INTERACTIONS OF TIME DEPENDENT, THERMAL AND HYGROSCOPIC CHARACTERISTICS AND RUNNABILITY PROPERTIES OF PAPER

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ABSTRACT

For purposes of design and optimization of industrial processing, many of the physical properties of paper have to be taken into account. The knowledge of the mechanical properties as part of the physical ones must be extended by considering hygroscopic, thermal, optic and electric material aspects. The knowledge on these properties will support the paper processing and converting industry in optimization of their processes and quality of paper.

In previous projects at the Institute of Engineering Design (LMK, Ruhr-University Bochum, Germany) a complete material law was developed and implemented into a finite element system [37]. This model contains also yielding and hardening which are major mechanical properties of paper.

Our ongoing research on the physical properties of paper determines thermal and hygroscopic material behavior. The determination of these physical parameters can be divided into a constructive, an experimental and an analytical part which was performed iteratively.

At first a usual test chamber with temperature control for general material testing was used for determining mechanical properties of paper in the three main directions under all process relevant temperature conditions. In this setup the climate conditions reach the limit for paper testing very soon. Therefore, an individual test chamber was conceived and developed to perform the relevant test environment for the analysis of paper under process relevant thermal and humid climate conditions. Secondly, mechanical experiments were performed in order to find the physical paper behavior, beginning with the mechanical properties of heated and moistured paper. Finally a mathematical formulation of hygro-mechanical behavior of paper was determined.

From our data we found correlations between moisture content and runnability properties. The humidity influence on the elastic modulus and on the strain properties of paper were determined with tensile tests in machine direction (MD) and compression tests in thickness direction (ZD).

Improving this knowledge of general physical material parameters can lead to a realistic mathematical formulation of the physical force-deformation-behavior in paper

and paperboard grades in future. Principles, designs and settings of finishing and converting machines can be optimized by using material laws in process analyses, based on this advanced material knowledge. Furthermore, details of the expected runnability properties of established paper and paperboard grades can be determined.

INTRODUCTION

For purposes of design and optimization of industrial processing, the knowledge of the mechanical properties has to be extended. They are influenced by hygroscopic, thermal, time dependent and other physical and chemical material aspects. Main focus is given to the research of chemistry and composition aspects of the micro composite material called paper. This is done with the knowledge of basic transformation of the chemical effects to the physical properties. To get a wide step forward in paper fabrication, processing and converting the basic mechanical knowledge has to be improved. This will result in new possibilities for optimizing processes and production with increasing quality efforts.

In drying processes and in coating temperature is used to reduce the moisture content. In calendering it is necessary to modify the surface. Different converting and handling processes modify paper in various ways. In this paper the calendering process is representatively taken to display the complex physical key-interactions of paper.

Increasing temperature reorders, breaks down, and reconnects the elastomer's network of coating and of adhesive components in the paper surface to modify gloss, smoothness and opacity at decreasing line loads. The interaction of temperature with moisture leads to difficulties in the calendering process. There the thermo-hygroscopic influences increase with the use of soft rollers which cause longer contact in length and time between the calender rollers and the paper web (longer nips). The higher the average web temperature is, the less relative humidity remains and the relative humidity influence chemical effects and fiber softening directly and indirectly. Relative humidity is the physical measure for the moisture influence by its partial pressure of water vapor in its gaseous state. Transformed to paper physics the relative paper humidity respectively moisture is assumed as measure for the quantity of hygroscopic interactions. It depends nonlinearly on the local climate conditions.

Specific analyses of Wilkström [42] on coating layers show, that those thermo-mechanical effects are influenced by latex binders. There is a big difference in the thermo-mechanics of raw paper and coating. Calendering results in compressing the surface and changes the surface porosity. This surface modification changes the printability parameters extraordinarily. To get sufficient values in the thermo-mechanical description, Wilkström [42] suggests analyzing the uncoated raw paper and the coating layers separately.

Another important effect is the time dependence. High frequent loading changes the mechanical response of the paper and especially of coating material strength. The compressibility and the contact time decreases, with increasing loading frequency [11, 17, 18]. On the one hand, increasing production velocities modify the mechanical properties; on the other hand, they change the hygroscopic influence on the materials.

These complex interactions of mechanical behavior with time, moisture and temperature have to be analyzed experimentally, before generating a material description as a material law for the "real" paper behavior.

STATE OF KNOWLEDGE

Most of coated and uncoated papers become calendered to finish their surface [3]. Calender settings apply the thermal and mechanical paper loadings. Even the smallest irregularities in paper and coating thickness result in irregular deformations in the calender nips. Moisture can increase the effects. Local gloss, smoothness and porosity errors change the paper quality and reduce its printability when mottling, irregular surfaces and gloss errors appear [3].

Latest paper models can describe the complete mechanical material behavior [37]. They are implemented into finite element systems to support process optimization by discovering the physics behind and to give engineers the possibility of using paper's strength. Yielding and hardening are just two major mechanical properties which can be described accurately with present paper models.

Hygroscopic, thermal and time dependent effects extremely influence the mechanical properties. To predict their effects on the paper material, advanced physical experiments have to be done. Research work on these multi-physical effects are present research activities in the context with new experimental possibilities to measure thermo-mechanic-dynamic paper properties.

Time dependent effects

Time dependent material effects directly interact with the aim of increasing process velocities for raising the production. The basic knowledge on these time dependent effects is developed for elastomer materials. These effects can simply be adapted to coatings. For the total paper network detailed investigations have to be done to identify the specific physical effects and interactions.

The general frequency dependent visco-elastic deformation behavior of coatings in calendering [11, 17, 18] can be analyzed with the WLF-theory [41]. The material deformation at high frequencies at temperature T_1 behaves like the deformation at lower frequency and lower temperature. With superposition methods the material behavior can be analyzed for the high industrial frequencies.

Ishikawa et al. [11] analyzed the thermo-mechanic-dynamic behavior of coating layers with different pigment-volume-concentrations (PVC) and discovered three strength zones in coatings. The first one (①) is dependent on the gloss temperature and therefore also dependent on the loading frequency. The instable second (②) and third (③) zones seem to be dependent from the coatings surface micro structure. They disappear after initial loading and behave in analogy to the Mullins effect [14, 20], which describes the breaking down of the filler network of filled elastomers.

The analysis of the pigment volume concentration (Figure 1, middle) and the effect of different pigment sizes (Figure 1, right) of the coating display the range of present processing. The displayed time dependent analyze with its applied frequencies is insufficient to present calendering efforts. The maximal frequency of 30 Hz displays a calendering speed of 36 m/min at an assumed soft-nip length of 20 mm. Nowadays, loading-unloading frequency of single calender nips are much higher. Industrial calenders are working with velocities up to 900 m/min to give paper the necessary compression pulse. This increases the frequency by factor 25. As they exist in hard calender nips or at low normal induced pressures, smaller nips also increase the frequency values. The corresponding loading times of 0.1 ms up to 2 ms depend on the calendering speed, roller radius and roller material [7, 29].

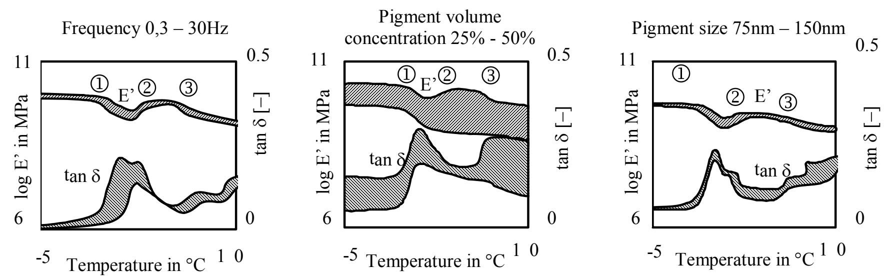

Figure 1 – Frequency's, pigment volume concentration's and pigment's particle size's influence on thermo-visco-elastic and thermo-mechanic coating behavior. Displayed are the effects on the pure elastic respectively storage Modulus E' and the loss factor tan δ (as measure for the energy loss of the hysteresis effects).[1]

This complex behavior is additionally complicated by the amplitude dependency of the frequency behavior [14, 20]. For paper, this behavior is known and analyzed for quasi-static loading under norm-climate conditions [37]. It will nonlinearly change with the applied load and its resulting strain.

The temperature and time dependence displayed in Figure 1 gives a first estimation of the influences and the focus concerning future research. Time and temperature dependent material properties have to be analyzed together to get process relevant material descriptions.

In opposite to quasi-static loading in thickness direction (ZD) paper behaves 20% stiffer at high frequent dynamic loadings (~ 160 Hz) up to 10 N/mm². This causes deformation differences of 25% at calendering relevant loads of 30N/mm² up to 50% at lower loadings of 5 N/mm². The plastic deformation is reduced up to 80% and the elastic deformation up to 30%. The elastic behavior remains constant above 10 N/mm² [4].

Feygin [4] developed a physical elastic-visco-plastic material description with the use of rheological models. The plastic deformations caused by the paper's fiber mesh are inserted starting at initial loading. Therefore the plastic deformations ε_p are assumed as time dependent to

$$\frac{E_0}{H}\varepsilon_p + T_d\dot{\varepsilon}_p = \varepsilon\left(\frac{\frac{E_0}{H}-1}{\frac{E_2}{E_1}+1}\right) \qquad \{1\},$$

where E_1 describes the retarded elastic modulus, E_2 is the plastic part of the elasticity modulus and H with

[1] Compared to experimental values of Kan [12], Mikkilä [18] und Prall [26], who measured elastic compression modulus E' of 9000 N/mm² up to 10000 N/mm², the data of Ishikawa et al. [11] show an error in the scaling. The displayed measurement values have to be MPa not Pa.

$$\frac{1}{H} = \frac{1}{E_0} + \frac{1}{E_1} + \frac{1}{E_2} \qquad \{2\}$$

calculates the long time elasticity modulus. Feygin calculates the time lag of the deformation T_d in the visco-elastic expression with the viscosity factor k to

$$T_d = k\left(\frac{1}{E_1} + \frac{1}{E_2}\right) \qquad \{3\}.$$

Finally the elastic-visco-plastic stress-strain-behavior is described with

$$\varepsilon + T_d \dot{\varepsilon} = \sigma \frac{1}{H} + T_d \frac{\dot{\sigma}}{H} \qquad \{4\}.$$

Rättö et al. [27] analyzed the time dependent mechanical compression behavior in the thickness direction with stress steered pulsation tests, relaxation and recovering experiments. The increasing of the pulsation frequency (load function: $\text{havsin}(x) = 0.5 \cdot (1 - \cos x)$) at high loadings (> 40N/mm²) reduces the plastic deformations up to 7%. The elastic deformations remain constant. The plastic deformation seems to be frequency dependent. With these analyses Rättö et al. [27] build up an analytical formulation for the time depending stress-strain-behavior of paper for loading

$$\varepsilon(t) = K(\sigma) + \int_{-\infty}^{t} J(\sigma_{,t} - \tau)\frac{\partial \sigma}{\partial \tau} d\tau \qquad \{5\}$$

and for unloading

$$\varepsilon(t) = K(\sigma_p) - K_r(\sigma - \sigma_p) + \eta \ln(t_p) + \int_{-\infty}^{t} J(\sigma_{,t} - \tau)\frac{\partial \sigma}{\partial \tau} d\tau \qquad \{6\}$$

Rigdahl et al. [31] analyzed the time dependent **in-plane tensile** behavior of paper at frequencies of 0.1 Hz up to 10 Hz at 0.3% initial pre-strain where an amplitude variation from 0.03% up to 0.18% is superposed. He determined that the loss factor of paper is increasing linearly and the elastic modulus is decreasing linearly with increasing strain amplitudes. The elasticity modulus in MD is dependent on the ZD-compressive and drying treatment. In cross direction he founds a bigger loss factor than in main direction.

Actual research activities at the University of Toronto, Pulp & Paper Centre, deal with the visco-elastic material behavior of coated papers [2] for calendering purpose.

A special approach of Mikkilä et al. [17, 18] suggests a WLF-related view on the gloss production at paper. He determines the calendering frequency as quotient of the web speed divided by the nip-length. The results show that there is an interaction between gloss temperature and frequency in analogy to the WLF-theory.

Temperature dependent effects

Temperature induces chemical changes and interacts with the moisture content of paper and coatings. Two major interactions are known. Firstly, for coating the chemical cross-linking and yielding can be determined as the major temperature influence. Secondly, the thermo-hygroscopic effects change the uncoated raw paper properties. The vice versa interactions also exist as not negligible effects. Additionally in previous production steps, thermal or humid treatments modify the material properties, as found by Suontausta [35] at hot and cold dried coating analyses.

In calendering temperatures between 20°C up to 250°C (experimental calendering setups) are used with high nip loads. The analyzed temperature range of Ishikawa et al. [11] from -50°C up to 150°C (Figure 1) maps the lower half of the calendering temperatures, starting at 80°C up to 180°C.

With thermo-mechanic-dynamic analyzes, Mikkilä [17, 18] found values of 12000 N/mm² up to 15000 N/mm² for the elasticity respectively storage modulus E' in the glass state (low temperatures and high frequencies) of coatings (clay, SB-latex). Under yielding conditions at low frequencies and high temperatures the elastic modulus E' reduce to 7000 N/mm² up to 10000 N/mm². In calendering the loss factor of coating increases up to 12%.

The loss factor displays the energy loss because of inner material damping behavior under dynamic loading. It's a measure from rheology and displays the energy consumption of inner friction and inner reorganization effects as a quotient of the lost elasticity divided by the stored elasticity.

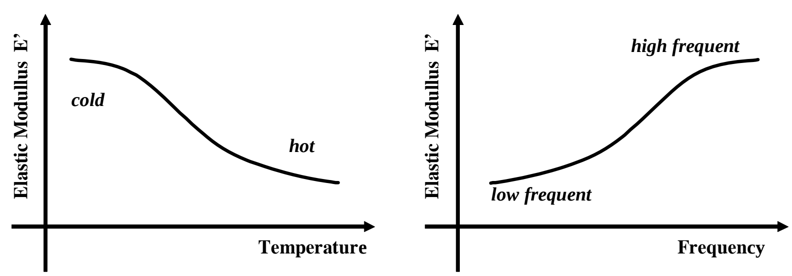

Figure 2 – Principle of the thermo-dynamic-mechanic material behavior according to the WLF-theory [41]. The material strength changes anti-symmetric by temperature and frequency.

These thermo-mechanical investigations are influenced by the thermal material properties of paper. Temperature coefficient, thermal conductivity and thermal diffusivity are the belonging physical measures.

Kerekes [13] analyzed the heat transfer between paper and solid surfaces. It is influenced by paper's thickness, density (bulk) and smoothness. He measured that a high temperature gradient does not produce a density gradient in the thickness direction [29].

A first estimation for the transient heat transfer in paper thickness in a calendering nip is given by Vreeland [28] with

$$T(z,t) = T_{ini} + \left(T_{heating} - T_{ini}\right) \cdot \left[1 - erf\left(\frac{-z}{2\sqrt{\alpha t}}\right)\right]. \qquad \{7\}$$

Therein, T is defined as temperature at the thickness coordinate z at time t with the initial web temperature T_{ini} and the thermal diffusivity α with

$$\alpha = \frac{k}{\rho \cdot C_p}, \qquad \{8\}$$

depending on the thermal conductivity k, the density ρ and the specific heat capacity C_p. The paper specific microstructure allows the dependence of the thermal conductivity with the density. The applied nip load in calendering reduces the pore volume and increases the thermal diffusion. Additionally, the contact pressure leads to an increasing contact width. The appearing thermal conductivity k_a can be defined as

$$\frac{1}{k_a} = \frac{1}{h_p \cdot t_\theta} + \frac{1}{k} \qquad \{9\}$$

with the present paper thickness h_p, the heat transfer rate t_θ and the thermal conductivity k [28].

Latest research activities at the Technical University of Darmstadt [44], Germany, have increased the knowledge on the thermal properties of paper by using the light flash method [19].

At experiments of Zhao [44] opacity and reflection of paper are minimized by a graphite coating layer which effect is finally substracted. Then the temperature is applied on the one side by Xenon light and measured on the opposite side by infrared sensors. The thermal properties are determined in assumption of constant behavior in analogy to Parker et al. [24] with an assumed heat capacity c_p of $c_p = 1{,}35 \frac{J}{kgK}$.

The analyses of Zhao [44] are performed in the temperature range of 20°C up to 200°C. They show that there is a maximum at 70°C for thermal diffusivity and conductivity. Calendering reduces the thermal diffusivity proportional to the thickness reduction by 30%. It reduces the pore volume and with it also the amount of air inside with its high thermal diffusivity ($k_{20°C} = 19 \frac{mm^2}{s}$, $k_{250°C} = 62 \frac{mm^2}{s}$ [32]). Fibers and fillers remain with their relatively high temperature diffusivity ($k_{wood} \approx 0{,}12 \frac{mm^2}{s}$, $k_{fillers} \approx 0{,}5 \frac{mm^2}{s}$ [25]).

The thermal conductivity of paper is smaller than $0{,}1 \frac{W}{mK}$ [44] (compare: $\alpha_{air} = 0{,}024 \frac{W}{mK}$, $\alpha_{wood} \approx 0{,}135 \frac{W}{mK}$, $\alpha_{fillers} \approx 1 \frac{W}{mK}$). Zhao [44] shows that calendering has a quantitative but no qualitative influence on the thermal conductivity in the calendering related temperatures of 80°C to 200°C. In this range the thermal conductivity is decreasing about 10%. It is also dependent on the applied calendering load. It shows a minimum at a line load of 100 N/mm² ($\alpha_p(100 \frac{N}{mm}) < \alpha_p(0 \frac{N}{mm}) < \alpha_p(200 \frac{N}{mm})$). This machine related behavior is important for further analyses of the related paper properties.

Specific calendering experiments of Rounsley [33] at LWC-paper with high temperature gradients of 200°C show a linear interaction of gloss (hunter) and temperature and a second order dependence of the PPS-roughness with a minimum at 160°C to 180°C. These manipulations produce modifications in layers up to 5-10µm by thermal loading at 6% moisture content. The hygro-thermal interactions could not be discovered with this mixed analyze, as temperature change the paper humidity.

Another temperature induced process in paper is hornification [43]. It affects the water retention ability of the fibers, their flexibility and their swelling by collapsing of pores and merging micro-fibrils. Hornification takes place at temperatures above 120°C and at moisture contents below 30%. If the web temperature is below 120°C, the mechanical resistance could be recovered by moistening.

Curling is also a temperature dependent phenomenon. Smallest irregularities in production over the thickness or cross-section profile of paper produce inertial mechanical and geometrical changes causing curl [43]. The temperature effects can be component specific [21].

Moisture dependent effects

Paper behaves hygroscopic. The moisture content of paper is the amount of water which exists in a paper sample. It depends on manufacturing, processing, finishing and the environmental climate. Values for dried papers differ from 2% up to 12 %, depending on its later use, on the containing materials (especially the hygroscopic cellulose fibers) and on the production process. The moisture content is measured in weight percentage related to the totally dried paper sample. Detailed methods are standardized in TAPPI T412, ISO 287 and SCAN P4. They are very time consuming and sensitive to handling. In general industrial analyses a paper sample is assumed as dry when it is hold under 105°C for minimum 24 hours.

Moisture in paper is interacting with process and environment climates. The storages for paper are climate controlled, and so is the environment of the most production and processing lines.

The difficulties related to the moisture dependent effects are located in the processes itself. High temperatures at calendering for example decrease the moisture content in paper. At calendering the moisture loss is dependent on the roller's surface temperatures. At LWC-Paper Suontausta [34] found that an increasing of the calendering temperature from 100°C to 150°C produces 1,2% moisture loss. Nip loads, process velocity and thermal paper properties also influence the moisture loss in an important but insufficient known way.

Steaming is used to reduce the drying effects in calendering, but steam is just applied to the surface region. At process velocities there is only a very small time gap to influence paper. Effects to surface and inner structure remain unknown. Laboratory experiments show that increasing moisture influences compressibility, thickness and smoothness strongly [7]. Shoe calenders (wide nip calenders) are used in cardboard calendering. They give best results with high temperatures and active steaming at low compressive loadings [30].

Both, coating and uncoated raw paper show hygro-mechanic force deformation behaviour. In experiments with single coated fine papers ($CaCO_3$) containing 3% to 8% moisture, Engström [3] could prove the interaction between thickness compression and moisture by analyzing the ratio between the plastic deformation of the coating layer $\varepsilon^{pl}_{coating}$ and the compression of the raw paper ε^{pl}_{paper}. This ratio increases with increasing moisture. Additionally the used coating (Clay, SB-latex) behaves more plastic when moisture is present [3].

Present activities of paper and coating specific research focus on modification, adaptation, optimization and manipulation of the processing materials (fibers, fillers, binders, pigments) to find and integrate new micro- and nano-technological effects. But the analyses of Mikkilä [17, 18] prove, that there is a huge undiscovered potential in rheo-mechanical and thermo-mechanical analyses for paper finishing and converting processes.

By fundamental analysis the influence of moisture on the mechanic material behavior could be found. In the literature only a few fragmentary aspects to the basic hygro-mechanical properties were found. The general opinion to moisture effects is the loss of strength. This general view is insufficient to present efforts. The fundamental physical interactions have to be discovered.

Moisture influences the two main fiber properties: **Elasticity modulus** and **glass transition temperature**. Therefore its influence on paper as micro composite material is important. Moisture has to be well distributed and well dosed across the web surface to prevent blackening or mottling. Applied temperatures can cause moisture gradients in transport direction or along the paper thickness. These effects onto profiles [33] have to be taken into account.

An increasing amount of water cause **plastification** of the cellulose fibers and dissolves the ion- and hydrogen bonds. The usage of water soluble binders enhances the effect and cause migration phenomena.

Swelling and **dwindling** characterize the volume increase respectively decrease through adsorption respectively desorption of water. Paper shows prominent swelling behavior in-plane and out-of-plane. The dwindling is prominent in machine direction but in cross and thickness direction it is assumed as negligible. The forces caused by these phenomena are considerable. The water adsorption from the ambient air is caused by capillary condensation and emerging chemical bonds. It shows its minimum dimensional change at 40-60% air humidity.

Creeping is mechanically defined as visco-elastic-plastic phenomenon. Byrd [1] developed a simple logarithmic approach $\varepsilon(t) = a + b \cdot \ln(t)$ for the time depending strain $\varepsilon(t)$ to describe the creeping of paper for initial creeping strains [10]. Therein the material specific parameters a and b depend on the material, on the creeping strain and on the air humidity [9]. The hyperbolic approach by Haraldsson [8] for the stress depending on strain and time has the formula $\sigma(t,\varepsilon) = \left(\frac{t}{t_0}\right)^n \cdot c_1 \cdot \tanh\left(\frac{c_2}{c_1} \cdot \varepsilon\right)$, with the process and material constants t_0, n, c_1 and c_2. Urbanik [36] analyzes a material formula $\frac{dX_C}{dt} = \mu \cdot \left|\frac{dX_H}{dt}\right|$, where he separates the hygroscopic strain X_H from the creeping strain X_C. The experimental determined creeping coefficient μ is supposed as time dependent. Other models for various air humidities are developed by Störemark and Fellers [43].

Discussing the state of knowledge on the moisture influence of paper, the dissertation of Nyman [22] has to be mentioned as the most advanced approach concerning the hygro-static and hygro-dynamic paper analyze. His continuum-mechanical approach for finite shell elements founds on a separation of the deformation behavior into elastic and non-elastic stretches. With these assumptions the hygroscopic deformation can separately be inserted into the stiffness and the load vector. Finally he uses the mass balance to generate a general adsorption and desorption description for the moisture movement through paperboard materials. This publication is specifically developed for transportation and storage purpose and not applicable to general paper processing and converting efforts.

GENERAL MECHANICAL BEHAVIOR

The general **in-plane** material behavior is predicted by the elastic-plastic **loading and reloading** formulation [37, 38] on the base of the phenomenological material behavior of Paetow [23]. The loading and reloading behavior for positive strain $\partial\varepsilon \geq 0$ was carried on by separating elastic and plastic strain in MD-tension $\varepsilon_{11} = \varepsilon_{11}^{el} + \varepsilon_{11}^{pl}$ (respectively CD with index $_{22}$ which behaves analogically) considering the load history dependent parameters mutable initial modulus ${}^{mod}E_{11}$ and mutable tensile stress limit ${}^{mod}\sigma_{11}$. Both depend on the material constants and on the plastic strain.

$$ {}_{up}\sigma_{11} = {}^{mod}E_{11}\left(1 + \frac{{}^{mod}E_{11}}{{}^{mod}\sigma_{11}}\left(\varepsilon_{11} - \varepsilon_{11}^{pl}\right)\right)^{-1}\left(\varepsilon_{11} - \varepsilon_{11}^{pl}\right) \qquad \{10\} $$

The **unloading** behavior's formulation base on the elastic strain dependent tangent modulus ${}^{tan}E_{11} = f(\varepsilon_{11}^{el})$ which behaves linearly at increasing stress ${}_{un}\sigma_{11}$. It depends on the failure-buckling-stress σ_{11}^{min} in MD pressure (respectively CD with index $_{22}$) and on σ_{11}^{max} the point of maximal tensile stress and strain. This leads to the phenomenological unloading behavior described with an exponential formulation [37] containing the experimentally determined and load history dependent material parameters C_{11} and D_{11}:

$$ {}_{un}\sigma_{11} = \frac{D_{11}}{C_{11}} e^{\left(C_{11}\varepsilon_{11}^{el} - 1\right)} . \qquad \{11\} $$

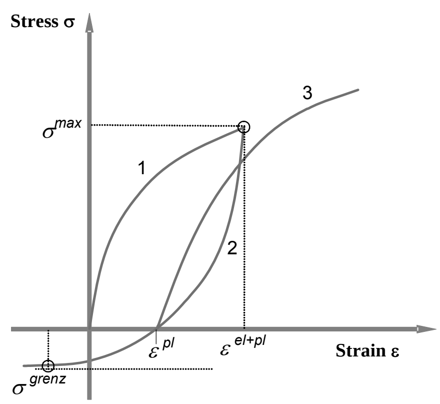

Figure 3 – General stress-strain behavior in MD resp. CD; Curve 1 loading, starting at the undeformed state; Curve 2 unloading phase from σ^{max} up to buckling at $\sigma^{grenz} = \sigma^{min}$; Curve 3 reloading [38].

Out-of-plane the elastic-plastic strain for ZD-compression effects by cyclic loading tests in thickness direction (ZD) have to be separated in pure elastic and in elastic-plastic deformations. Therein the elastic-plastic strain is defined by $\varepsilon_{33} = \varepsilon_{33}^{el} + \varepsilon_{33}^{pl}$. Without considering MD-ZD-shear interaction the ZD-compression behavior can be analyzed at small elastic deformation solely, where the nonlinear elastic behavior is defined as $\varepsilon_{33} = \varepsilon_{33}^{el}$. The qualitative behaviour is equivalent to the MD-unloading behavior (Figure 3), therefore the appropriate approach is an exponential one containing the load history dependent material parameters A_{33} and B_{33}.

$$\sigma_{33} = \frac{B_{33}}{A_{33}}\left(e^{\left(A_{33}\varepsilon_{33}^{el}\right)} - 1\right), \qquad \{12\}$$

The nonlinear elastic-plastic ZD-compression behavior $\varepsilon_{33} = \varepsilon_{33}^{el} + \varepsilon_{33}^{pl}$ must consider additionally the coupling to MD-ZD- respectively CD-ZD-shear interactions. A detailed view on the interactions would go too far. It can be found at Welp et al. [37, 38].

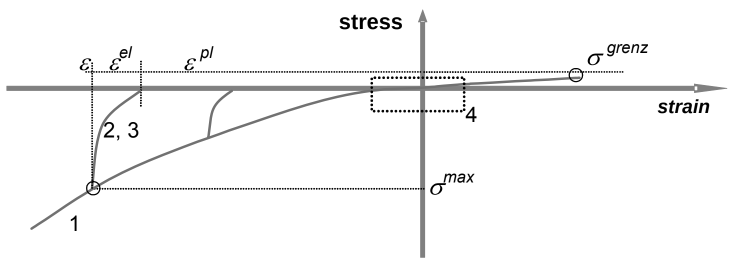

Figure 4 – General stress-strain behavior in ZD [38]; Curve 1 displays quasi-static loading; Curves 2 and 3 sketches unloading and reloading to the point of return $\sigma^{\max} = \sigma_{33}^{\max}$. Area 4 is set as pure elastic zone.

RHEO-MECHANICAL BEHAVIOR

With a universal testing machine the quasi-static range below 0.4 Hz is analyzed for the in-plane paper behavior.

This fundamental analyze discovers that paper stiffness increase with raising loading frequencies. Two hardening zones can be detected, where the stiffness increases considerably. One is located at frequencies below $0.05/s$, the other one depend on the applied strain. At high strain rates close to the failure point in MD the stiffness gradient increases at loading frequencies of about $0.2/s$. If the loading is about 30% of the tensile breaking strain the increasing gradient of stiffness is lower over the regarded frequency range. The second hardening zone is estimated to be above $0.4/s$.

This coupling of the force-deformation behavior with the loading time is mapped in Figure 5. The paper behaves strongly time respectively frequency dependent to the maximum applied displacement as representative of the loading amplitude. With an increasing applied strain loading the stiffness gradient of paper increases and the starting frequency of the second determined zone of hardening by frequency decreases.

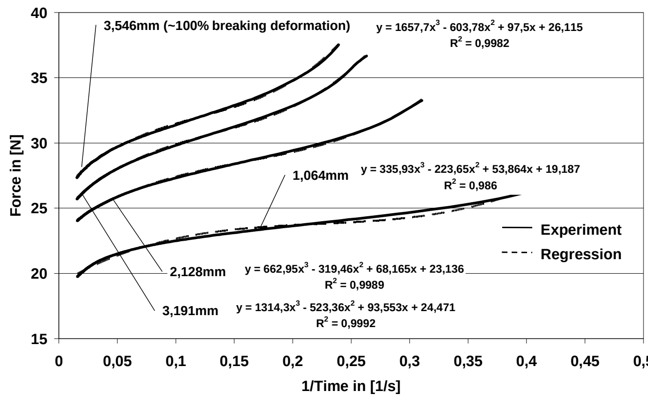

Figure 5 – Rheo-mechanical behavior – In-plane tension of uncalendered double coated WFC-paper (170g/m²) at different displacements displayed against the inverse of the loading time as representative for the loading frequencies.

To analyze time dependent mechanical properties in the process range with 500 Hz up to 1000 Hz special dynamic-mechanical-testing analyzer are needed.

THERMO-MECHANICAL BEHAVIOR

The first experiments were performed with a standard climate chamber. It was cooled with liquid nitrogen. This cooling device for temperature regulation and the hygro-thermal interaction influences the moisture content of the papers reasonable, even at low temperatures paper becomes dehumidified. That's why these standard setups are only possible to measure non process relevant paper properties at undefined dry moisture contents and at a quasi-static time range.

Analyzing the force-deformation behavior in thickness direction we found the tendency of an increasing stiffness with increasing temperatures (20°C up to 160°C) at double coated WFC-paper. Concerning thermal effects from 20°C up to 80°C we determine a scattering of 30% with big errors caused by humidity. From 80°C up to 160°C the values are more accurate and paper shows a scattering below 10%. Both ranges display the tendency of increasing stiffness.

HYGRO-MECHANICAL BEHAVIOR

To get first experiences with hygroscopic effects on mechanical properties, experiments at a universal testing machine were performed at the LMK under standardized temperature of 20°C. The paper samples are manually moistured. The water is applied by sprayed water. After a conditioning time of 24h we can assume the moisture content as constant over the papers cross section.

It is known that the hygro mechanical behaviour depends on the moisture content of the paper. Analyzing the moisture retention of calendered and uncalendered papers we discover that they change in calendering with the thermo-mechanical processing. At rotogravure printing papers and at the heavy WFC-paper (double coated) the equilibrium moisture content increases with calendering about 5-7% related to the uncalendered value. The light WFC-Paper (double coated) behaves different. Its moisture retention behavior decreases after calendering about 7%.

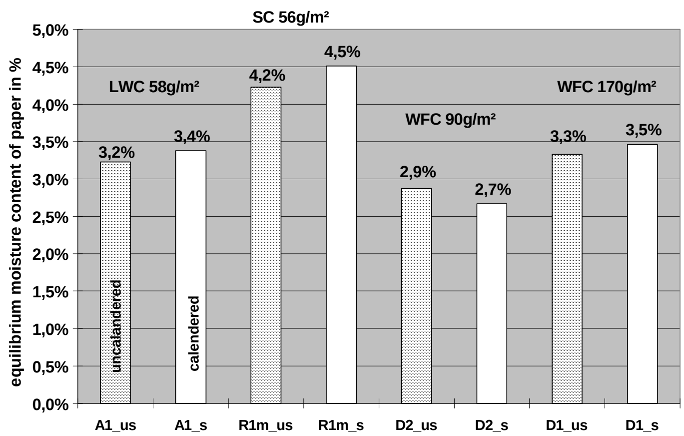

Figure 6 – equilibrium moisture content of uncalendered and calendered paper grades [39].

To extract the **in-plane humidity influence** on the paper's mechanical behavior tensile tests were performed with manual spray-moistening and conditioning for minimum 24 hours papers. To analyze the hygro-mechanical behavior the force-deformation behavior is transformed to the stress-strain behavior and evaluated by its fundamental mechanical measures: the initial elasticity modulus and the fitted stress limit. To determine the general behavior and to display interactions concerning the load history uncalendered and calendered papers were analyzed in MD and in CD [43].

The evenly applied moisture supports an excellent view on the hygro-mechanical paper properties.

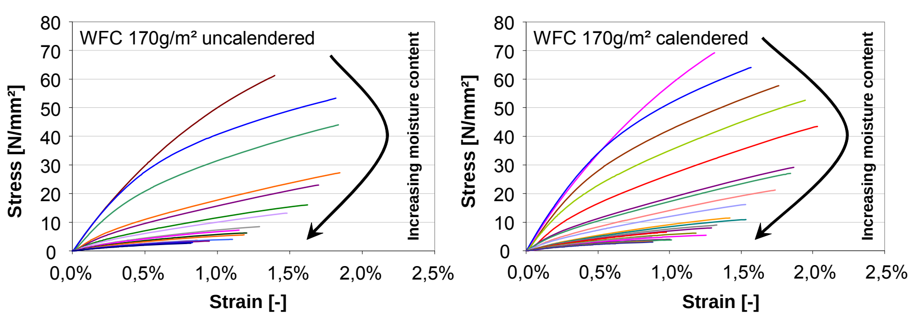

Figure 7 – In-plane moisture influence on the MD-stress-strain behavior of double coated WFC-paper. Moisture contents:
0.72% – 41.45% uncalendered, 0.61% – 45.16% calendered.

Up to 9% humidity in **uncalendered** paper the MD-stress-strain-behavior displays an increasing failure strain from 1.3 to 1.8%, whereas the MD-failure-stresses halves from 60N/mm² to 30N/mm². At higher moisture contents the failure stresses and strain decreases. The degressive curvature reduces with increasing moisture and the curves become more linear. At 20% moisture content the MD-failure-strain reaches dried paper's failure strain with 1.2% [43].

Compared to uncalendered papers the failure stresses and strains are higher at **calendered** paper. Qualitatively calendered paper behaves equal to uncalendered.

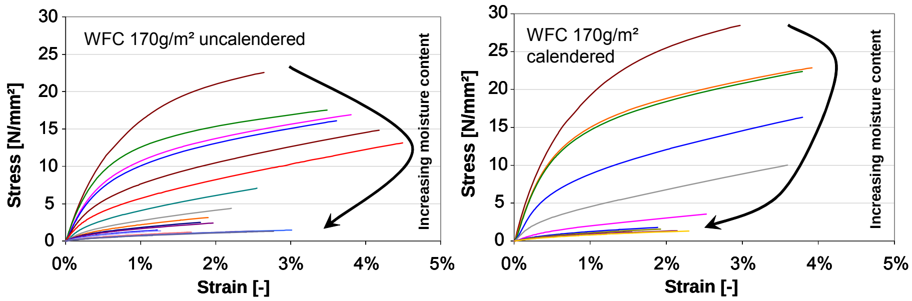

Figure 8 – In-plane moisture influence on the CD-stress-strain behavior of double coated WFC-paper. Moisture contents:
0.90% – 40.36% uncalendered, 0.84% – 35.96% calendered.

Qualitatively the hygro-mechanical behavior is equal in CD and MD. As known from the general material behavior in-plane, it shows about two times higher strains in CD. The according failure stresses are half of the MD-values.

To all results the dried sample behavior is remarkable. Presumably influenced by drying heat in its mechanical behavior, dried paper shows a qualitatively slight different behavior which will be much more visible in the following pictures.

To describe the moisture dependent paper behavior mathematically the fundamental material parameters of equation {10} have to be analyzed moisture dependent. In the following the moisture dependence of the initial elasticity modulus and the mathematical stress limit is analyzed on the presented data.

The **initial tensile elasticity modulus** depends on the initial gradient of the stress-strain-diagram. It is a measure for the initial stiffness of paper.

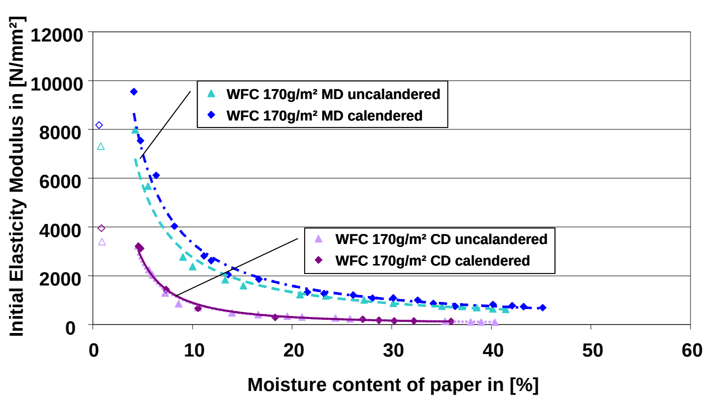

Figure 9 – In-plane moisture influence on the initial elasticity modulus of double coated WFC-paper. Experiments and exponential regression. Dried values displayed in open shapes.

Analyzing the initial elasticity modulus (Figure 9), the dried values differ strongly from the curvature. Therefore they aren't taken into account for the regression. Future analysis with the presented climate chamber will close this initial gap caused by the thermal influences of the drying procedure [43].

The initial elasticity modulus is strongly moisture dependent. It degressively decreases up to 20% moisture content until it nearly stays constant. Its behavior is qualitatively equal in MD and CD. Calendering increases the values slightly.

The experimental values show a potential curvature. Therefore a potential formulation {13} is determined depending on the moisture content C_{11}^{hygro} to describe the hygro-mechanical dependency of the initial elasticity modulus. The coefficient A_{11}^{hygro} and the exponent B_{11}^{hygro} are constants determined in laboratory experiments. Excluding the dried valued, it results in an excellent matching with the experimental values, compare Figure 9.

$$ {}^{\text{mod}}_{hygro}E_{11}^{ini} = A_{11}^{hygro} \cdot m^{-B_{11}^{hygro}} \qquad \{13\} $$

The **mathematical tensile stress limit** displays the maximum value of stress at an infinite strain. It is strongly moisture dependent and decreases degressively with increasing humidity to a value of 10N/mm². The curvatures of the calendered paper run below the calendered curves. In CD the values of the mathematical stress limit is less than a third of the MD values [43].

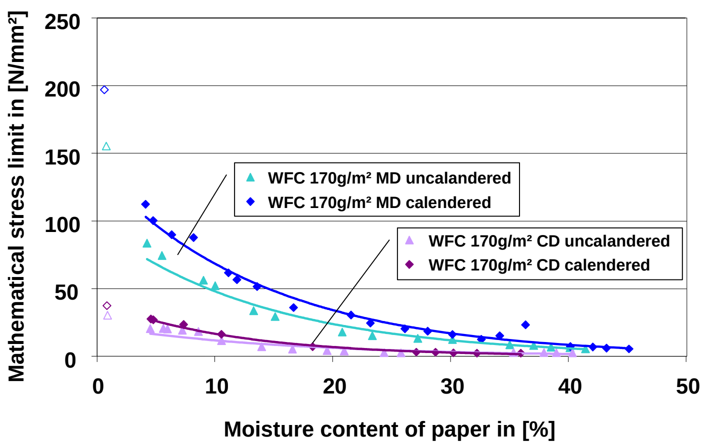

Figure 10 – In-plane moisture influence on the CD-stress-strain behavior of double coated WFC-paper. Moisture contents:
0.90% – 40.36% uncalendered, 0.84% – 35.96% calendered.

In opposite to the initial elasticity modulus the dried values follow the curvature of the calculated tensile stress limit qualitatively well. Despite of this, the thermal heating interacts with the mechanical properties. So the dried values are neglected for the first.

A potential curvature maps this moisture m dependent material property properly, compare Figure 10. Therein the coefficient C_{11}^{hygro} and the exponent D_{11}^{hygro} are material specific constants which have to be calculated from laboratory experiments.

$$ {}^{\text{mod}}_{hygro}\sigma_{11}^{ini} = C_{11}^{hygro} \cdot m^{-D_{11}^{hygro}} \qquad \{14\} $$

With these phenomenological assumptions for the moisture dependent initial elasticity modulus and the moisture dependent calculated tensile stress limit, we present a new mathematical description for the hygro-mechanic tensile behavior:

$$ {}_{hygro}^{\mathrm{mod}}\sigma_{11} = {}_{hygro}^{\mathrm{mod}}E_{11}\left(1+\frac{{}_{hygro}^{\mathrm{mod}}E_{11}}{{}^{\mathrm{mod}}\sigma_{11}}\left(\varepsilon_{11}-\varepsilon_{11}^{pl}\right)\right)^{-1}\left(\varepsilon_{11}-\varepsilon_{11}^{pl}\right) \qquad \{15\} $$

From now it is possible to predict the hygroscopic paper the mathematical description is now available until tensile break of paper.

CONCLUSIONS

The state of knowledge on the mechanical paper properties shows that research has to continue to match the industries efforts. The quadruple of existing nonlinear mechanical material descriptions, time dependent properties, thermal interactions and hygro-mechanical behavior of paper offer a wide range in research activities. Increasing knowledge on paper physics push technology development and progress.

The general, time dependent, thermal, and hygroscopic characteristics and their interactions are merged to display the urgent efforts in paper physics. The possibilities of present technologies in machine engineering and controlling as well as quality efforts force progress in describing the interactions of multiphysical material behavior.

Present technologies are not able to generate appropriate climate process and handling conditions. To perform analyses beyond these limits, a climate chamber is presented to perform the necessary testing environment. The short introduction on climate generation displays the complexity and the necessity of multiphysical consideration when dealing with paper.

Experiments are presented to the single paper characteristics. The general material behavior is described mathematically. Time dependent effects are exemplary displayed in the low frequency range. The effort to consider high frequencies is revealed. Thermal effects cannot be analyzed with present climate setups. First experiments give an estimation of their potentials.

Focus is set on the hygroscopic paper characteristics. Hygro-mechanics is most important due to the material’s inertial hygroscopic and hydrophilic components. The fundamental tensile mechanics are analyzed according to their moisture dependence.

Finally we suggest a new potential approach considering the humidity of paper under tensile loading.

OUTLOOK

The interaction of moisture and temperature is strong as displayed for air humidity in Figure 1. To analyze paper properties in its production and handling climates correctly, the moisture influence have to be taken into account. Paper humidity has to be stabilized, when temperatures are changed and vice versa. Additionally their dependence on time has to be regarded.

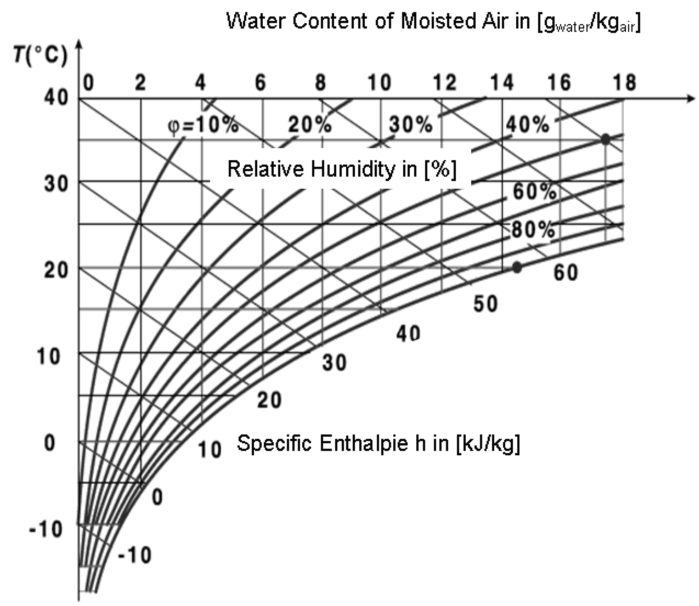

Figure 1 – Mollier diagram: Relative humidity and its amount of water at moderate temperatures.

Such analyze needs a special climate chamber, where the necessary thermal and hygroscopic conditions can be extended from the norm climate. The thermal and hygroscopic process related paper characteristics have to be determined in the climate range of -30°C (cold-storage-house) up to 300°C (hot calendering process) with 0% up to 100% humidity. The extreme range in temperature and moisture cannot be performed in present testing environments. Therefore a climate chamber is started to build up to reach these climate conditions.

Climate regulation

For paper physics climate regulation has to be classified as a two parameter closed loop control. Therein each parameter affects the other one by a strong interaction. In case of parallel temperature and moisture regulation an increasing of the temperature decreases the moisture, because water has a four times higher specific enthalpy compared to air. Remaining moisture constant the exponential increasing of the water content with temperature results in a raising influence. Both parameters have to be controlled parallel to work efficiently.

To display the complex interactions in regulations the dependencies of the climate chamber are mapped below for an applied closed loop control. Through sensors, the output values y_i of the system is fed back to the desired values w_i. The controller detects the difference between reference and output values to change the input signal u_i to the system. The inputs affect both output values in different ways. So the input values have to be divided into the signal values s_{ij}.

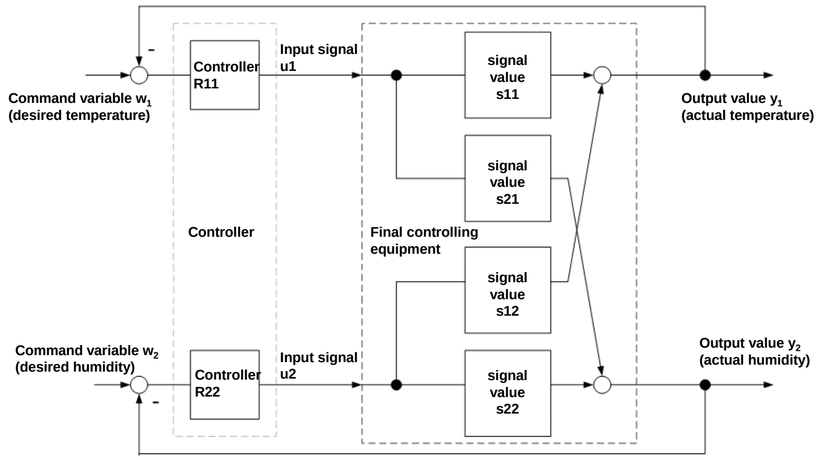

Figure 2 – Two parameter closed loop control for climate regulation.

Each input signal influences both output values. The control path depends mathematically on four differential equations respectively transfer functions. The exact determination of each function and the control unit design are in progress. A theoretical pre-calculation is not possible because of missing information concerning the behavior of the valves, convection, heat loss, and heat distribution.

Humidity sensors

The extreme climate conditions cause difficulties, especially for the air humidity detection above 95°C. There is only one experimental miniaturized humidity sensor found, who can work in the necessary temperature range up to 300°C.

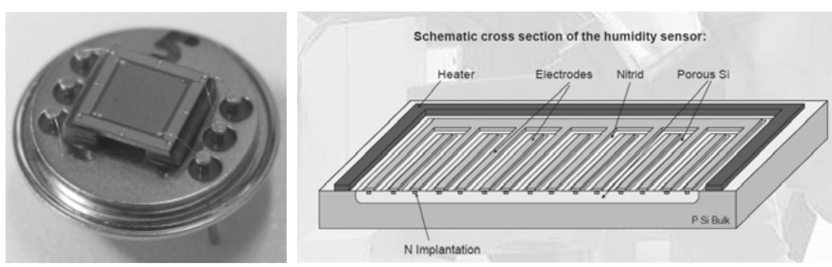

Figure 3 – Photo and principle of the humidity sensor by Furtwangen University [5].

This sensor is developed at Furtwangen University in cooperation with the Research Institute for Technical Physics and Materials Science (MFA) in Budapest. It consists of highly porous silicon (active surface per volume: 250m²/cm³ up to 400m²/cm³) with an integrated heating to prevent electric short-circuit by water. The detected capacity is the measure of this humidity sensor. From 0% up to 100% humidity it changes about 5000%.

ACKNOWLEDGEMENTS

The authors would like to thank the German Pulp and Paper Association (VDP-Verband deutscher Papierfabriken e.V.) for supporting this research. Further thanks are given to the involved manufactories (StoraEnso – Reisholz, UPM-Kymmene – Augsburg and UPM-Kymmene – Dörpen) for supplying the needed materials and the students Ms Wysgol and Mr. Kehl for their student works.

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Name & Affiliation	**Question**
Balaji Kandadai, Kimberly Clark	On one of your graphs you showed compression strain at different temperatures. Was a single test performed at each temperature?
Name & Affiliation	**Answer**
V. Niebuhr, Ruhr-University Bochum	You see different colors – each color is one temperature. Each color displays one sample tested in the universal testing machine.
Name & Affiliation	**Question**
Balaji Kandadai, Kimberly Clark	Did you try repeating the same test? I saw the curve shifting to the left in this case for the same temperature if you were to do the test again.
Name & Affiliation	**Answer**
V. Niebuhr, Ruhr-University Bochum	Yes, of course. You see lots of blue curves inside, green curves inside, yellow curves and so on. You see definitely that the spread of the curve changes – the distribution changes. This distribution, in my opinion, is generated by the undefined climate in this chamber. We cannot say what moisture content the paper has inside the chamber. We cannot say the temperature is right, but we may be able to say the general shift in average is an expression of temperature as influence. The sensors for temperature and moisture content in the typical climate chambers are insufficient. It was not possible to use the usual climate chamber because of the moisture content inside. Instead we cooled the chamber with liquid nitrogen, this is a dry environment. That way the moisture decreases. There is a steady flow outside because of the regulation.
Name & Affiliation	**Question**
Marko Jorkama, Metso Paper	One important property for the runnability and other processes is the paper friction coefficient. This could be the friction coefficient between the paper or web and the roller, or the web layer to layer friction coefficient. Have you considered studying the dependence of the friction coefficient on moisture or temperature?
Name & Affiliation	**Answer**
V. Niebuhr, Ruhr-University Bochum	Our aim is to also consider friction. We have built a friction machine testing setup which we can use in the climate chamber. I have a climate chamber constructed. The regulation is missing, so there are a lot of things to do. We must be able to run this climate chamber properly and perform tests concerning the hygro-mechanical effects before we can say anything regarding the friction. We need to monitor and control the moisture content inside the paper. The moisture content of the air is what is measured and controlled in most chambers. We have to interlink the

moisture of the air with the moisture content of the paper. Then we can proceed to friction tests.