

manuscript

## **Simoloyer<sup>®</sup>: major characteristics and features**

H. Zoz

Zoz GmbH, D-57482 Wenden, Germany

### **abstract**

The present paper, summarized upon originally request of General Motors, describes the major characteristics and features of the Simoloyer, certainly the most suitable and advanced device for High Kinetic Processing in laboratory as well as in industrial scale. The summary gives main criteria in kind of headlines that are later described in brief and one by one (pp 1-4). References are given on page 5, appendix I (pp. A1/1-A1/10) gives corresponding figures ordered at the end of each chapter. Materials processing examples including characterization are given in the epilogue.

### **summary**

#### **Simoloyer<sup>®</sup>:**

- 1. a system made for MA/HEM/RM in a dry process for industrial application**
- 2. supplies 3 times higher relative velocity of grinding media than conventional devices**
- 3. can not only be operated but also charged and discharged under vacuum/inert gas**
- 4. allows processing without dead-zones & negative gravity influence**
- 5. can be operated by Cycle Operation**
- 6. can be operated at high temperature due to separated cooling/heating systems**
- 7. is fully software operated, allows parameter measurement, control and history**
- 8. is technically scalable from 0.5 to 900 liters processing chamber capacity**
- 9. can be operated in batch, semi-continuous and auto-batch mode (carrier gas)**
- 10. is available with tools by stainless steel/Stellite, WC-Co and Si<sub>3</sub>N<sub>4</sub>**

#### **1. Simoloyer, a system made for MA/HEM/RM in a dry process for industrial application**

The Simoloyer is made for Mechanical Alloying (MA), High Energy Milling (HEM) and Reactive Milling (RM). It was maybe born 10 years back at Krupp Pulvermetall GmbH at Essen, Germany. This distinguishes the technology from many other mills, in particular from attritor mills that were decades ago made as mixers for paint industry.

What is needed for MA, HEM and RM which can be summarized as High Kinetic Processing (HKP), is energy transfer from some milling tools into (powder)material. In case of mills, this is a kinetic route where here the energy transfer is required to be very high [1-3].

(Figures for this chapter see appendix I, p. 1.)

## **2. Simoloyer supplies higher relative velocity of grinding media than conventional devices**

The most efficient route to reach a high kinetic energy transfer can easily be understood via a high relative velocity of grinding media. This is the key for the process. The Simoloyer allows with up to 14 m/s an almost 3 times higher relative velocity than any other known device [4-7].

The conclusion is not only, that HKP can be performed much faster, often it is the only way to perform this at all since e.g. effecting materials structure can require a determined energy level (egg-cocking story: 3 minutes at 100 °C supplies much less energy than 20 days at 70°C but in the last case the egg only becomes old...). This is not limited e.g. to activation energy level of solid state reactions, it also can refer to particle/grain collapse of then brittle materials [8].

Here it is very important to know, that the process in the Simoloyer is based on collision, not on shear and friction of res. between grinding media.

*(Figures for this chapter see appendix I, p. 2.)*

## **3. Simoloyer can be operated, charged and discharged under vacuum/inert gas**

Since it is in the nature of HKP, that large and active surfaces are produced and often anyway very reactive materials are processed, the kinetic of the oxidation reaction is tremendously increased [9]. This means for the process as well as for the safety of human and equipment, that next to processing, also charging (loading) and discharging (unloading) must be performed under controlled condition. Here the Simoloyer is the only known system that is equipped with suitable air-lock systems that allow fully controlled material transfer in small laboratory scale as well as in large industrial scale.

Since the equipment is made for industrial purpose at the end, it can not be a solution to place the device in a glove-box which often happens e.g. with planetary ball mills and shaker mills.

*(Figures for this chapter see appendix I, p. 3.)*

## **4. Simoloyer allows processing without dead-zones & negative gravity influence**

Based on first historical and second technological-difficulty reasons, most rotary ball mills for dry operation are based on a vertical impeller arrangement principle. The reason is the same as for the kitchen mixers: gravity keeps the product inside the container res. keeps the product away from the rotary seals of the drive shaft if there are some. On the other hand gravity effects in that way that the apparent powder concentration (density) during processing inside the container/vessel is increasing with the distance from the top. This leads to disadvantageous dead-zones and caking effect shown by Courtney et al. in 1993 [10]. The solution go horizontal sounds much easier than it is, since in the horizontal system, it is very difficult to keep the usually very fine and often very abrasive product away from the rotary seal. The Simoloyer does supply these features for long term operation and by this avoids effects like non-equally processed products.

Another typical dead zone is usually found in the load- and unloading systems of rotary ball mills, if there are some. If the system can not be opened to outside for loading/unloading, usually a screen-grating where product can pass and grinding media not, is used. This is closed by a following valve. The dead-zone is here between screen grating and valve because product can remain or go there and therefore is not inside the active volume of the mill. By unloading the processed material is then polluted by non- or less-processed material. The Simoloyer is equipped with an Ask-type draingrating which

avoids these effects [11] and is like the Simoloyer protected by US- and other patents [12].

*(Figures for this chapter see appendix I, p. 4.)*

## **5. Simoloyer can be operated by Cycle Operation**

In 1996, Zoz, Magini, Froes et al. [13] showed that Ti-based materials can be successfully processed by HKP without any process control agents. A major process engineering barrier of this and other extremely ductile materials is the effect of dead-layers, which means that the material sticks everywhere inside the mill, in particular at the inner surface of the vessel. The Cycle Operation procedure [14] which is meanwhile also covered by patents and today created by the MALTOZ®-Simoloyer-Operating software, did increase the powder yield in case of the processed Ti-24Al-11Nb material from < 5 % to 80 % [13]. Later in 1999, Chung, Kim et al. showed that Cycle Operation procedure compared to constant operation also can have tremendous influence on particle size reduction, grain refinement as well as grain-growth behavior after heat treatment [15].

Still there are only attempts to explain the mechanism where the idea is that the repeated interruption of kinetic, which Cycle Operation does, breaks the balance of deformation, fracture and welding in the process. Why this technique sometimes effects the process like or better than various PCA's but without pollution by the PCA itself, is not at all fully understood.

Today Cycle Operation is becoming an important tool for the successful processing of many ductile base materials like e.g. Ti-Al, Ti-Ni [16], Ni-Al [17], Cu-W [18], Ag-SnO<sub>2</sub> [19], Fe-Co [15] and in particular for rapid processing of ductile metal flakes like Au-, Ag-, Pd-, Ta-, Ti-, Ni-, Al-, Cu-, Fe- and Zn-flakes [20-22].

*(Figures for this chapter see appendix I, p. 5.)*

## **6. Simoloyer can be operated at high temperature due to separated cooling/heating systems**

Next to the wanted energy transfer into powder which may lead to surface energy increase, lattice transformation, a high defect structure by deformation, e.g. atomic dislocations etc., HKP usually produces a lot of heat [23]. At this point it should be noticed that Zoz et al. in 1999 showed that the energy balance in case of HKP can reach a lever of up to 50 % of energy in system [24] which is e.g. compared to conventional milling techniques (< 5 %) unexpectedly high.

For simple technological reasons, the heat must be transferred out of the system which means the Simoloyer must be and is equipped with very efficient cooling systems. In advance of a later European materials project [25], Chaffron, Chen and Martin did show that a maintained milling temperature (in this case around 110°C) can increase chemical transformation velocity in a milling process tremendously [26]. In the same project, Zoz, Ren and Spaeth in 1999 showed a decrease of processing time of the same material system under the same temperature from 30 hours in a laboratory frame grinder to 27 minutes in the Simoloyer [19]. Cycle Operation has been an indispensable tool here. From that point on, the cooling systems of the Simoloyer had been redesigned and are today separated, so that the technically necessary cooling can always be supplied to the device but the vessel / the process must not be cooled at the same time. Additionally it can be heated by built-in electrical heat sheets.

*(Figures for this chapter see appendix I, p. 6.)*

## **7. Simoloyer is fully software operated, allows parameter measurement, control and history**

Most of res. all milling processes, including those in commercial application, are not controlled at all. Usually not more is known than what has to be done in order to receive an existing effect which is nothing else than a principle of trial and error. Also by the Simoloyer, no engineered process can be fully controlled. What the Simoloyer does and here is supplied by the operating software (MALTOZ) is a control of some of the parameters like rotational speed, time (e.g. Cycle Operation) and some temperatures. Other temperatures, torque and total energy-consumption are only recorded [24]. Work on controllable energy input in correlation to an insitu energy balance measurement and calculation are still in progress. The software operated system supplies complete milling history and important safety features for human and equipment. In fact some parameters might be recorded for unknown reasons today, however, the philosophy here is that each known parameter might be important for the conclusion of all, with respect to the goal of reproducibility by control.

*(Figures for this chapter see appendix I, p. 6.)*

## **8. Simoloyer is technically scalable from 0.5 to 900 liters processing chamber capacity**

An important criteria for any kind of technical process is reproducibility. In particular in the field of mechanical milling, and the more in HKP, scaling up of won results by calculating corresponding parameters is in general impossible. What is valid is, that it is in any case much easier to forecast capabilities with respect to quality and quantity if there is no change of the processing principle. In other words it must be considered to be disadvantageous if laboratory scale exploration of a materials system is performed e.g. in a shaker mill, medium scale field tests are tried to cover e.g. by a planetary ball mill and finally a commercial production should be realized in a drum ball mill. Here the Simoloyer shows up an easier and safer way by availability of laboratory scale systems from 0.5 to 2 liters volume, medium scale systems of 5-20 liters volume and 100 to 400 liters volume for commercial application. The 900 liter-system is under construction. It is important, that all different sizes correspond to the same geometrical relations with similar kinetic and energy input per volume. Furthermore all unit-sizes are equipped with quick-changeable grinding units that are for the same Simoloyer base unit available in different sizes (up to CM20) as well as with different features e.g. ceramic units, heat-able or semi-continuous units where the grinding unit itself is the closed system that carries the vacuum or inert gas and may be stored under it until using this system res. materials-system again.

*(Figures for this chapter see appendix I, p. 6.)*

## **9. Simoloyer can be operated in batch, semi-continuously and auto-batch mode (carrier gas)**

Due to long processing times from 3-30 hours, MA and corresponding processes are batch-operations. Since in several materials systems, the Simoloyer could short cut processing times tremendously to 10-90 minutes, a continuously operation could be considered where the limit today is expected at around 10 min processing time. Within a large national funded project in Germany, a semi-continuously operating Simoloyer device is explored [27].

In 1998, Mizutani et al. showed that Cu- and Ag-flakes can be produced continuously using depression mode [21]. In February 1996 already Fukuda and Zoz set up the general idea of tangential unload path of flakes of a horizontal chamber using Stooke's law [28].

Later in 1998, Zoz et al. suggested the compression mode [29]. In 2001, Kaupp et al. showed that the Simoloyer can be successfully used for rapid performance by Reactive Milling of environmentally benign quantitative reactions without solvents and wastes in continuously operation [30]. Rhee et al. explore an auto-batch technique for Samsung Corning in the same year [31].

The principle of compression mode is based on a closed carrier-gas circuit where starting material is injected outside the grinding chamber into the carrier gas flow. The multiphase flow then passes the chamber on an tangential route and the product is insitu separated and classified.

In case of other barriers that hinder a continuously process, the same carrier gas can be used for an automatic res. semi-automatic operation which means a carrier gas is used to transfer the starting material into the vessel and after processing and again in a closed circuit is used to transfer the material out of the vessel where the so build multiphase flow is separated insitu by common classifier technique. This auto-batch procedure is to be used for larger quantities in industrial scale only.

*(Figures for this chapter see appendix I, p. 7.)*

#### **10. Simoloyer is available with tools by stainless steel/Stellite, WC-Co and Si<sub>3</sub>N<sub>4</sub>**

By the development of MMC, CMC and in particular CCC, the contamination of the product by the milling tools became more and more important. From 1997-99, Zoz et al. developed the Ceramic Simoloyer within a national funded project [32]. The goal here has been to find solutions where ceramic components can be used as milling tools under high shock loads in HKP. It has been shown, that most critical is not the vessel, since it is not exposed to high kinetic, not the rotor since it is exposed to high kinetic but has a small surface, most critical is the grinding media, since it is exposed to high kinetic and has a large surface. E.g. for a long term it has not been possible to successfully use WC-Co-materials as the tools because of non-availability of suitable grinding media [33]. Today rotors made by WC-Co bulk-parts and vessels lined with WC-Co bulk-plates can be used with Co-rich grinding media. Alternatively WC-Co can be exchanged to Si<sub>3</sub>N<sub>4</sub> where the grinding media is still problematic.

Here it should be noticed, that often ceramic materials can be processed using metal-based milling tools without critical contamination of the product. This is caused by the effect that the grinding media is coated by the product itself [34].

*(Figures for this chapter see appendix I, p. 8.)*

#### **Epilogue:**

The Simoloyer can not produce any nanomaterial. What can be done and performed very rapidly, cost-effective and in large scale are nano-structured materials.

The Simoloyer can produce extremely rapidly, cost-effective and in large scale ductile metal flakes, however, the morphology of these materials often is different from those that are known and commercially produced in drum ball mills. For many applications, further polishing operation must be applied which may be done in the Simoloyer as well (under low kinetic).

The Simoloyer can be used for extremely rapid particle size reduction of brittle solids.

Examples of materials and characterization are given in the appendix.

*(figures for this chapter see appendix I, p. 9-10)*

## References

- [1] J.S. Benjamin, Metall. Trans., Vol. 1, 2943 (1970)
- [2] J.S. Benjamin, T.E. Volin, Metall. Trans. Vol. 5, 1929 (1974)
- [3] H. Zoz, H.U.Benz, K. Hüttebräucker, L. Furken, H. Ren, R. Reichardt, Stellite bearings for liquid Zn-/Al-Systems with advanced chemical and physical properties by Mechanical Alloying and Standard-PM-Route, Part I, Metall Vol. 54, 11/2000, pp. 650-659, 2000
- [4] H. Zoz: *Performance of the Simoloyer*, 4<sup>th</sup> International Conference on Powder Metallurgy in Aerospace, Defense and Demanding Applications, eds. P.S. Goodwin, R.B. Schwarz, in Anaheim, Los Angeles, USA, May 08-10, 1995
- [5] H. Zoz, D. Ernst, R. Reichardt, High Energy Milling / Mechanical Alloying / Reactive Milling, 3rd International Symposium of the school of chemical engineering, University of Mexico City, May 1998
- [6] R.M. Davis, B. McDermott, C.C. Koch, Mechanical Alloying of Brittle Materials, Metall. Trans. Vol. 19a, 2867 (1988)
- [7] N. Burgio, A. Iasonna, M. Magini, S. Martelli, F. Padella, IL NUOVO CIMENTO, Vol. 13 D, No. 4 (April 1991)
- [8] H. Zoz, H.U.Benz, G. Schäfer, M. Dannehl, J. Krüll, F. Kaup, H. Ren, R. Reichardt, High Kinetic Processing of Enamel, part I, cooperative project 09-8-4413, Zoz GmbH, Pfaudler Werke GmbH, Degussa-Hüls AG, Wendel GmbH, Miele & Cie. GmbH & Co, International Symposium on Metastable, Mechanically Alloyed and Nanocrystalline Materials, Ann Arbor, Michigan, 2001
- [9] C. Suryanarayana, Guo-Hao Chen, F.H. Froes, *Milling Maps for Phase Identification during Mechanical Alloying*, Scripta Metallurgica et Materialia, Vol. 26, 1727 (1992)
- [10] R.W. Rydin, D. Maurice, T.H. Courtney, Milling Dynamics: Part I. Attritor, Metall. Trans. Vol. 24a, pp175-185 (1993)
- [11] D. Ernst, H. Weiss, H. Zoz, *Structure and Properties of Nanophase Materials*, TMS, eds. B.T. Fultz, C.C. Koch, P.J. Maziasz, R.D. Shull, 1995
- [12] US-Patent No. 5,678,776 (1997) # DE 195 04 540.8, Ask-draingrating
- [13] H. Zoz, D. Ernst, H. Weiss, M. Magini, C. Powell, C. Suryanarayana, F.H. Froes, *Mechanical Alloying of Ti-24Al-11Nb (at%) using the Simoloyer<sup>®</sup>* Metall Vol. 50, 09/96, pp. 575-579, 1996
- [14] H. Zoz, D. Ernst, *Mechanical Alloying using Cycle Operation - A New Way to Synthesize CMB-Materials*, 5<sup>th</sup> International Conference on Advanced Particulate Materials and Processes, West Palm Beach, Florida 1997, Proceedings
- [15] J.Y. Chung, J. Kim, Y.D. Kim, *Formation of Nanocrystalline Fe-Co Powders Produced by Mechanical Alloying*, Dept. of Metallurgy and Materials Science, Hanyang University, Ansan, Korea, 1999
- [16] H. Zoz, D. Ernst, I. S. Ahn, W.H. Kwon, *Mechanical Alloying of Ti-Ni-based Materials using the Simoloyer<sup>®</sup>*, TMS Annual Meeting 1997, eds. C.M. Ward-Close, F.H. Froes, S.S. Cho, D.J. Chellman: *Synthesis/Processing of lightweight Metallic Materials*, (Proceedings 1997)
- [17] Y. I. Jang, J. S. Kim, I. S. Ahn, Y. D. Kim, and Y. S. Kwon: *Spark-Plasma Sintering of Mechanically-alloyed NiAl Powder and Ball-milled (Ni+Al) Powder Mixture*, J. Korean Powder Metallurgy Institute, Vol. 7, No 3, 2000
- [18] J. H. Yoon, M. J. Suk, J. S. Kim, and Y. S. Kwon: *Morphological Change of W Particle during Liquid-Phase Sintering of the Mechanically Alloyed W-Cu Composite Powder*, Proceedings of PM2000, Kyoto, Japan
- [19] H. Zoz, H. Ren, N. Späth: *Improved Ag-SnO<sub>2</sub> Electrical Contact Material Produced by Mechanical Alloying*, Metall Vol. 53, 07-08/99, pp. 423-428, 1999
- [20] H. Zoz, R. Reichardt, H. Ren, H.U.Benz, Ductile Metal Flakes based on [Au], [Ag], [Cu], [Ti], [Al], [Ni] and [Fe] by High Energy Milling (HEM) – Part I, PM<sup>2</sup>Tech '99 Conference MPIF/APMI, June 20 – 24, Vancouver, Proceedings 1999
- [21] H. Zoz, D. Ernst, T. Mizutani, H. Okouchi, *Simoloyer<sup>®</sup>\* CM100s, semi-continuously Mechanical Alloying in a production scale using Cycle Operation-Part I*, Metall Vol. 51, 10/97, pp. 568-572, 1997
- [22] R. Savin, H.U. Benz and H. Zoz, Zn and Zn-Ni-flakes for anti-corrosives, unpublished exploitation report 05.02.-08.02.2000, Palm Springs, CA
- [23] P.G. McCormic, H. Huang, M.P. Dallimore, J. Ding, J. Pan, *The Dynamics of Mechanical Alloying, Structural Applications of MA*, ASM, eds. J.J. Barbadillo, F.H. Froes, R.B. Schwarz, 1993
- [24] H. Zoz, R. Reichardt, H. Ren, Energy Balance during Mechanical Alloying, Measurement and Calculation Method supported by the Maltoz<sup>®</sup>-software, PM<sup>2</sup>Tech '99 Conference MPIF/APMI, June 20 – 24, Vancouver, Proceedings 1999
- [25] Report of Brite Euram Project BE-95-1231, *Novel processing technique for AgSnO<sub>2</sub>. electrical contact Materials*, 1996-1999
- [26] L. Chaffron, Y. Chen, G. Martin, Ann. Chim. Fr. (1993) 18, pp. 395-402
- [27] TPW-project/State of NRW/Germany # 25072000, Continuously and monitored flake-production by HEM with aerodynamic separation & classification and rapid particle size reduction of brittle solids (aux. target, enamel) [2000-2003]
- [28] Authors discussion with T. Fukuda, October 1996, University of Siegen, Germany
- [29] H. Zoz, D. Ernst, R. Reichardt, *Simoloyer<sup>®</sup>\* CM100s, semi-continuously Mechanical Alloying in a production scale using Cycle Operation-Part II*, Metall Vol. 51, 09/98, pp. 521-527, 1998
- [30] G. Kaupp, J. Schmeyers, M. R. Naimi-Jamal, H. Ren, H. Zoz, Reactive milling with the Simoloyer: environmentally benign quantitative reactions without solvents and wastes, unpublished, submitted May 2001
- [31] D.J. Rhee, H.H. Choi, I.Y. Lee, H.U. Benz, H. Zoz, investigation of auto-batch operation for Samsung Corning, 04-07, 2001, unpublished report
- [32] TPW-project/State of NRW/Germany # 25072000, Development of the Ceramic Simoloyer [1997-1999]
- [33] H. Ren and H Zoz, Ceramic Powder using High Energy Milling, Interceram, Vol. 49 / 1-2000 pp 24-34
- [34] CRAFT Project BE-S2-3506 Ctr BRST-CT98-5490: Advanced Hard Coatings based on Ceramic Nanocomposites, 1998-2002

## Simoloyer<sup>®</sup>: major characteristics and features, appendix I A1/1

### 1. Simoloyer: a system made for MA/HEM/RM in a dry process for industrial application



Fig. 1a: CM01 for pilot testing



Fig. 1b: CM20, e.g. for production of nano-SiC composites at Zoz



Fig. 1c: CM100, e.g. for production of nanocrystalline metal-hydrides

### High Energy Milling / Mechanical Alloying / Reactive Milling:

#### The Principle

- the collision is the primary event for the energy transfer
- for an optimum impact is valid:

$$E_{kin} = \frac{1}{2}mv^2$$

m: mass of the ball

v: relative velocity of the ball

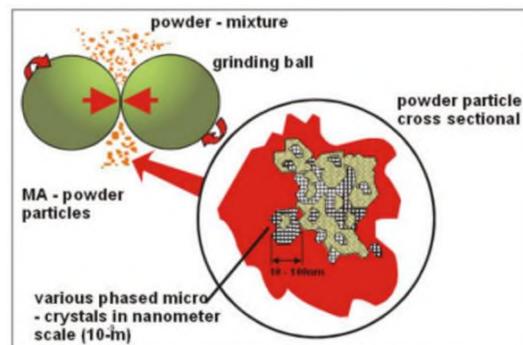


Fig. 2: principle of HEM/MA/RM

### **challenge: high relative velocity of grinding media**

High Kinetic Processing, Applications, Products		
High Energy Milling	Mechanical Alloying	Reactive Milling
surface, shape, particle size (geometry)	alloy (pseudo)	+ chemical reaction
<ul style="list-style-type: none"> <li>• Flakes (Particle Deformed Powder)</li> </ul>	<ul style="list-style-type: none"> <li>• Nanocrystalline Materials</li> </ul>	<ul style="list-style-type: none"> <li>• Contact Material</li> </ul>
<ul style="list-style-type: none"> <li>• Particle Coating (LPS, S)</li> </ul>	<ul style="list-style-type: none"> <li>• Amorphous Materials</li> </ul>	<ul style="list-style-type: none"> <li>• Nanocrystalline Materials</li> </ul>
<ul style="list-style-type: none"> <li>• Nanocrystalline Materials</li> </ul>	<ul style="list-style-type: none"> <li>• Oxide Dispersion Strengthened Alloys</li> </ul>	<ul style="list-style-type: none"> <li>• Mechanochemistry</li> </ul>
<ul style="list-style-type: none"> <li>• Highly Dispersed Phased Materials</li> </ul>	<ul style="list-style-type: none"> <li>• Iron and Oxide based Magnetic Materials</li> </ul>	<ul style="list-style-type: none"> <li>• Solid state synthesis</li> </ul>
<ul style="list-style-type: none"> <li>• Soft Magnetics</li> </ul>	<ul style="list-style-type: none"> <li>• Bearing Materials containing Solid Lubricants</li> </ul>	<ul style="list-style-type: none"> <li>• Hydride - Dehydride</li> </ul>
<ul style="list-style-type: none"> <li>• Particle size reduction (e.g. enamel)</li> </ul>	<ul style="list-style-type: none"> <li>• Ceramic Metal Composites (MMC, CMC, MMC, CCC)</li> </ul>	<ul style="list-style-type: none"> <li>• Activation (Catalysts)</li> </ul>

Fig. 3: principle of HEM/MA/RM

technical data subject to alterations

## Simoloyer<sup>®</sup>: major characteristics and features, appendix I A1/2

### 2. Simoloyer<sup>®</sup> supplies higher relative velocity of grinding media than conventional devices

#### HEM / MA / RM -Devices in use

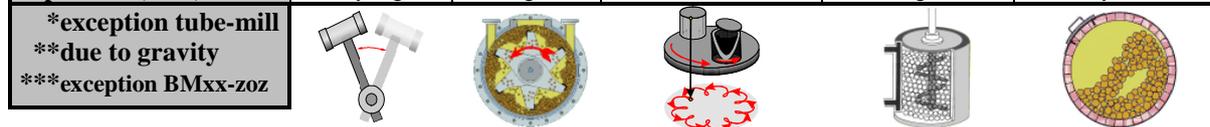
Modell nach: H. Zoz, 50 Aniversario de la Fundacion de la Esique

3<sup>rd</sup> Intern. Symp. of THE SCHOOL OF CHEMICAL ENGINEERING, May 27-29, 1998

dimensions economically and logically limited typical parameters, approximate data, partly estimates, partly calculated:

[1], [6], [7], [8], [10], [11], [12]

Device	Shaker Mill (spex)	Simoloyer <sup>®</sup> (Zoz)	Planetary Ball Mill (various)	Attritor <sup>®</sup> (union process)	Drum/Ball-Mill (various)
properties	brand				
Energy Impact relation	10	08	05	04	01
Friction / Sheer relation	low	low	medium	medium - high	high
Collision relation	high	high	medium	low - medium	low
Kinetic, Processing	very fast	fast	medium	medium	slow
Influence of gravity	little	very little	little	difficult	needed!
max. Diameter [m]	0.08	0.9	0.2	1	3
max. Total Volume [l]	0.2	400	8	1000	20000
max. relat. velocity [m/s]		16	5	4.5 - 5.1	x - 5
Specific Energy [kW / l]	--	0.55 (- 3)	--	0.1 (- 0.75)	0.01 - 0.03
Continuous process (dry)	impossible	Yes, semi	impossible	difficult	impossible*
Scaling up	no	yes	no	yes	yes
max. batch [kg]	0.2	250	2	250	12000
Contamination	low	low - high	low	low - high	low
Vacuum	possible	- 10 <sup>-4</sup> hPa	possible	poor	poor
Discharging	very difficult	easy	very difficult	medium	easy
Airlock	impossible	yes	impossible	difficult**	impossible***
Temperature control	very difficult	possible	difficult	possible	possible
Investment (costs)	low	very high	low	high	low
Operation (costs)	very high	high	low	high	very low



- [1] H. Zoz, D. Ernst, H. Weiss, M. Magini, C. Powell, C. Suryanarayana, F.H. Froes, *Mechanical Alloying of Ti-24Al-11Nb (at%) using the Simoloyer (Zoz - horizontal rotary ball mill), Part I*, ISMANAM 96 / Rome, Proceedings
- [6] H. Zoz: *Mechanical Alloying, Powder Metallurgy in Aerospace, Defense and Demanding Applications*, 1995, eds P.S. Goodwin, R.B. Schwarz
- [7] H. Zoz, D. Ernst, I. S. Ahn, W.H. Kwon, *Mechanical Alloying of Ti-Ni-based Materials using the Simoloyer*, TMS Annual Meeting 1997, eds. C.M. Ward-Close, F.H. Froes, S.S. Cho, D.J. Chellman: *Synthesis/Processing of Lightweight Metallic Materials*, (Proceedings 1997)
- [8] Brite-Euram Project BE-95-1321, *Novel Processing Technique for Ag<sub>3</sub>SnO<sub>3</sub> Electrical Contact Materials*, 1996 - 1999, International Materials Research Congress, Cancun, Mexico, 1997
- [10] H. Zoz, D. Ernst, T. Mizutani, H. Okouchi, *Simoloyer\* CM100s, semi-continuously Mechanical Alloying in a production scale using Cycle Operation - Part I (\* Zoz - horizontal rotary ball mill)*, *Advances in Powder Metallurgy & Particulate Materials - 1997*, Proceedings of the PM'97 Conference, Chicago, Vol. 2, p. 11-35, 1997
- [11] R.M. Davis, B. McDermott, C.C. Koch, *Mechanical Alloying of Brittle Materials*, *Metall. Trans.* Vol. 19a, 2867 (1988)
- [12] N. Burgio, A. Iasonna, M. Magini, S. Martelli, F. Padella, *IL NUOVO CIMENTO*, Vol. 13 D, No. 4 (April 1991)

Fig. 4: comparison of various milling devices

Low Kinetic Processing - shear and friction -



Fig. 5a: Rollermill RM1 with transparent vessel RBG02

- collision - High Kinetic Processing

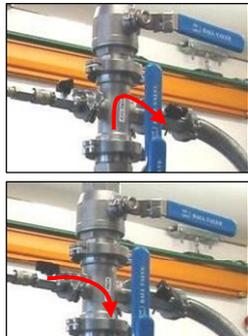
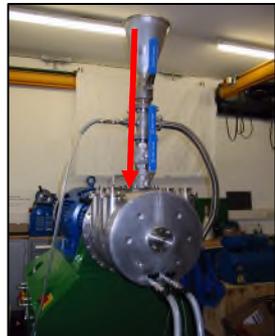
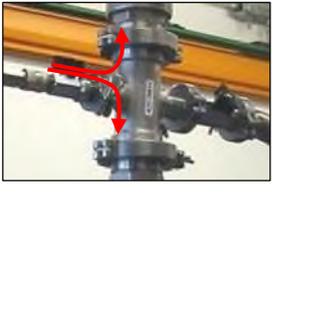


Fig. 5b: Simoloyer<sup>®</sup> CM01 with transparent vessel 01-21

technical data subject to alterations

## Simoloyer<sup>®</sup>: major characteristics and features, appendix I A1/3

### 3. Simoloyer can be operated, charged and discharged under vacuum/inert gas

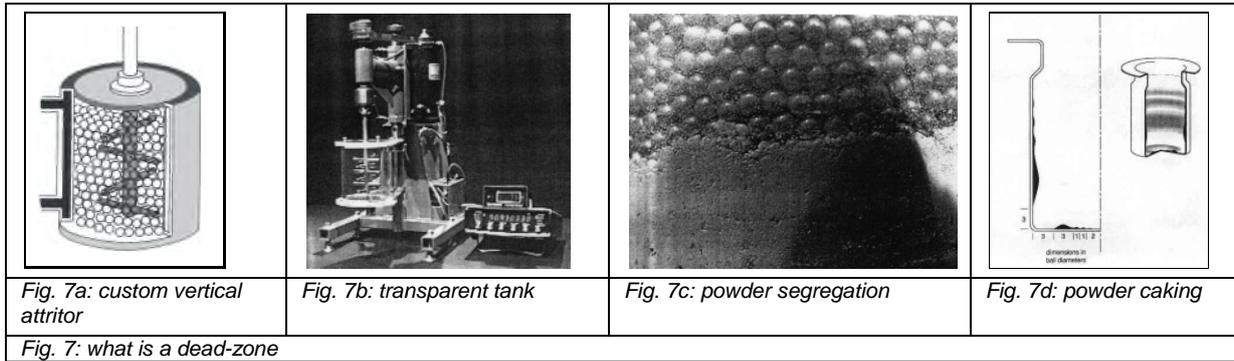
			
<p><i>Fig. 1: loading of the starting material into the valve-container inside a glove-box</i></p>	<p><i>Fig. 2a: closed and loaded valve-container connected</i></p>	<p><i>Fig. 2b, c: evacuation (air-lock, grinding unit) and refill of inert gas</i></p>	<p><i>Fig. 3: transfer of the starting material into the grinding chamber</i></p>
<p><b>Operation position:</b></p>			
			
<p><i>Fig. 4: processing under inert gas or vacuum</i></p>	<p><i>Fig. 5a: unloaded valve-container disconnected, sampling unit connected and evacuated</i></p>	<p><i>Fig. 5b: turning from Charging/Operation into Discharging Position</i></p>	<p><i>Fig. 5c: discharging sample under inert gas or vacuum</i></p>
<p><b>Discharging position:</b></p>			
			
<p><i>Fig. 6a: new/cleaned valve-container connected and evacuated (incl. air-lock)</i></p>	<p><i>Fig. 6b: refill of inert gas into air-lock and valve-container (may be repeated)</i></p>	<p><i>Fig. 7a: discharging product under inert gas or vacuum</i></p>	<p><i>Fig. 7b: loaded valve-container disconnected, ready for further processing</i></p>

## Simoloyer®: major characteristics and features, appendix I A1/4

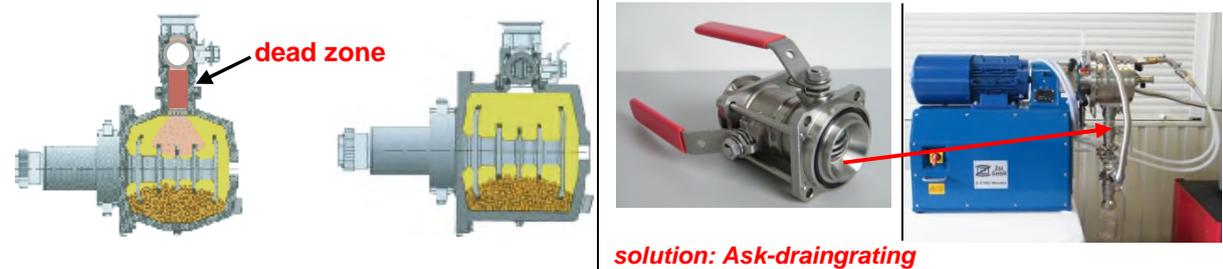
### 4. Simoloyer allows processing without dead-zones & negative gravity influence

What is a dead-zone?

- [1] an area in the process where grinding media motion is significantly lower than in average in the process;
- [2] an area in the process where the product (powder) density is significantly higher than in average in the process and leads to the caking effect;

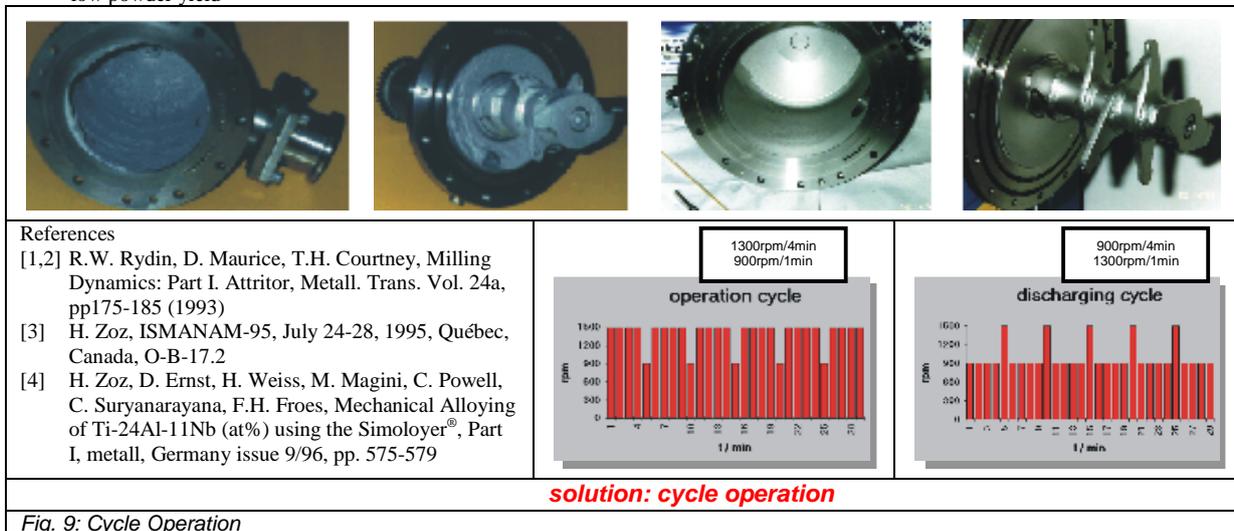


[3] an area in the process where grinding media can not go but powder;



What is a dead-layer?

- [4] a phenomena that occurs during high kinetic processing of CMB-materials due to their high ductility and adhesion tendency;
  - powders stick to the milling tools;
  - a large amount of powder is stored in layers on the vessel inner surface;
  - no further processing takes place;
  - sensitive change of concentration in the remaining free powder
  - low powder yield



*Fig. 9: Cycle Operation*

technical data subject to alterations

## Simoloyer®: major characteristics and features, appendix I A1/5

### 5. Simoloyer can be operated by Cycle Operation

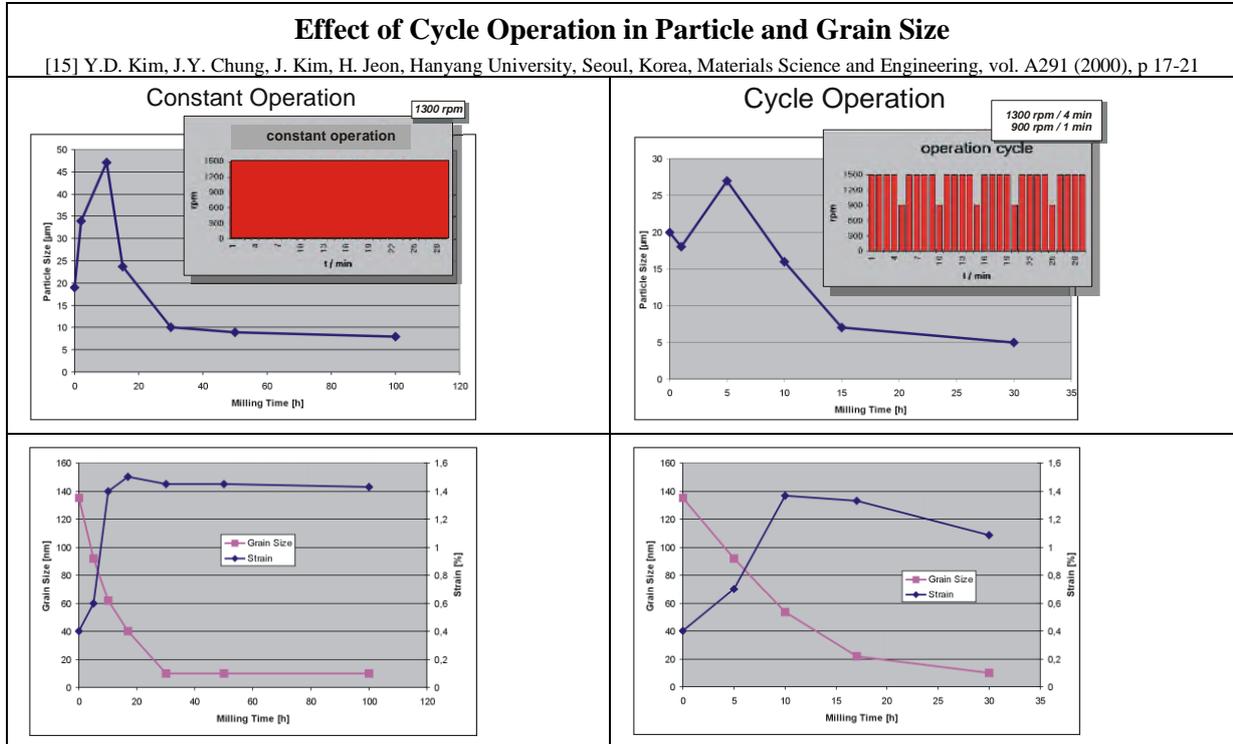


Figure 10a-d: Process parameter (constant-, cycle-operation)

### Simoloyer CM100s1, production of ductile metal-flakes (Ag, Cu) in batch-operation mode

[21] H. Zoz<sup>1</sup>, D. Ernst<sup>1,3</sup>, T. Mizutani<sup>2</sup>, H. Okouchi<sup>2</sup>;

<sup>1</sup>Zoz GmbH, D-57482 Wenden, Germany. <sup>2</sup>Fukuda Metal Foil & Powder Co. Ltd., Kyoto, Japan. <sup>3</sup>University of Siegen, D-57068, Siegen, Germany



Fig. 11a, -b: starting copper powder

Fig. 11c: Simoloyer CM100

Fig. 11d, -e: result of processing (3 min)

Fig. 11f: Maltoz3\*-SOP

Operation parameters:

contents: 200 kg of grinding media (100Cr6, Ø5mm), 20 kg of basic copper powder,

processing: 3 min. at 430 rpm (11,5m/s) → Process capability

discharging: 5 min. cycle (3 sec at 350 rpm, 13 sec at 65 rpm)

charging, handling, store: 10 min. approx.

full cycle: about 20 minutes approx.

→ 60 kg/h → 600 kg every day!

→ 400kg/h → 4t every day

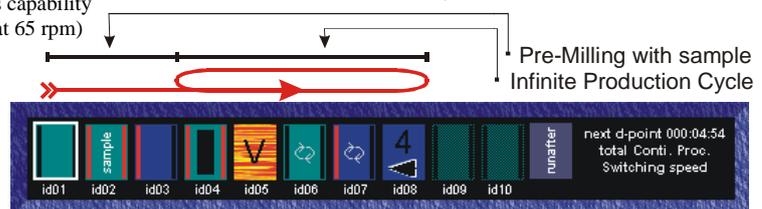


Fig. 11g: Maltoz®-program

technical data subject to alterations

## Simoloyer<sup>®</sup>: major characteristics and features, appendix I A1/6

### 6. Simoloyer can be operated at high temperature due to separated cooling/heating system

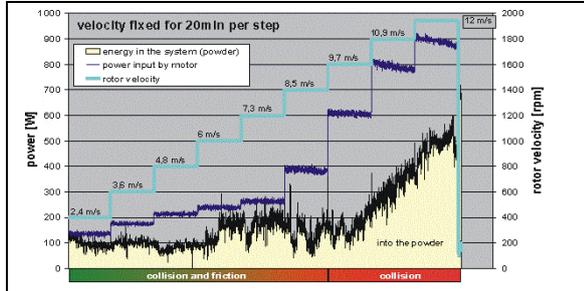


Fig. 12a: energy balance during High Kinetic Processing



Fig. 12b: cooling block KB100 (Simoloyer CM100)



Fig. 12c: heat-able grinding unit & control unit at CM01

### 7. Simoloyer is fully software operated, allows parameter measurement, control and history

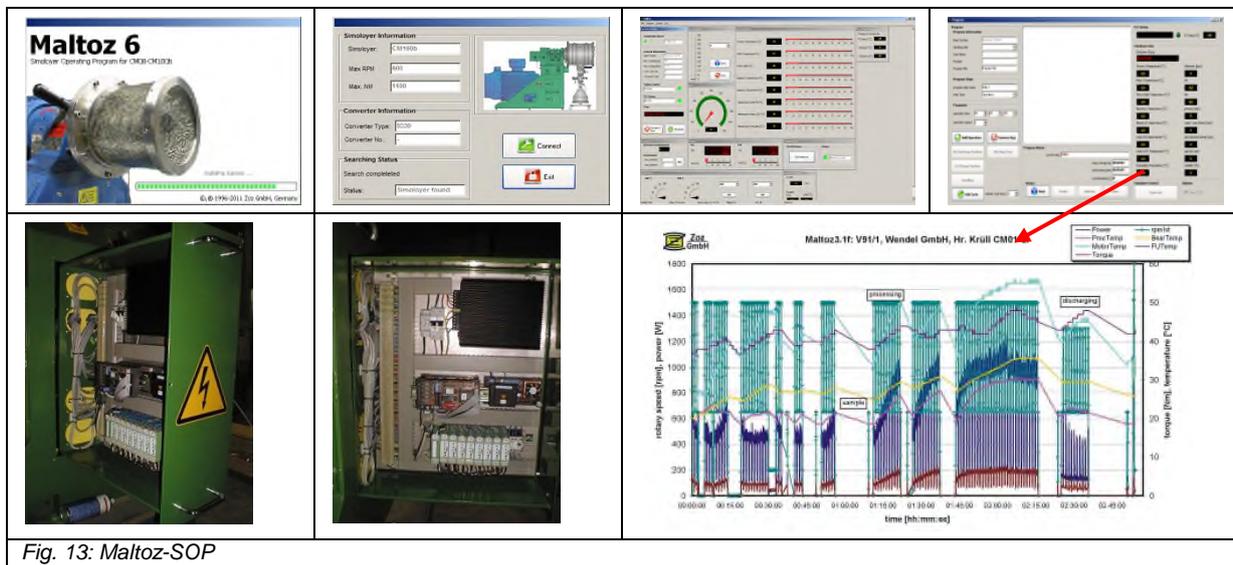
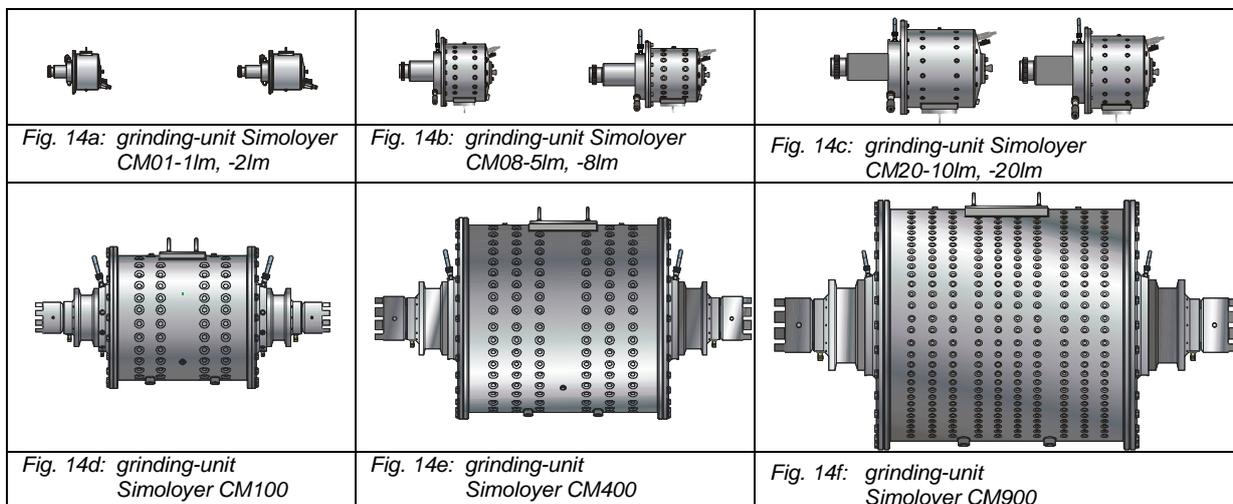


Fig. 13: Maltos-SOP

### 8. Simoloyer is technically scalable from 1 to 900 liters processing chamber capacity



technical data subject to alterations

## Simoloyer<sup>®</sup>: major characteristics and features, appendix I A1/7

### 9. Simoloyer can be operated in batch, semi-continuously and auto-batch mode (carrier gas)

#### Simoloyer CM01-2Is1, attempt of semi-continuous operation using compression mode



Fig. 15a: CM01-s1

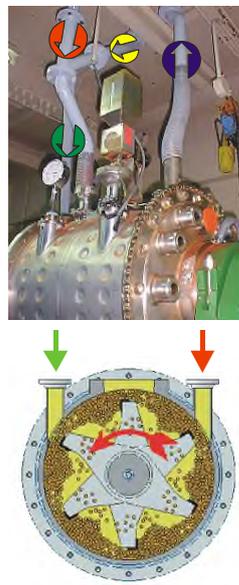


Fig. 15b: CM100-s1

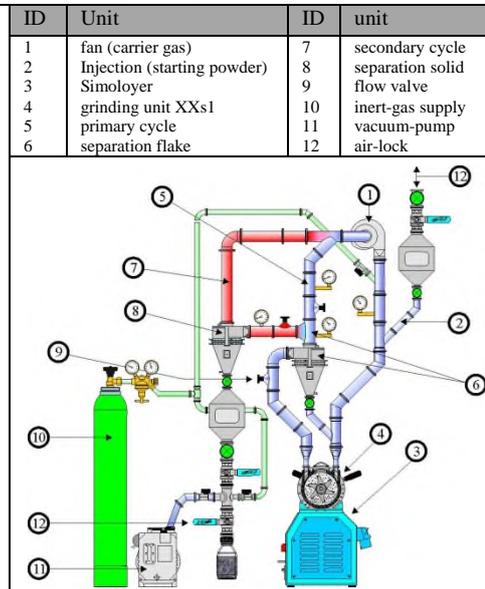


Fig. 15c: principle of CM01-s1

#### Simoloyer<sup>®</sup> CM20 (auto-batch configuration)

ID	unit	function
01	container	starting powder (multi)
02	rotary-vane-feeder	starting powder charging (multi)
03	side-channel-turbine	Carrier gas drive
04	automatic ball-valve	starting powder/airflow charging
05	grinding chamber	milled powder
06	automatic ball-valve	powder down/air up
07	cyclone	aerodynamic powder separation
08	rotary-vane-feeder	milled powder discharging
09	container	milled powder container

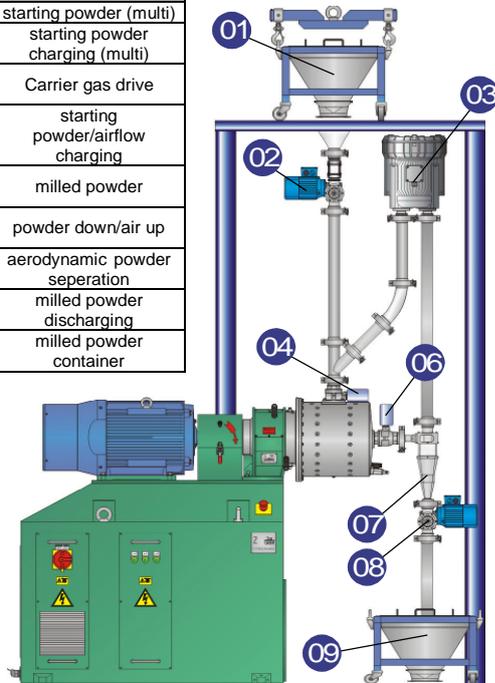


Fig. 16a: CM20-s2 (autobatch-configuration)

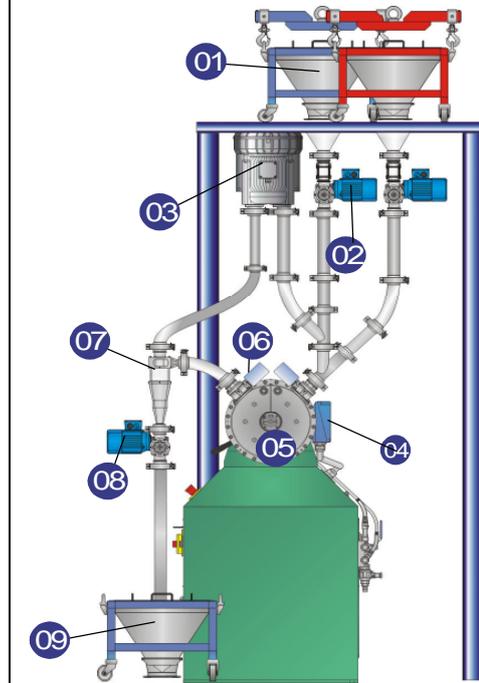


Fig. 16b: CM20-s2 (autobatch-configuration)

technical data subject to alterations

## Simoloyer<sup>®</sup>: major characteristics and features, appendix I A1/8

### 10. Simoloyer is available with tools by stainless steel/stellite, WC-Co and Si<sub>3</sub>N<sub>4</sub>

Simoloyer<sup>®</sup>, examples of different types of grinding-units and grinding media



Fig. 17a: Grinding unit W01-2Im-xx



Fig. 17b: Grinding-unit W01-2Im-SiN



Fig. 17c: Grinding unit W01-2Im-SiN



Fig. 18a: Grinding unit W01-2Im

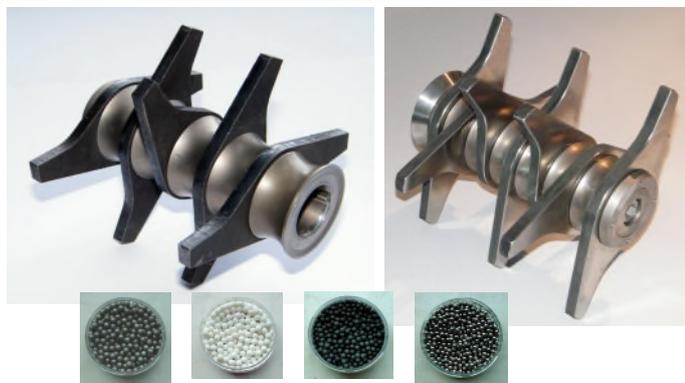
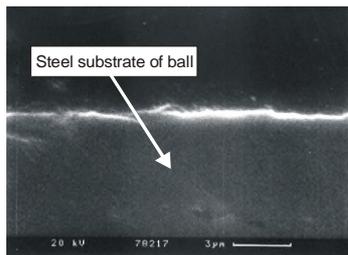


Fig. 18b: different types of rotors and grinding media for grinding units

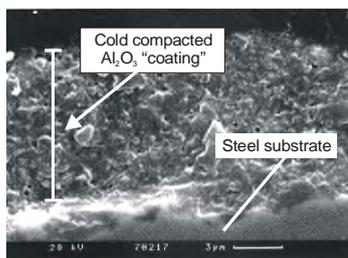
Simoloyer<sup>®</sup>, self-coating effect of grinding-media (→ reduction of abrasion)

### Cold Compacted "Coatings" on the grinding media grindig media - 100Cr6 (self) coated by:

Al<sub>2</sub>O<sub>3</sub>-5SiC



Cross section of a steel ball before milling

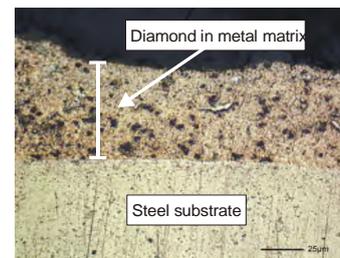


Cross section of a ball with cold compacted "coated" Al<sub>2</sub>O<sub>3</sub> layer after milling

synthetic diamond in metal matrix



100 Cr 6 Mahlkugel mit CuTi10 mit 2,4µm Diamant



Cross section of a ball with cold compacted diamond composite layer after milling

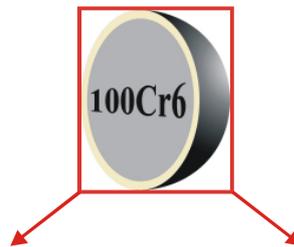


Fig. 19: coating of grinding media by Al<sub>2</sub>O<sub>3</sub> and diamond, cross-section and light-microscopy

technical data subject to alterations