Mechanical pulping

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Defibration mechanisms and energy consumption in the grinding zone – a lab scale equipment and method to evaluate groundwood pulping tools

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Abstract: Groundwood pulping is a process that employs large machines, making them difficult to use in research. Lab scale grinders exist, but even though they are smaller, the sizes of the grinding stones or segments make them cumbersome to exchange and tailor. This study presents a method and an apparatus for investigating the detailed mechanisms and the energy requirements behind the fibre separation process. A well-defined grinding tool was used at three different temperatures to demonstrate that the equipment can differentiate levels of energy consumption and defibration rates, confirming the well-known fact that a higher temperature facilitates defibration. It is also shown how the equipment can be used to study the influence of grinding parameters, exemplified by the effect of temperature on the way fibres are separated and the character of the produced fibres. A key feature of the equipment is the use and evaluation of small grinding surfaces, more readily designed, produced, evaluated and studied. This reduces both the cost and time necessary for testing and evaluating. At the same time, a technique to produce well defined grinding surfaces was employed, which is necessary for repeatability and robust testing, not achievable with traditional grinding stones.

Keywords: computed tomography; energy efficiency; groundwood pulping; lab scale equipment; test method.

Introduction

Groundwood pulping employs large machines where logs are fed onto a large rotating grinding stone. During the process, water is continuously supplied to the grinding zone, both to help transporting liberated fibres and to keep temperature stable. The process itself generates heat that elevates the temperature up to, or in pressure groundwood pulping even above, 100 °C (Steenberg and Nordstrand 1962). This is advantageous as high temperatures soften the wood cell structure, and particularly the lignin holding the fibres together, reducing the energy required to liberate individual fibres. The liberation process itself builds on repeated mechanical contact with grinding asperities in the rotating stone and the mechanisms have been studied extensively (Atack 1971). Altering the grinding tool surface may change the contact situation and the fibre liberation process. So, when designing new grinding tools, it is important to study the mechanisms in detail, to understand how the different tools influence the mechanisms.

Groundwood pulping is a process where trials in industrial machines are difficult, and even inappropriate. Not only because of the sheer size of the parts makes it difficult, but also due to the inability to control the environment and process parameters, as well as adjusting the properties of the tool and the wood specimen. In studies aimed at increasing the understanding of the grinding processes, lab scale tests are thus attractive as these avoid the use of full-scale equipment and tools and allow better control of the test conditions. Research using small grinders have been performed previously, investigating e.g. the effects of temperature (Steenberg and Nordstrand 1962) and atmospheric pressure (Atack et al. 1981), modification of conventional grinding stones (Lucander et al. 1985, Björkqvist and Lucander 2001, Nurminen et al. 2018), grit morphology (Enström et al. 1990, Sandås and Lönnberg 1990, Sandås 1991a, 1991b, Lönnberg et al. 1996, Tuovinen et al. 2008, Tuovinen and Fardim 2015), fatigue pretreatment (Salmi et al. 2012a), differences between heartwood and sapwood (Bengs and Lönnberg 1994), glass transition (Blechs Schmidt et al. 1986) and wood alignment

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Figure 1: A) The pressure chamber with the miniature lathe inside, holding a cylindrical wood specimen, and the heated water container in the bottom. B) An illustration of the tool holder mechanism.

(Heinemann et al. 2016, Saharinen et al. 2016). Tailored equipment has also been used to investigate influences of specific mechanisms, such as fatigue (Uhmeier and Salmén 1996, Salmi et al. 2012b, Moilanen et al. 2017), fibre peeling (Tuovinen et al. 2009) and fracture energy (Koran 1981). Commonly, however, studies deal with large wood volumes rather than with volumes at the scale of individual fibres. This work presents a methodology where tools, designed at the fibre scale, are used in an equipment that allows defibration mechanisms to be studied at the fibre level.

In order to facilitate studies on defibration mechanisms and the performance of new grinding surfaces, while at the same time reduce the size of the tool to make it more efficient, a new lab scale equipment utilising tools with non-stochastic surface structures has been developed. It achieves a high degree of control of many process parameters and it allows easy exchange of tool and wood specimen. Further, during the process it is possible to measure the grinding force and by analysing the wood specimen after the test, the volume of wood that has been transformed into fibres can be measured. Altogether, this makes the test suitable to judge the process efficiency when different tools are working different wood specimens. As such, it has proven to be a very useful tool when trying to understand the defibration process itself and when trying to improve the efficiency of the process, either by changing the parameters or by changing the tool geometry.

Experimental

The test equipment

Like in the real application, grinding is conducted in water. However, as high temperatures are also desired, above

the boiling point of water at atmospheric pressure, the test needs to be done at elevated pressure. The central part of the developed test equipment nicely fits within a pressure chamber designed to handle pressures up to 7 bar, see Figure 1a. Such pressure would cause the boiling point of water to rise to approximately 170 °C at 100 % RH, which is well above the temperatures used in industrial processes. A typical temperature however, e. g. 110 degrees only requires a pressure of 2 bar.

The heat and moisture during a test is provided by a water bath in the bottom of the chamber, slowly heated with a computer controlled resistive heater that takes the chamber and the instrumentation inside to the test temperature in about an hour. The software controlling the equipment and monitoring the process was developed in National Instruments LabView.

Inside the chamber, a test setup resembling a miniature lathe is placed, with a shaft connecting it to a motor outside the chamber. This allows a piece of wood to be rotated in the chamber. The tool holder mechanism, shown schematically in Figure 1b, presses a tool via a spring force onto the wood specimen. The holder is outfitted with force sensors consisting of strain gauges glued to thin steel plates and covered with protective polymer coatings, allowing registration of both the normal and tangential forces affecting the tool. A thermocouple is placed inside the chamber to measure the temperature. All sensors are connected to a computer using National Instruments CompactDAQ modules, to monitor and save the data. The forces can be registered at up to 50 kHz and the temperature data at 14 Hz.

Tools

The well controlled grinding tool surfaces were produced using a process route, outlined in Figure 2, developed pre-

Figure 2: The route for producing the grinding surfaces. Starting from a silicon wafer (1), a patterned mask is applied (2). The wafer is then anisotropically etched (3) and the mask is removed (4). A polycrystalline diamond film is grown on top of the silicon (5) and thin metal films are subsequently deposited (6). The wafer with the coating stack is soldered upside down to a steel plate (7) and the silicon is removed (8).

Figure 3: A tool with parallel sharp ridges across the tool surface shown from (A) above where everything visible is diamond and (B) in cross section where the bottom part is the rigid steel plate (the thin solder joint is not distinguishable from the steel).

viously (Gåhlin et al. 1999). A mask with a pattern of the desired surface structure is placed on a silicon wafer and the stack is anisotropically etched in potassium hydroxide (KOH). After removing the mask, a negative mould of the desired grinding surface is achieved. On this, a film of polycrystalline diamond is deposited using hot filament chemical vapour deposition (HF-CVD). The diamond is subsequently coated with a thin adhesion layer of titanium and a thicker layer of nickel. The nickel allows the stack to be soldered to a steel plate, which gives mechanical support to the finalised tool. Lastly the silicon mould is removed by etching, revealing the grinding tool stack with asperities of the desired geometry. In Figure 3 an example of this kind of well-defined tool surface is shown. The whole tool is 5×5 mm, with ridges stretching across the tool width.

Wood specimen

Norway spruce (*Picea abies*) is the most commonly used species in Swedish pulping today. The samples used in this work came from an approximately 50 years old tree in the middle of Sweden. To preserve the wood, log pieces were stored in a freezer from right after cutting. From the log, 10 cm slices were cut, and the slices were split into quarters, Figure 4a. Each quarter piece was then turned into a cylinder and finally ground using a 180 grit silicon carbide paper to give a well-defined surface. This procedure assures the circumference of each cylindrical wood specimen exposes regions both perpendicular and tangential to the growth rings in the wood, see Figure 4b. In a pulp mill, grinding takes place irrespective of the growth rings and there is no way to study their influence on the defibration process.

Figure 4: A) A piece of the log, sectioned in quarters to get four samples similar in growth ring pattern, and a roughly cut quarter ready for turning into a cylinder. B) An illustration of how growth rings are oriented in each cylinder and the direction of rotation.

Analysis

Force measurements

During the test, the normal and tangential forces are registered. The tangential force typically shows a small cyclic variation in harmony with the rotation of the wood cylinder. Although the wood specimen has been shaped into a cylinder in a lathe before the test, the increased temperature deforms it slightly to make it non-circular. During grinding, the tool position is not fixed and the tool will follow the shape of the wood specimen. This means that the non-circular shape will essentially be maintained despite the grinding during a test.

However, also this cyclic variation could be filtered out and, in principle, the force measurements should allow a grinding energy to be deduced directly. But the data cannot be used straight away. Since the strain gauges are located inside the chamber the normal force is set to the desired value before heating commences and the tangential force is zeroed before contact is established. Thereafter, their values are affected by both the initial mechanical strain and by strains induced by the changing environment. Changes in temperature cause the threads in the strain gauges to expand or contract, but as the strain gauge configurations are full Wheatstone bridge circuits, only the zero point of the measurement is changed. Other reasons for changes as temperature (and moisture) is changed is the expansion of the wood itself or of the materials surrounding the strain gauges, such as coatings protecting the strain gauges and the connecting wires from the moist environment. To handle these effects, two corrections are made to the force measurement data. At the end of the heating stage, just before the grinding test is initiated, the normal and tangential forces are reset to the values they had before heating. The changes during the actual grinding, where the temperature is stable, is compensated for by assuming that the process, and therefore the forces,

reaches a steady state during the latter part of the test. This procedure was found to give more reliable force measurements than, the perhaps more direct way, logging the forces for a while prior to grinding and assuming that behaviour will prevail also during grinding. Thus, any linear slope in the tangential force data during the latter part of the test is considered to be due to changes in the materials surrounding the strain gauges and the slope is thus removed from the whole data set, see Figure 5. This procedure preserves initial changes in the forces occurring before a steady state is achieved.

In tests with a truncated wood cylinder, i. e. when one side of a cylinder had been cut flat, it was proven that when the tool disengaged from the wood and passed the flat portion of the cylinder the force values indeed showed a drift at a scale similar to the slope found in the grinding tests. This motivates the procedure described above. Moreover, repeated analysis of the tool and wood surfaces after several increasingly longer tests have shown that steady state develops after a short initial stage, during which a slightly higher tangential force is usually recorded.

Wood cylinder

After a test, a track has formed in the wood cylinder. The depth of the track varies along the circumference, due to differences in fibre removal rate caused by the growth ring pattern. To allow for comparisons, measurements need to be done at a specific location, e. g. the position where the growth rings are perpendicular to the surface. A small segment of the track, about 10 mm along the track, is cut out from the cylinder and dried in room atmosphere (20 °C and ~50 % RH). The track is then coated with a thin gold/palladium film and imaged using a Scanning Electron Microscope (Zeiss Leo 1550) to investigate the mechanisms of fibre separation.

Figure 5: A) Original tangential force data from a 120 seconds long test showing a periodic variation caused by the rotation of the wood cylinder and a slope due to thermal effects. B) Original data with a fitted trendline and corrected data.

Figure 6: A) Stacking of images to increase contrast. B) Resulting image with track area marked in white.

Computed tomography (CT) is then employed to give a quantitative assessment of the track cross sectional area and ultimately a track volume. The CT equipment used was a Bruker Skyscan 1172 μ CT, running at 53 kV source voltage and 188 μ A source current. X-ray images are acquired during sample rotation, 998 radiographs over an angle of 180 degrees is saved with a resolution of $1.97 \times 1.97 \mu\text{m}/\text{pixel}$. From the radiographs a 3D reconstruction of the sample is made using the Bruker software NRecon. 500 slices of the track are selected from the reconstruction and resized to have half the size of the original image, resulting in a resolution of $3.94 \times 3.94 \mu\text{m}/\text{pixel}$. To enhance the difference between wood and void, 10 adjacent images are combined into one by selecting the brightest pixel at each position, see Figure 6. This results in 50 images, in which the track cross section areas are selected manually using Adobe Photoshop and the pixels counted and translated to an area. An average of the areas from these 50 images thus represents the track area in a length of just under 2 mm of the track.

Grinding energy calculations

To estimate an energy consumption, the recorded tangential force data and the measured grinding track areas are used. The energy consumption is calculated as the work, W , performed by the tangential force per dry mass fibres.

The work is performed by the tangential force, F_T over the sliding distance during a test, s ,

$$W = F_T \times s \quad (1)$$

And this can be written as,

$$W = F_T \times \omega r t \quad (2)$$

Where ω is the angular velocity, r is the average radius of the wood piece, and t is the test time.

The measured grinding track areas, A , are used to calculate the volumes of removed fibres, V ,

$$V = A \times 2\pi r \quad (3)$$

Figure 7: A) Tangential forces from three tests at different temperatures. B) Measured cross section areas where the points represents a series of 50 measurements along a 2 mm section of the circumference. C) Calculated median energy spent, error bars show upper and lower quartiles.

And, by combining Equations (2) and (3), the volume specific energy consumed, E_V , can be calculated,

$$E_V = \frac{W}{V} = \frac{F_T \times \omega t}{A \times 2\pi r} \quad (4)$$

For the demonstration in this work, ω is 2π rad/s and t is 120 s, and Equation (4) can be rewritten as,

$$E_V = 120 \times \frac{F_T}{A} \quad (5)$$

To calculate the mass specific energy consumption, the wood density, ρ , is used,

$$E_m = \frac{E_V}{\rho} \quad (6)$$

For the calculations in the demonstration section, a wood density of 400 kg/m is assumed.

This allows for comparisons between tests with different tools or different parameters when testing in this lab scale equipment, but the energy consumption should not be used for direct quantitative comparisons with industrial data.

Demonstration

To illustrate the results possible to obtain with the equipment, a set of experiments were performed at three different temperatures: 70 °C, 90 °C and 110 °C, with Norway spruce specimens being 86 mm in diameter and rotating at a speed of 60 rpm. The normal force applied on the tool, the grinding surface shown in Figure 3, was 10 N and the test duration was 120 seconds. During the tests the forces were measured at 25 kHz, but averaged and stored at 25 Hz. The temperature data was measured and stored at 10 Hz.

The tangential force recorded during the tests was slightly lower at the highest temperature than on the two lower temperatures, as shown in Figure 7. Sections of the grinding track were extracted at the position where the growth rings were perpendicular to the surface, i. e. in the top part of Figure 4b, to be analysed and compared. The removed volumes, Figure 7b, increase with temperature which is in accordance with previous research. The force and the volume can be used to calculate the specific energy required for defibration as shown in Figure 7c. The result that the energy requirement decreases with increasing temperature confirms the well-known fact that increased temperature facilitates defibration, which is generally accredited to thermal softening.

SEM images of the surfaces after grinding, Figure 8, show that at higher temperatures the track gets a cleaner less cluttered appearance, where fewer fibres are still attached to the surface. This indicates that the separation and removal of fibres are indeed easier at higher temperature and thus leaving fewer partially removed or fractured fibres.

The small volume of fibres produced during the test directs the analysis of the fibres to morphology studies of individual fibres. The volume is simply too small to allow the use of more conventional methods, like freeness etc. At the lower temperatures, the fibres are damaged and cut into shorter fragments. At the high temperature, 110 °C, fibres are longer and partially fibrillated, see Figure 9.

Remarks on the test and the analysis

The equipment was demonstrated for a rotational speed of 60 rpm, which with a wood cylinder diameter of 86 mm

Figure 8: SEM images of the track after tests at A) 70 °C, B) 90 °C and C) 110 °C showing less damage to the fibres with increasing temperature.

Figure 9: Examples of fibres separated at 110 °C showing a mixture of long and short fibres.

gives a peripheral velocity of 0.27 m/s. Although this can be increased somewhat, the equipment is not designed to operate quite at the speeds used in industrial grinding, such as 10–30 m/s. More importantly, the frequency of deformation has been shown to have a strong influence in fatigue processes and defibration of wood (Salmén 1984). In the present work, the short distance between the ridges make the contact frequency very high and comparable to that caused by the grinding stone profile in industrial grinding.

In industrial grinding, a substantial part of the power supplied to the process is converted into heat that increase the temperature of the process. In contrast, the equipment and tools used here make the friction power from grinding insignificant (a few Watts) in comparison to the power supplied for heating the equipment. This means the temperature in the grinding zone is fully dictated by the ambient temperature and with the moist pressurized steam it can be considered stable throughout a grinding test.

The grinding time is admittedly quite short, 2 minutes. One reason for this is the normal force on the tool is provided by a spring, and the force will decrease as the tool grinds a track in the wood. The intention of using short

times is to restrict the decrease during a test to a maximum of 5% from its initial value. With the spring used, this corresponds to a track depth of 2 mm, which essentially only allows for a few minutes of grinding, depending on the rate of defibration. However, the subsequent analysis is highly resolved, making the limited grinding time acceptable as differences between different grinding conditions are clearly distinguishable.

Although necessary for adequate testing, the hot and moist environment is challenging for the equipment. In particular, it is not ideal conditions for the strain gauges. These are protected from most effects of the moisture by the coatings described above, but the coatings themselves are affected by the environment and they will mechanically affect the strain gauges. This results in drift in the measured forces during grinding, which further motivates short tests to minimise this effect.

Compensation for drift was made in an unusual way as described above. The reason is the heating is performed with the tool in contact with the wood. During heating, relaxation in any susceptible material such as the fastenings and sealants of strain gauges will occur to reduce stresses. Once the grinding commences, both the tangential and the

normal force are changed. This new situation means the materials are once again taken away from equilibrium and the materials start to adjust accordingly. This is likely the reason why monitoring the drift in forces for a while before grinding in order to predict and compensate for drift during grinding was not very successful. Actually, it resulted in very erratic compensations. The chosen path, using the drift during the second half of the experiment where steady state prevails, proved much more successful.

In industrial grinding the geometries of the wood and grinding stone are the opposite of the experimental setup presented here, with a rotating cylindrical stone and wood logs pressed to it. This industrial geometry can be arranged for also in this test equipment, but the reversed setup selected for the lab test has the advantage of reducing the required size of the grinding tool, making it easier to produce and tailor these. Moreover, as a stationary tool will meet growth rings of the rotating wood specimen at different angles, the mechanisms and rates of fibre separation will differ depending on the location along the circumference of the wood specimen. The selected reversed setup enables parallel investigations on such influences from the changeable nature of the wood in a single test. On an even finer scale, i. e. on the scale of a growth ring, mechanisms will change when a particular asperity grinds in purely early- or latewood, and even depending on the relative direction of the gradient in fibre density through a single growth ring. The presented experimental setup and methodology allows also such details to be studied.

Very likely, there will be variations between tests with the same parameters. This is a foreseen consequence of the variation of the inherent nature of wood as an organic material. Each individual test is performed at a specific location of the wood specimen by moving the tool in the axial direction of the cylinder. Different locations present their individual density of growth rings, presence of rays and amount of resin, etc. and this will cause an unavoidable scatter in any result.

The setup allows for collection of the fibres for further analysis. The collected fibres are specific to each specific test condition, but of course they will reflect an average over the whole circumference of the wood specimen. It should be noted that the tools and processes used here are for demonstration purpose and do not represent an optimised setup for producing fibres of a specific character.

This method provides great opportunities to investigate the influences from tool surfaces on mechanisms and the corresponding specific energy consumption. The process to produce tools presented in this work provides ample opportunities to design the surface to produce fibres for different scenarios, e. g. long undamaged fibres or

chopped or worked fibres. But to do this, further studies are necessary to understand how grinding parameters influence the mechanisms and the process when using well-defined grinding tools.

Conclusions

- The presented method offers unique possibilities for both studying the details of defibration mechanisms using different tool designs and measuring the corresponding energy efficiency.
- The tangential and normal forces acting on the tool are monitored during the grinding motion and the amount of removed fibres in the track is measured with high accuracy to allow calculation of the energy spent on the actual defibration.
- The setup gives easy access to the wood specimen after trials, offering opportunities to investigate the surface of the wood to study the detailed defibration mechanisms.
- High precision grinding tools with extreme control of the size and position of the asperities, enables detailed studies of the influence of asperity shape and relative position on the defibration process.
- The use of small and cheap grinding tools facilitates manufacturing and comparisons between different tool designs.

Suggestions for further studies

Studies of the fundamental influence of load are already in progress. Studies on how to design tools to promote defibration and control the flow of separated fibres out of the contact zone has also been initiated. But the question of how to optimize the tools is complex and will require much further work. Asperities may be given different shapes and distributions and different types of asperities may be combined to achieve desired mechanisms. In addition to such questions is also that of upscaling the tools. Early tests suggest it should be possible to produce curved tools. This would allow tools with a cylindrical shape, which would make the grinding contact more similar to today's grinding processes and the tools more suitable for adaptation in industry.

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