubes where we use polymer-tubes with included Cu-wire and fit them with KF-adapters. Booth kind of tubes are shown in figure 13.

6. Testing of CAD with enamel (particle size reduction, light-weight powder)

The initial testing of CAD with the TGD20a has been performed in Germany under the support of Toyota staff and then later the unit was shipped to Japan.
Of course for the first time testing, we did choose an easy to process, easy to discharge and a well understood material (in HKP). And this is enamel which is a glass-material [12-13].

6.1 Preparation of testing material and comparative batch process

In order to receive results in conventional discharging by gravity and centrifugal force by the rotating rotor, we started with the initial preparation (grinding) of the to be tested material. Enamel-frits in the size of 4-8 mm were processed in a CM20 Simoloyer with a grinding unit W20-20lm which was loaded with 25 kg of grinding media and 500 g of the glass-frits only because this material is quite light and for the later testing we wanted to use a glass-container DN50-500ml where this size can only contain 500 g of processed enamel powder.
Table 2 gives the detailed parameters for the initial processing of the enamel and figure 14 inside this table an image of the starting enamel-frits:

process parameters of batch process for initial preparation of the material		
device	Simoloyer CM20, 15 kW, Maltoz 3.2	
grinding unit	W20-20lm (20 liter, modular type b, water-cooled)	
grinding media	chromium steel, 100Cr6, 5mm, 25 kg	
processed material	enamel frits No. 02 (Pemco), 500 g	
product/ball weight ratio	1:50 (because of glass-container)	
rotational speed	450 rpm, refers to 7 m/s approx.	
processing time	30 min	
operation mode	constant, no cycle	
atmosphere	no, air	
discharging	draingrating As-20	white-enamel frit (02)

Table 2: parameters of batch process for initial preparation of the enamel-frits (& figure 14)

In order that the glass powder would not see any structure change during the discharging tests, we processed the material at quite low kinetic at about 7 m/sec only , but for a much to long time at 30 min where 5-10 minutes already would have been enough to receive the final particle size < 6 µm.
We produced a number of batches and this was always the starting material for the discharging tests. At any discharging test, we loaded 500 g of processed glass-material and distributed this in the grinding chamber and the grinding media by a kind slow pre-mixing always at the same parameters for

3 min at a rotational speed of 250 rpm of the rotor in order to simulate a preceding processing (before the discharging test).

The reference discharging test in conventional condition (test 01) was done at 200 rpm and 100 % powder yield was achieved after 1500 s which is equal to 25 min. It must be noticed, that in conventional discharging, the main-port of the grinding unit with the adapted container is in bottom-position (since we need to use gravity).
As an image of this configuration, figure 3b can be used since this is almost identical. The following table 3 gives the additional parameters for the comparative discharging test in batch processing as far as they vary and exceed the ones given in table 2 (base parameters).

additional parameters of comparative discharging test in batch processing (test 01)	
device, media, condition	>> base parameters glass-powder
rotational speed discharging	200 rpm, refers to 3 m/s approx.
position of grinding unit	main-port in bottom position
discharging 100 % yield after	25 min
operation mode	constant, no cycle
discharging	draingrating As-20, glass-container DN50-500ml

Table 3: additional parameters of comparative discharging test in batch processing

The given yield data was received online which means we stopped the time and after each interval did determine the volume with respect to the mark at the glass-container.

This procedure was suitable since the entire main assembly sheet and so the cyclone and the glass-container was vibrating which did lead to a quite flat and horizontal level in the glass-bottle. Additionally, at each measurement-point we took a picture from the bottle and used digital measurement on the PC-screen.The total volume at the end at 100 % was measured and then vis-à-vis transferred to the received data for every fix-point. Figure 15 shows the measurement-set up and figure 16 shows a representative series of pictures taken during one discharging test.

Figure 15: measurement set-up for the discharging tests

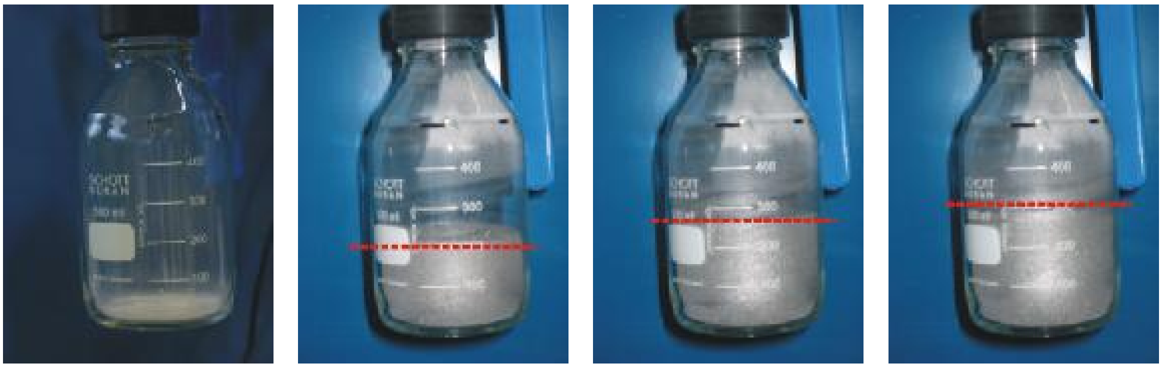


Figure 16: series of pictures for digital measurement of the powder yield at each fix-point

6.2 CAD-tests at varied rotor velocity

In the following testing of CAD with the TGD20a, the main-port of the grinding unit with the adapted piping to the TGD20a is in top-position. This has been chosen at first, since in later auto-batch mode, we would not be able to turn the grinding unit any longer.

In the first test of CAD with the TGD20a (test 02), we varied the rotational speed of the rotor at 100 % power of the turbine. In case of a rotational speed of 200 rpm, a 100 % powder yield is achieved after 20 min. In the next test we used a rotational speed of 400 rpm (test 03) at 100 % power of the turbine and received a 100 % powder yield after 9.5 min.

Table 4 gives the additional parameters for the CAD-test at varied rotor velocity as far as they vary and exceed compared to the base parameters.

additional parameters for CAD-test at varied rotor velocity (test 02-03)	
device, media, condition	>> base parameters glass-powder
pre-mixing	250 rpm, 3 min
SKV180-DN50 (turbine)	100 % (power)
by-pass	yes (100 % open)
vibration module	yes, 100 % power
rotational speed discharging	200 rpm, refers to 3 m/s approx. (test 02)
rotational speed discharging	400 rpm, refers to 6 m/s approx. (test 03)
position of grinding unit	main-port in top position
discharging 100 % yield after	20 min (test 02)
discharging 100 % yield after	9.5 min (test 03)
operation mode	constant, no cycle
discharging	draingrating Ask-20, TGD20a

Table 4: additional parameters for the CAD-test at varied rotor velocity

In result, the doubled rotational speed did lead to an about 2 times shorter discharging time which is surprising because one major target and expectation here is, to lower the rotational speed in order to reduce the kinetic impact during discharging. However, here it shall be pointed out, that the by-pass in this testing series was decided to always kept fully open (see chapter 7).

Compared to the conventional discharging in bottom position, the CAD-method did lead to an only 25 % shorter discharging time at 200 rpm which is not that much surprising, since the glass-powder is relatively easy to discharge. At 400 rpm the discharging time was 2.6 times shorter.

Figure 17 gives the diagram of the powder yield of the above tests 02-03 compared to the conventional discharging test 01 over time and volume:

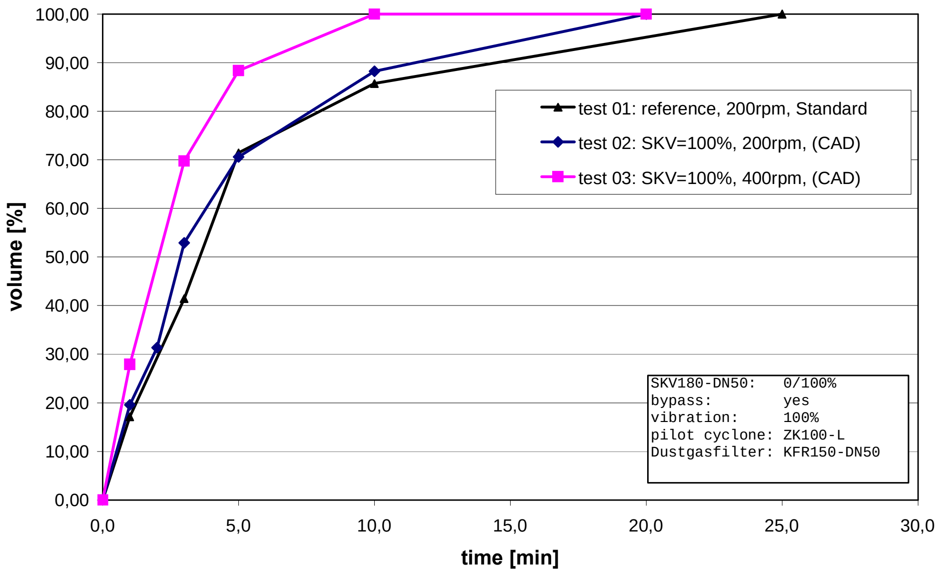


Figure 17: powder yield of tests 01-03 (conventional and CAD)

6.3 CAD-tests at varied carrier-gas flow and rotor velocity

In the next step we compared different carrier-gas flow-rates at different rotational speeds of the rotor. Therefore we use the before test-results 02-03 and performed the test 04-05 at 50 % carrier-gas flow-rates (50% powder of the turbine) where in test 04 the rotational speed was 200 rpm and in test 05 it was 400 rpm.

additional parameters for CAD-test at reduced turbine power (test 04-05)	
device, media, condition	>> base parameters glass-powder
pre-mixing	250 rpm, 3 min
SKV180-DN50 (turbine)	50 % (power)
by-pass	yes (100 % open)
vibration module	yes, 100 % power
rotational speed discharging	200 rpm, refers to 3 m/s approx. (test 04)
rotational speed discharging	400 rpm, refers to 6 m/s approx. (test 05)
position of grinding unit	main-port in top position
discharging 100 % yield after	29 min (test 04)
discharging 100 % yield after	20 min (test 05)
operation mode	constant, no cycle
discharging	draingrating Ask-20, TGD20a

Table 5: additional parameters for the CAD-test at reduced turbine power and varied rotor velocity

Table 5 gives the additional parameters for the CAD-test at reduced turbine powder and at varied rotor velocity as far as they vary and exceed compared to the base parameters.

In test 04 at 50 % carrier-gas flow-rate and at a rotational speed of 200 rpm, a 100 % powder yield is achieved after 29 min. In test 05 at 50 % carrier-gas flow-rate and at a rotational speed of 400 rpm, a 100 % powder yield is achieved after 20 min.

In result, at 50 % carrier-gas flow-rate, the doubled rotational speed did lead to an about 1.5 times shorter discharging time which has been 2 times shorter at 100 % carrier-gas flow-rate.
Figure 18 gives the diagram of the powder yield of the above tests 04-05 compared to the tests at 100 % carrier-gas flow-rate 02-03 over time and volume:

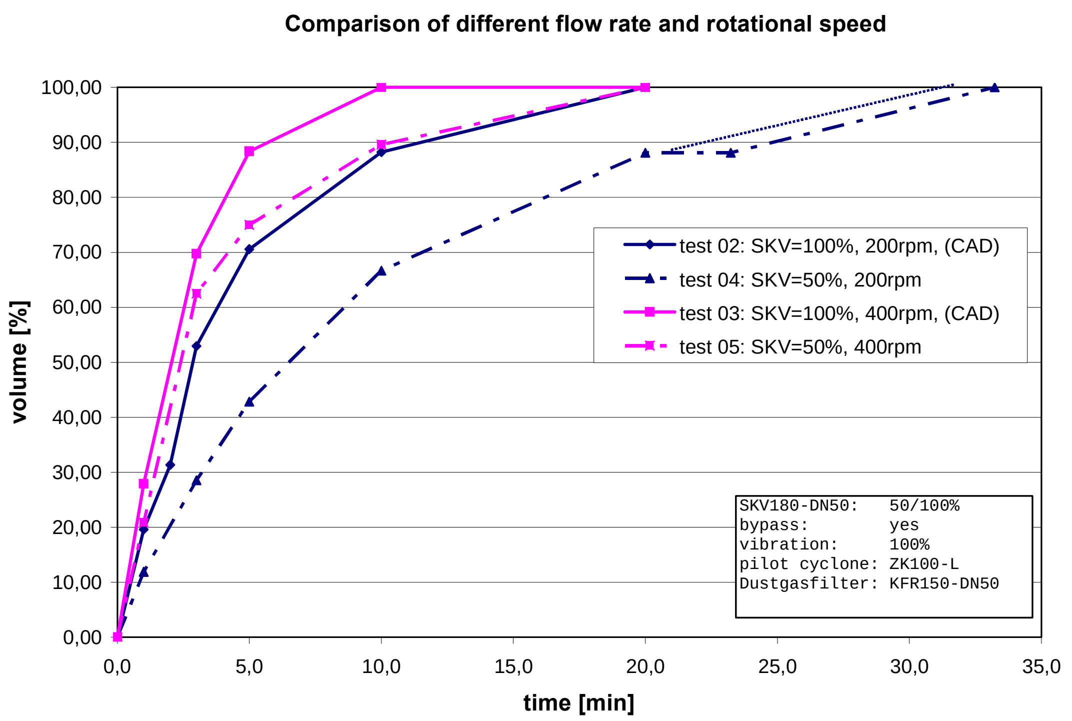


Figure 18: powder yield of tests 02-05 (varied gas-flow and rpm)

In result, always the tests at 400 rpm are superior, which means under the here determined circumstances (other parameters), the effect of rotational speed is higher that the effect of gas-flow (in the dedicated limits).

7. Investigation of auxiliary devices

7.1 Investigation of the by-pass (short-cut of grinding chamber)

The idea of the bypass is to allow a different carrier-gas flow-velocity and a different carrier-gas flow-volume in the grinding chamber and in the TGD20a (in particular cyclone and piping).

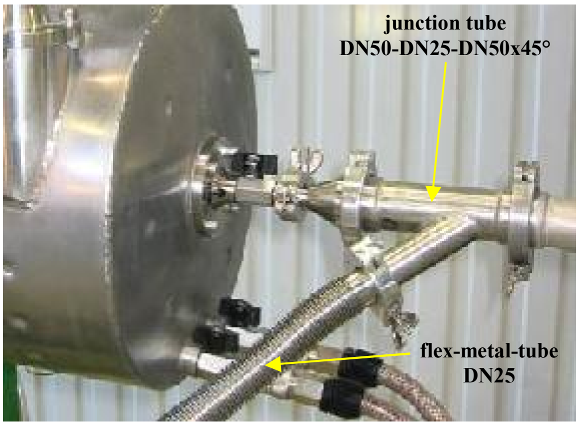


Figure 19: by-pass configuration at grinding unit

Therefore in front of the side adapter where the gas-flow enters the grinding unit and after the Ask-draingrating, where the multiphase-flow exits the grinding unit, each a junction tube DN50-DN25-DN50x45° was always installed and both junctions are connected with a flex-metal-tube DN25 and an adapter DN25-G1/2-DN25 (with ball-valve).
Since we expected, that we need a higher multiphase-flow-velocity in the cyclone and since the highest gas-flow-velocity in the piping is anyway favorable in order to avoid any sedimentation in the tubes, for all the CAD-test before, the by-pass was always fully open. Now in the next test it will be closed and results compared. The by-pass configuration is shown on figure 19. CAD-test 06 was performed, where the by-pass was closed and all other parameters were same as for test 02.

additional parameters for CAD-test with closed by-pass (test 06)	
device, media, condition	>> base parameters glass-powder
pre-mixing	250 rpm, 3 min
SKV180-DN50 (turbine)	100 % (power)
by-pass	no (100 % closed)
vibration module	yes, 100 % power
rotational speed discharging	200 rpm, refers to 3 m/s approx. (test 06)
position of grinding unit	main-port in top position
discharging 100 % yield after	30 min (test 06)
operation mode	constant, no cycle
discharging	draingrating Ask-20, TGD20a

Table 6: additional parameters for the CAD-test with closed by-pass

Table 6 gives the additional parameters for the test 06 as far as they vary and exceed compared to the base parameters.

In test 06 at 100 % carrier-gas flow-rate and at a rotational speed of 200 rpm, a 100 % powder yield is achieved after 30 min. In case of test 02 at the same parameters but with the opened by-pass, this was already achieved after 20 min which is 1.5 times faster.

Figure 20 gives the diagram of the powder yield of the above test 06 compared to the test 02 over time and volume:

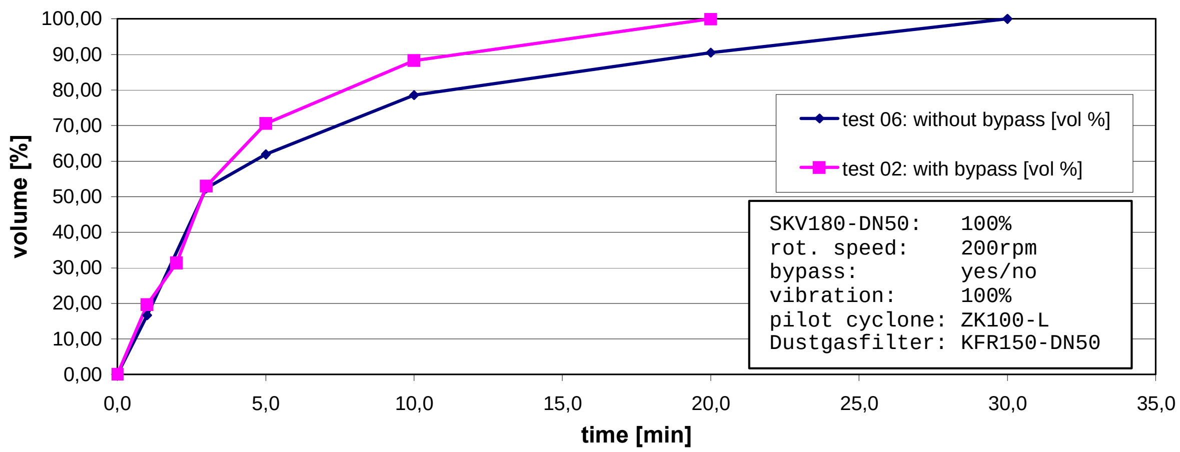


Figure 20: powder yield of tests 02 and 06 (varied by-pass mode)

In result, for the here tested glass-material, the by-pass seems to be important since it shortens the discharging time significantly.

7.2 Investigation of different side-adapters (gas-flow-in)

Since during testing, we notice a severe temperature increase at the piping reduction (DN40-DN16) in front of the side adapter at the grinding unit where the carrier-gas enters the same, we tried first and provisionally a draingrating RD-NW16-DN25 which is originally a dead-zone free loading-valve for the HV-Simoloyer (similar to vertical attritor) and which has not a free gateway of DN16 but of DN25. Then, since the results show an improvement, a new side-adapter RK-DN25-G1/2 was designed and manufactured. Figure 21a-c shows these devices in detail:

Figure 21: side-adapter RK-DN16-G3/8 (a), draingrating RD-NW16-DN25 (b), side-adapter RK-DN25-G1/2 (c)

In the following CAD-test 07, the side-adapter RK-DN25-G1/2 was tested under the same conditions as test 06 but with the replaced side-adapter which means the carrier-gas flow-rate was 100 % at a rotational speed of 200 rpm and with a closed by-pass.
Figure 22 gives the diagram of the powder yield of the above test 07 compared to the test 06 over time and volume:

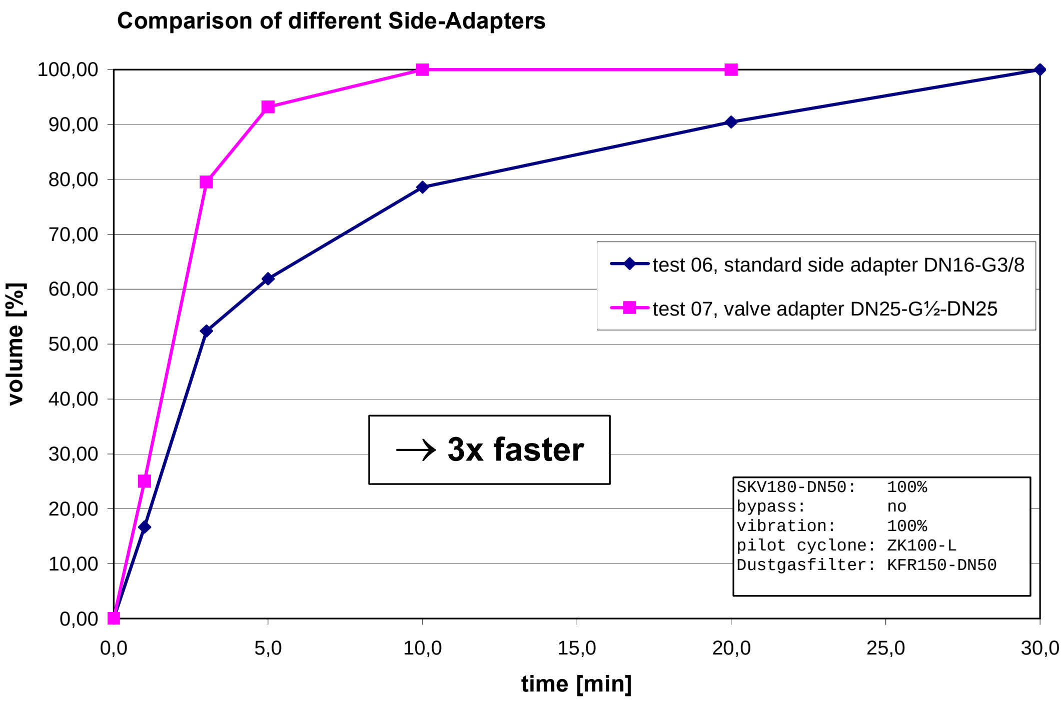


Figure 22: powder yield of tests 06 and 07 (varied side-adapter)

In result of test 07, a 100 % powder yield is achieved after 10 min. In case of test 06 at the same parameters but with the smaller side-adapter, this was achieved after 30 min which is 3 times longer.

In result of comparison, it has been very important to replace the side-adapter into the type RK-DN25-G1/2 since this shortened the discharging time by factor 3 !
Even the other parameters in terms of rotational speed and by-pass mode were not the better found ones, the test 07 did result in the shortest discharging time that was achieved in all tests up to this point.

7.3 Investigation of the heat-exchanger

Since we expected, that due to compression and expansion of the carrier-gas at the side-channel-turbine, the gas temperature would be significantly increased, TGD20a was equipped with a heat-exchanger WT40-500. In the following, we investigated if the heat-exchanger is necessary.
Therefore we did some testing where we varied parameters that significantly effect the carrier-gas flow which is the turbine power, the by-pass mode and the side-adapter type where here, we used the draingrating RD-NW16-DN25 (figure 21b), since this can be adjusted for different free gateways. We measured the temperature always at the gateway-tube of this draingrating (see figure 23) since here we still have the smallest gateway of the entire gas-system res. the largest reduction of cross-section of the same. And of course we did all these tests each at the water-cooling of the heat-exchanger (see figure 24) being active and alternatively being disconnected.

Figure 23: draingrating RD-NW16-DN25 with temperature measurement

Figure 24: heat-exchanger WT40-500

Furthermore, we did the temperature measurements when performing the first initial CAD-tests with the metal-powder (see chapter 8) since the thermal conductivity of the metal is significantly larger and therefore we decided to test the heat-exchanger at in so far real conditions because TGD20a is intended to be used for metals at first.

Figure 25 gives the diagram of the temperature development at various conditions under discharging of metal-powder that can due to confidentiality not be described in further details:

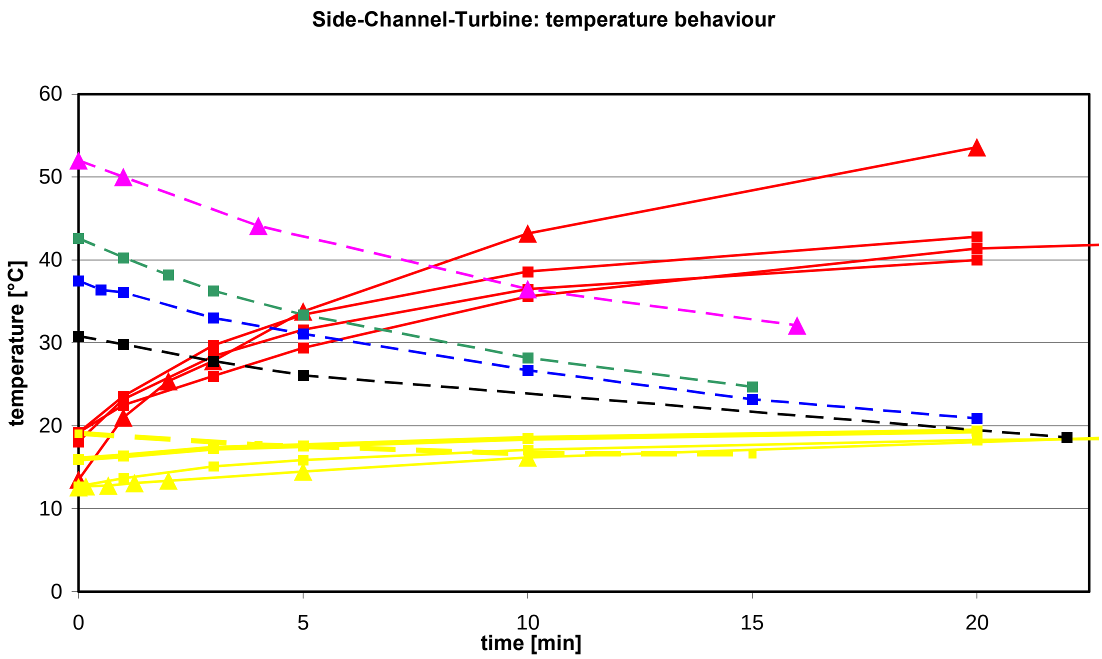


Figure 25: temperature development at various conditions

The following table 7 determines each symbol and explains every single temperature curve:

Temperature development at various conditions, symbols and comments					
IDl	Symbol	turbine	cooling	by-pass	side adapter
R1		100 %	off	yes	DN16
R2		100 %	on	yes	DN16
R3		100 %	on	no	DN16
R4		100 %	on	yes	DN25
Y1		50 %	off	yes	DN16
Y2		50 %	on	yes	DN16
Y3		50 %	on	yes	DN16
Y4		50 %	on	yes	DN16
Gr		25 %	on	yes	DN16
Bu		5 %	on	yes	DN16
Ba		3 %	on	yes	DN16
LR		0 %	on	yes	DN16

Table 7: symbols of temperature measurement in figure 25

The red curves R1-R4 show the temperature development starting between 12°C and RT at a turbine power of 100 % where only in case of R1, the cooling is switched off and only in case of R3 the by-pass is closed. Only in case of R4, the larger gateway of DN25 was used.

In result, during the ongoing discharging tests, the temperature increases after 20 min without cooling (R1) of the gas-flow to about 55°C and the slope of the curve gives the expectation of a further increase with the time. The same condition but with cooling (R2) leads to about 40°C and the slope has almost reached horizontal angel which means a further increase is not expected. The difference in case of R3 and R4 where by-pass and side-adapter have been changed is marginal in the range of 5°C and in slope follows R2.

The yellow curves Y1-Y4 show the temperature development at a turbine power of 50 % starting between 12°C and RT where only in case of Y1 the cooling had been switched of. However, all curves do not exceed RT at any time.

The green, the blue and the black curve (Gr, Bu and Ba) show the cooling effect of the heat-exchanger from starting points at higher temperature between 30 and 45°C at 25, 5 res. 3 % turbine power and reach RT after about 20 minutes. The light red curve (LR) shows the same but at no cooling and starting from about 52°C and at no turbine operation. The slope is similar to Gr, Bu and Ba which is simply explained by the fact, that there is no gas-transportation inside the system that would increase the temperature reduction faster.

In result, at 100 % turbine power, the heat-exchanger is necessary since without it, a temperature of about 60°C might be expected, where 55°C is already critical. In fact it shall be considered to use a heat-exchanger with higher exchange-capacity, e.g. a WT40-1000. At a turbine power of 50 %, the heat-exchanger is not considered to be necessary.

7.4 Investigation of the vibro-support system

Of course we also wanted to verify if the vibro-support system that is assembled on the main assembly sheet of TGD20a is suitable and here in particular we wanted to find out, if 4 engines work best or maybe a reduced number gives better results. For this test we simply fixed a sieving-capsule of a vibrating-screen SW28-VA-N at the vibrating main-assembly-sheet and tested the sieving effect with some standard reference powder at different numbers of engines and at different balance condition of the same. The best results we achieved at 50 Hz two unbalanced motors and therefore we rebuilt the TGD20a correspondingly.

8. Testing of CAD with metal-alloy (soft-MA, heavy powder)

In the next we describe the testing with metal-powder which is on of the targets for the final application of TGD20a. First tests have been done in Germany with iron powder ASC100.29 (Hoeganaes) and then later in Japan with Atomel 250M-100 (Kobe Steel) as a typical reference metal-powder for the tests. Both materials are pure iron and were of nearly same particle size.

We have not done any reference testing in conventional discharging mode in Germany. For this we used the information that has been received in earlier operation of the CM20 Simoloyer with the same

metal powder, where these results were actually not satisfying and this is why the here reported co-operation was finally set up. The yield in conventional discharging after batch processing was in the range of 75 % after a discharging time between 30 minutes and 1 hour which was considered to be to long for the set-up of a pilot production.
In Germany, we tested the TDG20a at different turbine power (50 and 100 %), at different grinding unit positions (main-port bottom and on top) as well as the effect of the by-pass and we used different Discharging Cycles which means Cycle Operation was always used.
In Japan, we tested the TDG20a at different grinding unit positions (main-port bottom and on top) as well as the effect of the by-pass and we always used Cycle Operation but did not change the Cycle itself.
The initial preparation of the material was done similar to the procedure with the glass-material.

8.1 CAD-tests at varied grinding unit position, by-pass mode and discharging cycle

In this test we compared different grinding unit positions (main-port bottom and on top) as well as the effect of the by-pass and we used different Discharging Cycles. The turbine power was always 100 %.
In test 10, we used a Discharging Cycle at 600 rpm for 5 s and 200 rpm for 5 s which means the rotational speed of the rotor was always going up and down between 200 and 600 rpm for 5 s each 5 s. The by-pass was open and the main-port of the grinding unit in top position.

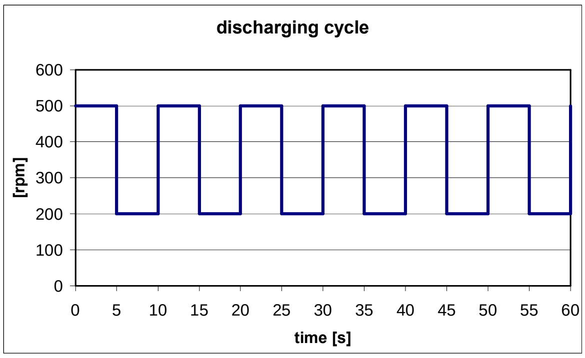


Figure 26: Discharging Cycle 500rpm-5s / 200rpm-5s

In test 11 we used the same parameters as in test 10 but the Discharging Cycle was composed at 500 rpm for 5 s and 200 rpm for 5 s which means in comparison, that the high-level was 100 rpm lower than in test 10.
Figure 26 give a visual example of the Discharging Cycle used at test 11:
In difference to test 11, in test 12, the bypass was closed and in test 13 we additionally changed the position of the grinding unit which means we turned the main-port into bottom position.

The parameters for the CAD-test 10-13 are given in table 8:

parameters for the CAD-test 10-13 (varied cycles, by-pass and position)	
device	Simoloyer CM20, 15 kW, Maltoz 3.2
grinding unit	W20-20lm (20 liter, modular type b, water-cooled)
grinding media	chromium steel, 100Cr6, 5mm, 25 kg
processed material	metal-powder (Germany), 500 g
product/ball weight ratio	1:50 (because of glass-container)
pre-mixing	250 rpm, 3 min
SKV180-DN50 (turbine)	100 % (power)
operation mode	Cycle Operation
atmosphere	argon, preceding evacuation
by-pass	varied
vibration module	yes, 100 % power
Discharging cycle	600rpm-5s / 200rpm-5s (test 10)
Discharging cycle	500rpm-5s / 200rpm-5s (test 11-13)
position of grinding unit	varied
discharging	draingrating Ask-20, TGD20a

Table 8: parameters for the CAD-test 10-13 (varied cycles, by-pass and position)

Test 10, at the Discharging Cycle with the higher top-level, the open by-pass and the main-port in top position, after 10 min discharging did lead to a yield of 93 %.
Test 11, at the Discharging Cycle with lower top-level did lead to a yield of 85 % after the same time which is 8 % less. Test 12, where thy by-pass was closed, did lead to a yield of 90 % which is again 5 % more and test 13, additionally with the main-port in bottom position, did lead to a yield of about 100 % after 10 minutes which is the best achieved result. It shall be noticed, that the data in % is rounded to a full number.

Figure 27 gives the diagram of the powder yield of the above tests 10-13 over time, volume and calculated proportion (percentage of load):

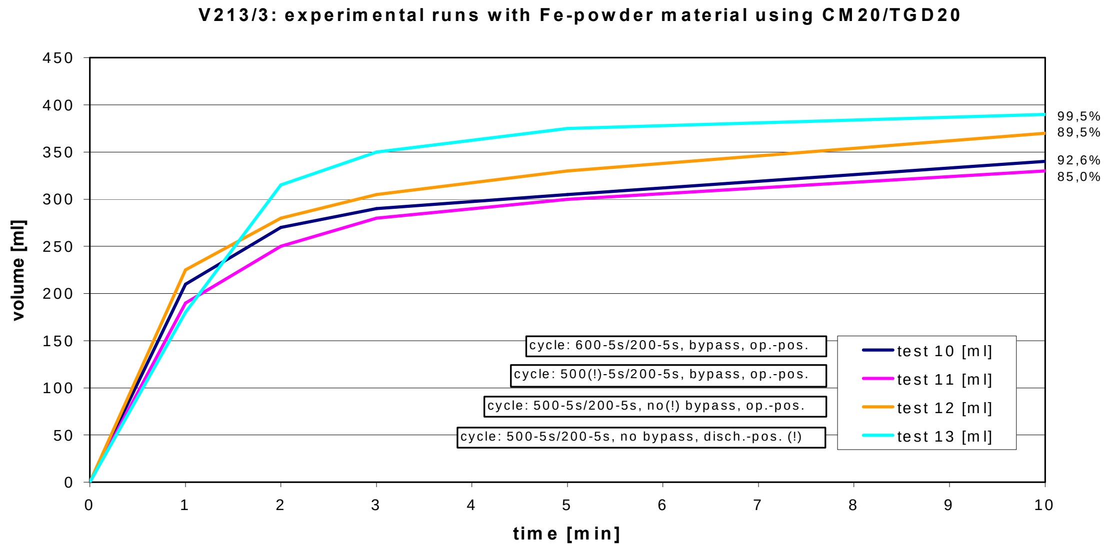


Figure 27: powder yield of tests 10-13 (varied cycles, by-pass and position)

In result, in case of the here tested metal-powder, the by-pass does not play a that important role as in case of the light-weight glass-powder where the difference was reported much more significant.
Slightly more important here is the position of the main port, in other words the effect of the gravity, since the best results were achieved in bottom position.
It is important to notice, that all 4 CAD-tests did lead to far better results than achieved earlier in conventional discharging where a yield of 75 % was achieved after more than 30 minutes discharging. Here, 75 % are achieved already after an average time of 2-5 minutes which is about 10 times faster and the total yield is improved from 75 % to 100 % if the parameters at test 13 are applied.

8.2 CAD-tests at medium turbine power and varied rotational speed

In this test series we compared different rotational speed at medium turbine power which was 50 %.
In test 14 we used constant operation at 200 rpm and in test 15 we changed to 400 rpm.

additional parameters for CAD-tests at medium turbine power (test 14-15)	
device, media, condition	>> base parameters metal-powder
pre-mixing	250 rpm, 3 min
SKV180-DN50 (turbine)	50 % (power)
by-pass	yes (100 % open)
vibration module	yes, 100 % power
rotational speed discharging	200 rpm, refers to 3 m/s approx. (test 14)
rotational speed discharging	400 rpm, refers to 6 m/s approx. (test 15)
position of grinding unit	main-port in top position
discharging 100 % yield after	30 min (test 14)
discharging 100 % yield after	20 min (test 15)
operation mode	constant, no cycle
discharging	draingrating Ask-20, TGD20a

Table 9: additional parameters for the CAD-tests at medium turbine power

Table 9 gives the additional parameters for the tests 14-15 as far as they vary and exceed compared to the base parameters:

In test 14, a rotational speed of 200 rpm, did lead to a yield of about 100 % after 20 min discharging. In test 15, a rotational speed of 400 rpm, did lead to a yield of about 100 % after 30 min discharging.

Figure 28 gives the diagram of the powder yield of the above tests 14-15 over time and volume where in case of test 14, there was a time-break for some technical reason which means the identifies 180 s must be deducted from the total time:

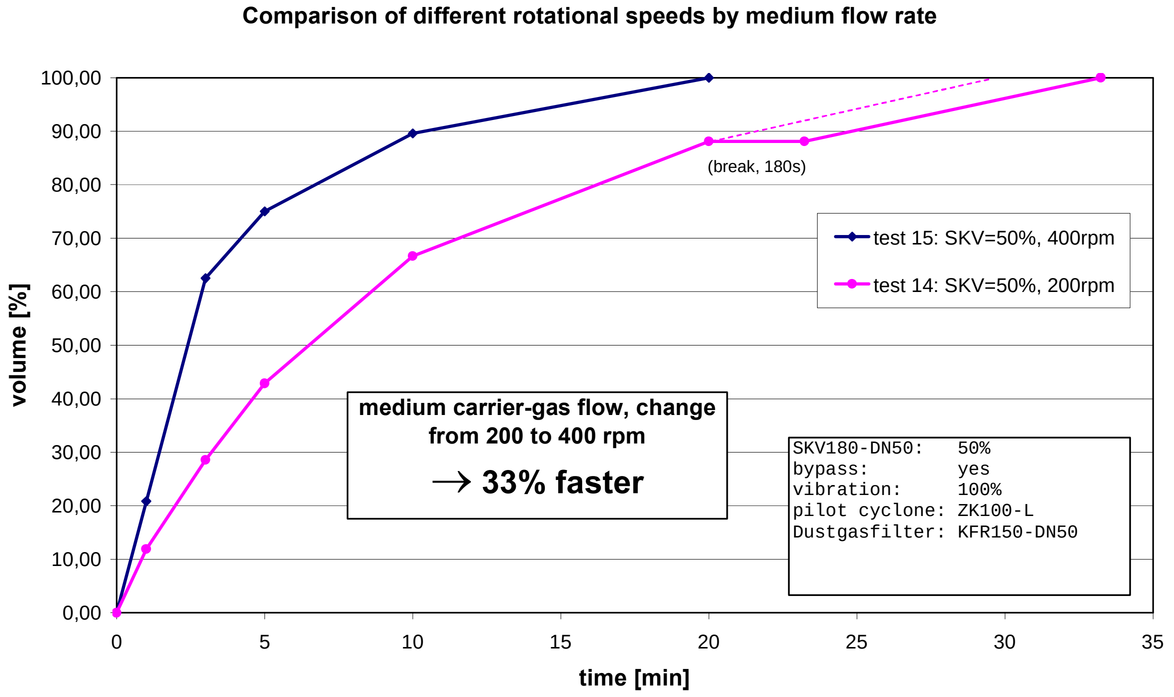


Figure 28: powder yield of tests 14-15 at medium turbine power

In result, in case of the tested metal-powder at 50 % turbine power, a yield around 100 % can be reached after 20 min at higher rotor velocity at 400 rpm and at lower velocity at 200 rpm after 30 min which is 33 % longer time.
In result, also here the rotational speed plays an important role and also important is, that a yield around 100 % can be reached, which was not possible in conventional discharging.

8.3 CAD-tests in Japan at 100 % turbine power and Discharging Cycle

In this test series we used always 100 % turbine power and a Discharging Cycle at 500 rpm for 5 s and 200 rpm for 5 s which means the rotational speed of the rotor was always going up and down between 200 and 500 rpm for 5 s each 5 s (see figure 26). We compared different grinding unit positions (main-port bottom and on top) as well as the effect of the by-pass.
The given yield data was again received online which means we stopped the time and after each interval did determine the volume with respect to the mark at the glass-container and additionally glued a mm-scale on the glass-bottle. This procedure was suitable since the entire main assembly sheet and so the cyclone and the glass-container was vibrating which did lead to a quite flat and horizontal level in the glass-bottle. Additionally, at each measurement-point we took a picture from the bottle and used digital measurement on the PC-screen.

Figure 29a+b: measurement set-up for the discharging tests in Japan

The total volume at the end at 100 % was measured and then vis-à-vis transferred to the received data for every fix-point. Figures 29 a and b show the measurement-set up and figure 30 shows a representative series of pictures taken during one of the discharging test.

Figure 30: series of pictures for digital measurement of the powder yield at each fix-point

In test 20, the main-port of the grinding unit was in top position and the by-pass closed. In test 21 the grinding unit was in the same position, but the by-pass open. In test 22, the main-port of the grinding unit was in bottom position and the by-pass closed. In test 23 the grinding unit was in the same position, but the by-pass open.

Table 10 gives the additional parameters for the tests 20-23 as far as they vary and exceed compared to the base parameters:

additional parameters for CAD-tests at 100 % turbine power (test 20-23)	
device, media, condition	>> base parameters metal-powder
pre-mixing	250 rpm, 3 min
SKV180-DN50 (turbine)	100 % (power)
by-pass	varied
vibration module	yes, 100 % power
Discharging Cycle	500rpm-5s / 200rpm-5s
position of grinding unit	varied
operation mode	Cycle Operation
discharging	draingrating Ask-20, TGD20a

Table 10: additional parameters for the CAD-tests at 100 % turbine power

Figure 31 gives the diagram of the powder yield of the above tests 20-23 over time and volume.

In test 20, where the grinding unit was in top-position and the bypass closed, a yield of about 100 % was achieved after 9 min discharging. In test 21, where the grinding unit was in top-position and the bypass open, a yield of about 100 % was achieved after 10 min discharging. In tests 22 and 23, where the grinding unit was in bottom-position and the bypass closed (22) respectively open (23), both of the given curves almost match to each other and a yield of about 100 % was achieved after 5 min discharging.

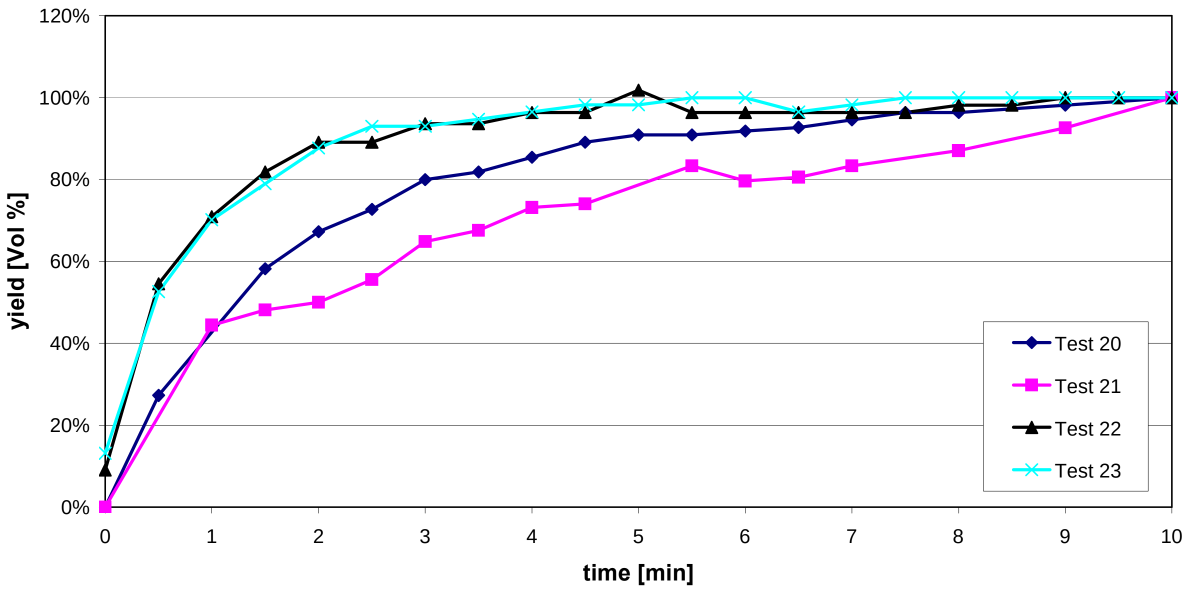


Figure 31: powder yield of tests 20-23 at 100 % turbine power

In result, for metal-powder, the by-pass effect was not that significant in both positions grinding unit, except in test 20 the result with the by-pass closed was about 10 % better compared to test 21 where additionally in the time-window between 1 and 10 minutes the yield development was much better.

In total, the grinding unit could be completely discharged already after 5 minutes which is more than just a remarkable result (compare chapter 8.1, last paragraph).

Conclusions

In conclusion, the principle and the process of HKP have been explained and applications have been given. In detail, the different routes batch, auto-batch and semi-continuously processing have been discussed. The discharging procedures related to the different routes have been compared with respect to their major technical difficulties. Idea and principle of Carrier-gas Assisted HKP and of Carrier-gas Assisted Discharging (CAD) have been explained.
The technology of the new developed CAD-unit TGD20a as an auxiliary device for a CM20 Simoloyer has been tested and described in detail which covered a by-pass, different side adapters, a heat-exchanger and a vibro-support system.
A number of tests are reported, where the effects of rotational speed, cycle operation, grinding unit position, side adapter gateway, by-pass mode, turbine powder have been investigated.
The results of the tests in Germany based on glass-powder did lead to a powder yield close to 100 % after less than 10 minutes already. For the tests based on metal-powder, 10 minutes discharging time could be determined for a full powder yield respectively.
The results of the tests based on metal-powder did lead to a powder yield close to 100 % after 5 minutes already in the in so far investigated ideal condition. In conventional discharging route, only about 75 % after 30-60 minutes could be achieved which means the new technology is an important step forward and enables us to consider and use the Simoloyer at HKP for industrial production in advanced batch and auto-batch mode. Very important to notice is, that the significant improvement in discharging time (and total yield) does not only lead to important quantitative achievements since the by this needed less kinetic impact during discharging also must lead to better quality standards.

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