# A method for measuring the in-plane compressive strength and the compression behavior of coating layers

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**ABSTRACT:** Cracking at the fold is a quality defect sometimes observed in coated paper and board. Although tensile and compressive stresses occur during folding, test methods to measure the compressive strength of a coating have not been available. Our objective was to develop a method to measure the compressive strength of a coating layer and to investigate how different mineral coatings behave under compression. We used the short-span compressive strength test (SCT) to measure the in-plane compressive strength of a free coating layer. Unsupported free coating films were prepared for the measurements. Results indicate that the SCT method was suitable for measuring the in-plane compressive strength of a coating layer. Coating color formulations containing different kaolin and calcium carbonate minerals were used to study the effect of pigment particles' shape on the compressive and tensile strengths of coatings. Latices having two different glass transition temperatures were used. Results showed that pigment particle shape influenced the strength of a coating layer. Platy clay gave better strength than spherical or needle-shaped carbonate pigments. Compressive and tensile strength decreased as a function of the amount of calcium carbonate in the coating color, particularly with precipitated calcium carbonate. We also assessed the influence of styrene-butadiene binder on the compressive strength of the coating layer, which increased with the binder level. The compressive strength of the coating layer was about three times the tensile strength.

***Application:*** Knowing the compressive and tensile strengths of a coating layer gives a better understanding of the response of coated paper to folding.

Cracking at the fold is an aesthetic defect that can occur when a coated paper or paperboard is folded. This defect may be merely cosmetic, but it can lead to complete paper failure at the location of the fold. When the paper is folded, the coatings on the inside and outside of the fold are subjected to different stress levels. The outside of the fold is subjected to a tensile stress, whereas the inside of the fold undergoes compression.

Many studies of the cracking mechanism at the fold have been published. Colley [1,2] investigated the crease-cracking tendency of lightweight coated magazine papers. Guyot et al. [3] presented four different models of behavior of coated papers depending on the degree of damage after folding. They suggested that fold-crack resistance is influenced by the coating penetration into the stock and by the mechanical characteristics of the coating inside the fold. Barbier et al. [4] investigated the folding of coated paper numerically using a finite element method. Salminen et al. [5] studied the balance between fold cracking and bending stiffness using a finite element model. Jopson and Towers [6] suggested that cracking is likely to occur in the outer layer if the compressive strength of the sheet, especially of the coating layer, is too great in relation to its tensile strength. When cracking at the fold is a problem in coated papers, strength properties of the coating layer influence cracking. Therefore, it is important to know how the coating composition affects the compressive and tensile strengths of the coating.

On a macroscopic level, the coating layer is a composite; hence, the strength depends on the mechanical properties of the binder, the pore structure, and the total adhesive area between binder and pigment. Water and solvents can weaken the structure of the coating layer by changing the mechanical properties of the binder or by weakening the interfacial bonding between the pigment and binder. On a microscopic level, the coating layer is composed of pigment-binder-pigment adhesive joints. The strength of these individual joints depends on the mechanical properties of the binder and on the surface chemical properties of the binder and the pigment.

Some studies regarding coating layer strength have been published. Parpaillon et al. [7] investigated the in-plane tensile strength of kaolin and ground calcium carbonate coating layers. They found that the tensile strength increased as the amount of latex increased. Kaolin gave a tensile strength twice as high as calcium carbonate, at the same latex level.

Lepoutre and Rigdahl [8] related the stiffness of a coating layer to the coating porosity and pigment shape. They studied a wide range of pigments of different shapes and discovered that isometric ground calcium carbonate decreases the elastic modulus more than the needle-shaped mineral, and that the stiffness of the coating layer is determined by the coating po-

| Pigment | Type | Shape | Median, d50, µm (Coulter LS) | Median, d50, µm (SediGraph 5100) |
|---|---|---|---|---|
| Capim SP (Imerys) | Kaolin | Platy | 2.632 | 0.6 |
| Hydrocarb 60-LV (Omya) | Ground calcium carbonate | Round | 1.992 | 1.46 |
| Opacarb A40 (Speciality Minerals) | Precipitated calcium carbonate | Aragonite | 0.681 | 0.42 |

***I. Particle size of the pigments used in the experiments. (Imerys, Paris, France; Omya, Oftringen, Switzerland; Specialty Minerals, New York, NY).***

rosity and the elastic moduli of the binder and pigment. Inoue and Lepoutre [9] and Okomori and Lepoutre [10] studied the out-of-plane strength of a coating layer. They found that the z-direction strength increases with increasing pigment size and increasing latex content. Husband et al. [11,12] studied the effect of pigment particle shape on the tensile strength of a coating layer. They found that the stiffness and in-plane tensile strength increase as the shape factor of the clay particle increases.

Most of the studies dealing with coating layer strength consider the effect of pigment and binder on tensile strength. The strength of a coating layer depends on the pigment used in the coatings, and on the volume of binder in the coating. Because latex is more expensive than inorganic pigment, the amount of latex used generally is kept as low as possible. As a result, other properties, such as pigment shape, glass transition temperature of latex, or porosity of the coating layer become more attractive for affecting strength.

Jopson et al. [6] suggested that the short-span compressive strength test (SCT) method could be used to measure the compressive strength of a coating layer, but this was not tested. Alam et al. [13] measured the compressive strength of a coating color, but they used a cubic sample for the measurements. SCT is a tool to measure the compressive strength of paperboards, but it also can be used with thinner paper samples. The challenge during this measurement is to prevent buckling of the test piece during a test using a very short span, usually 0.7 mm. Because of the arrangement of a sample, the SCT is considered to be a more reliable test method for compressive strength determination [14].

## MATERIALS AND METHODS

We divided our experiments into two parts. In the first part, we developed a suitable method for making free-standing coating layers for our tests and tested to see whether the SCT method was suitable for measuring the compressive strength of the coating layer. In the second part, we studied the effect of the choice of pigments and styrene-butadiene binder glass transition temperature ($T_g$) on compressive strength.

### *Materials*

The type of pigment, binder choice, and amount influence the strength of a coating layer. These were the main variables considered when choosing the materials for our tests. A focus of the study was on investigating how the pigment shape affected the compressive strength of the coating layer.

| Latex | Company | $T_g$ |
|---|---|---|
| DL980 | Styron | -7°C |
| DL966 | Styron | 20°C |

***II. Glass transition temperatures of lattices (Styron, Berwyn, PA, USA).***

We selected the pigments so that their sizes and size distributions were as similar as possible and only their shape was different. However, finding a precipitated calcium carbonate with the same size as the other inorganic pigments was difficult. We chose one grade in which the length of the individual particles was similar to that of a typical general-purpose kaolin. A scanning electron microscope was used for this selection. At this stage, only a visual evaluation was made. **Table I** summarizes the median sizes of the pigments. The difference in the kaolin medians is due to the shape of the kaolin. According to the manufacturer (Imerys; Paris, France), the aspect ratio of the kaolin was 25.

To study the effect of each binder's $T_g$, we selected two functionalized carboxylated styrene-butadiene latices (**Table II**) differing mainly in the styrene/butadiene ratio. We used 10 parts of latex per 100 parts of pigment. Sodium carboxymethyl cellulose was used as a thickener in all the coating colors.

### *Methods*

Coating color layers were created using an RK coater (RK PrintCoat Instruments; Royston, Hertfordshire, UK). The substrate chosen was cellophane with a thickness of 20 µm. The film was easy to separate from the substrate after drying. A problem was that the coating layer had a tendency to curl during drying. The dry coating thickness was about 100 µm. This was achieved by applying a wet film with a thickness of about 200 µm. The dry coating layer had to be thick enough for the compressive strength test without buckling. We calculated that a 100 µm film was sufficient for the mea-

surements. Films were dried for 3 min under a hot air stream (190°C) and then left at room temperature for at least 2 h. After drying, the cellophane was peeled off, the coating films were cut into 15 mm wide strips, and the thickness of each strip was measured.

The compressive strength was measured in an Lorentzen & Wettre (AB Lorentzen & Wettre; Kista, Sweden). compressive strength tester. The compression rate was 3 mm $min^{-1}$ and the free span was 0.7 mm. The tester gave the maximum compressive force per unit width of the test piece expressed in kN $m^{-1}$. To obtain the compressive strength as a force per unit area (MPa), the measured value was divided by the thickness of the test piece. Twenty measurements were made for each sample to calculate a mean value and standard deviation.

The tensile strength was measured using 15 mm wide strips and an extension rate of 10 mm $min^{-1}$. The strength was calculated by dividing the load (N) at break by the cross-sectional area of the sample. For each sample, the tensile strength was measured 10 times to calculate the mean value and standard deviation. All the experiments were carried out at 23°C and 50% relative humidity. (For this report, coating strength exclusively refers to the tensile and compressive strength, not coating picking strength.)

## RESULTS

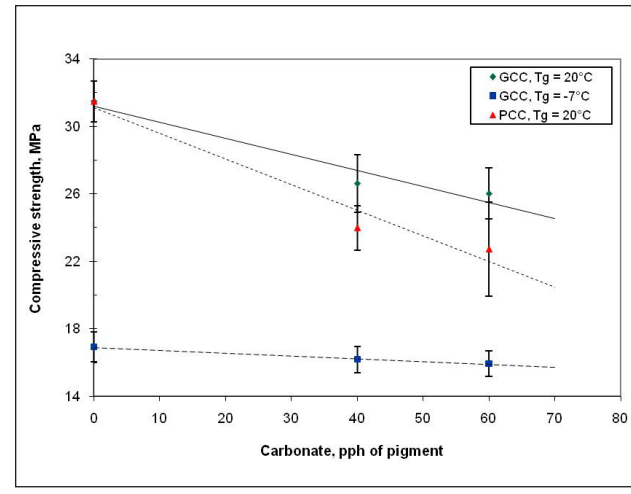
### *Test method for compressive strength*

To our knowledge, no earlier attempts have been made to measure the compressive strength of a coating layer using the SCT method. Our first step was to determine the reliability and reproducibility of the SCT method. We made three different sets of the coating layers, measured the compressive strength of each set, and compared the results (**Table III**). Statistically, the mean values were not significantly different, indicating good test reliability.

### *Compression behavior of coating layers*

Having found the SCT method to be suitable and reliable, we used it to measure the compressive strength of coating layers with different pigments and latices. We also measured the tensile strength of the coating layer to compare it to the compression strength and see if they behaved in the same way.

Plate-like kaolin pigment gave a stronger coating layer than spherical or needle-like calcium carbonate pigment. The results (**Fig. 1**) demonstrated that a pure kaolin coating had high compressive strength, and that addition of calcium carbonate to a coating color reduced strength. The compressive strength decreased considerably when the relative proportion of calcium carbonate increased. The needle-like precipitated calcium carbonate (PCC) gave a lower compressive strength than the spherical ground calcium carbonate (GCC).




***1. Compressive strength as a function of carbonate in coating. Latex level 10 pph by weight of pigments. Error bars are one standard deviation.***

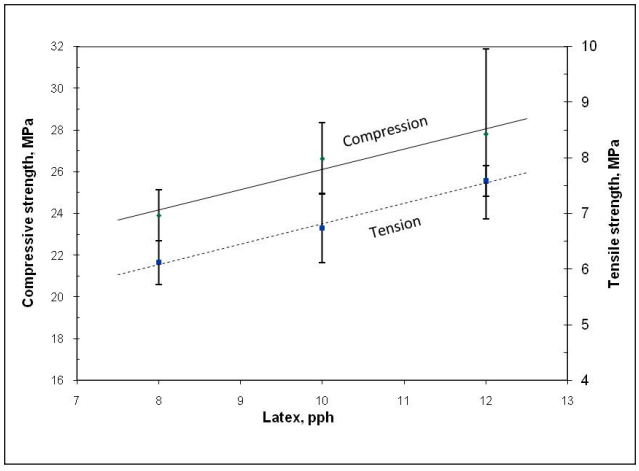
The results demonstrated that the latex with a high $T_g$ gave high compressive strength (Fig. 1). The harder latex (i.e., latex with high $T_g$) film was subjected to larger changes under the compressive stresses than the soft latex when calcium carbonate was added to the coating color.

The binder in a coating color holds pigment particles together, binds the coating to the paper, and makes the coating layer strong. When the binder level increases, the strength of the coating layer also should increase. **Figure 2** shows that the in-plane compressive and the tensile strength increased linearly when the latex amount was increased from 8 pph to 12 pph.

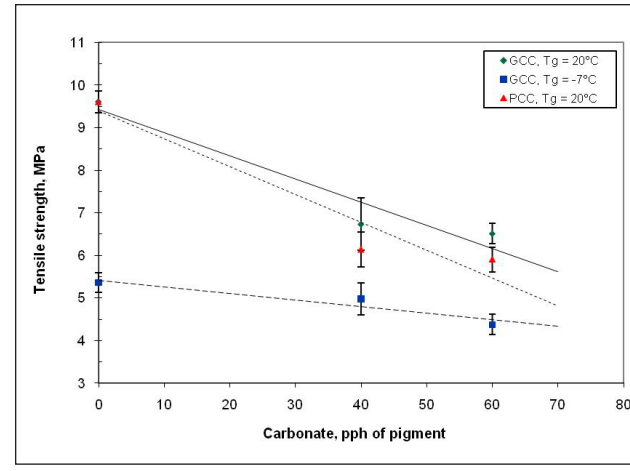
The tensile strength of the coating film was studied to determine whether the coating behaved the same as under compression. **Figures 1-3** show that the tensile strength with added calcium carbonate was similar to compressive strength. The platy kaolin gave the highest tensile strength, as expected from previous works. The tensile strength decreased when calcium carbonate was introduced into the coating color. The effect on tensile strength was significant up to about 40 pph of calcium carbonate. After that level, the ten-

| | Measurement 1 | Measurement 2 | Measurement 3 |
|---|---|---|---|
| Compressive strength, MPa | 30.42 | 30.57 | 30.20 |
| Standard deviation, MPa | 2.49 | 1.58 | 2.32 |

***III. Mean and standard deviation of three independent sets of measurements.***




*2. Compressive and tensile strength as a function of latex content. Kaolin 100 pph, latex $T_g$ = 20°C.*

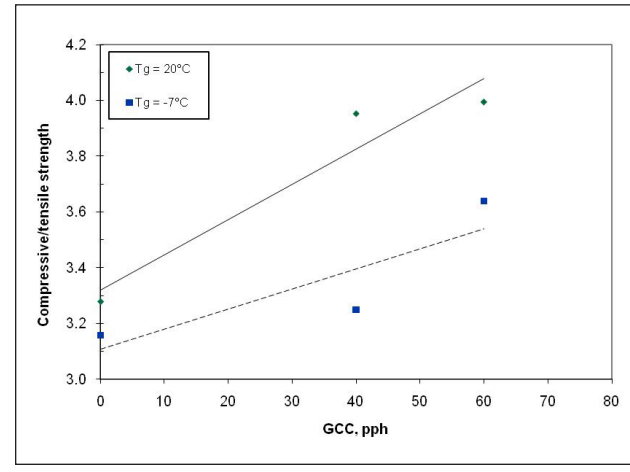



*3. Tensile strength as a function of carbonate in the coating. Latex level 10 pph by weight of pigments.*

sile strength reached a plateau; further increasing the carbonate had little effect on strength.

As with the compressive strength, the harder latex gave a stiffer coating layer than the softer latex. With the harder latex, the drop in tensile strength when carbonate was added to the coating was greater than with the soft latex.

**Figure 4** shows the ratio of compressive to tensile strength. Results illustrated that the compressive strength of the coating layer was three times higher than the tensile strength with kaolin-based coatings. The increase in the ratio with calcium carbonate added to the coating occurs because the compressive strength decreased less than the tensile strength.




*4. Ratio of compressive strength to tensile strength. Latex level 10 pph by weight of pigments.*

## DISCUSSION

This study has shown that it is possible to make free coating films with some limitations, and that the SCT method, which is normally used for paperboard, is suitable for measuring the in-plane compressive strength of the free coating films. The small standard deviation from replicated sets of measurement demonstrated that the method is reproducible and that the results obtained can be considered reliable.

In-plane compressive strength was highest for the kaolin-based coating and decreased when calcium carbonate pigment was added. According to earlier studies [11,12], platy kaolin particles give a stronger coating layer, with respect to tensile strength, than does a blocky or spherical particle pigment. Results from our study show that platy kaolin presumably creates a structure that can best support compressive load. In the blade coating process, the blade geometry influences the coating layer properties [15,16]. The kaolin particles can become orientated along the machine line (in-plane) direction and create a compact structure with a low void fraction. Tensile strength will increase if the kaolin particles are orientated in the in-plane direction, but poor alignment of kaolin particles will reduce the tensile strength [17].

Our results demonstrated that the particular calcium carbonates evaluated reduced the in-plane compression strength as they were introduced to the coating color. The effect was similar with GCC and PCC grades, but with needle-like PCC, the decrease was greater. GCC consists of more or less spherical particles. When GCC is added to the coating, the kaolin cannot pack tightly. The pore size and pore volume of uncalendered coatings is highest with a blend of 70% clay and 30% GCC [18]. This means that the pore size, and also the pore shape, might affect strength. Pore size and shape might also account for the substantial drop in compressive strength, when the specific type of calcium carbonate is added to the coating color. With an addition of up to 40% calcium carbonate, the drop in strength was comparatively large. However, further experimental data are needed to explore the breadth of this conclusion. This means that the structure of a pure kaolin coating is strong, but that the strength can easily be lost by adding other types of pigment to a coating.

The results from our study showed that the particular PCC used gave a lower strength than GCC. That might be explained by the poor packing of the needle-like PCC particles, which gives a more porous structure. A similar result was demonstrated by Okomori and LePoutre [10], who showed that acicular PCC gave a higher void fraction than prismatic PCC particles. The z-direction strength was less with acicular PCC than with prismatic PCC [10]. PCC particles are more fragile than GCC particles, and this may lower the tensile strength of the coating layer. The needle-like shaped PCC particles might need more binder than do spherical GCC particles to create as strong a structure. More binder would create a less porous structure and give greater strength.

The strength of a coating layer depends on the pore structure, the association between a pigment and a binder, and on the binder properties. Earlier studies [7,11] showed that the type of binder and the amount of binder have a great effect on the strength, and that the tensile strength increased when the amount of latex was increased. Results from our study show that the compressive strength also increases when the amount of latex is raised. More latex particles provide more binding power so that the pigment particles are better bound together and strength increases. The latex amount also affects the porosity of the coating layer. More latex better fills coating pores, and thus the strength is greater.

The glass transition temperature greatly affects the tensile strength of the coating layer [7]. The tensile strength increases when $T_g$ increases. In our study, the compressive strength increased with $T_g$. Even a small change in $T_g$ had an influence on the compressive strength. Latex with a lower $T_g$ creates a ductile and elastic film, which means that by choosing the latex carefully, the desired strength and balance of all other properties in the coating can be obtained.

The harder latex had a greater effect on the compressive strength when calcium carbonate was added to a kaolin-based coating. Changes when calcium carbonate was introduced were greater than with the soft latex.

The compressive strength of the coating layer was about three times its tensile strength. The ratio of compressive to tensile strength was smallest with an all kaolin coating. Adding more carbonate to a coating color increased the ratio of compressive to tensile strength because the carbonate reduced the compressive strength proportionately less than the tensile strength. The shape of the pigment particle had more effect on in-plane than z-direction tensile strength [19]. The aligned platy coatings gave high in-plane strength. However, the strength was rapidly decreased when specific carbonates were introduced to the coating color. We hypothesize that aligned platy coatings have more resistance to compression strength than round GCC, but are weaker under tensile stress.

To avoid a cracking problem when folding the paper, the compressive strength of a coating should not be too high in relationship to its tensile strength. As our results showed, calcium carbonate affects the compressive strength less than the tensile strength. The inside of the fold determines resistance to cracking at the fold [3]. Knowing the compressive and tensile strengths of the coating layer could make it possible to optimize the coating layer mechanical properties in favor of folding. Further study is needed to determine the relation between these strengths and the cracking at the fold.

## CONCLUSIONS

We have developed a method to measure the in-plane compression strength of a coating layer. Our results showed that the SCT method can be used with free standing coating layers. The accuracy and reproducibility of measurements were good.

Our study further shows that pigment shape has a substantial effect on the compressive behavior of a coating layer, which confirms earlier findings. The specific calcium carbonate grades used in our study decreased the compressive strength more than the tensile strength. Platy kaolin pigment gave the best in-plane compressive strength for the pigments we studied. Use of calcium carbonates in a coating color reduces the in-plane compression strength. An effective way to change the compressive strength is by changing the latex $T_g$. The compressive strength increases when $T_g$ rises. $T_g$ also affects the stiffness of the coating layer; higher $T_g$ gives a stiffer layer. **TJ**

## ACKNOWLEDGMENT

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## ABOUT THE AUTHORS

We chose this topic because we did not find published research about in-plane tensile and compression strength of coating color, although they both have important roles in fold cracking of coated paper. This research shows the behavior of the coating layer on compression and supports the findings made in previous research about in-plane tensile strength of the coating layer.

The most difficult aspect of this research was how to show the different behaviors of individual pigments under tension and compression. This was done by choosing common pigments with different shapes and mixing them together in specific proportions.

One of the most interesting findings was discovering the different behaviors of pigments on tension and compression (i.e., how pigment shape affected the coating layer tensile and compressive strengths).

The results of this study will help mills to know how the different pigments and binders affect the strength of the coating layer and potentially adjust the coating color recipes to avoid cracking at the fold. The next step is to study the relation between tensile and compressive strength and cracking at the fold.

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