2000 TAPPI JOURNAL PEER REVIEWED PAPER

INFLUENCE OF TEMPERATURE AND TYPE OF COATING LATEX ON THE OUT-OF-PLANE COMPRESSION BEHAVIOUR OF COATING LAYERS

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ABSTRACT

Coated papers are often calendered at high roll temperatures. Isolated coating layers without any base paper or other substrate present were here compressed in a platen press at two temperatures, 23 and 150ºC. Four different types of coating latices were compared with respect to their influence on the compressive properties in the thickness direction of the coating layer; a styrene-acrylate, a poly(vinyl acetate), and a high and a low T_g styrene-butadiene latex. The compression of the coating layers was found to be strongly related to the thermal softening of the coating latex.

The surface roughness of the coating layer had a substantial influence on the compression behaviour of the coating layers. This was revealed by a series of model experiments using consecutive loading sequences. A woodfree paper was blade coated with coating colours containing each of the studied latices. When these papers were calendered in a pilot calender at roll temperatures of 37 and 145ºC, there was a clear relationship between the development of paper gloss and the thermal softening of the latices.

INTRODUCTION

Almost all pigment-coated paper products are calendered after the coating operation in order to improve their surface smoothness and printing properties. The calendering technique used is then adapted to the target paper properties for each grade. For coated products, common techniques are high-temperature soft calendering, super calendering and, in the case of matte-coated grades, soft/soft calendering (i. e two unheated polymer rolls). In order to achieve the benefits of a temperature-gradient calendering, the roll temperature used for soft calendering has increased both for coated and uncoated grades.

For uncoated paper products, a high roll temperature is used to selectively soften the paper surface and thereby achieve a higher degree of surface deformation and less compaction of the inner structure of the paper. A prerequisite for this is that a temperature gradient develops in the thickness direction of the paper as a result of the high roll temperature and the short residence time in the calendering nip. The softening of the paper surface fibres refers here to the thermal softening of the wood polymers; lignin, hemi-cellulose and cellulose.

The same mechanism of temperature-gradient calendering applies in principle for coated products. The coating layer is a structure built-up primarily of three components; pigments, binder (latex or starch) and air-filled pores. The pigments are usually inorganic materials, i.e. clay and calcium carbonate, whose mechanical properties are not significantly affected by the temperature. On the other hand, the mechanical properties of the coating latices exhibit in general a relatively strong temperature dependence.

This work focuses on the effect of temperature on the out-of-plane compression behaviour of coating layers containing different types of coating latices. The behaviour is compared with results from pilot-scale high-temperature calendering of coated paper. For completeness, it should be mentioned that the softening of the wood polymers in the base sheet under the coating layer also plays a role in the case of temperature-gradient calendering of coated products, cf. Vreeland et al. (1989). This is not however considered here.

The experiments were performed on isolated coating layers (at temperature equilibrium) without any base paper or other substrate present, using a platen press with a heated pressure head. In order to be able to study the compression properties of the coating layers without any disturbing influence of the surface roughness, the coating layers were extraordinarily thick (1-2 mm). The coating layers were preheated to temperature equilibrium before they were compressed. Four different coating compositions were tested, differing in the type of latex binder used. The influence of the surface roughness will be discussed separately.

EXPERIMENTAL

Preparation of the Coating Layers

The coating layers were prepared using a casting technique. The dimensions of the mould were 160 x 160 mm with a depth of 3 mm. Since the coatings tend to shrink considerably in the plane during drying, the adhesion between the coating layer and mould surface had to be minimised, to prevent the thick coating layer from cracking into small pieces. This was achieved by placing a Teflon surface in the bottom of the mould (cf. Kan et al., 1997). In order to avoid distortion during the final stage of the drying and to obtain an even thickness of the coating layer, it was necessary to place a perforated plate over the mould. The coating layers were dried slowly at room temperature for two days to avoid excessive cracking.

The dry coating layers were cut to square test specimens (45 x 45 mm) and then ground to an even thickness with a sufficiently low surface roughness. The thickness variations were small in comparison with the total thickness of the test specimens, the coefficient of variation being about 0.1%.

Testing Methods

The compressibility of the coating layers was evaluated using an MTS-equipment (Material Testing System) as a platen press, the deformation of the test specimen in the thickness direction and the pressure applied to the surface were measured simultaneously as a function of time. The MTS-equipment was equipped with a pressure head that could be heated to a pre-determined and controlled surface temperature. In these trials, the temperature was either 23 or 150ºC.

The coating layers were subjected to a haversine pulse of 3 s with a maximum pressure of 30 MPa, after an initial pressure pulse of 80 kPa had been applied until temperature equilibrium was reached (1 to 3 minutes). During each compression measurement, the temperature of both sides of the coating layer was monitored. The coating layers were also subjected to a creep-recovery test, where a maximum pressure of 30 MPa was applied as rapidly as possible (~23 ms) and then kept constant for 350 s. The recovery of the coating layers was followed for another 350 s after unloading.

The softening behaviour of the pure latex films was evaluated with a Perkin Elmer Dynamic Mechanical Analyzer (DMA) (Hagen et al. 1994). A dynamic tensile force with a frequency of 1 Hz was applied to the latex film which had a thickness of about 60 μm. The storage modulus of the latex film was sampled every 1/3 of a degree from 25 to 200ºC. For some latex films, the measurement was interrupted before 200ºC was reached, due to extensive thermal softening.

The thickness of the papers was measured with an instrument developed at STFI (Fellers et al. 1986) and the grammage according to SCAN-P 6:75. The average paper gloss was evaluated according to Tappi T480 with a Lehmann instrument (LGDL-07-LABOR). All measurements were performed at 23°C and 50% RH.

The Composition of the Coating Layers

The compositions of the coating colours are given in ***Table 1***. The solids content of the coating colours was 63% and the pH was adjusted to 8.5 with NaOH.

***Table 1**. The coating formulations.*

	pph[b)]	Product name	Supplier
Calcium carbonate	60	Hydrocarb 90	Omya Plüss-Staufer
Kaolin clay	40	SPS	EEC International
Latex	12 [a)]	see table 2	Raisio Chemicals
CMC	0.8	Finnfix-10	Metsa Speciality Chemicals

a) an extra test series was made with coating layers having only 10 pph of the high T_g styrene-butadiene latex.
b) pph - weight parts/100 weight parts of pigment

Four latices with substantial differences in softening behaviour were used in these studies; a poly(vinyl acetate), a styrene-acrylate and two styrene-butadiene latices with a high and a low glass transition temperature, ***Table 2***.

***Table 2**. The latices used and their glass transition temperatures (T_g) as stated by the supplier.*

Notation	Component	Product name	T_g, ºC
S/B1	styrene-butadiene	Styronal 805	+25
S/B2	styrene-butadiene	Styronal 328	-19
PVAc	poly(vinyl acetate)	Raisional 1116	+28
S/A	styrene-acrylate	EP 5157	+28

Pilot Calendering

A woodfree base paper (MoDo Paper AB, Husum Paper Mill, Husum, Sweden) was coated with coating colours containing the four different types of latices (Wikström et al. 1999). The grammage of the base paper was 80 g/m^2. It had an ash content of 16% and was internally sized. The paper was coated once on each side at 900 m/min using a blade coater with an applicator roll in the pilot coater. The coating was performed at the Coating Technology Centre in Raisio, Finland and the coat weight was 12 g/m^2 on each side. The paper was then calendered in STFIs pilot calender with a soft nip (heated steel roll and polymer-covered 70 ºSh D roll) at temperatures of 37 and 145ºC. The line load was 315 kN/m and the machine speed 100 m/min.

RESULTS AND COMMENTS

The Thermal Softening Behaviour of the Pure Latex Films

Figure 1 shows the storage modulus of the pure latex films at different temperatures. The softening behaviour of the S/B2 diverged markedly from that of the other types. The S/B2 had a comparatively low storage modulus at room temperature and the modulus was only slightly reduced when the temperature increased. The S/B1, PVAc and S/A all had a higher storage modulus than S/B2 at low temperatures, but when the temperature reached about 40ºC these three films softened appreciably, leading to a higher storage modulus for the S/B2 at temperatures higher than 50 - 60ºC. Of these three, the S/B1 had the highest storage modulus, when the temperature exceeded 60ºC, followed by the S/A and the PVAc. When the temperature reached 120ºC, the S/A exhibited another drop in modulus. The modulus of the S/A-latex film could therefore not be measured at temperatures higher than 120ºC (due to the limitations in the resolution of the load cell). Similar behaviour was observed for the PVAc and the S/B1 at temperatures of 170ºC and 190ºC, respectively, but not for the S/B2.

The softening behaviour of the pure latex films can be considered as the potential for the different latices to contribute to a thermal softening of the coating layer (e.g. during temperature-gradient calendering with a hot soft nip). The S/B2 should thus have the lowest potential in this respect, since it exhibited no marked decrease in the storage-modulus in this temperature range.

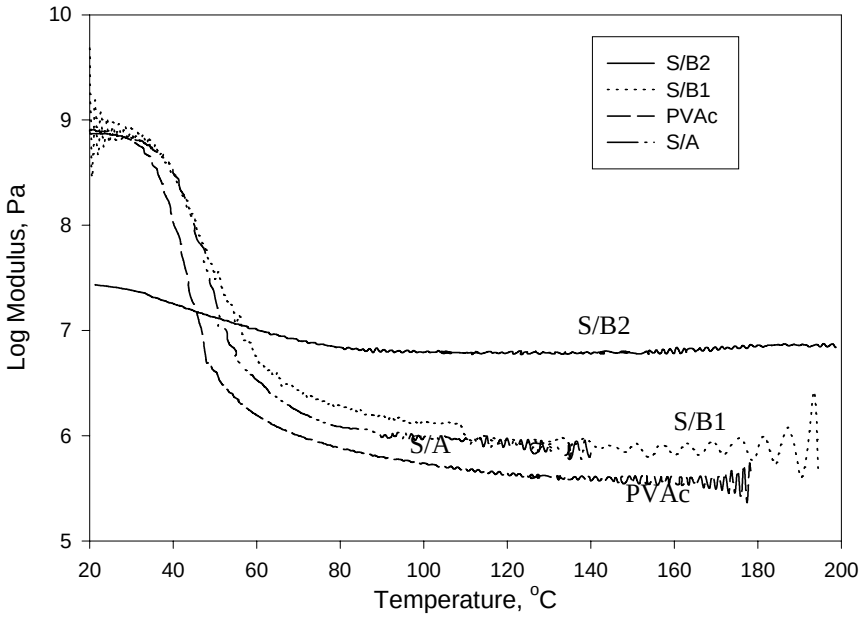

Figure 1. *The softening behaviour of the pure latex films illustrated by the temperature-dependence of the storage modulus at 1 Hz.*

The Compressive Behaviour of the Thick Coating Layers

It can of course be questioned whether or not the properties of the thick (1-2 mm) moulded coating layers are representative of those of the thin (5-20 µm) layers on paper products. The structure of the thin coating layers on paper probably differs in many respects from that of the thick layers, due in particular to the differences in the manufacturing process. The industrial paper coating operation involves the complicated flow of a pigment suspension at high shear rates and extensions. The drainage of the liquid phase into the base paper, the drying conditions and the degree of deformation during the process all affect the structure of the final coating layer, e.g. the binder distribution in the coating layer and orientation of the pigment particles (Engström 1994 and Gane et al. 1995). In order to check, at least to some extent, whether the thick coating layers are representative of the thin layers, their densities and porosities were compared. ***Table 3*** shows the densities and the porosities of the coating layers calculated from measurements of the grammage and the thickness of the test specimens, and the proportions and densities of the different components. The calculated porosity of the thick coating layers varied from 25 to 30%. The porosity of thin coating layers of similar composition has been reported to be in the range of 25-35% (Lepoutre and Hiraharu 1989, Eriksson and Rigdahl 1993). This would indicate that, in this sense, the thick coating layers are to some extent representative of thin coatings.

Table 3*. The density and porosity of the thick coating layers. The standard deviation (s.d.) refers to the difference between single test specimens within each group.*

Notation	Density, kg/m^3	s. d., kg/m^3	Calculated porosity, %
S/B1 (with 10 pph latex)	1626	10	30
S/B1 (with 12 pph latex)	1674	13	27
S/B2	1710	6	25
PVAc	1637	12	30
S/A	1691	9	27

Figure 2 shows the out-of-plane compression behaviour of the coating layer containing 10 or 12 pph of S/B1-latex when subjected to a load pulse with a maximum pressure of 30 MPa for 3 s at room temperature. The result of single loading cycles at different specimens is shown in the graph. Clearly the compression behaviour of the coating layers depended on the amount of latex. The coating layer with 10 pph S/B1-latex exhibited a higher deformation at a given stress than the layer containing 12 pph of the same latex; this was especially pronounced when the compressive stress exceeded 20 MPa. The compression modulus of the coating with the lower binder content was lower. This behaviour is due partly to a higher porosity (lower density) of the layer containing 10 pph S/B1 than of the layer with 12 pph latex. The densities were 1625 and 1675 kg/m^3, respectively, and more porous structures are simply mechanically weaker. In a sense, the compression behaviour parallels the behaviour in tension for pigmented coatings of this kind. An increase in the latex content improves the tensile strength and the tensile modulus in the plane (Parpaillon et al 1985). This behaviour was discussed in terms of a better stress transfer between the pigment particles in the coating structure at higher latex contents.

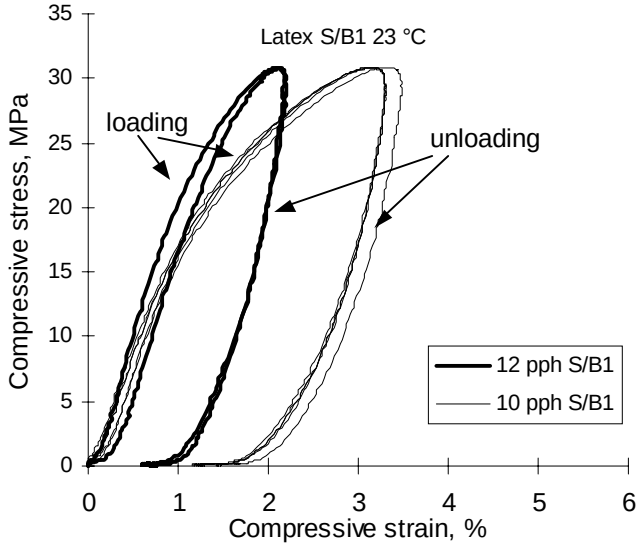

Figure 2. *The compressive stress as a function of the compressive strain at room temperature for coating layers with 10 and 12 pph of S/B1-latex.*

Figures 3a-d show the compression behaviour of the coating layers compacted at 23 and 150ºC. Three of the coating layers exhibited quite a pronounced deformation at the higher temperature.

The deformation behaviour of the two S/B-containing coating layers differed markedly from each other. In the case of the S/B1-containing coating layer, both the maximum and the permanent deformation (after unloading) increased about four times when the temperature was raised from 23 to 150ºC. In case of the S/B2-containing coating layer, neither the maximum nor the permanent deformation changed with temperature. Compared to the others, the pure S/B2 latex film also exhibited only a small change in the storage modulus when the temperature was increased, cf. Figure 1. There was obviously a relation between the out-of-plane compressive behaviour of the coating layers and the storage modulus of the pure latex films. The S/B2-containing coating layer was softer than the others, i.e. had a lower storage modulus and deformed more at a given compressive stress, at room temperature, which can be expected since the T_g-value of the latex polymer (-19ºC) was much lower than those of the others, 25 - 28ºC, Table 2.

The PVAc-film exhibited the strongest temperature dependence of the storage modulus. The corresponding coating layer was also stiffer than the others in compression (out-of-plane) at room temperature and exhibited the largest compaction at 150ºC, Figures 3a-d.

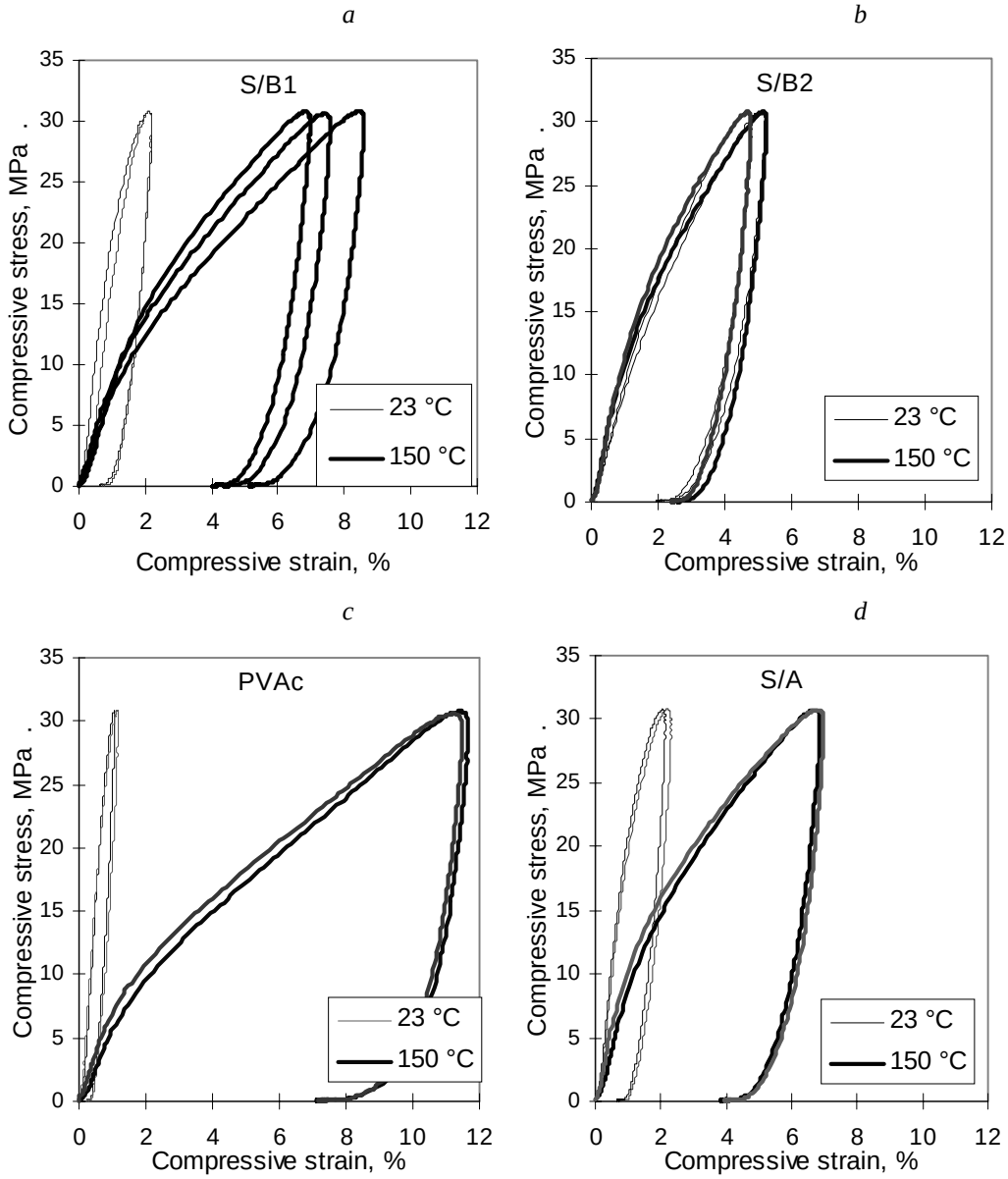

Figure 3a-d. *The compressive stress vs. the compressive strain for the coating layers containing the different types of latices at 23 and 150ºC. The graphs represent single loading cycles at different specimens.*

In order to simplify a comparison of the temperature dependence of the compression behaviour of the coating layers, the compressive stiffness during loading was determined, ***Figure 4***. The compressive stiffness is here defined as the slope of a linear regression of the loading curve in the stress range 0 - 30 MPa. Actually, a secant modulus is then used as a measure of the compressive stiffness. Again, it is very clear that the compression behaviour of the S/B2 layer was hardly influenced by the temperature, whereas an increase in temperature had a very strong effect on the stiffness of the layers containing the other latices.

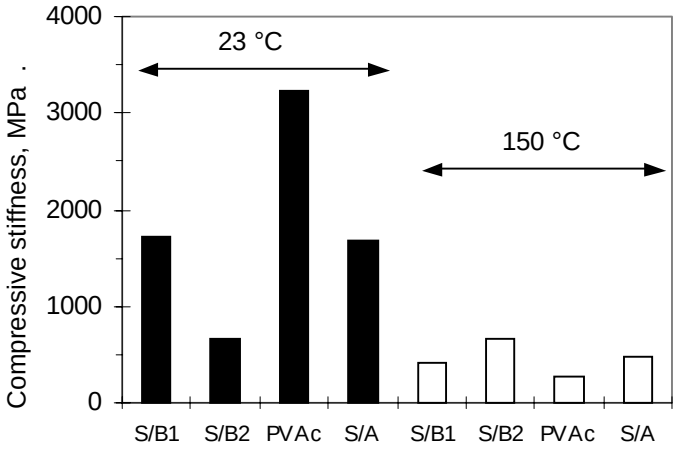

Figure 4. *The compressive stiffness of the coating layers at temperatures of 23 and 150ºC.*

Creep-Recovery Behaviour of the Thick Coating Layers

Creep-recovery tests can provide information on the deformation behaviour of a coating layer subjected to a short load pulse. These tests clearly revealed that the deformation of the thick coating layers was time-dependent. ***Figure 5b*** shows the compressive strain when the S/A-containing coating layer was subjected to the pressure pulse shown in ***Figure 5a***. The maximum pressure, 30 MPa, was applied as rapidly as possible, ~23 ms, and was then kept constant for 350seconds. The strain after 350 seconds was about 4% at a temperature of 23ºC and about 10% at 150ºC. These strains can be compared with the maximum compressive strains obtained with the haversine pulse (pulse time 3 s) shown in Figure 3d; 2 and 6%, respectively. For clarity it should be mentioned that the pulse times during calendering are significantly shorter than the times used here, of the order of a millisecond or shorter.

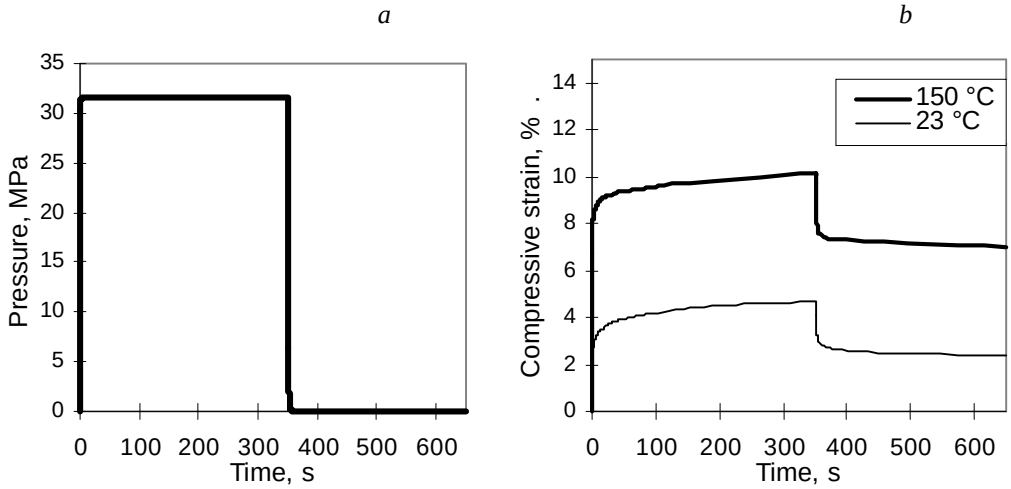

Figure 5a & b. *a) The applied pressure pulse during the creep-recovery test. b) The corresponding compressive creep behaviour of the S/A-containing coating layer at temperatures of 23 and 150ºC.*

In the creep-recovery test, the maximum pressure was applied within 23 ms. ***Figure 6*** shows the compressive strain 30 ms and 350 s after the application of the load, and also the maximum strain after approx. 1.5 s when the 3 s haversine pulse was used. The maximum pressure was 30 MPa in all cases. The coating layers exhibited a substantial creep deformation at both 23 and 150ºC. The compression was greater at the higher temperature even

after 30 ms loading, except for the S/B2-containing coating. This is an important observation in this context, since the dwell time in a calender nip is of the order of milliseconds or less.

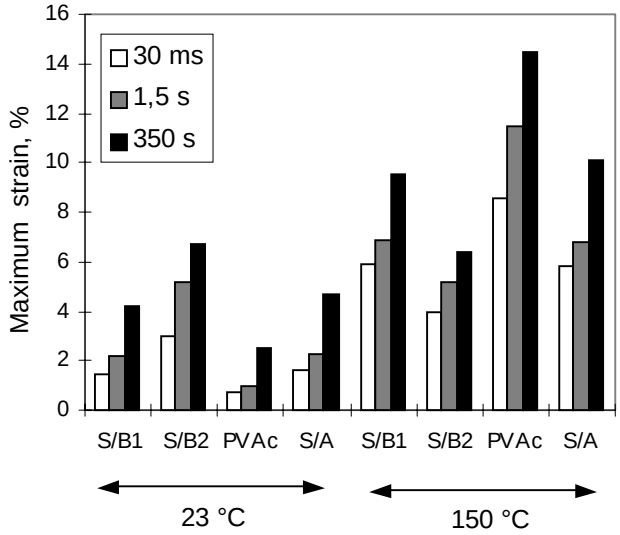

Figure 6. *The compressive strain of the coating layers after 30 ms, 1,5 s (haversine pulse) and 350 s loading, respectively. The applied pressure was 30 MPa in all cases.*

Influence of the Surface Roughness on the Compressive Behaviour

Besides influencing properties such as stiffness, gloss and brightness, the mass distribution and the surface roughness of the coating layer affect its deformation behaviour in a calender nip. The cross-section of a paper coated with the PVAc-containing coating colour (***Figure 7)*** clearly illustrates that there is a substantial variation in both the thickness and the topography of the coating layer.

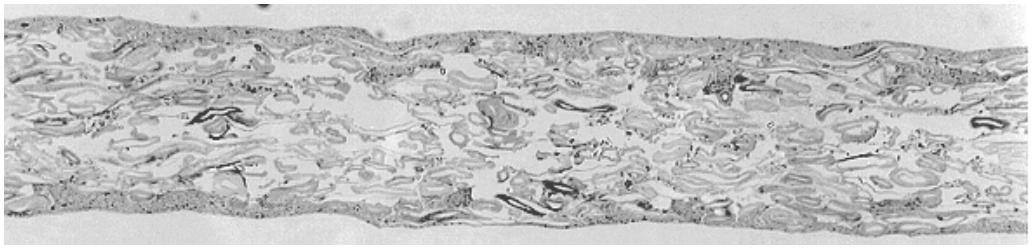

Figure 7. *A cross-section of the woodfree paper coated with the PVAc-containing coating. The coat weight was 12 g/m^2 on each side of the paper.*

Thin coating layers with a thickness of about 50 µm were also produced. ***Table 4*** shows the coefficient of variation of the thickness for the thick and the thin S/B1 coating layers and also for the paper coated with the S/B1-containing coating colour. The thin moulded coating layer had a coefficient of variation of the thickness, which was of the same order of magnitude as that of the coated paper.

***Table 4**. The average thickness and the corresponding coefficient of variation of the thick and thin moulded coating layers, and of the coated paper, when the S/B1 latex was used as binder.*

	Average thickness, µm	**Coefficient of variation, %**
Thick coating layer	1696	0.12
Thin coating layer	50	4.9
Coated paper	109	4.6

There are of course large and important differences between the thin moulded coatings and the coated papers with regard to how they deform when subjected to a pressure pulse. The compressive behaviour of a coated paper is probably in the first place determined by the mechanical properties of the base paper, and the boundary layer between the coating and the base paper is also of importance. Nevertheless, model experiments can be useful to clarify the importance of the surface roughness for the deformation behaviour in the nip.

Figure 8 shows the behaviour when a thin and the thick coating layer have been exposed to a series of five haversine pulses of 3 s. The temperature of the pressure head and of the coating layer was 23ºC and the maximum pressure was 30 MPa. The thin coating layer in Figure 8 exhibited a much higher deformation during the loading from the first pulse than in the subsequent four pulses. In the case of the thicker coating layer, there was only a small difference between the compressive strain from the first pulse and that from the following pulses. This observation emphasizes that when a coated paper passes through a series of nips, i.e. in a supercalender or in a multi-nip soft calender, most of the reduction in the surface roughness and increase in the gloss is achieved in the first nip (Lee 1982 and Morin et al. 1996).

When a coated paper is compressed in the first nip, the applied load is concentrated to the topographic peaks of the coating layer. The large strain associated with the first pressure pulse is due to the preferential deformation of the peaks in the coating surface, i.e. to a reduction of the surface roughness. This pronounced deformation takes place irrespective of the temperature and the large deformation of the coating layer during the first load pulse may appear to be less affected by the temperature than the deformation caused by the subsequent pressure pulses. As a consequence, the first nip should perhaps be softer than the forthcoming nips in a multi-nip calender in order to reduce stress concentrations that cause gloss and porosity variations in the coating layer.

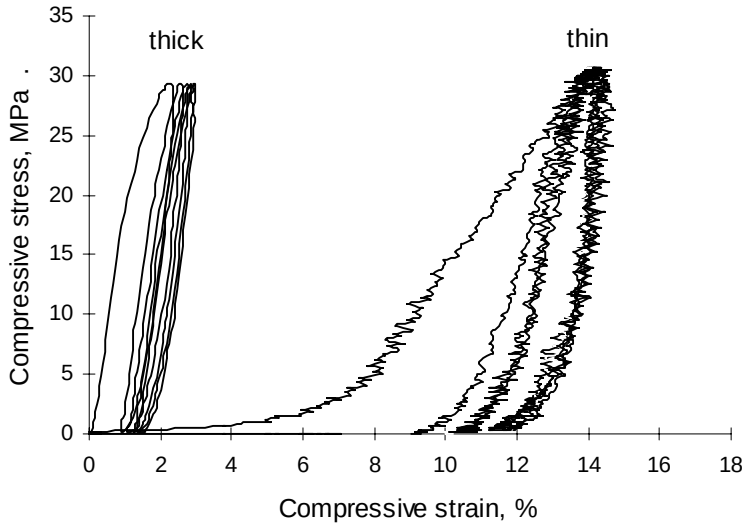

***Figure 8.** The compressive stress vs. the compressive strain for the thin and thick S/B1-containing coating layers compressed in a series of five haversine pulses of 3 s at room temperature. The maximum stress was 30 MPa.*

A Comparison with Pilot Calendering

Woodfree papers coated with the coating colours that contained the four types of latices were pilot calendered. The calendering conditions were as far as possible adapted to correspond to the conditions used in the platen press experiments. The line load was 315 kN/m and the papers were calendered in a soft nip (one passage). The machine speed was 100 m/min and the temperature of the heated roll was 37 or 145ºC. Under these conditions, the maximum pressure and the dwell time in the nip are estimated by Hertz contact theory to be 22 MPa and 11 ms, respectively.

Figure 9 shows the paper gloss of the coated papers before and after calendering. The gloss is here considered to be an indication of the degree of surface compression of the coated papers. When the paper was calendered at 37ºC, the paper coated with the S/B2-containing coating colour had the highest gloss and the paper coated with the PVAc-containing coating colour had the lowest. This is in accordance with the measured stiffness of the coating layers in Figure 4 (cf. also Figure 1). At room temperature, the S/B2- and the PVAc-containing coating layers had the lowest and highest stiffness respectively. When calendered at 145ºC, the gloss of all the papers increased, the lowest gloss being obtained with the paper with the S/B2-containing layer, that also had the highest stiffness at higher temperatures (cf. Figure 4 and Figure 1).

There is evidently a relation between the thermomechanical properties of the pure latex film and the compression behaviour of the coating layer in a heated calender nip.

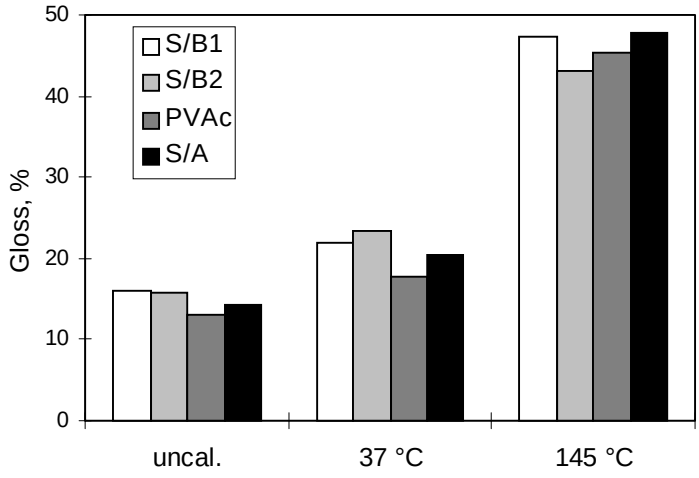

Figure 9. *The paper gloss before and after calendering. The paper was calendered in a soft nip at a roll temperature of 37 or 145ºC.*

FINAL REMARKS

The measurements were here performed with an even temperature distribution in the coating layers. In high-temperature calendering in practice, the situation is more complicated. The temperature is not evenly distributed due to the short dwell time in the calender nip and to the thickness irregularities of the coating layer. Nevertheless, this work reveals that there is a connection between the thermomechanical properties of the latex binder and the compressive stiffness of the coating layer that significantly affects the deformation of the coating layer in the calender nip.

The woodfree papers coated with coating colours containing the four types of latices were also test printed after being calendered under different conditions. The print result and the paper surface properties are described elsewhere (Wikström et al. 1999). It was found that the type of coating latex affected the print result and the surface properties not only by its influence on the calenderability, but also by its effect on the coating structure. The latter effect is not directly related to the calendering operation. Nevertheless, a proper understanding of the relation between the thermomechanical properties of different coating binders and their effect on the response of the coating layers to high temperature-gradient calendering is certainly important.

The deformation behaviour of coating layers compressed at high temperatures is also of interest for the development of constitutive models of coated layers. Such models should be very useful when attempts are made to control the deformation of the surface layers during calendering. However, to achieve such a model for coating layers more experiments have to be performed. The in-plane material properties of the coating layers, for example, have to be characterised. Material testing in-plane also provides an opportunity to evaluate the effect of the degree of anisotropy on the deformation behaviour and also differences in material properties between tensile and compressive loading.

A more fundamental method of modelling the mechanical response of a coating layer would be to use a micro-mechanical approach taking into account the main components of the coating layer; pigment, air and binder. The development of a micro-mechanical model would start from evaluating the constitutive behaviour of a pigment and a latex. The evaluation may be facilitated by the fact that the pigment (at least some of the pigments used) and the latex are probably mechanically isotropic. A second step would then be to determine the configuration of the pigment and the latex when a coating layer is formed. This means that the roles of the particle size distribution, the orientation of particles and the latex distribution need to be clarified. In the last step, a micro-mechanical FEM-model (Finite Element Method) would be developed based on the geometry from the second step and the constitutive behaviour from the first step. The FEM-model of the coating layer can then be subjected to a loading case that can be experimentally followed, i.e. the compressive test performed in this work.

ACKNOWLEDGEMENTS

The authors thank Raisio Chemicals, especially Jan-Erik Theirfolk and Jörgen Lindholm, for their scientific and experimental support. Sune Karlsson, Jolanta Borg and Anne-Marie Olsson are thanked for their experimental assistance and Dr J. A. Bristow for the linguistic revision of the manuscript.

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Keywords
Binder, calendering, coated paper, compression, latex, surface structure

Received for review: February 20, 1999
Accepted: January 27, 2000

This paper was accepted for abstracting and publication in the August issue of TAPPI Journal.