

# **High Energy Milling of Temperature-Sensitive Intermediates - Scale-up to Production**

## **1. Introduction**

Pigmented intermediates like colour concentrates which are ultra-fine ground in agitated media mills have to fulfil constantly increasing quality demands with respect to particle fineness or coloristic properties, for example. For producing the desired quality characteristics, a particularly high volume-specific power input is frequently required, which can be handled in special mills in the development laboratory. As modern formulations are increasingly temperature-sensitive, the transfer of the wet grinding to production scale often becomes difficult, if not impossible. The following contribution analyzes the basic problems of scale-up. It will be shown that there is a substantial potential of achieving a quality increase by agitated media milling with high power density using new mill constructions which are capable of a linear scale-up. The operation with high power density results furthermore in a considerable increase in the economic efficiency of the production process.

## 2. Definition of mass-specific energy input and power density

### 2.1 Mass-specific energy input

The quality of dispersion or grinding in an agitated media mill is usually a function of the energy applied to the mill base.

$$\text{Quality} = f(\text{mass-specific energy}) \quad [1]$$

In pass operation, the mass-specific energy input is calculated as the quotient of the net power consumption of the grinding chamber and the product mass flow:

$$E_{m,\text{pass}} = \frac{P - P_0}{\dot{m}} \left( \frac{\text{kWh}}{\text{t}} \right) \quad [2]$$

$P$  : [kW] power absorption of motor

$P_0$  : [kW] no-load power (operation without beads)

$(P - P_0)$  : [kW] net power absorption of grinding chamber

$\dot{m}$  : [t/h] product mass flow

$E_m$  : [kWh/t] mass-specific energy input

In recirculation operation, the net power absorption is integrated over the grinding time and the integral is related to the batch quantity:

$$E_{m,\text{recirc}} = \frac{\int_0^{t_{\text{grind}}} (P - P_0) dt}{m} \quad [3]$$

If, for a particular application, the mass-specific energy input required to obtain a defined quality is known from a laboratory or pilot trial, the production mass flow is obtained from:

$$\dot{m}_{\text{prod,pass}} = \frac{P - P_0}{E_m} \quad [4]$$

$$\text{or: } \dot{m}_{\text{prod,recirc}} = \frac{m}{t_{\text{grind}}} = \frac{\int_0^{t_{\text{grind}}} (P - P_0) dt}{E_m \cdot t_{\text{grind}}} \quad [5]$$

The quantity that can be produced per unit of time is, therefore, directly proportional to the power applied to the grinding chamber ( $P - P_0$ ). In this general context, the statement is only valid if agitated media mills are operated within reasonable parameter ranges. This aspect will not be dealt with in any further detail here.

## 2.2 Power density in the grinding chamber

With many demanding dispersing and grinding tasks, the desired quality characteristics can be obtained only if, as an additional condition, a certain intensity of load is exceeded. A macroscopic, readily quantifiable number for a mean of the load intensity is the volume-specific power density  $P_V$  of the grinding chamber.

$$P_V = \frac{P - P_0}{V_{MR}} \quad [6]$$

$P_V$  : [kW/l] volumetric power density

$V_{MR}$  : [l] volume of grinding chamber

The term high power density means hereinafter values of:

$$P_V \geq 2 \text{ kW / l} \quad [7]$$

Examples of application in which desired product properties of heat-sensitive formulations may be obtained only if a relatively high power density threshold is exceeded are:

- Intensive grinding of aqueous pigment preparations with the quality target of maximizing the colour strength.  $\rightarrow t_{\text{max}} \leq 35 - 40 \text{ }^\circ\text{C}$
- Producing chips grade with liquid printing inks and pigment concentrates for coating applications. The desired quality characteristics, apart from colour strength, are often gloss and transparency.  $\rightarrow t_{\text{max}} \leq 50 \text{ }^\circ\text{C}$
- Dispersing of black pastes for industrial coatings, particularly automotive paints with the quality target of "jetness".  $\rightarrow t_{\text{max}} \leq 40 \text{ }^\circ\text{C}$

- True grinding of ultra-lightfast polycyclic pigments in the nanometer range with a view to maximizing colour strength and gloss by means of particle size distributions in the range of 20 – 80 nm. →  $t_{\max} \leq 40 \text{ }^\circ\text{C}$
- Microdispersing of UV-curable printing ink concentrates. →  $t_{\max} \leq 45 \text{ }^\circ\text{C}$

### 3. Scale-up-problems in full space mills with jacket cooling only

#### 3.1 Power density as a function of mill size

Figure 1 shows a schematic of a conventional full space agitated media mill. The product is fed in at the front end of the grinding chamber. Mechanical agitation is applied to the mixture of mill base and grinding media by means of an agitator of any design. Depending on the flow rate and viscosity, the mill base will exert greater or lesser drag forces on the grinding media with a tendency to transport the beads in the direction of the grinding media retaining device at the other end of the grinding chamber, where they become concentrated.

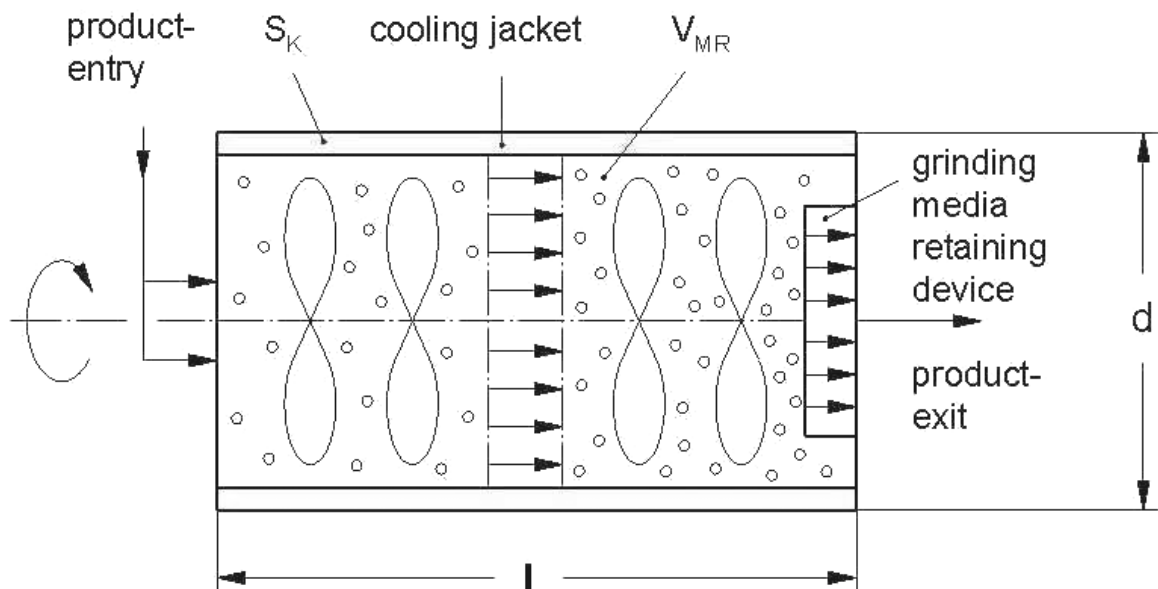


Fig. 1: Schematic representation of a full space mill with jacket cooling

In conventional full space mills, only the outer surface of the jacket is available as a cooling surface. In a laboratory mill of this type with a volume of 1 l, experience has shown that an agitation power of about 2 kW can be applied without overheating

temperature-sensitive products. The maximum realizable power density is therefore about  $P_V = 2 \text{ kW / l}$ .

For products which can be produced on a laboratory scale and which require a power density during grinding of up to  $2 \text{ kW / l}$  in order to develop the desired quality characteristics, the transfer to industrial-scale production becomes all the more difficult the larger the volume of the full space mill gets, as shown by the following relationships:

The jacket cooling surface is calculated from:

$$S_K = \pi \cdot d \cdot l \quad [8]$$

The grinding chamber volume, ignoring the agitator volume, is

$$V_{MR} = \pi \frac{d^2}{4} \cdot l \quad [9]$$

Thus the volume-specific cooling surface

$$\frac{S_K}{V_{MR}} = \frac{4}{d} \quad [10]$$

becomes smaller in inverse proportion to the increasing diameter of the grinding vessel. The values are particularly unfavourable in the case of large-volume, short and stout types of mills.

Assuming a geometric scale-up specification  $l / d = \text{const.}$ , it follows

from equation 8:  $S_K \sim d^2$  and from equation 9:  $V_{MR} \sim d^3$

and from equation 9 inserted into equation 8:

$$S_K \sim V_{MR}^{2/3} \quad [11]$$

If development work is carried out on a pilot mill with the cooling surface  $S_{K,pilot}$  and the grinding chamber volume  $V_{MR,pilot}$  and if production is to be transferred to an industrial-scale mill with  $V_{MR,prod}$ , then the following applies for the cooling surface ratio, according to equation 11:

$$S_{K,prod} = S_{K,pilot} \cdot \left( \frac{V_{MR,prod}}{V_{MR,pilot}} \right)^{2/3} \quad [12]$$



If, for example, the production-scale execution has a 10 times larger grinding chamber volume, the cooling surface according to equation 12 is greater only by a factor of  $10^{2/3} = 4.6$ . In order to prevent product overheating, the power density must be reduced accordingly to less than half since the power input, assuming an identical heat transfer, may only rise at most proportionately to the increase in the cooling surface:

$$P - P_0 \sim S_K \sim V_{MR}^{2/3} \quad [13]$$

$$(P - P_0)_{prod} = (P - P_0)_{pilot} \cdot \left( \frac{V_{MR, prod}}{V_{MR, pilot}} \right)^{2/3} \quad [14]$$

Equation 10 inserted into equation 13 gives as scale-up specification for the permissible power density:

$$\frac{P - P_0}{V_{MR}} \sim \frac{S_K}{V_{MR}} = \frac{4}{d} \quad [15]$$

The power density that can be realized in a full space mill decreases in a proportion of  $1 / d$  with the mill diameter for larger mills.

In order to avoid product overheating, large full space mills must be operated with an increasingly low power density. One of the consequences of this is that the productivity based on grinding chamber size falls, and so does the efficiency; moreover, it is no longer possible to produce demanding products once the power density has fallen below a critical level. One possible, though disadvantageous solution would be to operate a fairly large number of small full space mills in parallel in order to achieve the high product output desired.

### 3.2 Estimate of the required grinding chamber volume

Pilot plant trials show that a specific energy  $E_m$  has to be supplied in order to obtain a desired product quality. An industrial-scale production rate of  $\dot{m}_{prod}$  (t/h) is required. The power to be supplied to the production-scale mill is obtained from equation 2:

$$(P - P_0)_{prod} = \dot{m}_{prod} \cdot E_m \quad [16]$$

The minimum grinding chamber size to be considered can be estimated from equation 14 after conversion and use of equation 16:

$$V_{MR,prod} = V_{MR,pilot} \cdot \left( \frac{\dot{m}_{prod} \cdot E_m}{(P - P_0)_{pilot}} \right)^{3/2} \quad [17]$$

## 4. Small-volume high-performance mills with defined internal grinding media recirculation

### 4.1 Conditions for a linear scale-up

A reliable scale-up of agitated media milling with high power density with a reproducible product quality can be achieved only if the specific cooling surface and the power density realizable at a constant product temperature are independent of the mill size. Moreover, a heat transfer that does not depend on mill size must be guaranteed.

$$\frac{S_K}{V_{MR}} = \text{const} \neq f(\text{mill size}) \quad [18]$$

$$\frac{P - P_0}{V_{MR}} = \text{const} \neq f(\text{mill size}) \quad [19]$$

In applications with a relatively low energy requirement, a high realizable power density leads to a high mass throughput (corresponding to a high production rate), according to equation 4. Even with extreme operating parameters, therefore, the local bead distribution should be independent of the product throughput in order to be sure of preventing local product overheating due to grinding media hold-up in front of the separating device.

It follows from equation 19 that:

$$(P - P_0)_{prod} = (P - P_0)_{pilot} \cdot \frac{V_{MR,prod}}{V_{MR,pilot}} \quad [20]$$

On the basis of pilot plant trials, the mill volume required for industrial-scale production can be estimated from equation 20:

$$V_{MR,prod} = V_{MR,pilot} \cdot \frac{\dot{m}_{prod} \cdot E_m}{(P - P_0)_{pilot}} \quad [21]$$

#### 4.2 Structure and function of a multiple-zone Perl Mill with a non size-dependent power density

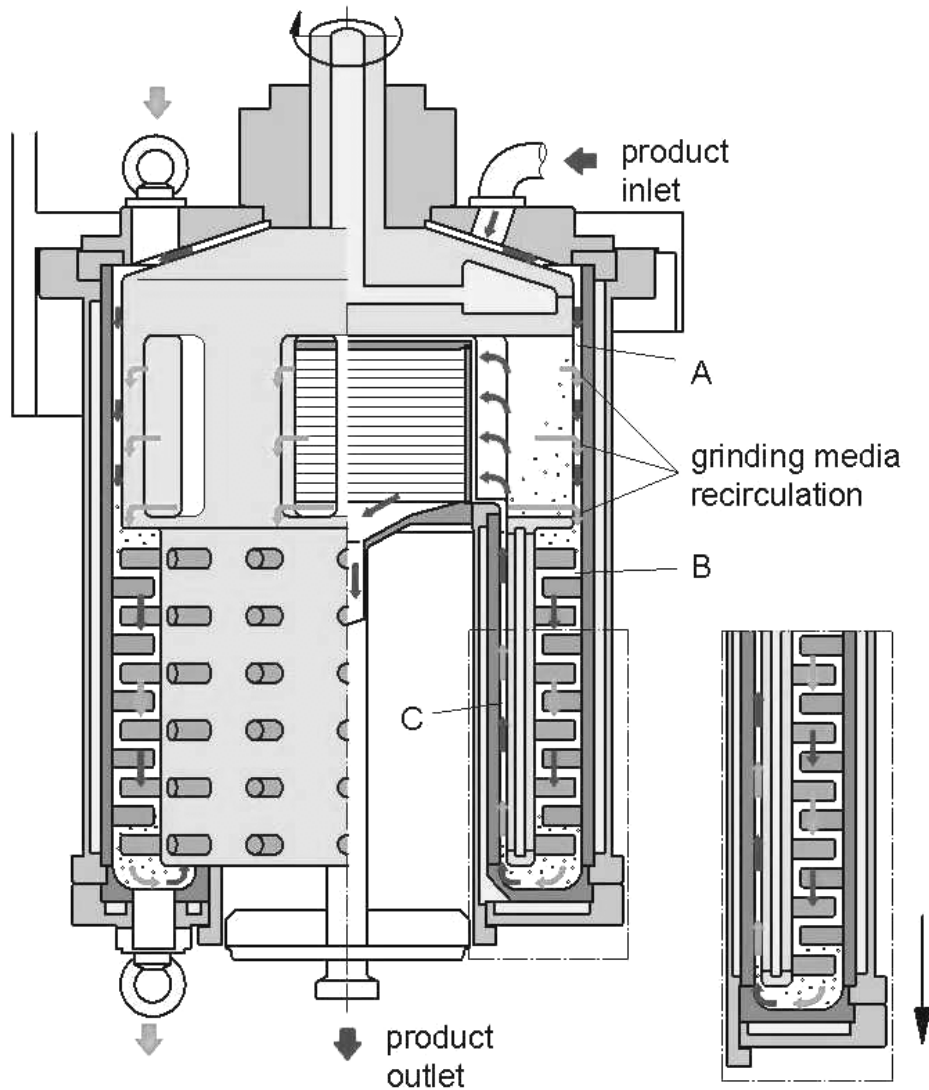


Fig. 2: Schematic of the double-cylinder multiple-zone Perl Mill Advantis

An example of a mill design with the above-mentioned features is the multiple-zone Perl Mill Advantis according to Figure 2 [1, 2, 3]. A hollow cylindrical rotor driven from the top is situated in a double-cylinder stator housing. The mill base flows in from the top, first through a cylindrical eddy current inducing gap in which intensive predispersing takes place. This is followed by intensive grinding due to fluidization of the grinding media in the peg-counterpeg zone of the outer grinding chamber, which is delimited by the outer stator cylinder and the outer rotor surface. After the mill base/grinding media mixture has been diverted in the bottom zone, shear grinding

takes place in the inner Couette gap between the inner rotor surface and the smooth-walled inner stator.

The grinding media carried along with the product flow is separated at the upper end of the inner grinding chamber by centrifugal force and arrives back in the eddy current inducing gap through slots in the rotor. As a result of this principle of defined internal grinding media recirculation, it is possible to obtain flow rates which are greater by more than one order of magnitude compared with full space mills of the same size.

In Figure 2, the structural scale-up principle is represented schematically. Retaining all the relevant dimensions, the process chamber in larger mills is merely extended to a greater diameter and lengthened axially. In contrast to conventional full space mills, the ratio of cooling surface to mill volume is independent of the mill size. For this reason, it is possible to realize non size-dependent power densities of up to  $P/V = 4 \text{ kW/l}$  with the same low product temperature on a laboratory scale and on an industrial scale.

### 4.3 Increasing productivity and/or quality

As an example of application, the dispersion of a transparent pigment paste in recirculation operation will be discussed below. In a multiple-zone high-performance mill on an industrial scale, the power density was varied in a controlled manner by increasing the rotor speed.

In Figure 3 the relative transparency is plotted against the relative grinding time as a quality characteristic. The standardization parameter used for time and transparency was the grinding time  $t_{\text{standard}}$  which is required to achieve the previous quality specification for the transparency  $x_{\text{standard}}$  with a power density of  $2.1 \text{ kW/l}$  in the grinding chamber. In view of the extraordinarily large cooling surface, the flow of coolant had to be reduced considerably in order to arrive as close as possible to the limiting temperature  $t_{\text{max}}$ . The relative transparency initially increases very quickly. By definition, the desired quality  $x_{\text{standard}}$  is reached after  $t / t_{\text{standard}} = 1$ . Only a slightly higher transparency can be obtained by prolonging the grinding time. A constant quality plateau of 104 % is soon reached.

Dispersion with a higher power density of  $3 \text{ kW/l}$  leads to a much more rapid increase in quality. The standard is reached after about 61 % of the original dispersing time. By continuing the recirculation operation, a much better transparency can be achieved. The final level is 27 % higher than the previous standard.

For an even higher power density of  $3.3 \text{ kW/l}$ , the third curve (triangles) is obtained. The dispersing time to achieve the standard quality can thus be reduced to below 50 %. If necessary, by prolonging the energy input at a very high power density a final transparency value of 137 % can be obtained compared with the previous standard.



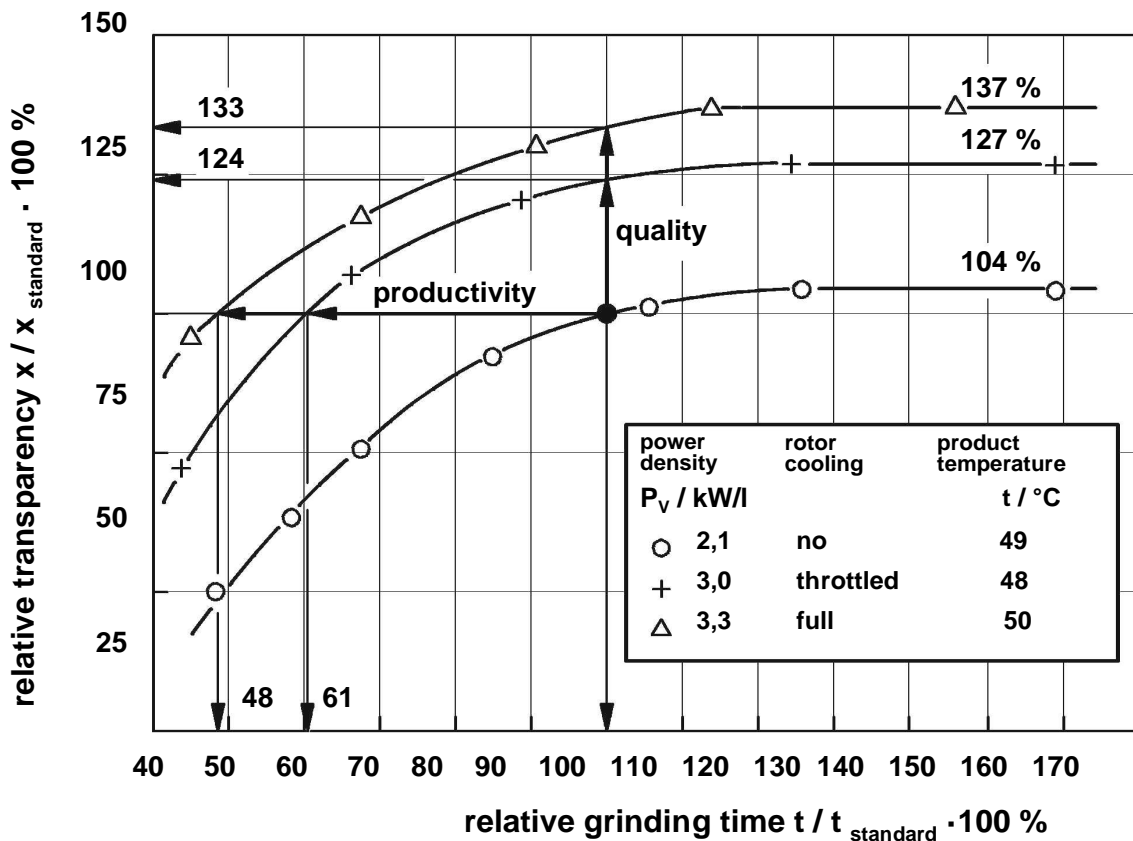


Fig. 3: Dispersing of a transparent pigment paste in recirculation operation with 3 power densities in the grinding chamber, transparency as a function of the grinding time

Figure 4 shows the relative transparency as a function not of the grinding time, but of the mass-specific energy input. The previous quality standard  $x / x_{\text{standard}} = 100 \%$  is achieved with a specific energy input of 540 kWh/t irrespective of the power density in the grinding chamber. Only when there is a further energy input do the three curves diverge. In the event of a higher power density, the further addition of energy is used to increase quality, whereas a power density of 2.1 kW/l scarcely permits any further increase in transparency. It should be borne in mind that even this lowest value could not be realized in full space mills on a production scale without overheating the product.

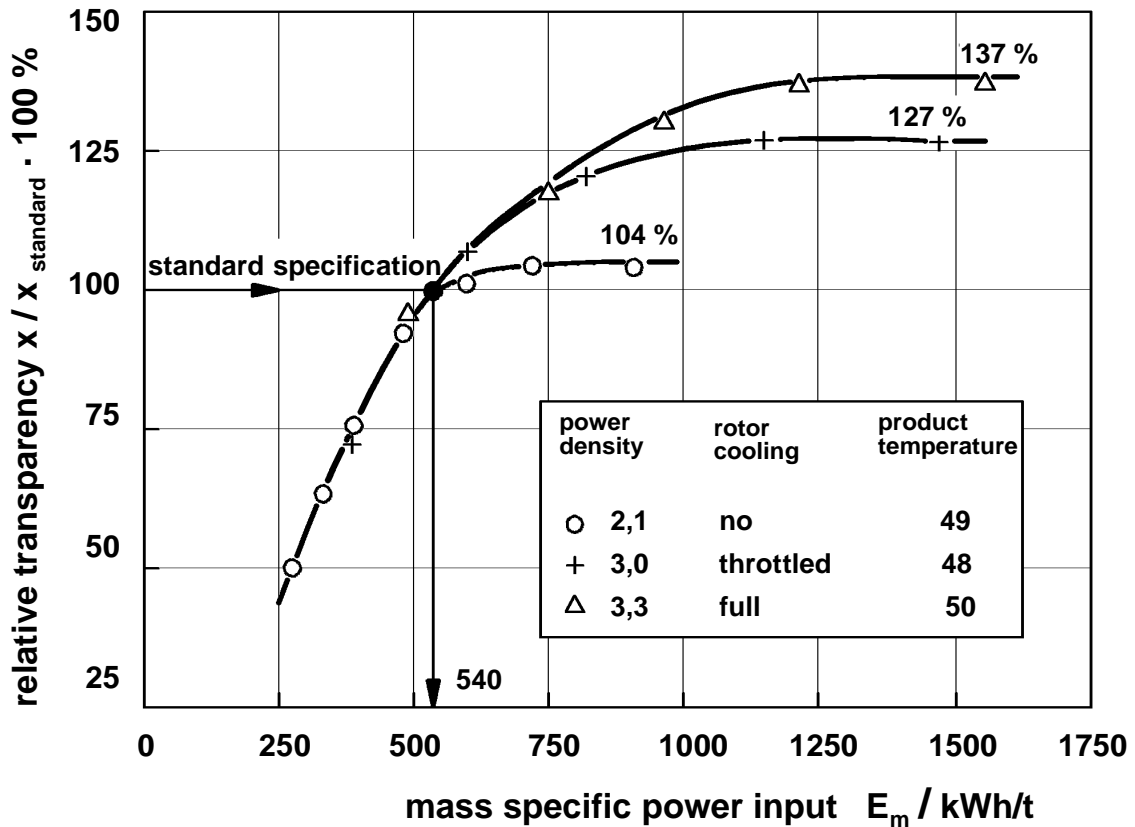


Fig. 4: Dispersing of a transparent pigment paste in recirculation operation with 3 power densities in the grinding chamber, transparency as a function of the specific energy input

To summarize, the findings of Figures 3 and 4 contain two options:

1. Whilst retaining the previous quality standard, the grinding time can be drastically reduced and thus the production rate can be increased by grinding with a higher power density. As a result, the overall economic efficiency of the plant is enhanced considerably.
2. By setting a higher power density, it is possible to obtain quality standards at a much higher level which could not be achieved with a lower power density even by prolonging the grinding time indefinitely (or by reducing the throughput in pass operation).

## 5. Example for scale-up

For grinding a heat-sensitive product with  $t_{\max} \leq 45 \text{ }^\circ\text{C}$ , the specific energy requirement both in a full space mill and in a high-performance mill on a pilot plant scale was determined at  $E_m = 200 \text{ kWh/t}$  in each case, despite the difference in power density. For industrial-scale production, a throughput of 500 kg/h is required. The design stages are summarized in the following table.

Table: Scale-up stages on the basis of pilot plant trials

<u>Pilot mill</u>			<u>full space mill</u>	<u>double-cylinder annular gap mill</u>
Grinding chamber	$V_{MR}$	[l]	5	1.1
Cooling surface	$S_K$	[m <sup>2</sup> ]	0.16	0.13
Max. net power for $t \leq t_{\max}$	$(P - P_0)$	[kW]	5	4.4
Power density	$\frac{P - P_0}{V_{MR}}$	$\left[ \frac{\text{kW}}{\text{l}} \right]$	1	4
Specific energy	$\frac{P - P_0}{\dot{m}_{\text{pilot}}}$	$\left[ \frac{\text{kWh}}{\text{t}} \right]$	200	200
Production rate	$\dot{m}_{\text{pilot}}$	$\left[ \frac{\text{kg}}{\text{h}} \right]$	25	22.5
<u>Industrial-scale production</u>			<u>full space mill</u>	<u>double-cylinder annular gap mill</u>
Specific energy	$\frac{P - P_0}{\dot{m}_{\text{prod}}}$	$\left[ \frac{\text{kWh}}{\text{t}} \right]$	200	200
Throughput	$\dot{m}_{\text{prod}}$	$\left[ \frac{\text{t}}{\text{h}} \right]$	0.5	0.5
Net power (equation 16)	$(P - P_0)$	[kW]	100	100
Grinding chamber	$V_{MR,\text{theo}}$	[l]	from equation 17: $5 \text{ l} \left( \frac{0.5 \cdot 200}{5} \right)^{3/2} = 447 \text{ l}$	from equation 21: $1.1 \text{ l} \left( \frac{0.5 \cdot 200}{4.4} \right) = 25 \text{ l}$
Selected size	$V_{MR,\text{actual}}$	[l]	500	26
Power density	$\frac{P - P_0}{V_{MR}}$	$\left[ \frac{\text{kW}}{\text{l}} \right]$	0.2	3.85

The table shows that even the pilot plant machine featuring the annular gap principle has a grinding chamber volume which is more than 4 times smaller compared to the full space pilot mill. The net power of 100 kW required for industrial-scale production can be applied to a commercially available 26 l mill with the same specific cooling surface and power density. In the case of the full space mill, the demand for an identical specific cooling surface leads to a virtually 20 times larger grinding chamber volume of more than 400 l.

The power density is reduced by a factor of 5 compared with the full space pilot plant machine and proves to be approximately 20 times lower than in the double-cylinder annular gap mill. It is often the case that the desired quality can no longer be achieved with such a low power density.

The fact that the 20 times smaller annular gap volume requires only a proportionately smaller grinding media charge and has a drastically reduced flushing agent consumption makes a considerable contribution towards enhancing the economic efficiency. The material loss during a product change is negligible.

## 6. Summary

With temperature-sensitive products, conventional full space agitated media mills on an industrial scale arrive at the limits of the scale-up potential. Double-cylinder annular gap mills, on the other hand, are capable of a linear scale-up. The high power density in the small-volume annular gap mills frequently leads to a major increase in the achievable product quality. Moreover, due to the high productivity obtained from a small grinding chamber, they make a substantial contribution towards reducing the operating costs and thus towards increasing the economic efficiency of the production process.

## 7. Literature

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