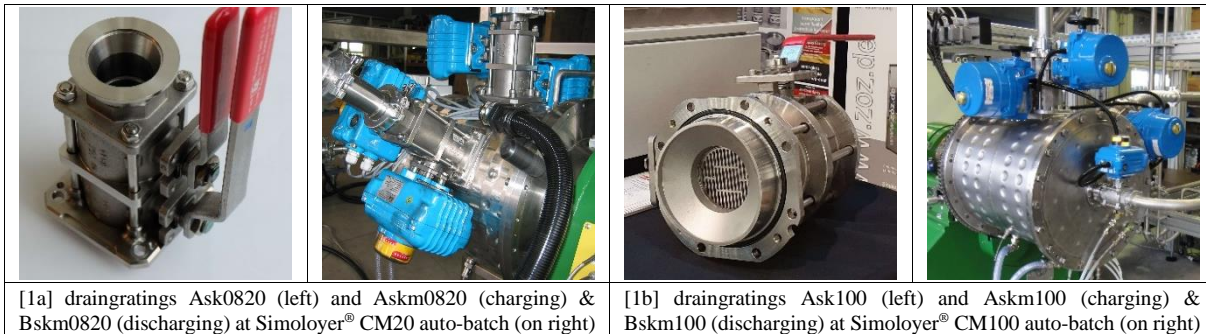


## HKP in the Simoloyer® Media Reload Processing (MRP)

High Kinetic Processing (HKP) in the Simoloyer® represents the most advanced technique for Mechanical Alloying (MA), High Energy (HEM) and Reactive Milling (RM) for making Nanostructures. Three general processing modes are addressed, namely the common batch-process (01), auto-batch (02) with automatic loading and unloading as well as the semi-continuous processing route (03) for insitu separation/classification by the adapted carrier-gas/multiphase flow circuit.



In all three modes, starting-powder material is charged into the Simoloyer® processing chamber that is loaded with grinding media (GM). The processed powder is subsequently discharged while GM remains in the chamber. Separation is provided by advanced draingrating systems with respect to atmosphere and/or handling mode from manual to fully automatic.

### Temp. Recovery & Product Flow

Except Reactive/HighTemp processing, grinding media stores processing heat that needs to be dissipated via cooling systems at vessel, flanges and sometimes rotor-blades and/or main-port circuits. In mode (3), additionally the carrier-gas can be utilized for temperature recovery.

Separation GM / product at discharging can represent a bottleneck in the product flow chart, particularly if product unloading shall be performed under vacuum or severely controlled atmosphere utilizing Ask-type draingrating [1a/b], Zoz-patent, as the only known solution (without dead zone).

### Media Reload Processing (MRP)

HKP at industrial manufacturing (repeated and fast processing) may require complete discharging/charging of product including GM in order to increase discharging efficiency (time and yield, economic) and to extract all heat that is stored by GM (heat-break, process technical) from the flow chart.

Complete discharging may also improve product access for CMB materials, such as highly ductile metals and composites.

Media Reload Processing (MRP), as a variant to batch- or auto-batch mode, also allows to discharge through a fixed main-port at 6 o'clock position [2-10b] without turning the grinding unit from charging- to discharging position. Thus auto-batch without carrier-gas becomes possible and carrier-gas assisted discharging can be replaced to some extent.

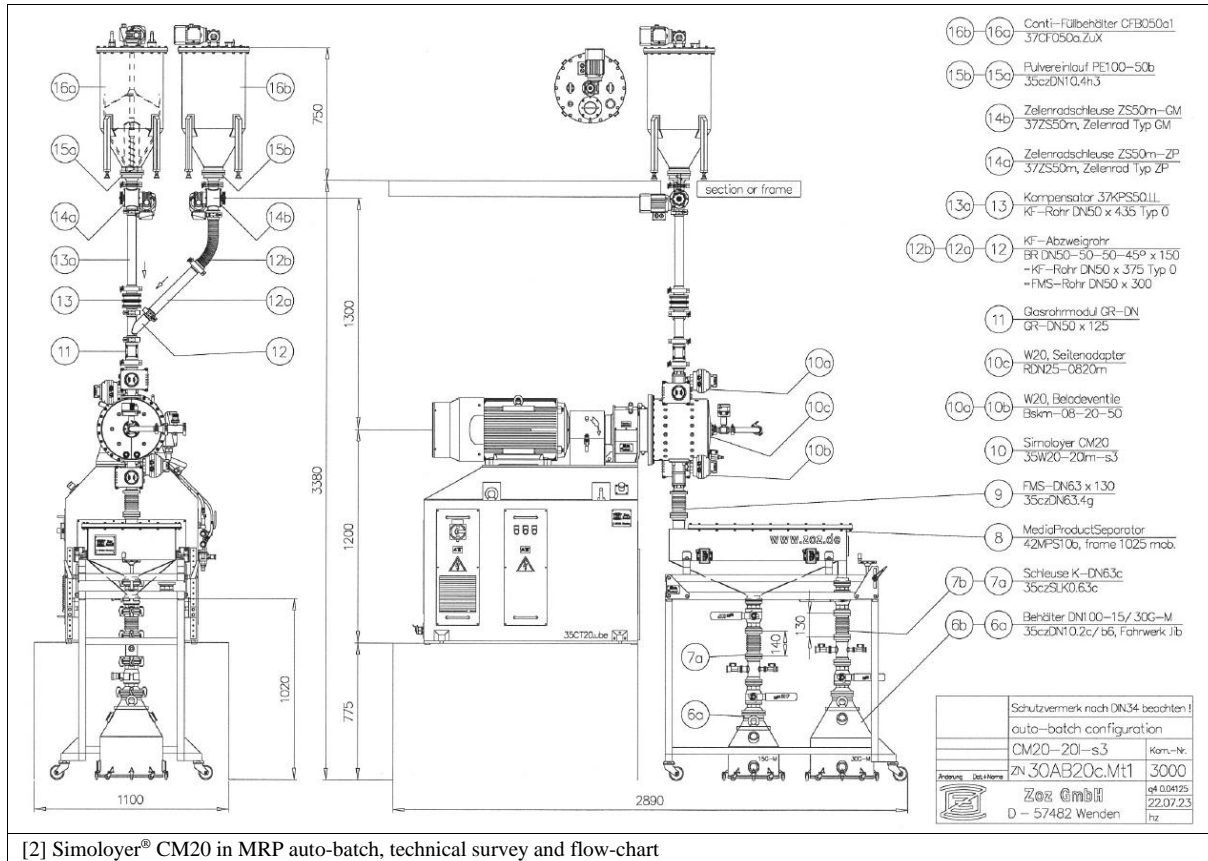
### MRP - loading/unloading at vessel

Charging/discharging of starting/processed powder + GM right at the processing chamber is done via two chargegratings Bskm [1b] [2-10a/b] at 6 o'clock and 12 o'clock position respectively. Type Bsk, on the contrary to type Ask, is appropriate for twin-direction, all transfer run by the Maltoz® control software.

### Separation GM/product, here MPS10b

The MediaProductSeparator MPS10b [3a-d] is made to collect the full multi-phase flow from the Simoloyer® CM20 processing chamber. GM will pass over a vibrating pan while powder product is passing through the same. GM is collected in the container (2-6b), powder product in the container (2-6a). Separation res. remaining time is controlled by the converter driven vibration intension as well as the adjustable pan-shift-angel. Classification can be observed through a complete transparent cover of MPS, the entire process can be operated under vacuum or inert gas.

## HKP in the Simoloyer® CM20 auto-batch at Media Reload Processing (MRP)



[2] Simoloyer® CM20 in MRP auto-batch, technical survey and flow-chart

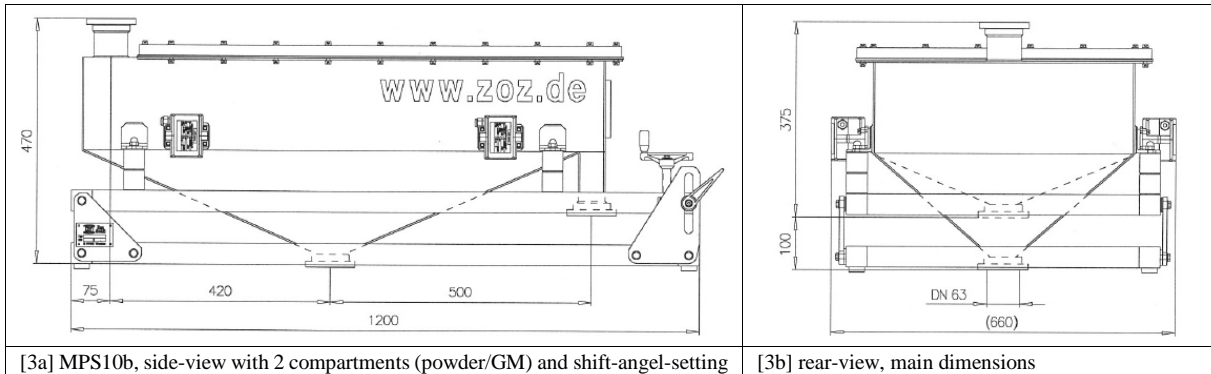
pos.	unit-definition	what for ?
06a	KF-Container DN100-15G-M, chassis 15b6	collecting powder product
06b	KF-Container DN100-30G-M, chassis 30b6	collecting grinding media
07a/b	Airlock K-DN63c (2x)	connecting MPS10b with containers 06a/b
08	MediaProductSeparator MPS10b	separation powder product / grinding media
09	FlexMetalTube DN63x130	connecting MPS10b with Simoloyer draingrating
10	Simoloyer CM20-20lm	HKP device in MRP auto-batch mode
10a/b	Chargegrating Bskm 08-20-50 (2x)	charging/discharging powder product + grinding media
10c	Sideadapter RDN25-0820m	automatic gas/vacuum-port
11	Transparent Pipe Module GR-DN50x125	visual control of charging process
12	KF-Junction Tube BR DN50-50-45°x150	merging starting powder + grinding media
12a	KF-Tube straight DN50x375 type 0	connecting charging line
12b	FlexMetalTube DN50x300	grinding media to rotary vane feeder
13	KF-Compensator KPS50LL	connecting charging line
13a	KF-Tube straight DN50x435 type 0	starting powder to rotary vane feeder
14a	RotaryVaneFeeder ZS50m-ZP	controlled transfer of starting powder
14b	RotaryVaneFeeder ZS50m-GM	controlled transfer of grinding media
15a/b	PowderInlet PE100-50b (2x)	connecting rotary vane feeders to CFB containers
16a	ChargingContainer CFB050a1, agitated	storage for starting powder (50l)
16b	ChargingContainer CFB050a1, not agitated	storage for grinding media (50l)

[T1] Simoloyer® CM20 in MRP auto-batch, main component list

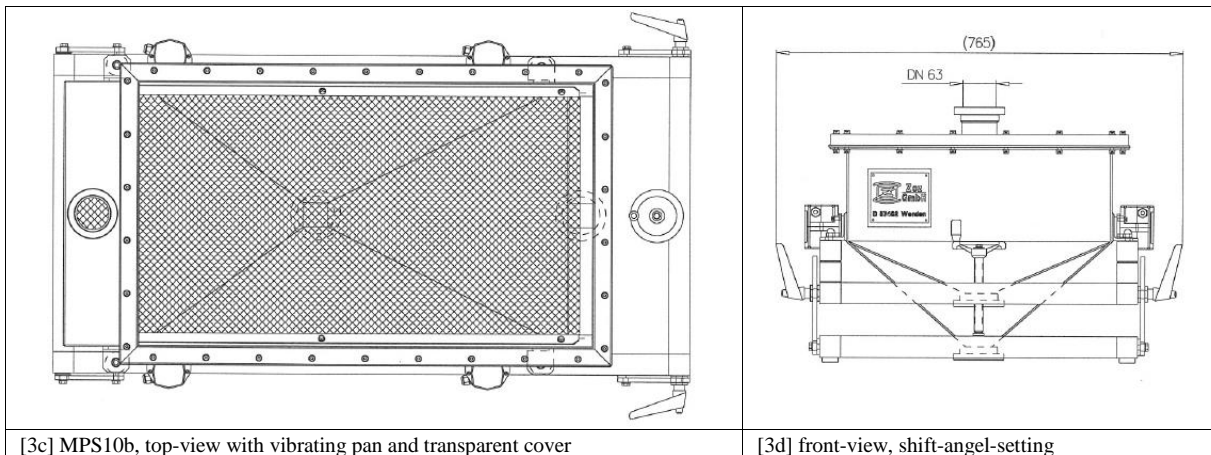
## MediaProductSeparator MPS10b

### after HKP in the Simoloyer® during auto-batch at Media Reload Processing

MediaProductSeparator MPS\* is made to collect and separate the full multi-phase flow (grinding media + powder product) particularly from the Simoloyer® processing chamber during auto-batch operation. Material flows in via the KF loading port DN63 on upper left [3a] onto the converter-driven vibrating pan [3a]. Grinding media (GM) will pass over towards the smaller collector-compartment on right while powder product is passing through into the larger collector-compartment on left [3a].



GM and powder product are forwarded and separately stored in corresponding containers or transfer pipelines connected each at KF unloading ports DN63. Separation res. remaining time “on screen” is controlled by choosing proper vibration intensity as well as the adjustable pan-shift-angle.



Classification can be observed through the transparent cover of MPS, entire process can be operated under vacuum or inert gas. Vibration- and shift-angle optionally controlled by Maltoz® software.

#### MRP – major advantages

MediaReloadProcessing for industrial manufacturing provides significant saving of discharging time at the Simoloyer®, which can easily become 20 times faster. By relocating the powder product separation from the complex Simoloyer® to the comparably simple MediaProductSeparator, cost reduction is achieved (a) in investment, (b) in operating cost and (c) in maintenance cost. Advantages not only relevant to large scale and auto-batch operation are described by practically eliminating the unavoidable impact by shear/friction/collision during common Simoloyer® discharging. This improves (d) the product quality also since the important powder/ball weight ratio is significantly changing with ongoing Simoloyer® discharging progress. If applicable, then MRP is less harmful to the powder product.

#### MRP – limitations

MediaReloadProcessing and so applying MediaProductSeparator does only promise success for discharging processes that in the Simoloyer® perform at low kinetic and high yield. MRP nor MPS cannot compensate e.g. sticking and agglomeration of powder material towards the inner vessel surface during HKP. Access can only be expected to so called “free powder product”.

## Rotary Vane Feeder ZS\*\*-GM\*

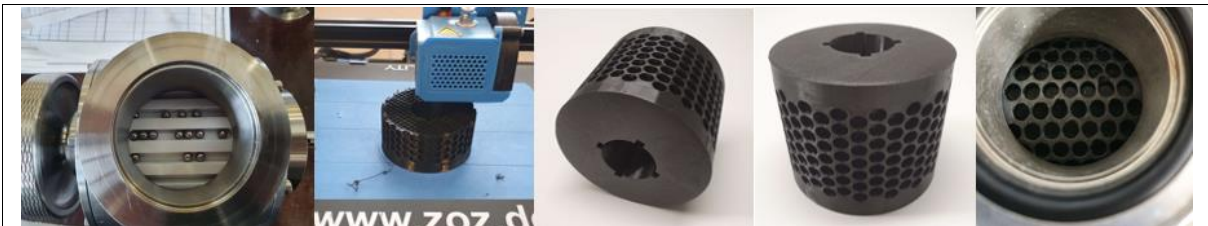
### precise auto-batch feeding of grinding media at Media Reload Processing

Media Reload Processing in auto-batch requires precise feeding not only of starting powder material but also of grinding media (GM) for each processing run. Rotary Vane Feeder ZS do represent to outside vacuum-tight cycle locks that to some limited extent can be used for packaging/portioning of processing components. Common star-feeders cannot transfer GM, resulting frequent blocking does harm equipment and process. Thus, a new feed-wheel strategy had to be developed.



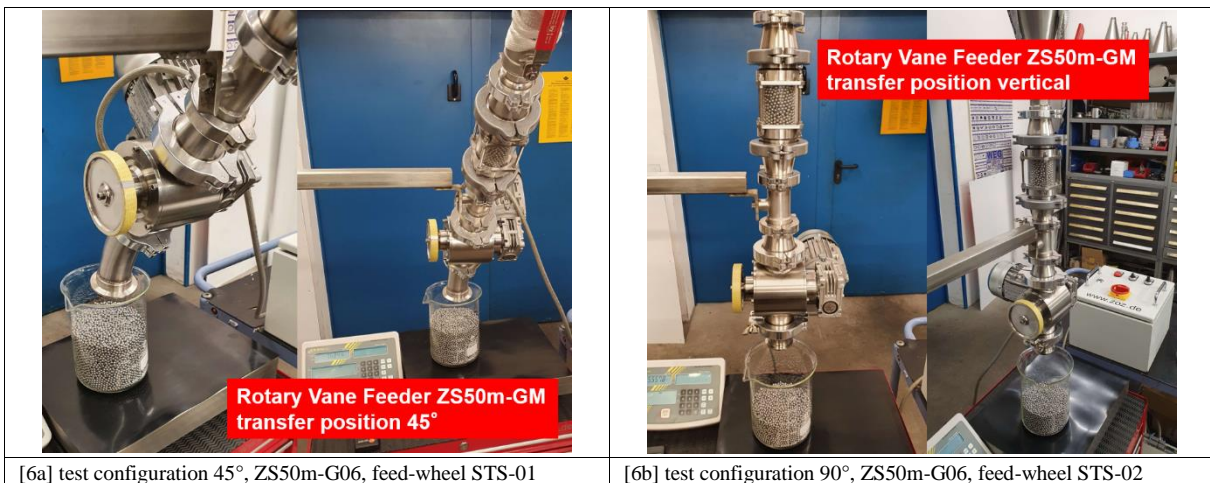
[4] star-feeders 37ZS50.ZR1 utilized in Zoz ZS Rotary Vane Feeders, adjusted mainly in material, seals & surface, in geometry only slightly

Significant geometric changes have been applied, particularly feed-wheels that have no star-shape anymore. E. g. the zebra-type feed-wheel performed better than the star-type but not at all acceptable as a commercial solution. Good results are achieved with the single-trap surface (STS) wheel that was made in multiple variants using rapid prototyping / 3D-printing PETG filament.



[5] feed-wheels, zebra-type (left) and 3D-printed STS-type, installed at ZS50m (on right)

The working-volume of ZS50m with common star-feeder 37ZS50.ZR1 is 96,99cm<sup>3</sup>, feed-wheel STS-50-GM06 at full load carries 245 steel-balls D4,762mm each 0,44g weight and 56,5414 mm<sup>3</sup> volume. In case of STS-feeder, the working volume should describe the “transfer-volume”, which equals to the volume of the 245 steel-balls, thus 13,85 cm<sup>3</sup> at  $\Sigma 107,8g$ . In result, the star-feeder transfer rate is 7x (7,003) higher compared to the STS-wheel.



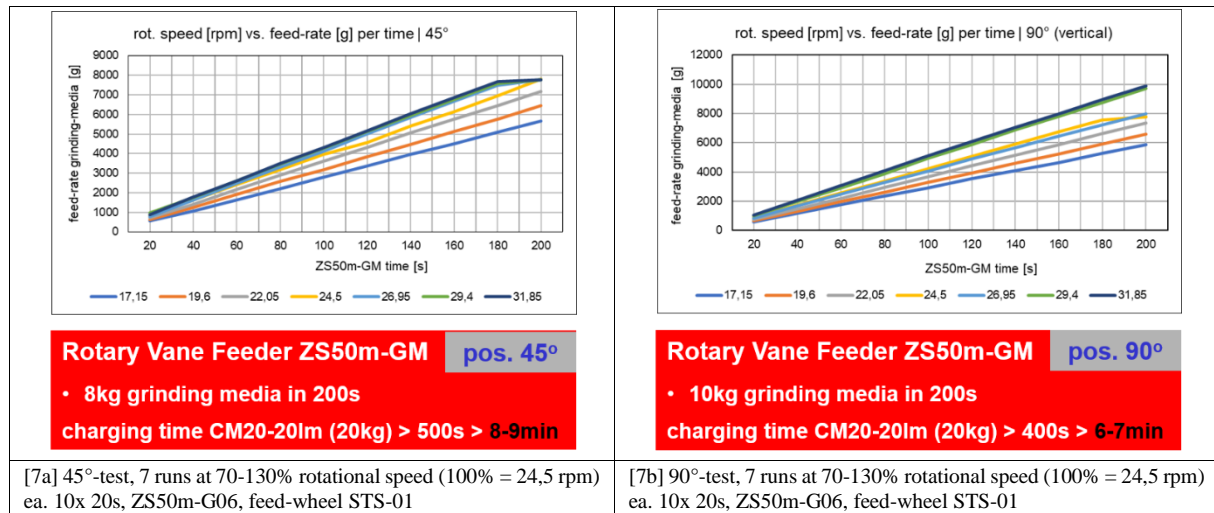
[6a] test configuration 45°, ZS50m-G06, feed-wheel STS-01

[6b] test configuration 90°, ZS50m-G06, feed-wheel STS-02

A rotary vane feeder ZS50m is fixed in adjustable transfer angel position [6a/b], grinding media is fed from a KF-Container DN40-G1-31 through KFA-Adapter DN50-DN40x45 and a Transparent Pipe Module GR-DN50x125. At 45°-test [6a], additionally a pipe-bend RBA-DN50-22.5° was installed at the ZSm flow-out port. Grinding media was collected in a 2.000ml glass-bin (Schott) continuously weighted on a high precision balance (Kern KFS-T).

## Rotary Vane Feeder ZS50m-GM06 pilot results of single trap surface (STS) feed-wheel at grinding media transfer

140 measures during 14 runs with 2 new STS-wheels in 2 transfer positions (45° and 90°) have been carried out. Each measure was taken after 20s, thus the total run-time was 1.400s/wheel equals to 23min20s, total test-time 46min40s. Rotational speed of the feed-wheel was varied from nominal 100% (24,5rpm) to 70, 80, 90, 110, 120 and 130% for each transfer position. Total transferred grinding media was 105,494kg equals to 239.759 steel balls.



Best results with respect to transfer load are achieved at 90° transfer position [7b], all 7 curves/lines are higher than the corresponding ones at 45° transfer position. The feed-rate is increasing with the rotational speed of the STS-wheel increasing.

	transfer position	Σ time [s]	Σ transfer [kg]	feed-rate [kg/h]	feed-rate [Δ-%]	feed-rate max. [kg/h]	feed-rate max. [Δ-%]	runs	data obtained	% N-speed deviation	
a	45°	1.400	50,382	129,554	+/-0	139,806	+/-0	7	ea. 20s	70-130%	
b	<b>90°</b>	1.400	55,112	<b>141,717</b>	+9,14	178,002	+27,32 <sup>x1</sup>	7	ea. 20s	70-130%	
Σ	a+b	2.800	105,494	135,635	-	14	14	14	-	-	
T2, mass transfer, feed-rate average and max.							<sup>x1</sup> > see mismatch M02				

Due to mismatches M01-M04, the general increase 45° to 90° can only be estimated. With regard to the total transferred mass in both angels (50,382kg vs. 55,112kg), it should be higher than (+9,14%) and lower than the measured max. value of (+27,32%). In any case it is significant, feeding angel, which describes particularly the flow-in angel at ZSm is to be recognized [T2].

M	mismatch	% N-speed [%]	speed [rpm]	curve	time	
01	45°	120	29,40	green	180s ff.	significant change of slope (-)
02	45°	130	31,85	black	180s ff.	significant change of slope (-)
03	90°	100	24,50	orange	180s ff.	significant change of slope (-)
04	90°	110	26,95	blue	all	blue lower than orange, should be vice versa
T3, mismatches with respect to [1a] and [1b]						

Even the working/transfer volume of STS-wheels is comparably small, the transfer capacity is impressive where at present status commercial success can only be predicted since strength and stability of the filament-printed structure is not at all acceptable for commercialization. However, weak material in this case is resulting in wear and damage determining lifetime but geometry is determining performance. STS-wheel-speed and transfer position are determining performance, too and should be further investigated, highest applied 130% of nominal speed at 90°-position seems not to be the limit. However, achieved transfer-performance parameters are more than good enough for commercialization.

## Rotary Vane Feeder ZS50m-GM06 pilot + next gen. filament damage at prototype, predicted next gen. commercial performance

After geometric performance of STS-type feed wheels is confirmed, commercial product availability can now be predicted. The next generation STS-wheels will be made by stainless steel which should solve the given wear/damage problems of the weak filament-structure. Following the common star feeder experience where each of the star heads represents a tube-stripper, STS-wheel (ZS50-scale) in V2A/V4A will be equipped with 6 tube-strippers made from PTFE. These provide pressure-shock resistance as well as wheel-in-tube wear, transfer tube/case will be chromium-plated which also represents an option at decades experience.

ZS50m-GM, wear of vane feeder drum (prototype PETG filament 1.7)					
	position	runs	total grinding media	total time	wear (locks)
(a)	45°	70	50.382g	23min20s	48/245 (19,6%)
(b)	<b>90°</b>	<b>70</b>	<b>55.112g</b>	<b>23min20s</b>	<b>09/245 (3,7%)</b>
(c)	45°+90°	140	105.494g (105,5kg)	46min40s	57/245 (23,3%)

**T4, next gen. VFD to be printed in 1.4301/1.4404 by KAMI/Seoul/Korea**

[8] components & test-wheels, (b) STS-wheel after all test-runs in 90°-position, (a) STS-wheel from 45°-position testing correspondingly

As for wear res. damage during test-runs with the two STS-wheels, the wheel (a) after 90°-position testing showed 48 damaged compartments (locks) res. 48 steel-balls remained in the compartments. Much less damage is observed at wheel [8b], where 9 damaged compartments/steel-balls were counted. Each wheel carries 245 compartments, thus for [8a] a damage rate of 19,6% and for [8b] a damage rate of 3,7% is recognized. The insofar poor stability of [8a] may explain mismatches M01 and M02 at 45°-testing. Mismatches M03 and M04 in 90°-testing could not be explained by the 9 unavailable compartments since M03/04 did NOT appear at the end of the test-run. Again, geometric performance can be stated as proven/given.

Based on the available data on both, the present pilot-test STS as well as previous testing/experience with all ZSm-units, minima performance can be predicted. Rotary vane feeders ZS\*\*m-GM\* will soon be appropriate to providing precise packaging/portioning of grinding media at Simoloyer® auto-batch operation utilizing MediaReloadProcessing:

unit-size ZS	ZS25	ZS40m-GM	ZS50m-GM	ZS63m-GM	ZS100m-GM
access surface E/A Δ-%	24	66,6	100	165,3	416,4
type GM est. minima	0,7 kg/min	2 kg/min	3kg/min	5kg/min	12,5 kg/min
for Simoloyer®	CM01 (2kg)	<b>CM08</b> (8kg)	<b>CM20</b> (20kg)	<b>CM20</b> (20kg)	<b>CM100</b> (100kg)
charging time (standard GU-size)	(3min) <b>too small</b>	<b>4min</b>	<b>6-7min</b>	<b>4min</b>	<b>8min</b>

T5, prediction of automatic GM loading time for Simoloyer® CM08 - CM100. ZS25 is too small, CM01 may be charged with ZS40 in 1min. For CM400 & CM900, ZS130 and ZS160 (KF-DN130/DN160) will be introduced.

Next results to be published upon availability.  
References to follow, once this project is done.

Zoz Group, 31.08.2023