

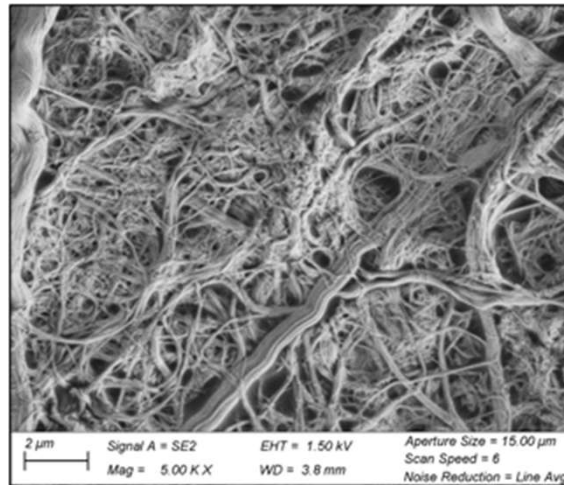
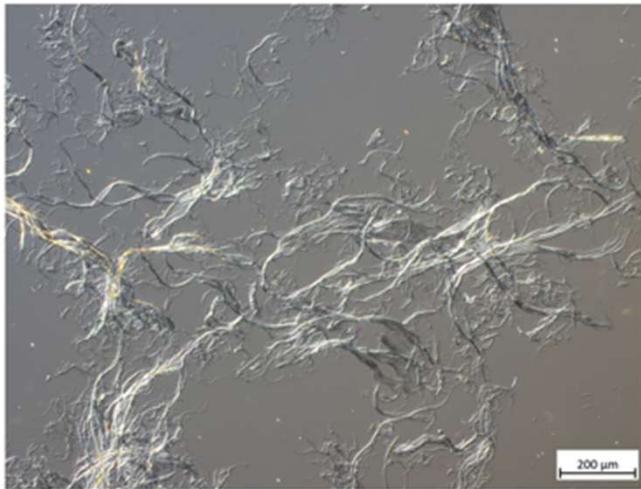


Industrial-Scale Production of Microfibrillated Cellulose Using Stirred Media Mills

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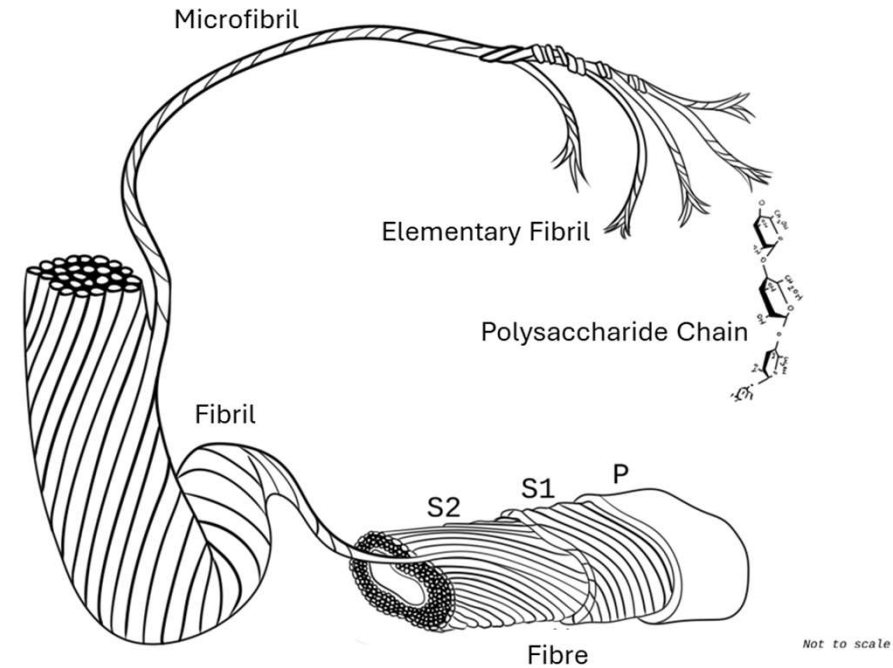
Introduction

- FiberLean sell **Stirred Media Mill Grinders** for the production of microfibrillated cellulose.
- **Entirely mechanical process**, no chemical or enzymatic pre-treatment.
- **Flexible operating conditions** enable tailoring product characteristics and optimising for a wide variety of fibre feedstocks.

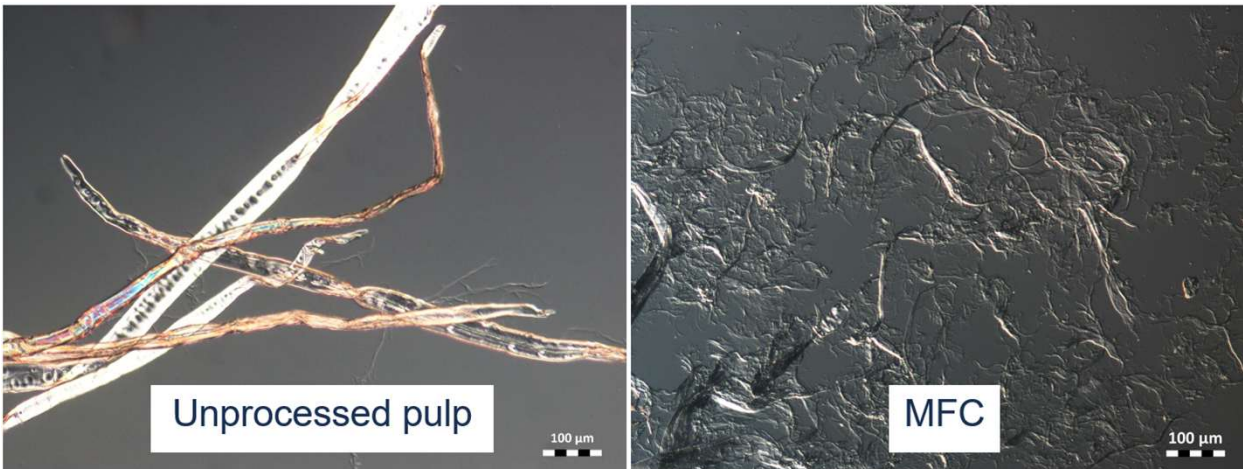


What is MFC?

- Micro-fibrillated Cellulose (MFC) is made from cellulosic feedstocks such as wood pulp, which are ***mechanically disintegrated*** to liberate their ***fibril and microfibril sub-structures***.
- This greatly increases specific surface area and fibril aspect ratio. ***Enhances bonding ability***.



- When added to the paper-making process: improved mechanical and barrier properties, reduced porosity, smoother surfaces.



MFC Applications

The strengthening and viscosifying properties of grinder MFC have shown benefits in applications such as:

- **Graphic paper:** filler increase / softwood reduction.
- **Folding box board:** chemical pulp reduction in outer layers.
- **White top liner:**
 - fibre reduction, improved optical properties from formation improvement and filler increase.
 - MFC wet-end coating to upgrade brown boxboard to WTL with minimal capex.
- **3D moulded objects:** fibre reduction by light-weighting, improved formation, reduced porosity.
- **Tissue:** fibre reduction by light-weighting.
- **Barriers:** MFC can form a layer which greatly improves oil and grease resistance and oxygen barrier properties for food packaging. MFC can also form a suitably smooth surface for water barrier application.
- **Specialty papers:** various (e.g. low porosity improves coating holdout; significant increases in wet web strength enables low GSM papers on machines configured for much higher GSM).

Pilot Plant Production Facility, and Product Form

Suspension (< 2% fibre solids)



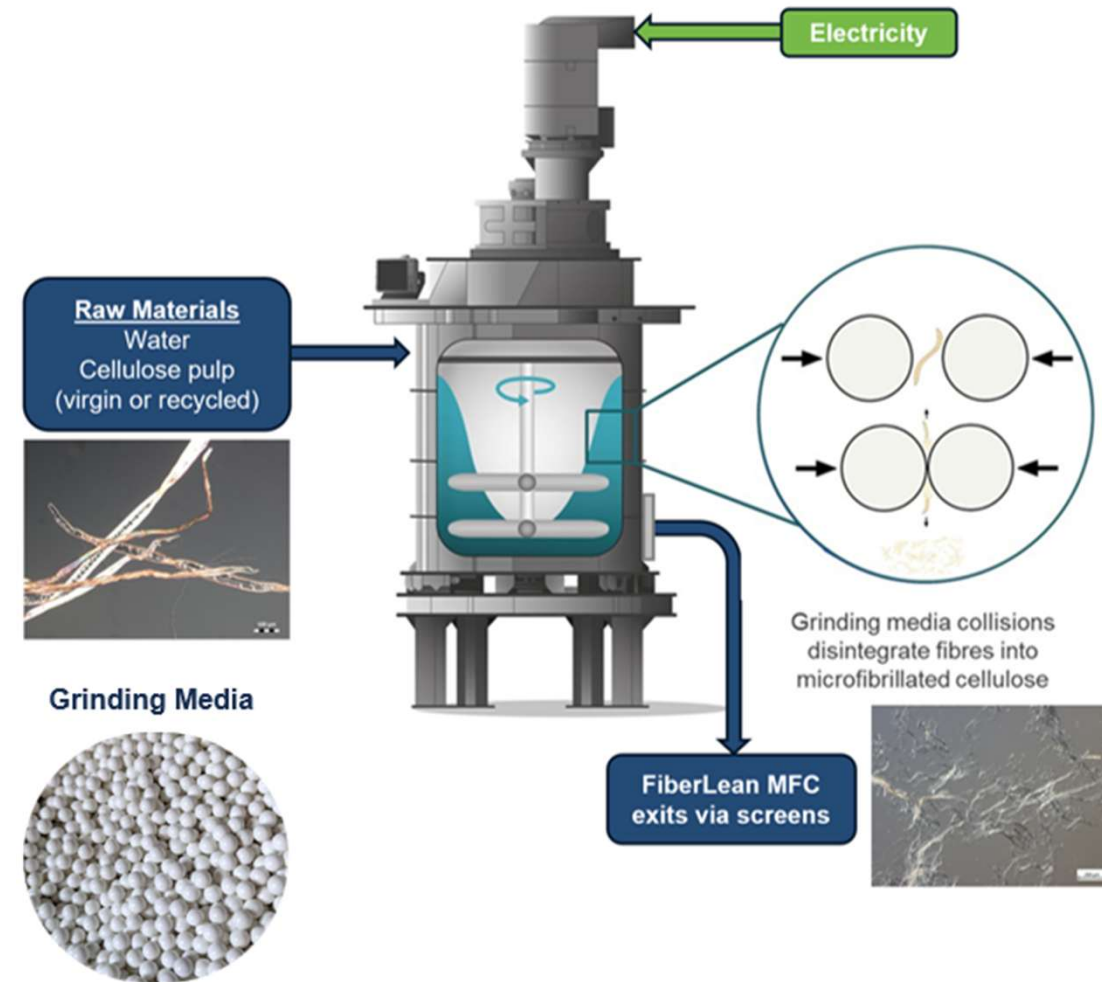
Dewatered Cake (10 – 20% fibre solids)



Production plant in the UK
**2000 dry metric tonnes / annum
of MFC capacity.**

Operational since 2013

Stirred Media Mill Grinder Working Principles



Stirred vessel, charged with water, pulp and **ceramic grinding media**.

Widely used in minerals and mining industry, adapted by FiberLean for MFC production.

Mechanical method, no chemical / enzyme pretreatment needed.

Fibrillation occurs when fibres gets trapped between colliding grinding media particles.

Grinding media are the 'working surfaces' for fibrillation; very high active surface area; permits high throughput and efficient production of MFC.

Continuous, single pass operation; energy input controlled by flow rate and motor power.

Established Products



G250

Capacity ~ 1200 dry metric tonnes of MFC per grinder per year. Appropriate size for most paper and board applications.

New Products



G175

Capacity ~ 400 dry metric tonnes of MFC per grinder per year. Small paper mills, tissue mills.



G125

Capacity ~ 100 dry metric tonnes of MFC per grinder per year. Speciality applications, moulded fibre etc.

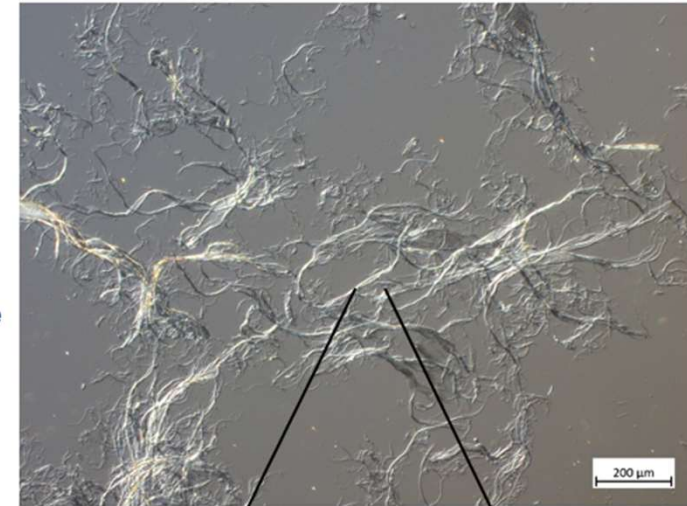
Example of Industrial Grinder Installed at Paper Mill



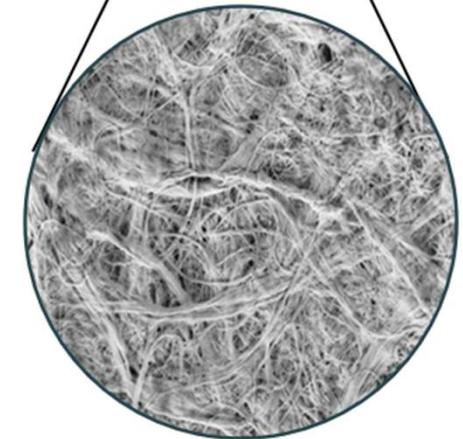
Morphology of Grinder Produced MFC

- Grinder-produced MFC is best described as '**surface-nanostructured macromaterial**'.
- Micrometre and nanometre scale fibrillar surface structure on a sub-millimetre scale macrostructure.
- **Fine** and long **micro and nano fibrils** in the **nanostructure** enhance **bonding** at fibre-fibre joints and with filler particles.
- A **coarse macrostructure** improves **bridging** between fibres and **improves MFC retention**.
- The objective is usually to efficiently generate a **high level of microfibrillation whilst retaining coarse macrostructure**.

Optical micrograph showing MFC coarse macrostructure



SEM showing MFC fibrillar micro and nano structure



Effect of Morphology: Bonding vs. Bridging

Bonding: strength of bonding at particular fibre-fibre joints. Related to **MFC nanostructure**.

Bridging: ability of MFC particles to overlap multiple fibres in a sheet. Related to **MFC macrostructure**.

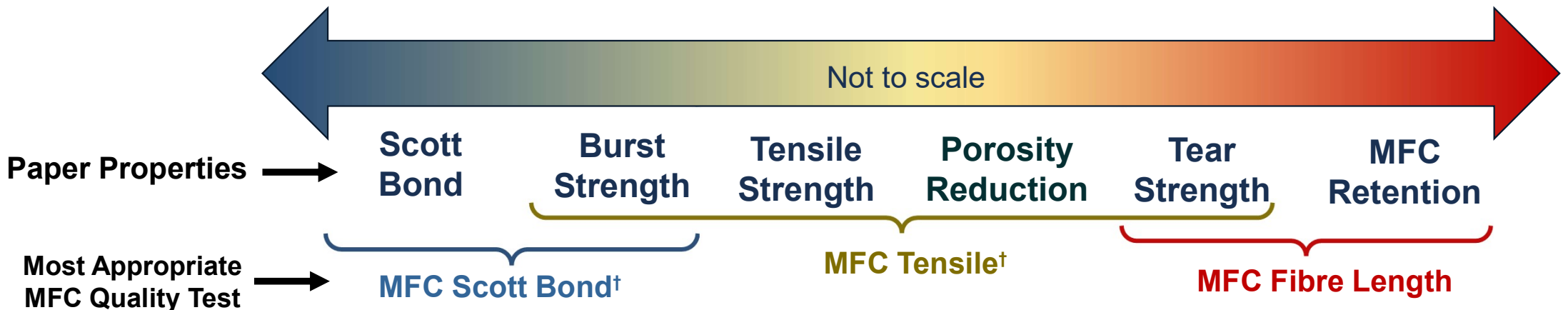
Different paper properties have different dependencies depending on the nature of the failure mechanisms. Improvements in bonding and bridging are **not necessarily mutually exclusive**. One can optimise for both by having a **high degree of fibrillation and a coarse macrostructure**.

Improved by Bonding

MFC with **high fibrillation surface area** desirable

Improved by Bridging

MFC with **Coarse macrostructure** desirable



[†]Requires dilution of MFC with something that weakens bonding e.g. mineral, to be meaningful

Grinder Optimisation Parameters

1. Machine Parameters

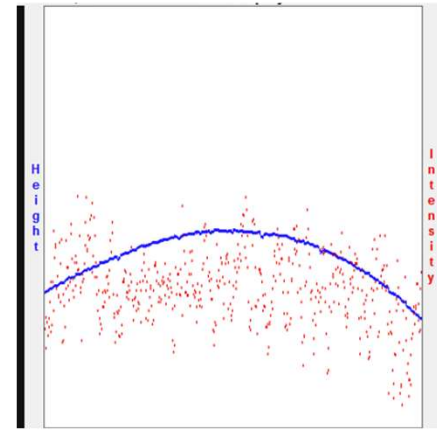
- Specific Energy Input ($\propto 1 / \text{flow rate}$)
- Impeller Speed
- Media Fill Fraction ('media fluidity')
- Impeller / Grinder Geometry

2. Grinding Media Parameters

- Density
- Elasticity
- Size
- Shape
- Surface Roughness

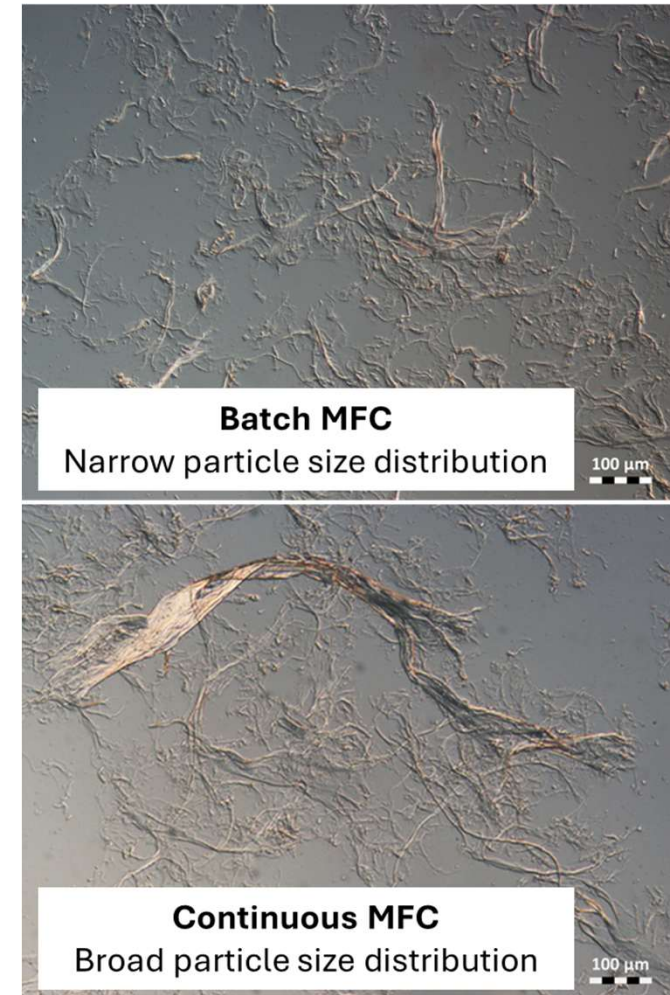
3. Feedstock Parameters

- Fibre Dimensions
- Fibre Strength
- Fibre Chemistry (Hemicellulose / Lignin)
- Solids Content



Batch vs. Continuous Operation

- Grinders can operate either in batch or continuous mode.
- **Batch mode:** Feed added, outlet valve closed, grinder run for a given time until target energy input is achieved. Product washed out afterwards.
- **Continuous mode:** Feed added and product extracted continuously, specific energy input controlled by flow rate.
- **Continuous mode gives much higher throughput** as it doesn't require the batch change time, but has a ***broad residence time distribution***.
- Consequently, continuous mode contains over- and under-processed material.
 - Not a problem for most papermaking applications, but size-sensitive applications can avoid the coarse under-processed fibre fraction by using batch mode.



Controlling Specific Energy Input

$$\text{Specific Energy Input} \left(\frac{kWh}{t} \right) = \frac{\text{Motor Power (kW)}}{\text{MFC Flow Rate} \left(\frac{t}{h} \right)}$$

- Motor **power** typically **kept constant** to maximise throughput (e.g. 300 kW).
- In continuous operation **specific energy input** is **controlled by MFC flow rate**.
- **High flow rate**: low specific energy input, **lower quality MFC**, but **high capacity**.
- **Low flow rate**: high specific energy input, **higher quality MFC**, but **low capacity**.

Example 1: Low Energy MFC

Control valve permits high flow rate

MFC residence time very low

Very low specific energy input

Lower product quality

Very high capacity

Feed flow rate controlled to match outlet flow rate

Example 2: High Energy MFC

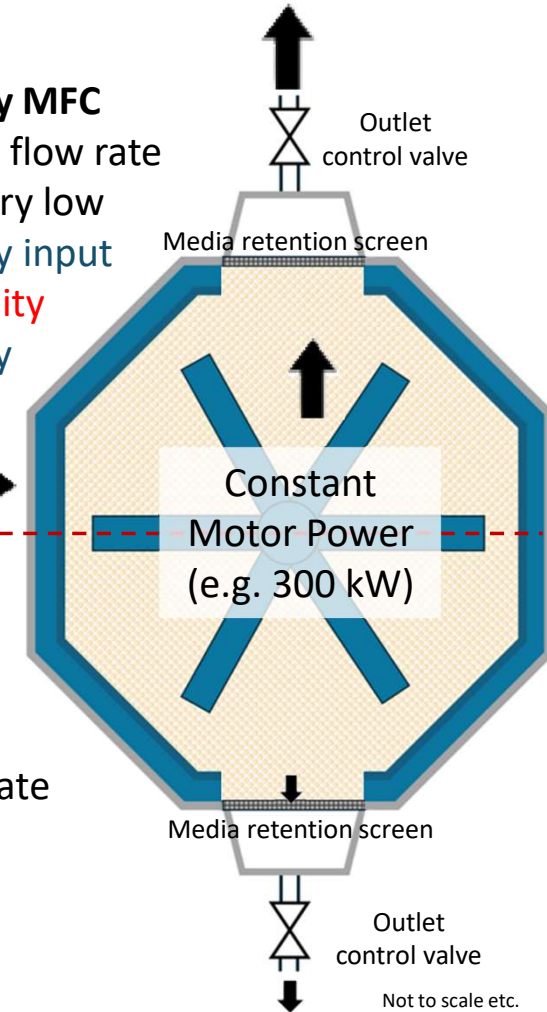
Control valve heavily restricts flow rate

MFC residence time increases

High specific energy input

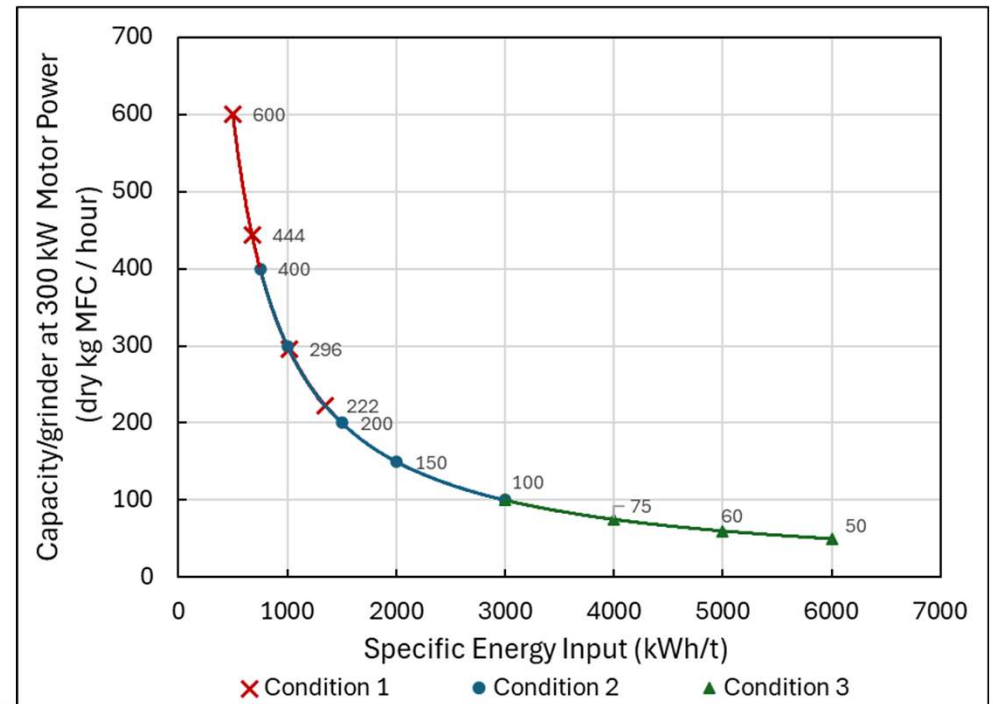
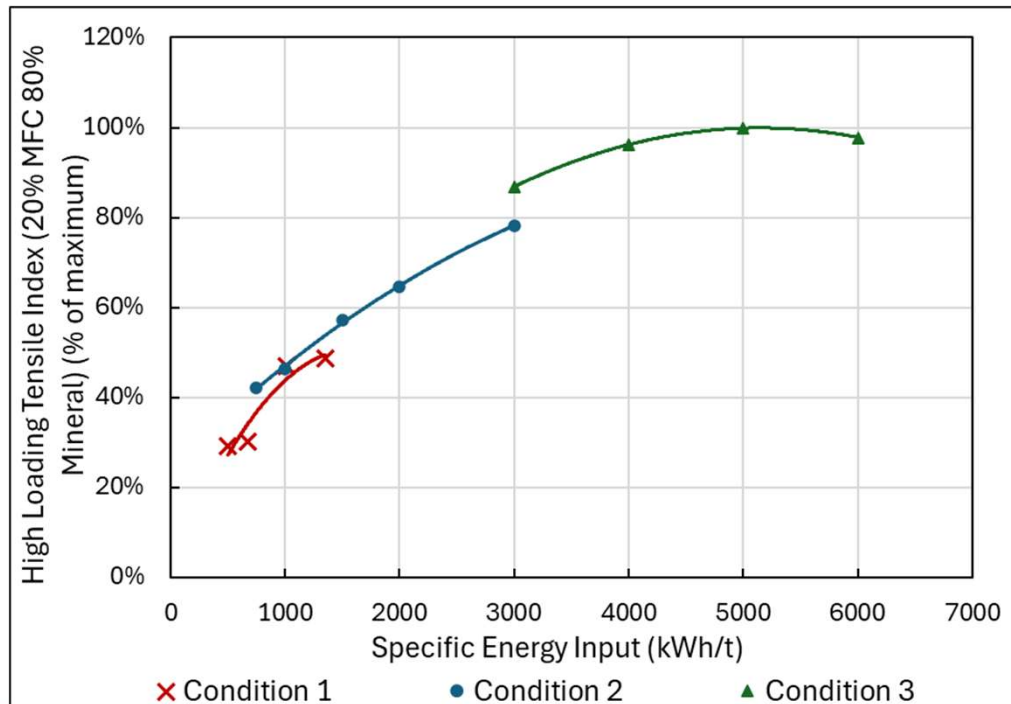
Higher product quality

Low capacity



Specific Energy Input vs. Quality and Capacity

- Trade-off between MFC quality and capacity.
- Best combination depends on papermaking circumstances and economics.
- Optimum process conditions also change with energy input.



What is the Optimum Energy Input?

- **High dose of Low Energy MFC:** < 1500 kWh/t (e.g. 8% dose at 500 kWh/t)
 - ✓ Efficient at improving many paper strength properties.
 - ✗ More severe effect on dewatering.
 - ✗ At low enough energy, so much of the furnish needs to be processed into MFC to achieve the strength improvement that dimensional stability / bulk problems can occur.
 - ✗ Higher volumes require more ancillary capex.
- **Medium dose of Medium Energy MFC:** 1500 – 3000 kWh/t (e.g. 2% dose at 2000 kWh/t)
 - ✓ Efficient at improving paper strength properties.
 - ✓ Dewatering / dimensional stability issues are minor.
 - Typical optimum range for most papermaking applications.
- **Low dose of High Energy MFC** > 3000 kWh/t (e.g. 1% dose at 4000 kWh/t)
 - ✗ Diminishing property improvement efficiency at increasing energy.
 - ✓ Optimum for some *barrier applications*.
 - Optimum for merchant MFC supply (due to shipping cost).

Two-Step Fibrillation Model

- Microfibrillation using a grinder can be considered analogous to **two chemical reactions in series**:

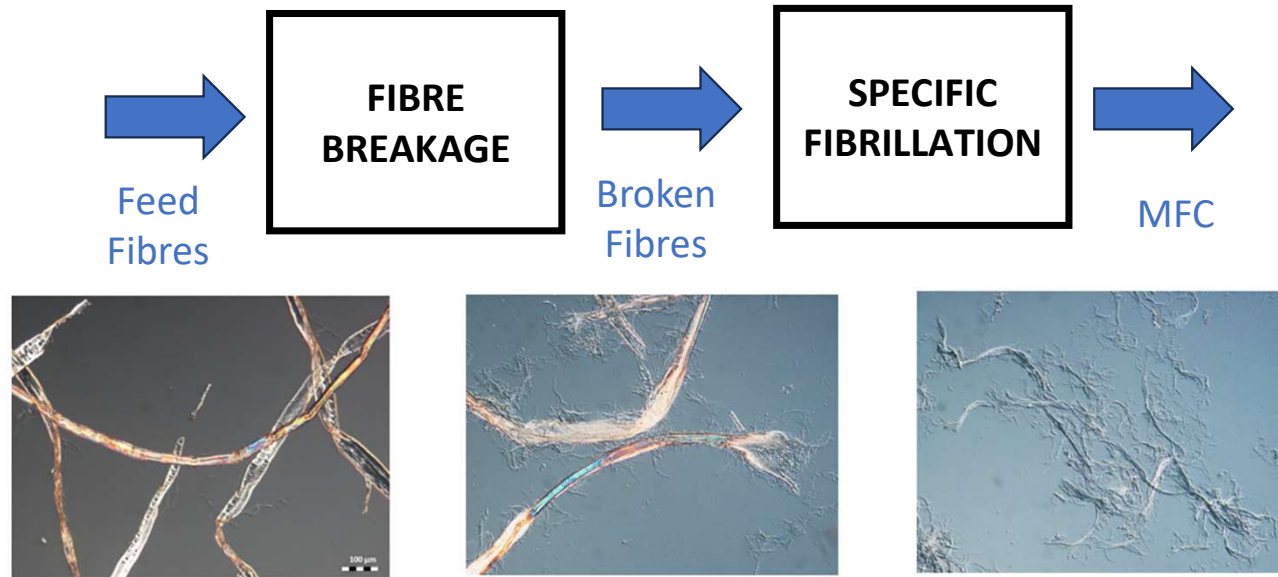
Step 1: Fibre Breakage

Grinding media collisions compress and cut fibre cross-sections.

Step 2: Specific Fibrillation

Further collisions and shear peel apart the fibrils at the breakage sites.

- **Fibre breakage requires more energy than specific fibrillation.**
- **Changing grinder operating parameters tends to affect both these steps differently.** For example, increasing impeller speed could improve fibre breakage at the expense of specific fibrillation.
- **Total fibrillation can be limited by either step.**



Process Optimisation: Stress Intensity Theory

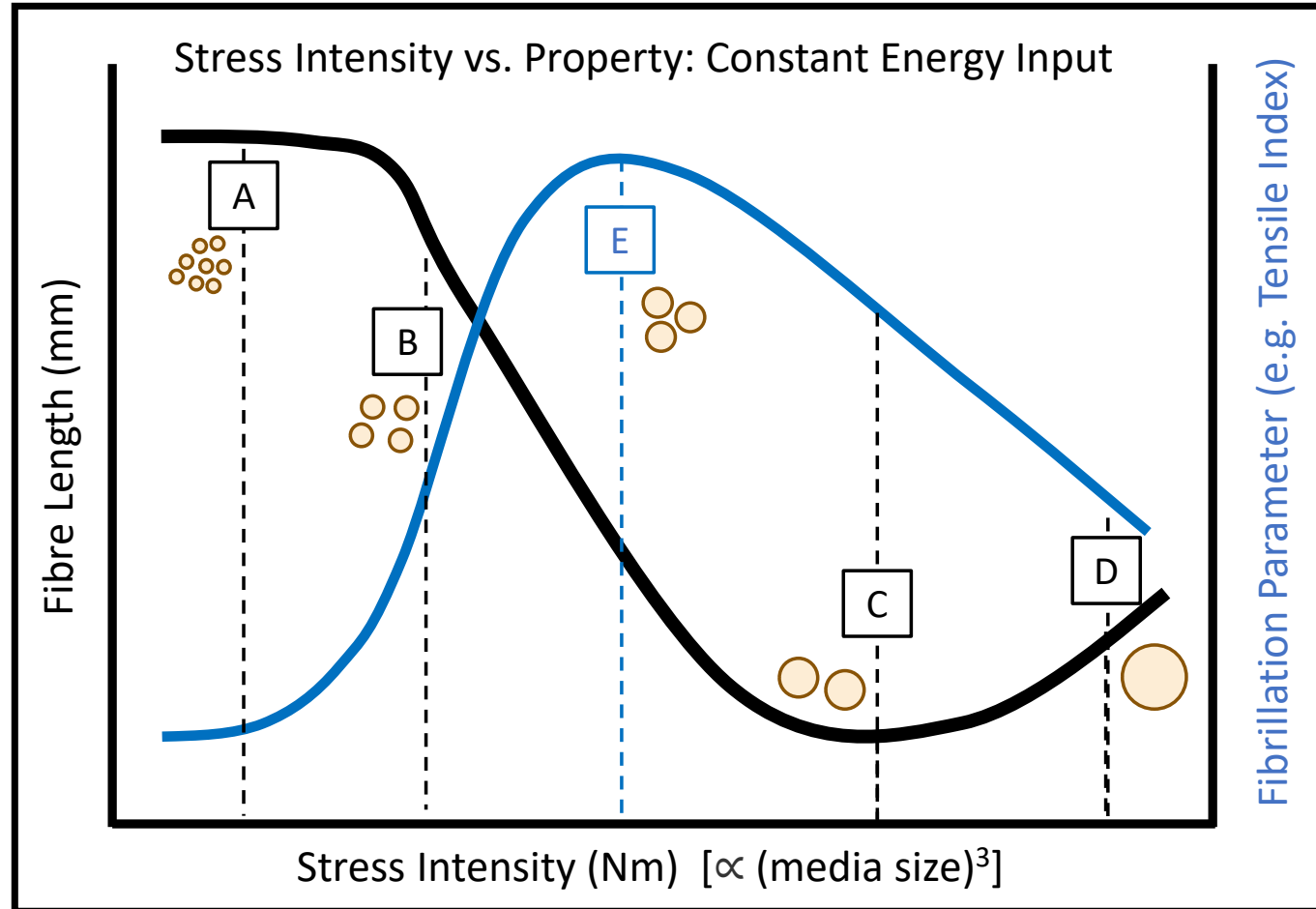
- ‘Stress intensity theory’ as formulated for stirred media milling of mineral particles^[1] can be modified to apply to cellulose grinding^[2]

$$E \propto SN \cdot SI$$

- E = Specific Energy Input
- SN = Number of collisions
- SI = ‘Stress Intensity’, \propto to average kinetic energy of a collision.

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

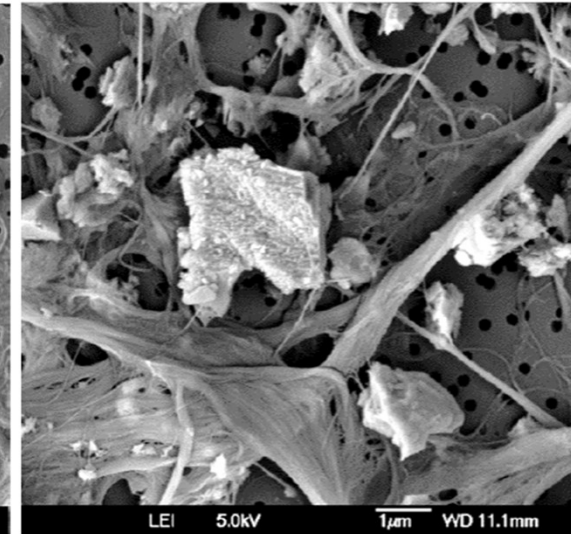
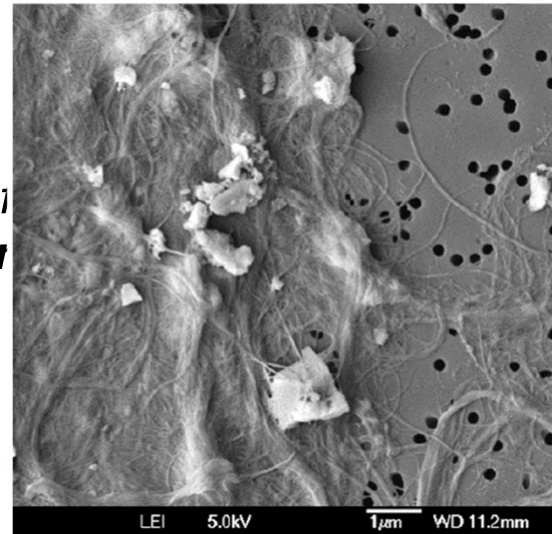
- ρ_{GM} = Grinding media density
- v_t = Impeller tip speed
- d_{GM} = Grinding media diameter



Relevant Fibre Properties

- **Intrinsic Fibre Strength (zero-span tensile index):**
Low zero-span tensile strength fibres have many pre-existing flaws in the cross section; ***a high value makes the fibre breakage step harder.***
- **Fibre Geometry:** thick-walled, high cross-sectional area fibres (i.e. softwoods) are harder to break in a single collision.
- **Hemicellulose Content:** Amorphous polysaccharide that separates cellulose microfibrils and provides a *preferred plane of breakage*. ***Makes the fibrillation step easier.***
- All three properties influence the relative difficulty of fibre breakage and fibrillation, and so ***change optimum media size and other grinding conditions.***

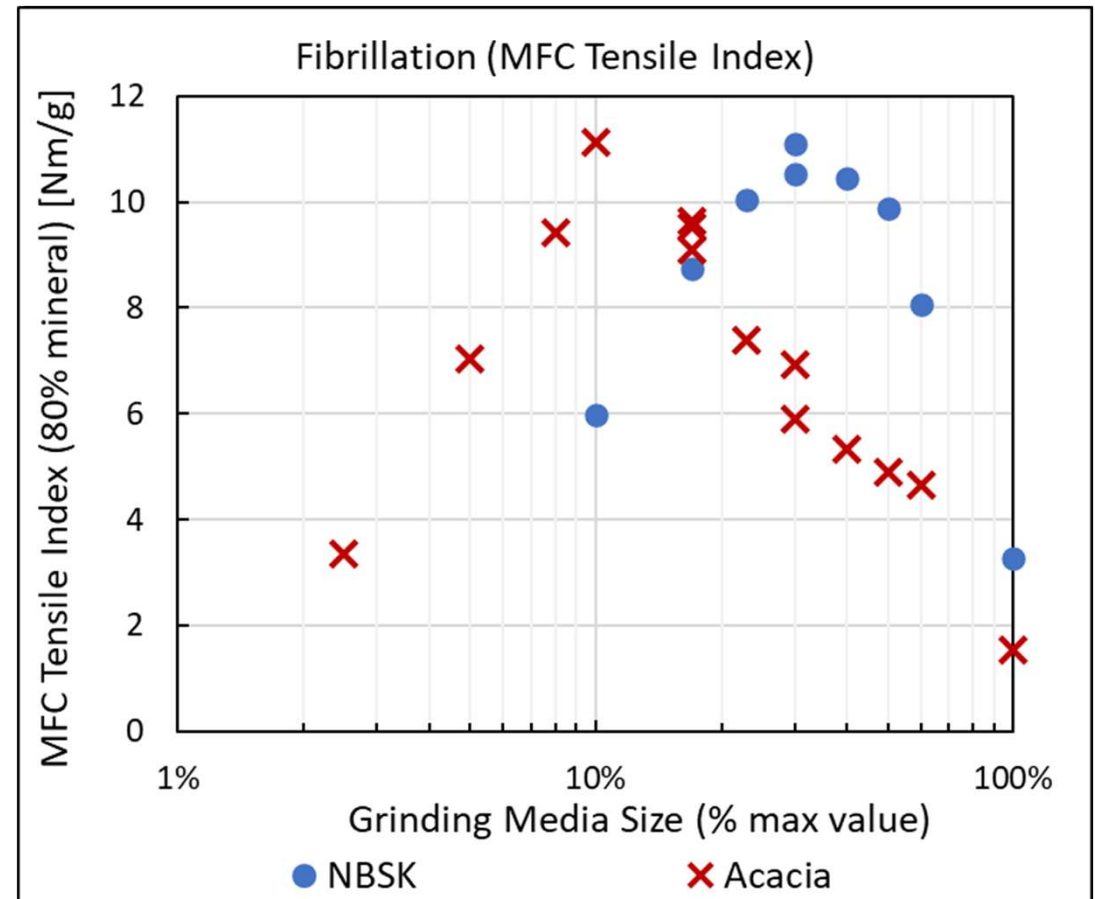
MFC – mineral composite produced from fibres with high hemicellulose content (left) and low hemicellulose content (right)



Influence of Fibre Properties on Optimum Conditions

- Comparing two fibre species at a fixed energy input:
 - **NBSK**: softwood, long fibres, thick cell wall, strong cross-sectional strength.
 - **Acacia**: hardwood, short fibres, thin cell wall, weak cross-sectional strength.
- Optimum media size for fibre breakage is significantly lower for Acacia than for NBSK.
- Optimum for fibrillation also lower for Acacia.
- **Good MFC can be made from weak fibres so long as appropriate conditions are selected.**

Fibre	Optimum media size for fibre breakage (% maximum)	Optimum media size for fibrillation (% maximum)
NBSK	50%	30%
Acacia	17%	10%



Conclusions

- **Stirred media mill grinders** are an efficient and high throughput method of **mechanically producing MFC** at industrial scale.
- Compared to alternative technologies, **grinders can generate high quality microfibrillation whilst maintaining a coarse macrostructure**, with particular benefit to certain paper properties that are improved by inter-particle bridging (e.g. MFC retention, porosity, tensile strength).
- Grinders typically operate in a **continuous** manner, but can operate in batch mode for size-sensitive applications.
- A grinder can produce a wide range of MFC qualities by varying the specific energy input (where there is a trade-off between quality and capacity).
- **Collision energies** within the grinder **can be varied by orders of magnitude** by changing numerous process parameters, e.g. grinding media size, impeller speed.
- This influences the balance of fibre breakage and fibrillation, and so can **be tuned to produce high quality MFC based upon feedstock properties**, or produce MFC of different morphologies depending on requirements.

Thank you for your attention

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