Technische Universität Braunschweig

iPAT Institut für Partikeltechnik

Wet grinding and dispersing

Fundamental Considerations

Carsten Schilde and Arno Kwade
Institute for Particle Technology
Technische Universität Braunschweig

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Content

Introduction

Question

- Which parameters can be changed if we want to design or optimize a stirred media milling process?

Important operating parameters of stirred media mills

1. **Operating parameters of the mill**
   - Grinding or dispersing time
   - throughput
   - Stirrer tip speed
   - Grinding media size
   - Grinding media material (density, elasticity and hardness)
   - Filling ratio of the grinding media
2. **Operation mode of the mill** (One or multiple passage mode, pendulum or circuit operation)
3. **Formulation** (composition of the suspension)
   - Solids concentration of the pigments
   - Fluid (water, solvents, resins and so on)
   - Additives or dispersing agents (Reduction of the viscosity and/or avoidance of reagglomeration or flocculation)
4. **Mill geometry**
   - Type of the mill
   - Size and dimensions of the mill

Main objectives

a) Product quality, which is defined among others by

- Particle size distribution, gloss, intensity of colour, transparency
- Product purity (no contamination by wear of mill and grinding media)
- No product degradation (for example by too high temperatures)
- Stability (against reagglomeration, flocculation, sedimentation and so on)

b) Economy, which is determined above all by

- Investment costs
- Operating costs (energy, cooling water, maintenance and so on)
- Production capacity
- Cleaning expenditure

Content

3.1 Description of grinding and dispersing results

- Product quality
- Productions capacity
- Results related to grinding time and specific energy

3.2 Stress mechanism and stress models

- Mill related stress models
- Product related stress models
- Relation between model parameters

3.3 General application of the stress models

- Particles between two grinding media
- Estimation of stress events and frequency
- Estimation of stress energy and intensity
- Comparison of stress intensity and particle strength
- Specific energy and efficiency factor

Description of product quality

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- Depending on the industry and on the product the quality is determined for example by:
  - Optical (visual) characteristics of the particles or pigments respectively (intensity of color, glace, transparency)
  - Mechanical characteristics of fillers
  - Storage stability of dispersions and suspensions
  - Reactivity of disperse solids because of larger surfaces
  - Sintering activity of ceramic materials
- Product properties are mainly determined by the physical properties, namely the particle size distribution (**property function**)

Property function: Transmission as function of median particle size

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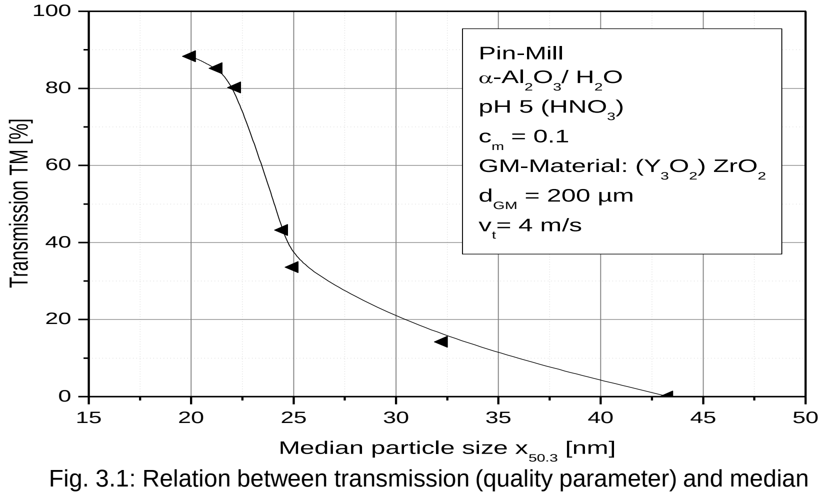

Fig. 3.1: Relation between transmission (quality parameter) and median particle size

Description of product quality

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  - Mechanical characteristics of fillers
  - Storage stability of dispersions and suspensions
  - Reactivity of disperse solids because of larger surfaces
  - Sintering activity of ceramic materials
- Product properties are mainly determined by the physical properties, namely the particle size distribution (**property function**)
- Particle size distribution is taken as main quality parameter
- Product quality is described by characteristic numbers (e.g. median size x_{50} and characteristic size x_{90})

Specific energy

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- Energy transferred into the grinding chamber related to the stressed product mass
- Batch operation:

$$E_m(t_C) = \frac{E(t_C)}{m_P} = \frac{\int_0^{t_c} (P(\tau) - P_0)\, d\tau}{m_P} \approx \frac{\bar{P} - P_0}{m_P} \cdot t_C \qquad (3.2)$$

- Continuous operation:

$$E_m = \frac{P_{stationary} - P_0}{\dot{m}_P} \qquad (3.4)$$

where P := mean power draw of the motor
P_0 := no-load power

Question

➢ Why is it important to minimize the specific energy consumption?

Basic equation of production rate

$$\dot{m}_P = \frac{m_{P,batch}}{t_{batch}} = \frac{P - P_0}{E_m} = \frac{P_{GC}}{E_m}$$

$$t_{Batch} = \frac{m_{P,Batch} \cdot E_m}{P - P_0}$$

where

$\dot{m}_P$	:= Solids mass throughput (solids mass flow rate)
$m_{P,Batch}$	:= Mass of a batch
t_{Batch}	:= Production time for a batch
P	:= Power draw of mill motor (gross power)
P_0	:= No-load power draw of mill motor (mill without content)
P_{GC}	:= Power transferred into grinding chamber (net power)
E_m	:= Specific energy for demanded product quality

Example 1
Determination of production capacity

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- The installed motor power of the mill is 25 kW with a no-load power of 5 kW for the stirrer tip speed under consideration.
- The specific energy requirement of your product (related on solids mass) for the given operating parameters is 100 kWh/t
- Which product mass of solids can you produce per hour with your mill?

1. net power: 25 kW – 5 kW =
2. Production rate:

$$\dot{m}_P = \frac{P - P_0}{E_m} =$$

Example 1
Determination of production capacity

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- The specific energy requirement of your product (related on solids mass) for the given operating parameters is 100 kWh/t
- Which product mass of solids can you produce per hour with your mill?

1. net power: 25 kW – 5 kW =
2. Production rate:

$$\dot{m}_P = \frac{P - P_0}{E_m} =$$

Example 2

- The demanded production rate is 1 t/h of solid product
- The specific energy requirement of your product (related on solids mass) for the given operating parameters is 100 kWh/t
- Which net power input must the mill or mills have under the assumptions, that the specific energy requirement is independent on mill size?

1. net power:

$$P - P_0 = \frac{E_m}{\dot{m}_P} =$$

Question

$$\dot{m}_P = \frac{m_{P,batch}}{t_{batch}} = \frac{P - P_0}{E_m} = \frac{P_{GC}}{E_m}$$

$$t_{Batch} = \frac{m_{P,Batch} \cdot E_m}{P - P_0}$$

- When do we get the maximum possible production rate?

Basic equation of production rate

- Maximum production rate is achieved, if
  - **power input into grinding chamber, $P - P_0$, is as high as possible**
  - **specific energy for production of certain product quality, E_m, is as low as possible.**
- Minimum specific energy decreases relative mill and grinding media wear to a minimum
- Power input and specific energy requirement depend on several operating parameters like v_t, d_{GM}, ρ_{GM}, c_m etc.
- **Problem:** Finding set of operating parameters, which cause the highest power input $P - P_0$ and the lowest specific energy E_m
- Equation is also basis for transfer of results from laboratory mill to a production mill (Scale-up)

Determination of power draw and specific energy

Power draw

- **Highest possible power draw** of a stirred media mill is restricted by
  - installed motor power
  - installed cooling capacity (in case of temperature sensitive product)
  - wear of mill and grinding media.
- Determination of operating parameters, at which the highest possible power draw is really transferred into the grinding chamber using **relation between power number and Reynolds number**

Specific energy

- Determination of operating parameters, at which the specific energy consumption for a certain product quality has a minimum value, using the so-called **stress model**

Determination of product quality as function of milling time and specific energy

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- Experiments give information how the product quality at different operating parameters depend on
  - Grinding and dispersing time
  - Specific energy.
- Experiments show how
  - Power draw
  - Throughput behavior
  - Cooling behavior
  - Wear behavior

  depend on the operating parameters.
- Different possibilities to run a grinding or dispersing test

Different possibilities to run a test

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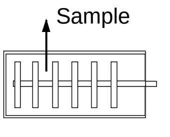
1. Batch operation



2. One Passage mode (continuous operation)

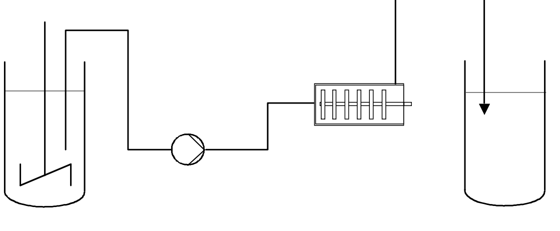


Fig. 3.2

Different possibilities to run a test

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3. Multiple passage

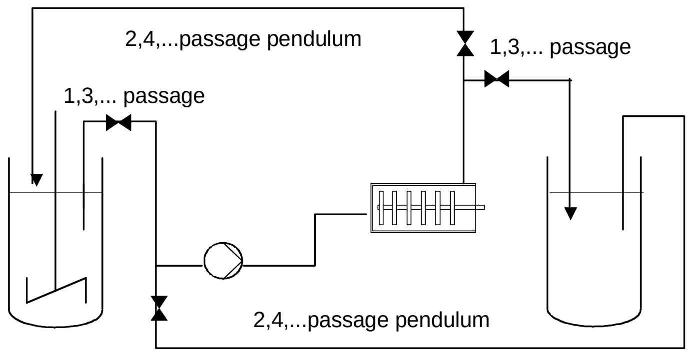

Fig. 3.2

Different possibilities to run a test

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4. Circuit operation (with stirred vessel)

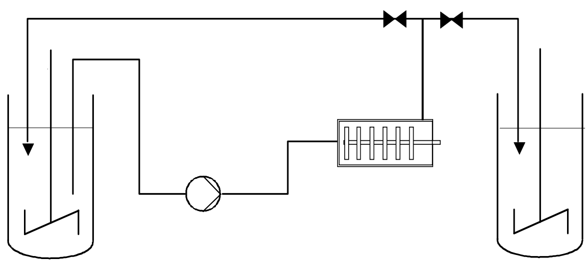

Fig. 3.2

Mean milling time for different operation modes

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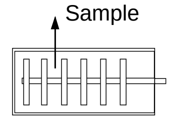
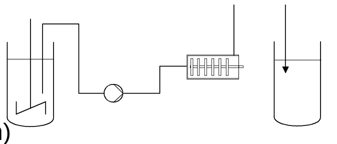
- Discontinuous operation (i.e. no flow through grinding chamber)

$$\bar{t} = t_{tot}$$



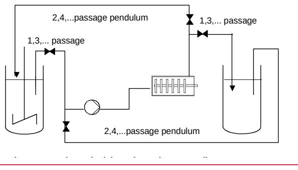
- Continuous operation (e.g. one passage mode)

$$\bar{t} \approx \frac{V_{GC} - V_{GM}}{\dot{V}_{Susp}} \qquad (3.3)$$



- Batch operation (circuit or pendulum operation)

$$\bar{t} = \frac{V_{GC} - V_{GM}}{V_{Susp,tot}} \cdot t_{tot} \qquad (3.6)$$



Specific energy for different operation modes

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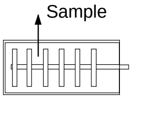
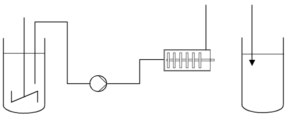
- Discontinuous Operation (i.e. no flow through grinding chamber)

$$E_m(t_C) = \frac{E(t_C)}{m_P} = \frac{\int_0^{t_c} (P(\tau) - P_0)\, d\tau}{m_P} \approx \frac{\bar{P} - P_0}{m_P} \cdot t_C \qquad (3.2)$$



- Continuous operation (e.g. one passage mode)

$$E_m = \frac{P_{stationary} - P_0}{\dot{m}_P} \qquad (3.4)$$



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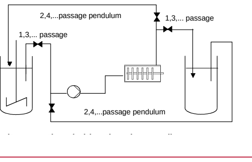
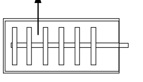


Example 3

Discontinuous operation

- Grinding time 20 min, Mass 1 kg, net power 2 kW
- Mean residence time: $\bar{t} = t_{tot} =$



- Specific energy:

$$E_m(t_C) = \frac{E(t_C)}{m_P} = \frac{\int_0^{t_c} (P(\tau) - P_0)\, d\tau}{m_P} \approx \frac{\bar{P} - P_0}{m_P} \cdot t_C =$$

Example 3

Continuous operation

- Volume flow rate of suspension 5 l/h,
  solids concentration by volume 20%, solids density 2 kg/l,
  mill volume 1 l,
  grinding media filling ratio 0.8, porosity of bulk grinding media 0.4
  net power at stationary operation 2 kW
- Mean residence time:

$$\bar{t} \approx \frac{V_{GC} - V_{GM}}{\dot{V}_{Susp}} =$$

- Specific energy:

$$E_m = \frac{P_{stationary} - P_0}{\dot{m}_P} =$$

Example 3

Batch operation (circuit or pendulum operation)

- Grinding time 10 h,
  solids concentration by volume 20%, solids density 2 kg/l,
  mill volume 1 l, suspension volume 50 l
  grinding media filling ratio 0.8, porosity of bulk grinding media 0.4
  net power 2 kW, Grinding time 10 h
- Mean residence time:

$$\bar{t} = \frac{V_{GC} - V_{GM}}{V_{Susp,tot}} \cdot t_{tot} =$$

- Specific energy:

$$E_m(t_C) = \frac{\int_0^{t_c} (P(\tau) - P_0)\, d\tau}{m_P} \approx \frac{\bar{P} - P_0}{m_P} \cdot t_C =$$

Question

- What diagrams would you plot after running the grinding or dispersing tests?

Product fineness as function of specific energy (linear scale)

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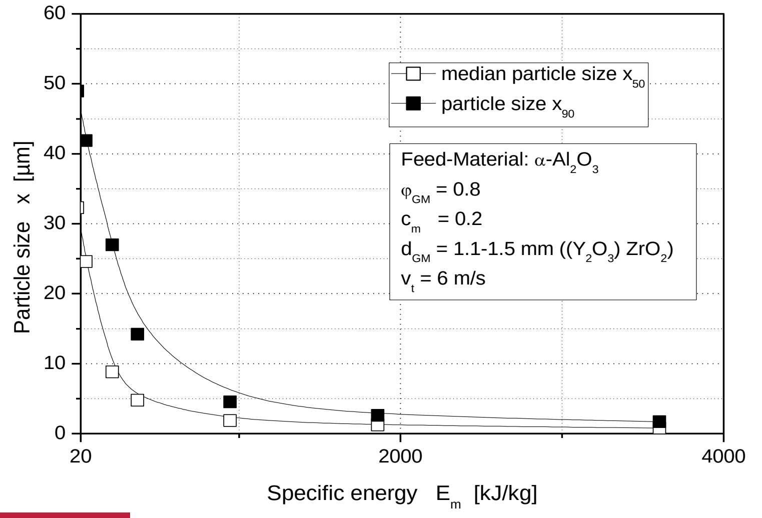

Fig. 3.3

Product fineness as function of specific energy (log-log scale)

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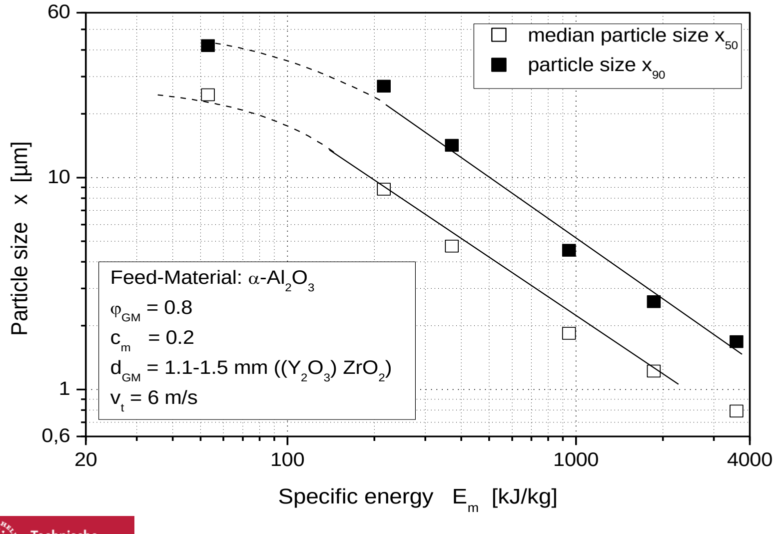

Fig. 3.4

Product fineness as function of grinding time (log-log scale)

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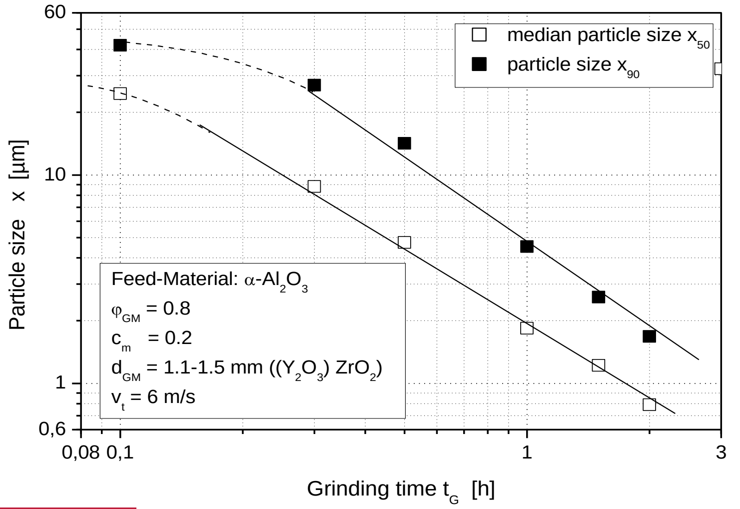

Fig. 3.5

Content

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3.1 Description of grinding and dispersing results

- •Product quality
- •Productions capacity
- •Results related to grinding time and specific energy

3.2 Stress mechanism and stress models

- •Mill related stress models
- •Product related stress models
- •Relation between model parameters

3.3 General application of the stress models

- •Particles between two grinding media
- •Estimation of stress events and frequency
- •Estimation of stress energy and intensity
- •Comparison of stress intensity and particle strength
- •Specific energy and efficiency factor

Resistance against fragmentation (dispersing and grinding)

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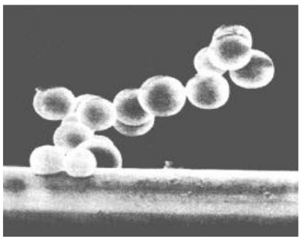
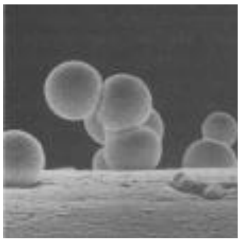
- Strength of particles (primary particles and particles collectives as for example aggregates) depends on
  - Particle structure
  - Binding and adhesion forces between nanoparticles
  - Size of the particle collectives and primary particles
    - Particle structure





K.Borho et al.: Produkteigenschaften und Verfahrenstechnik, Chem.-Ing.-Tech. 63 (1991), 8, pp. 792-808

Particle structures

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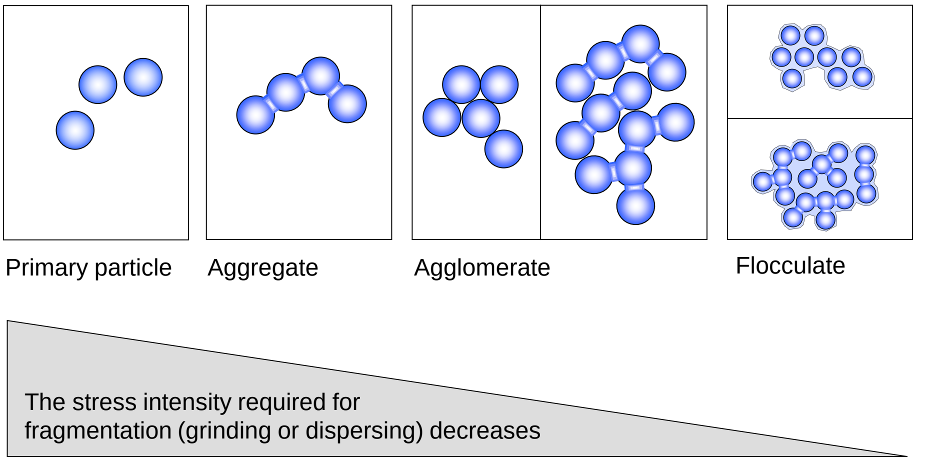

Resistance against fragmentation (dispersing and grinding)

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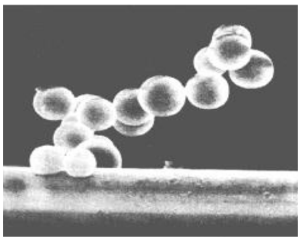

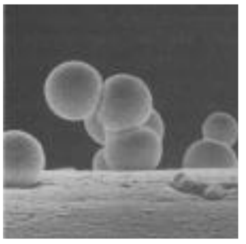

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Resistance against fragmentation (dispersing and grinding)

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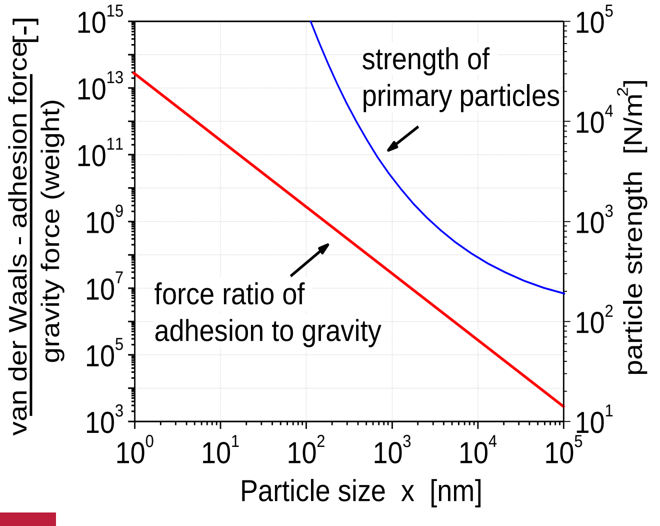
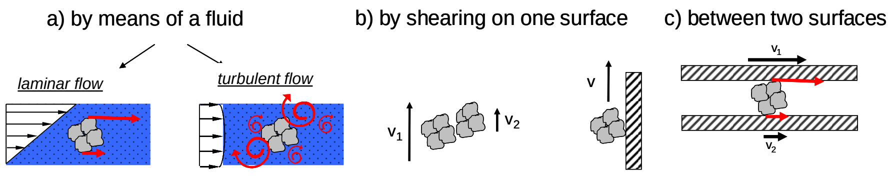


Fig. 3.6

Different stress mechanism

Shear stress



Compressive stress

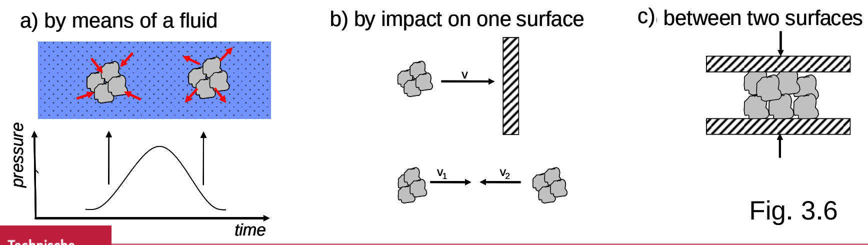


Fig. 3.6

Stress mechanisms in different dispersing machines

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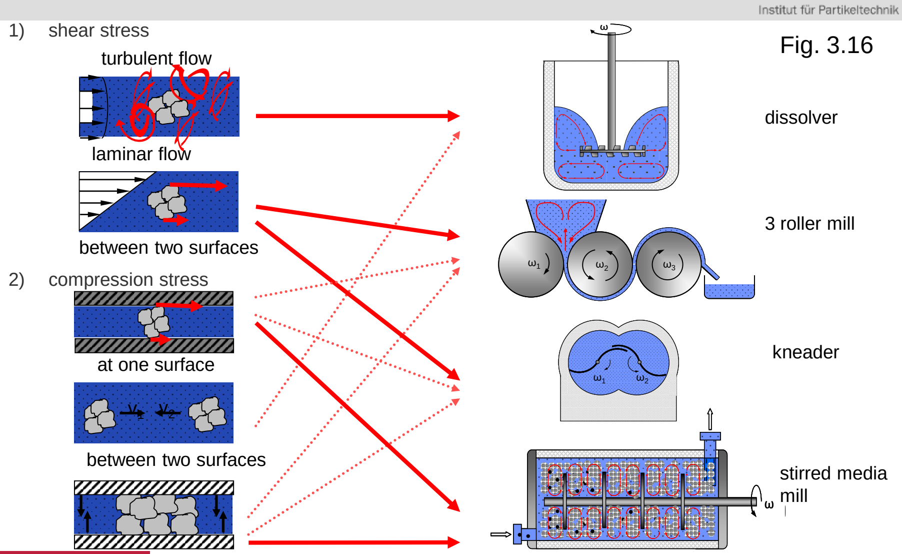

Fig. 3.16

Basic idea of stress models

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Practical example

- Smashing a stone with a hammer into pieces
- Different possibilities to hit the stone
  - Small or large hammer
  - Low or high speed
  - One or more hits

Two different points of view

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- What is the hammer doing?

I. How often does the hammer strike (independent on number of stones)?
→ Frequency of strokes

II. How strong are the strokes?
→ Energy of the hammer

- Mill related model

- What happens with the stone?

I. How often are the stone and the resulting fragments hit?
→ Number of hits

II. What are the intensities of the hits?
→ Specific energy supply

- Product related model

Mill related stress model

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- The grinding behaviour of a mill is determined by
  - the **type of stress** (e.g. impact or compression and shear)
  - frequency of strokes or stress events
    ➔ **stress frequency, SF_M**
  - the energy made available at each stress event
    ➔ **stress energy, SE**
- Total number of stress events: $\mathbf{SN_M = SF_M \cdot t_c}$ (3.13)

  where t_c is comminution time for a certain product quality
- Stress energy is not constant for all stress events
  ➔ Frequency distribution of the stress energy

Density function of stress energy

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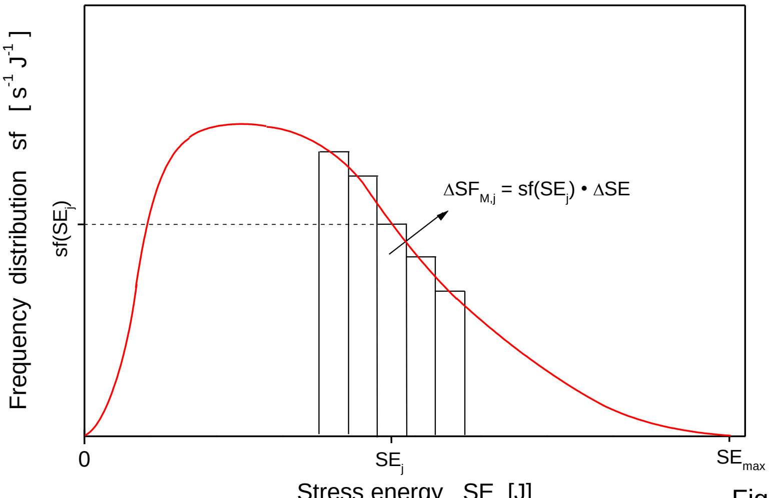

Fig. 3.7

Product related stress model

- The product quality and fineness achieved in a grinding or dispersion process is determined by
  - how the feed particles and the resulting fragments are stressed
    ➔ **type of stress** (e.g. impact or compression and shear)
  - how often each feed particle and the resulting fragments are stressed
    ➔ **stress number per feed particle, SN_F**
  - how high the specific energy or specific force at each stress event is
    ➔ **stress intensity, SI**
- Number of stress events and stress intensities are not constant for all particles and can only be characterized by distributions
- The stress intensity determines, how effective the specific energy transferred to the product is transposed into product quality and product fineness.

Effect of stress intensity on product quality for one stress event

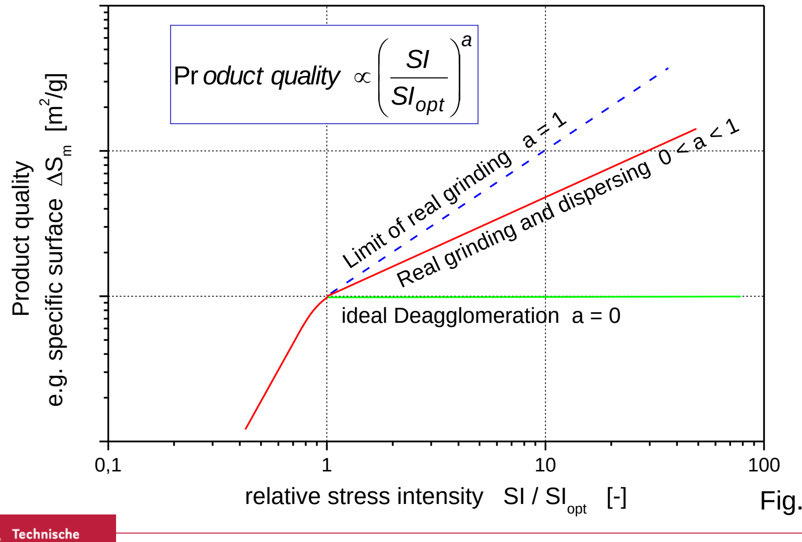

Fig. 3.8

Effect of stress energy on energy utilization

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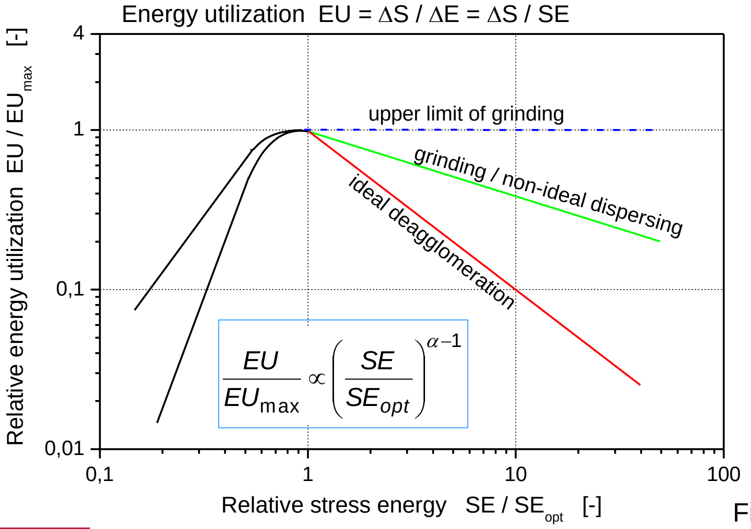
Energy utilization EU = ΔS / ΔE = ΔS / SE



$$\frac{EU}{EU_{max}} \propto \left(\frac{SE}{SE_{opt}}\right)^{\alpha-1}$$

Fig. 3.9

Macroprocess - Relation of stress energy and stress number with specific energy

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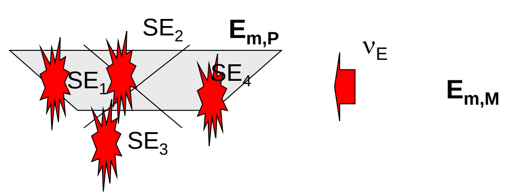

$$E_{m,P} = \frac{\sum_{i=1}^{SN_t} SE_i}{m_{P,tot}} = \frac{SN_t \cdot \overline{SE}}{m_{P,tot}} = \nu_E \cdot E_{m,M} \qquad (3.16)$$

where $E_{m,M}$:= Specific energy input into grinding chamber

$E_{m,P}$:= Specific energy transferred to product particles

ν_E := Energy transfer factor

Relation between model parameters and specific energy consumption

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Grinding and dispersing result is constant, if

- Specific energy $\mathbf{E_{m,P}} = \nu_E \cdot E_{m,M} \propto SN_M \cdot SE$

and

- Stress energy **SE**

are constant.

Power density

$$\frac{P_M}{V_{GC}} = \frac{P_P}{\nu_E \cdot V_{GC}} = \frac{SF_M \cdot \overline{SE}}{\nu_E \cdot V_{GC}} \qquad (3.18)$$

where

P_P	[J/s]	:= power available for the product
ν_E	[-]	:= energy transfer factor
P_M	[J/s]	:= power consumption of the mill

Content

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Number of stress events

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Number of stress events of each feed particle and the resulting fragments, SN:

$$SN = \frac{N_C \cdot P_S}{N_P} \qquad (3.23)$$

where N_c := Number of grinding media contacts

P_S := Probability, that a particle is caught and sufficiently stressed

N_P := Number of feed particles in the process

Probability, that a particle is caught and sufficiently stressed:

Desagglomeration/ Desintegration: $P_S \propto d_{GM}{}^2$ (3.25)

Real grinding: $P_S \propto d_{GM}$ (3.26)

Stress number

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Deagglomeration/Disintegration:

$$SN \propto \frac{\varphi_{GM} \cdot (1-\varepsilon)}{(1-\varphi_{GM} \cdot (1-\varepsilon)) \cdot c_v} \cdot \frac{n \cdot t}{d_{GM}} \qquad (3.28)$$

Real grinding:

$$SN \propto \frac{\varphi_{GM} \cdot (1-\varepsilon)}{(1-\varphi_{GM} \cdot (1-\varepsilon)) \cdot c_v} \cdot \frac{n \cdot t}{d_{GM}^2} \qquad (3.29)$$

where n $[s^{-1}]$:= rotational stirrer speed

t [s] := grinding or dispersing time

φ_{GM} [-] := filling ratio of the grinding media

ε [-] := porosity of the bulk grinding media

c_V [-] := solids concentration by volume

Energy and intensity of stress events

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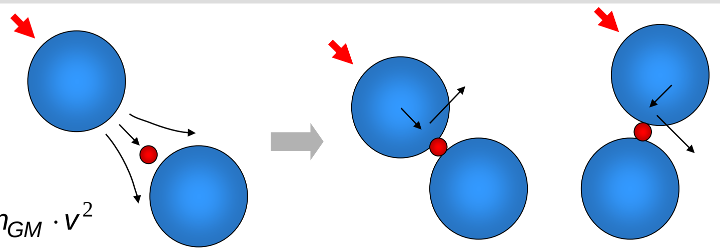

$$E_{kin} = \frac{1}{2} \cdot m_{GM} \cdot v^2$$

$$E_{kin} = \frac{1}{2} \cdot m_{GM} \cdot v^2 = \frac{1}{2} \cdot \frac{\pi}{6} \cdot d_{GM}^3 \cdot \rho_{GM} \cdot v^2$$

Kinetic energy is consumed for

- Displacement of the suspension between two grinding media
- Deformation of the grinding media during stressing
- Deformation and stressing of the product particles

Stress energy of the grinding media

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- Conditions
  - Only one particle is stressed intensively
  - The tangential velocity of the grinding media is proportional to the stirrer tip speed
  - The mill geometry and size is constant
  - The displacement of the suspension does not result in a decrease of grinding media velocity
  - The elasticity of the product particles is much higher than the elasticity of the grinding media
- Definition of the **stress energy of the grinding media**

$$SE \propto SE_{GM} = d_{GM}^3 \cdot \rho_{GM} \cdot v_t^2 \qquad (3.30)$$

- SE_{GM} is a **measure** for the real maximum stress energy in the grinding chamber

Example 4

- Calculation of stress energy of grinding media

a) Grinding media size 1 mm, grinding media density 6000 kg/m³, Stirrer tip speed 10 m/s

$$SE_{GM} = d_{GM}^3 \cdot \rho_{GM} \cdot v_t^2 =$$

b) Grinding media size 2 mm, grinding media density 6000 kg/m³, Stirrer tip speed 10 m/s

$$SE_{GM} = d_{GM}^3 \cdot \rho_{GM} \cdot v_t^2 =$$

Stress energy of products with low elasticity

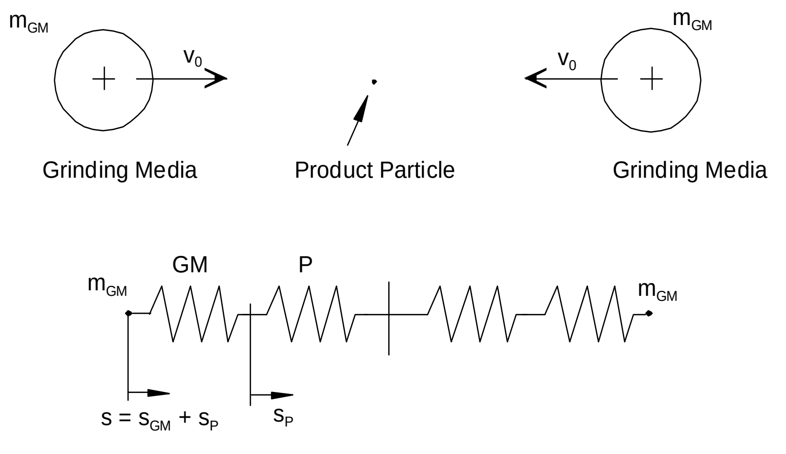

Fig. 3.13

Stress energy of products with low elasticity

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Measure for the stress energy transferred to the product:

$$E_{V,P,max} \propto SE_P = d_{GM}^3 \cdot \rho_{GM} \cdot v_t^2 \left(1+\frac{El_P}{El_{GM}}\right)^{-1} \qquad (3.32)$$

Part of the energy, which is transferred to the product particles:

$$E_{P,rel} = \frac{E_{V,P,max}}{E_{V,P,max}+E_{V,GM,max}} = \frac{El_{GM}}{El_P + El_{GM}} = \left(1+\frac{El_P}{El_{GM}}\right)^{-1} \qquad (3.34)$$

Effect of the Young modulus of grinding media on the part of energy transferred to the product

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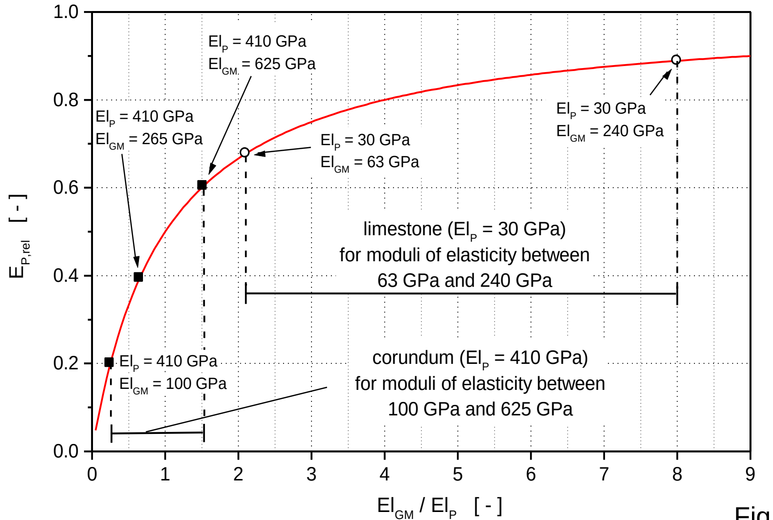
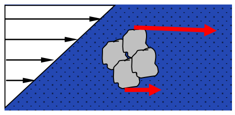


Fig. 3.14

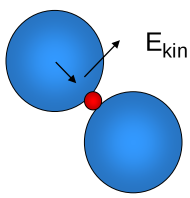
Stress intensity at two important stress mechanisms

- Shear stress in a fluid (laminar flow)



stress intensity $\propto \tau \propto c \cdot \eta \neq f(x)$

- Compression stress between two surfaces (grinding media)



$$E_{kin} = \frac{1}{2} \cdot m \cdot v^2 \propto d_{GM}^3 \cdot v_t^2 \cdot \rho_{GM}$$

stress intensity $\propto \frac{E_{kin}}{m} \propto \frac{E_{kin}}{x^3}$

Comparison of particle strength with stress intensity as function of particle size

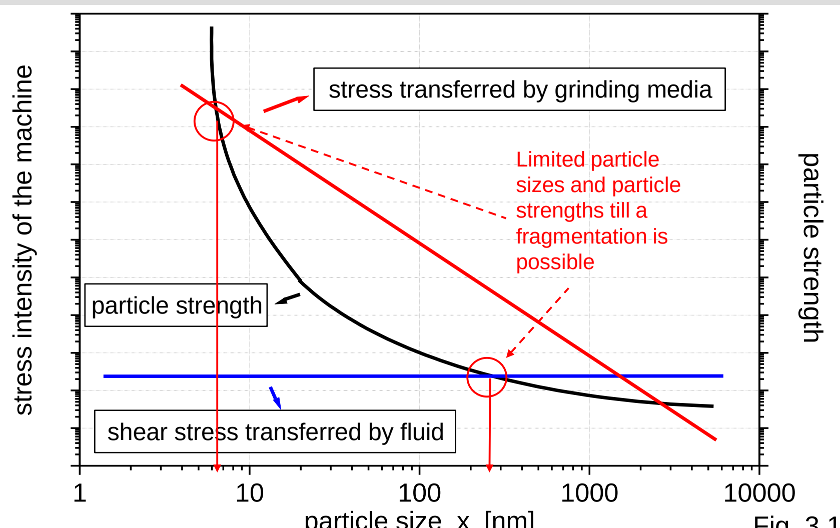


Fig. 3.15