Z-directional testing of paperboard in combined tensile and compression loading

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ABSTRACT: The out-of-plane properties of paperboard are important in several converting applications such as printing, sealing, creasing, and calendering. A juxtaposed tensile and compression curve in the z direction (ZD) will, however, appear to have a kink or discontinuity at 0 stress.

The purpose of the present work is to capture the continuous transition between tension and compression and to increase the understanding of the complex ZD properties of paperboard by cyclic testing. In this attempt to unify the ZD tensile and compressive behavior of paperboard, samples were laminated to the testing platens using heat seal laminate film. The method for adhering the samples was compared to samples that were laminated and glued to the testing platens. The edge effects of the cutting method were evaluated in compression testing with samples not attached to the testing platens. The flat slope seen in the initial part of the pure compression curve disappeared when the samples were laminated to the testing platens. The flat slope was instead replaced by a continuous response in the transition across 0 N. The stiffness in the transition region resembled the response in tensile testing. When the testing is cycled, the material exhibits a history dependence. Starting the cycle in either compression or tensile will show an effect on the stiffness at the transition, as well as the compressive stiffness. However, the ultimate tensile strength is unaffected.

Application: This study shows the material response of paperboard in the z direction to combined tensile and compression testing.

Although paper is occasionally treated as a two-dimensional material, converting applications such as printing, sealing, or calendering require the consideration of material properties in the out-of-plane direction of the sheet. The present study will focus on the material response in tension and compression. The compressive behavior is important for printing applications, where it affects the print quality, as well as for calendering, where the goal is to change the surface and or thickness of the material. The z direction (ZD) tensile behavior, especially ZD strength, comes into play in printing techniques such as offset, where tacky ink can cause delamination after the print nip shown by, e.g., Vaha-Nissi et al. [1]. The ZD tensile strength also plays a role in tape and glue applications, where it can be the ultimate limiting factor for the strength of a seal, i.e., when the joint is stronger than the delamination strength of the material. Further, the ZD strength likely has a role to play in delamination seen during creasing and folding, as well as during in-plane compression of thicker paperboards, to an extent.

In any tensile measurement method, the effect of the sample fastening needs to be considered. In the case of ZD tensile measurements of paper, the paper material is adhered to the flat surface of a testing platen. The adhesive strength must be larger than the internal strength of the paper tested. The influence of the glue or adhesive and its possible penetration into the paper structure must also be considered. The testing platens are fastened in a testing machine and pulled apart while the force is recorded. The force measurements provide the ZD strength of the material. Depending on the specific method, it is common to also attach an extensometer to the setup and record the displacement. By recording the displacement as well as the force, the ZD stiffness can be characterized together with the overall stress-strain behavior. Three methods can be worth noting here. First, the ZD tensile strength can be measured with methods detailed in standards such as SCAN-P 80:98 "Z-directional tensile strength" and TAPPI Standard Test Method T 541 "Internal bond strength of paperboard (z-direction tensile)." These methods use an adhesive tape between two plates that are separated while the force is recorded. This method can be automated and is typically used in auto-liner measurements. It is suitable for weaker paperboards and only indicates the strength of the material.

A second way is to use photo-mounting tissue instead of adhesive tape between the paper and the testing plate. The testing piece comprised of testing plates, photo-mounting tissue, and paper are cured in a hot air oven at 110°C for 1 h under a pressure of 0.23 MPa. After curing, the testing piece is conditioned for a minimum of 12 h in standard climate, which is deemed sufficient for moisture equilibration as shown by Girlanda and Fellers [2]. A drawback of

the method is the photo-mounting tissue that has both its own fiber structure and is heat and pressure sensitive, i.e., requires an even pressure during curing, which can be difficult to achieve.

A third way is to use heat-seal laminate film on the paper samples. The laminate film surface is then adhered to the testing platens by soaking a dummy paper (commonly tissue of some kind) in low viscous cyanoacrylate glue and letting the testing piece cure for 1 h under a small weight, a method described and evaluated by Andersson and Fellers [3]. The samples must be cut at least twice. First, the paper-laminate film combination must be cut in a size larger than the testing platen before gluing to prevent the low-viscous glue from permeating the paper on the sides of the test piece. After curing, the edges must be trimmed around the test piece. The method is time consuming and difficult to automate due in part to the many steps required to prepare each test piece. Some additional shortcomings of the lamination method concerning the possible permeation of the heat seal laminate and the heat transfer in a commercially available laminator has been discussed by Lucisano and Pikulik in an evaluation of the method [4]. Despite the shortcomings, the laminate method allows for testing samples with high ZD strength, while ensuring that the influence of the adhesive is minimized.

In the present study, ZD tensile tests are performed using the laminate method to establish a baseline for the tensile response of the paperboard. This study aims to combine tensile and compression measurements, and therefore the method used to adhere the samples to the testing platens had to be tailored to the test protocol and setup. The glue and dummy paper layers in the laminate method are removed, and the test piece comprised of only metal testing platens, laminate film, and paper are cured in an oven. Additionally, no pressure other than the weight of the upper testing plate is imposed during curing of the test piece.

The ZD tensile behavior of paperboards aimed at, e.g., liquid packaging, characteristically shows a linear elastic response followed by a short nonlinear behavior prior to peak strength and a cohesive post peak behavior. The initial response usually has an elastic modulus around 1%–10% of the in-plane ditto, as reported by Niskanen [5]. The initial stiffness is mainly dependent on network and fiber properties; the strength is mainly determined by the strength of the fiber-to-fiber joints rather than fiber strength; and the post peak behavior is mainly governed by fiber pullout (remaining bonds and friction) and is thus dependent on fiber length.

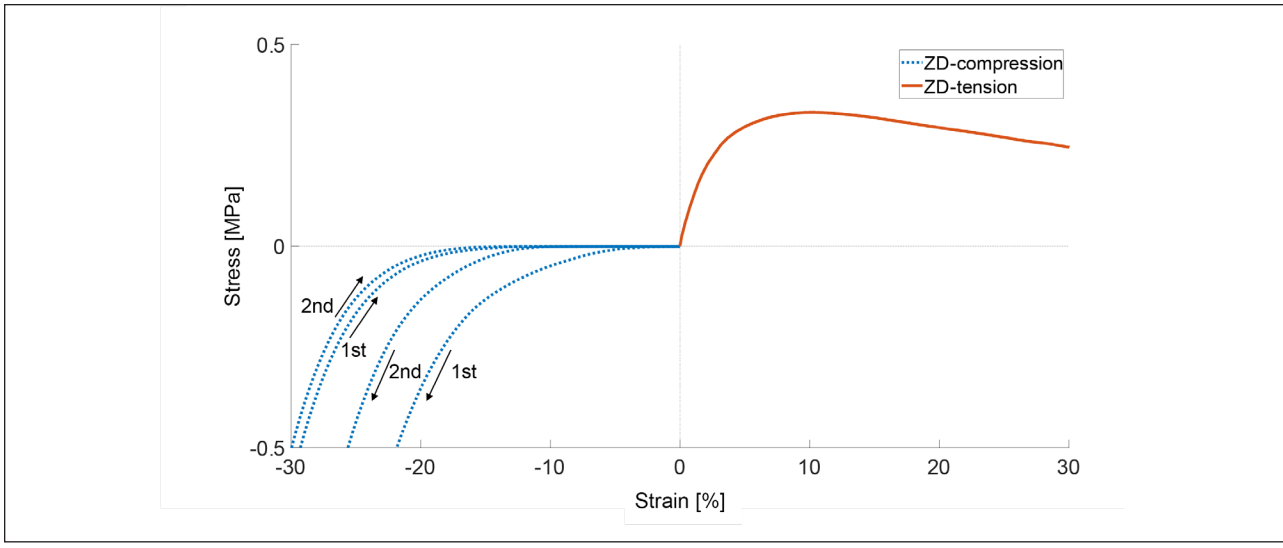
In comparison to measuring the tensile properties in ZD, the compression methods are somewhat more straightforward. The ZD compression is commonly measured by placing the paperboard between two flat and parallel surfaces (e.g., smooth, steel compression clamps) that are compressed together while recording force and displacement. When measuring compression, great care needs to be taken to assure the parallelism of the compressing surfaces, as even a small angle can have a large effect on the initial response of the curve, as investigated by Hagman [6]. Measuring stacks of paperboard mitigates the effects of misalignment but might obfuscate the initial response of the paperboard. Another common problem when measuring compression is how to account for edge effects. If the sample is bigger than the clamps, the actual affected area is somewhat indetermined. However, if the area of the sample is smaller or identical in size to the clamps, care must be taken to avoid edge effects caused by the method used to cut the samples into size, i.e., arched and uneven edges.

Both the tensile and compressive response in ZD becomes more pronounced, complex, and important with increasing paper thickness, especially if the thickness increase is combined with a stratification of the paper. Typically, a paperboard is stratified to achieve a bulkier middle part and denser outer parts to enable an "I-beam" profile that promotes bending stiffness without increased fiber usage. For a paperboard with a complex stratified ZD profile, the ZD response becomes an amalgamation of the properties of each ply. The ZD stiffness depends on the combination of layers. The ZD tensile strength, on the other hand, will be limited by the weakest layer.

The ZD compression behavior typically shows an initial flat slope before transitioning into a steeper slope. The initial flat slope of the ZD compression curve would hint at a discontinuity around zero when compared to the initial behavior of the tensile response, as illustrated in **Fig. 1**. The initial flat part of the compression behavior is decreased when the sample is adhered to the testing platens and is therefore mostly deemed an effect of surface roughness [7]. Due to the uncertainties regarding what affects the response in the initial region of the curve, the modulus is sometimes extracted from the beginning of the offloading part of the compression curve [8]. Regardless of the effect of thickness and texture, the main body of literature reports ZD behavior in compression from testing performed without adhering the samples to the testing platens. The initial ZD compressive behavior and the transition between tension and compression is rarely explored for paperboard. Only one recent example of the latter is known to the authors [9].

The nature of this transition could shed a light on the mechanisms at play in the initial compressive behavior. Since the material is coherent, the behavior over the transition should be continuous. The steeper slope after the initial plateau in compression is probably due to a combination of network compaction and inherent fiber response. Limited compression is recoverable, although not instantaneously. Repeated compression shows an asymptotically "stiffer" behavior, but this also appears to be recoverable for limited pressures, given time.

This paper sets out to test paperboard in combined tension and compression. The main hypothesis is that a continuous transition between tension and compression can be



1. Typical tensile and compressive behavior of paperboard (ZD: z direction). Note that the curves have been measured separately. The compression curves in the figure are from two consecutive compressions to a maximum pressure outside the range of the graph, and back up to 0 pressure. The numbered arrows indicate the compression cycle (1 or 2).

captured and that this transition will show that the initial behavior seen in compression testing is either an artefact of gradual contact due to thickness variations or an intrinsic response of the paperboard similar to what is seen in hyperelastic open foams. The aim of studying the ZD response in transitional loading is to bring further insights into the complex nature of the out-of-plane properties of paperboard. To enable the combined load case, the sample preparation, geometry, and test piece assembly had to be altered from more common practices of preparing ZD tensile and compression test. The effect of the alterations is investigated.

MACHINE SETUP FOR EACH LOAD CASE

To test the ZD response in both combined loading and in pure tensile and compression, three different machine setups were required to meet the demands of each load case.

ZD compression machine setup

The compression testing setup consisted of two steel compression clamps 35.7 mm in diameter, which is the same diameter as the testing platens used for tensile testing. These clamps were aligned using two three-jaw chucks mounted on an electrodynamic universal testing machine (E-UTM), as shown in **Fig. 2** (right image) The compression clamps are only held in place on the circumference, and the flat part of the cylinders are merely resting against the mount. This setup would not withstand the axial tension required for tensile testing. An extensometer was attached to the compression clamps to record the axial movement, and the force was recorded using a load cell rated for 5 kN. Care was taken to ensure proper alignment and centricity of the samples when inserting them between the clamps. The samples were compressed two times to 3 MPa at a rate of 0.02 mm/s.

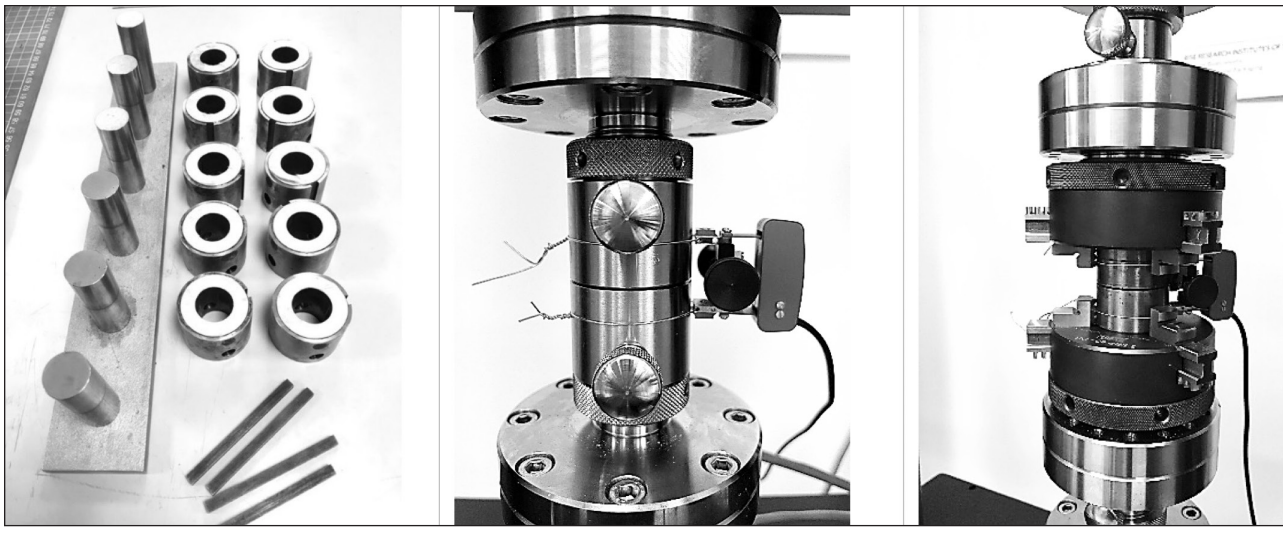
ZD tensile machine setup

The tensile testing setup consisted of two steel testing platens 35.7 mm in diameter (same as in compression) with the paper attached between them. The testing platens in turn are screwed on to a 30-cm-long aluminum rod of the same diameter as the testing platen. The rod and platen are screwed onto a 40 kN load cell. The setup is shown in **Fig. 3**. The rod is connected to the moving piston of a hydraulic universal testing machine (H-UTM) by inserting a thick pin through a wide hole in the rod. When the piston moves, the pin rests perpendicularly to another pin of the same diameter that is permanently attached in the hole in the rod. The ZD tensile setup is therefore not suited for compression tests. An extensometer is attached to the two testing platens to record the displacement during measurements. The test is carried out at a speed of 0.02 mm/s and continued well past break.

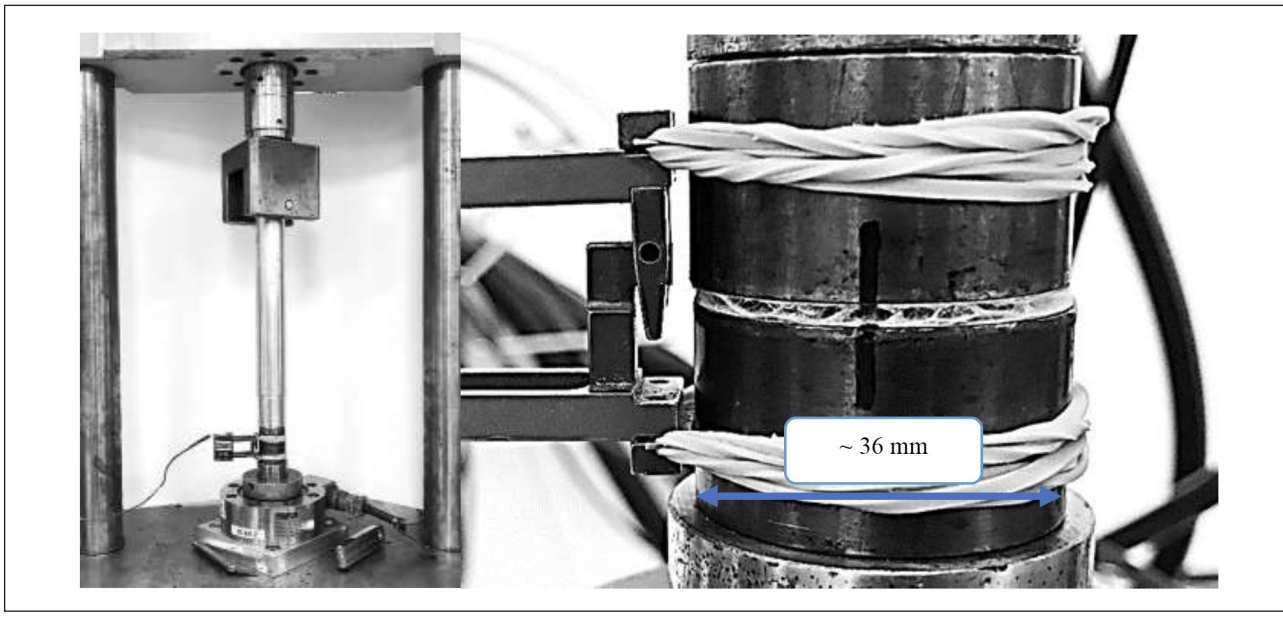
ZD tensile and compression machine setup

Neither the setup used for ZD tensile in Fig. 3 nor the setup used for ZD compression in Fig. 2 (right image) was adaptable to perform the opposite load case. Instead, a specialized setup was used, as shown in Fig. 2 left and middle images. To enable loading in both tension and compression, testing platens that could be mounted with a solid pin onto the E-UTM's mounting taps were required. As can be seen in Fig. 2 (left image), these are circular specimen holders (originally designed for torsion testing). An extensometer was attached to the sample holder. The testing was displacement controlled with a rate of 0.02 mm/s. Two different load protocols were tested, the first starting in compression and the second starting in tension.

For both protocols, a 0.2 MPa (140 N) tensile load limit was chosen for all cycles, with exception of the final load-



2. Left and middle: Setup for combined tensile and compression testing. Right: Compression testing setup.



3. Setup of the ZD tensile testing and a closeup of the test piece.

ing. This limit was chosen to ensure that the sample passed its initial thickness after each compressive load, while being well under the expected maximum load. It was recognized that this load level is beyond the elastic region in tension.

The compressive load was cycled to increasing maximum stress. The aim was to investigate if key features of the cyclic compressive behavior seen in free samples remained. Four features were of particular interest: 1) initial behavior; 2) incomplete recovery between consecutive loads; 3) load path of consecutive compressions to increased load levels, and 4) slope of the offloading.

In the first load protocol, starting in compression, only two load levels were used: 1 MPa (700 N) and 3 MPa (2100 N). Both levels were chosen to be well beyond the initial plateau behavior seen in free compression (Fig. 1). The higher load was repeated two times. The results from testing using the first protocol allowed for a better estimate of the boundary of the "initial" compressive behavior. The compressive load levels in the second protocol were adjusted, based on observations of the displacement from measurements using the first protocol. The second protocol includes a lower load level of 0.2 MPa (140 N) but skips the repeated 3 MPa load.

The first load protocol was:

1. Apply compression to 700 N.
2. Apply tension to 140 N.
3. Apply compression to 2100 N.
4. Apply tension to 140 N.
5. Apply compression to 2100 N.
6. Apply tension until break.

The second load protocol was:
1. Apply tension to 140 N.
2. Apply compression to 140 N.
3. Apply tension to 140 N.
4. Apply compression to 700 N.
5. Apply tension to 140 N.
6. Apply compression to 2100 N.
7. Apply tension until break.

MATERIAL AND SAMPLE PREPARATION

Three types of paper were chosen for their varied properties, two thicker multilayered paperboards and one thinner paper material. Here, they will be referred to as medium paperboard, heavy paperboard, and thin paper. All the materials were of commercial quality. The thin material was primarily selected for its high ZD tensile strength. This material was used to verify that the attachment between paper and laminate in the no glue method was sufficiently strong, with a clear fiber-surface at the break (no laminate seeping through). The two paperboards had similar ply structure but different grammages and densities. Grammage, structure, and thickness can be found in **Table I**. The different tests and the material used for each of them are also presented in Table I. The thicker, bulkier paperboard was utilized to assess the effect of the cutting method, since it was deemed to be more sensitive to edge damage than the paperboard with higher density.

The heat-seal laminate film used for both the reference and adjusted method is Perfex-DB 120(50/70) from GMP Co. Ltd. (Paju, Gyeonggi, South Korea). The film is double-sided and does not release any hazardous gases when heated.

Sample cutting, assembling, and aligning test pieces, and conditioning of the samples

The setup for combined ZD tensile and compression testing required a different sample geometry and attachment to the metal platens than in the glue based ZD tensile method. Four different sample geometries are used in this study, and they are outlined in **Table II**. The table also lists which cutting method was used and in which test the type of sample was used.

Sample cutting for edge-effect evaluation

To assess the possible edge effect of the sample cutting method and to provide reference data for the combined tensile-compression testing, pure compression testing was performed. Three methods were used to cut the samples:

- Laser cut: FLUX Beambox Pro (50 W) CO_2 laser (FLUX; Taipei City, Taiwan) at 20% power and 50 mm/s
- Cutting machine: Brother ScanNcut SDX1200 (Brother; Bridgewater, NJ, USA) at standard settings
- Manual die cut

The samples were cut into three different diameters: 40 mm, 36 mm, and 30 mm. They will be referred to as large sample, platen-sized sample, and small sample, respectively, as shown in Table II.

Assembling and aligning test pieces in ZD tensile tests

The paper material for testing was cut into 5 cm x 5 cm pieces, which in turn were put into a sandwich consisting of, from bottom: low grammage paper, two-sided heat seal laminate film, test material, two-sided heat seal laminate film, and low grammage paper. This stack of material was run through a laminator at 142°C, creating a closed laminate structure. By laminating over the edge of the paper material, creating a "closed bag," care is taken to avoid glue penetration from the sides. This structure was then attached to two cylindrical metal testing platens (diameter 35.7 mm ~ A = 1000 mm²) using a thin dummy paper soaked in low viscous cyano-acrylic glue (Permabond 941, Permabond; Pottstown, PA, USA) to finalize the test piece. The composition of the test piece is depicted in **Fig. 4**. The glue is allowed to cure for 24 h while the test piece is loaded with a dead weight of 500 g. Each test piece is aligned separately during the gluing of the metal platens to the sample piece (tissue, heat seal laminate, and paper). The sample piece was larger than the metal platens to avoid glue

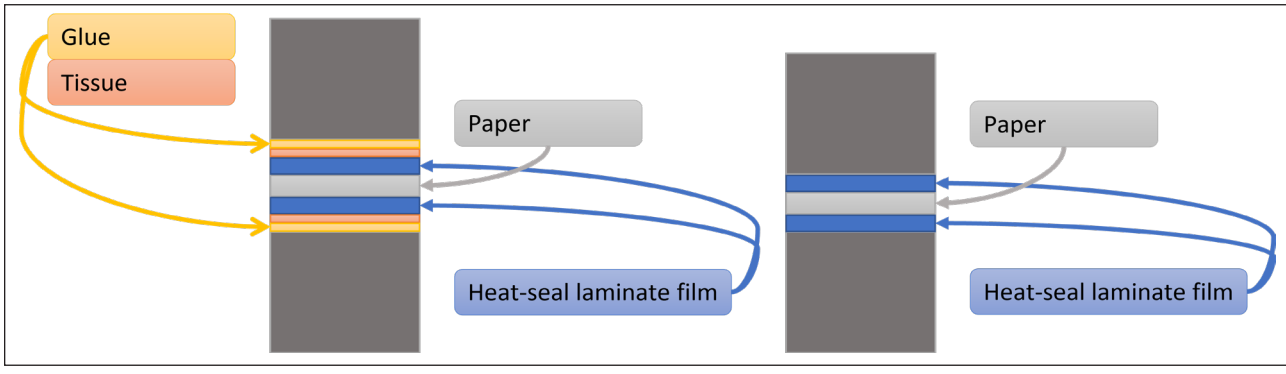
Material	Paper Properties				Test Methods				
	Grammage, g/m²	Ply	Structural Thickness, µm	Std. Dev., µm	ZD Tensile	ZD Tensile (no glue)	ZD Compression	ZD Compression Edge Effects	Combined ZD Tensile and Compression
Thin paper	60	Single	65.0	4.86		X			
Heavy paperboard	340	Multi	502.5	19.13			X	X	X
Medium paperboard	270	Multi	382.1	14.22	X	X	X		X

ZD: *z* direction.

I. The different tests and the properties of the materials used for each of them.

	Sample Properties				Utilized in:				
	Sample Shape	Outer Diameter	Inner Diameter	Approx. Area	Preparation Method	ZD Tensile	ZD Tensile (no glue)	ZD Compression	Combined ZD Tensile and Compression
Large sample		40 mm	NA	1250 mm²	Die cut			X	
					Machine cut			X	
					Laser cut			X	
Platen-sized sample		36 mm	NA	1000 mm²	Die cut			X	
					Machine cut			X	
					Laser cut			X	
					Hand cut around testing platens	X			
Small sample		30 mm	NA	700 mm²	Die cut			X	
					Machine cut			X	
					Laser cut			X	
Donut sample		35.5 mm	20 mm	700 mm²	Laser cut		X		X

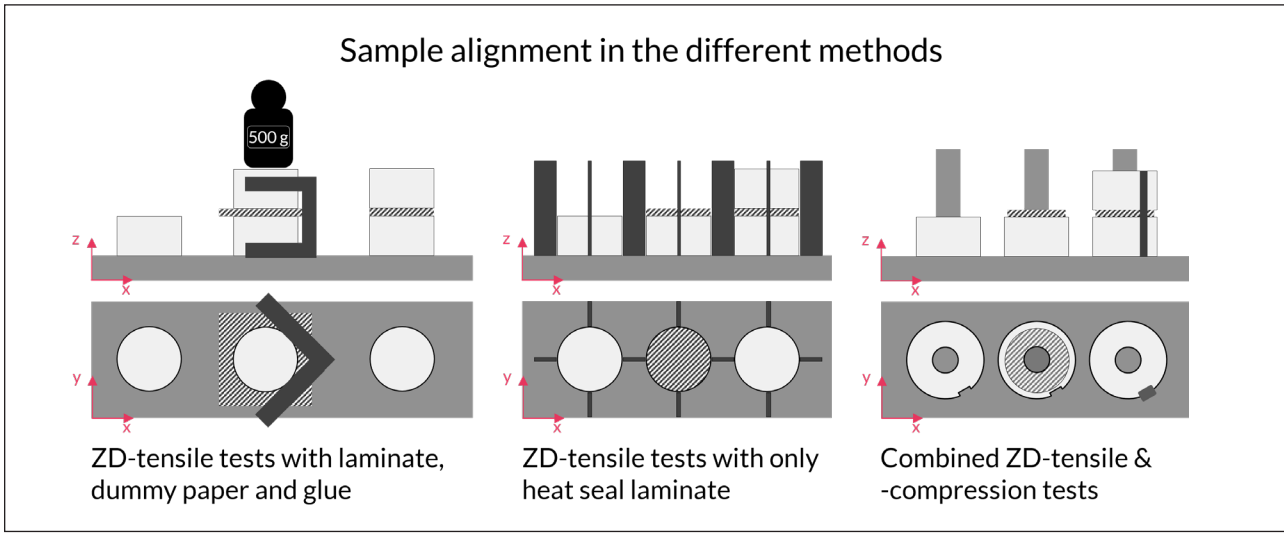
II. Sample properties.



4. The different test piece compositions in ZD tensile testing, showing the glue method to the left and no-glue method to the right.

seeping in, and the metal platens must be aligned one by one using a special holder while applying the glue, as illustrated in **Fig. 5**. After the glue was cured, excess material was cut away and the edges were trimmed by hand using a scalpel or razor. The test piece could then be mounted in the testing device.

In the second version of ZD tensile preparation, the dummy paper and glue were removed from the assembly, and the paper was adhered to the testing platens by only the heat seal laminate, as shown in Fig. 4. When assembling the paperboard and heat seal laminate for ZD tensile without the dummy paper and glue, the paperboard samples were cut to the same size as the platens and placed between them. The test pieces were placed in a holder to ensure alignment between platens and the samples, as shown in Fig. 5. The holder (that can contain up to 36 test pieces) was placed in the oven for 60 min at 142°C, and the test pieces were not removed from the holder until they had cooled.



5. The sample alignment for the different methods.

All ZD tensile tests were performed with the same metal testing platens and tested in the same machine and setup. Any misalignment from mounting the test pieces in the machine will therefore be the same.

Combined ZD tensile and compression samples

The paperboard samples and the heat-seal laminate film were laser cut to fit the testing platens, as illustrated in Fig. 4 and Fig. 5. The special setup used in the combined testing required using testing platens that have a hole in the middle for an alignment pin; therefore, these samples will be referred to as donut shaped. The specimen holders with the samples were placed in a stand with a centerpin during curing to ensure proper axial alignment, see Fig. 5 and Fig. 3. To ensure alignment when mounting the sample in the machine, a cutout has been made along the metal cylinders where a separate pin is placed so that the mounting holes will be aligned vertically. The alignment is required to enable mounting without imposing unwanted preloads. The test pieces are cured in the oven with the holder and alignment pin, and these are not removed until the test pieces have cooled down.

Climate and conditioning

All the tests were performed in a climate-controlled environment at 23°C and relative humidity (RH) of 50%. All the samples were conditioned for at least 24 h. The laser-cut samples were reconditioned before testing, and all other sample cutting was performed in the lab. The samples for the ZD tensile test were conditioned before they were sealed in the laminate pouches. Reconditioning after curing in the oven was tested using samples laminated between metal foil. The samples were weighed before and after curing, at 24 h intervals, to ascertain that they had reached a stable moisture level.

EVALUATION OF THE ADJUSTED SAMPLE PREPARATION

Reconditioning of test pieces

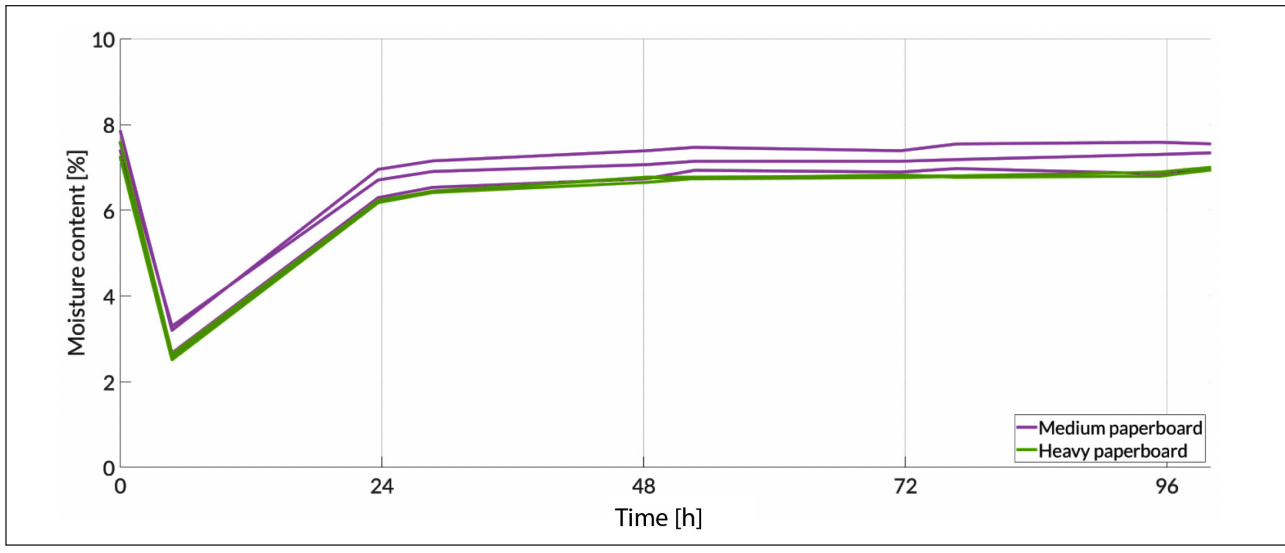
The extended time in the oven will dry out the samples; therefore, the time to recondition them was investigated. Samples of both the medium and heavy paperboard were attached to metal foils and cured in the oven with a metal test platen resting on top to apply the same weight as on the adjusted ZD tensile samples. The foil-paper samples were weighed before curing and every 24 h after curing. The moisture level in the samples were calculated as *(weight/dry weight)* — 1, after the weight of the laminate and metal foils had been deducted. The moisture level is shown over time in **Fig. 6**. The samples dried out significantly during the time in the oven. A lot of moisture is recovered during the first 24 h, but it takes around 72 h before the moisture content stabilizes.

The original moisture content was, however, never recovered (on average it ended up 0.4% lower). One possible explanation is that the samples were cut from a larger sheet that had been stored in an unconditioned environment and not pre-conditioned in a drier environment before measurements; i.e., the difference in moisture content is likely due to the regular moisture hysteresis seen in paper.

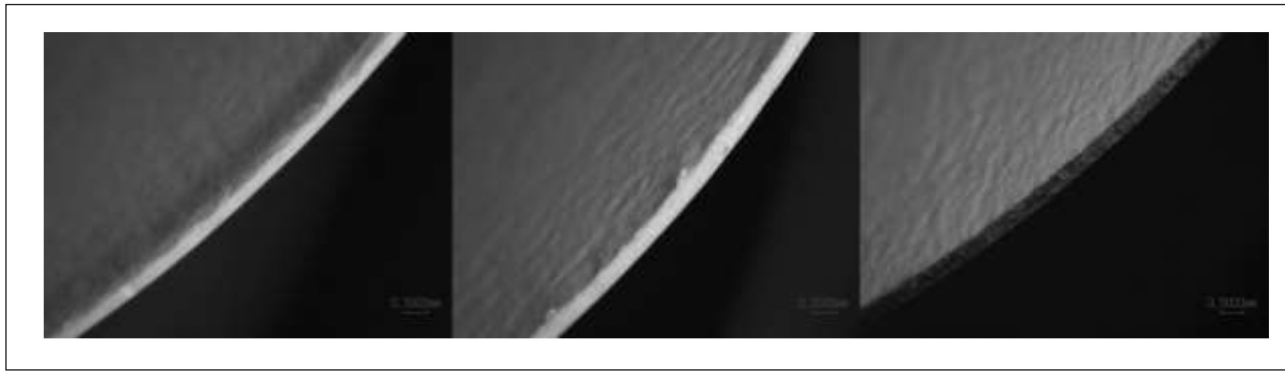
Two sets of control pieces were tested, one of which consisted of only metal foil and plastic laminate, which showed that these were stable in weight. The second set consisted of samples laminated and glued with dummy paper to the metal foil. These samples recovered their moisture fully within 24 h, indicating that the moisture is likely locked in the paperboard by the plastic laminate during the pass through the laminator.

Compliance testing of the mounting materials

Compliance testing was performed by assembling test pieces without paperboard; i.e., only laminate with or without



6. Reconditioning of the samples laminated to metal foil. "0 hours (h)" means that the samples were tested as soon as they had reached room temperature.



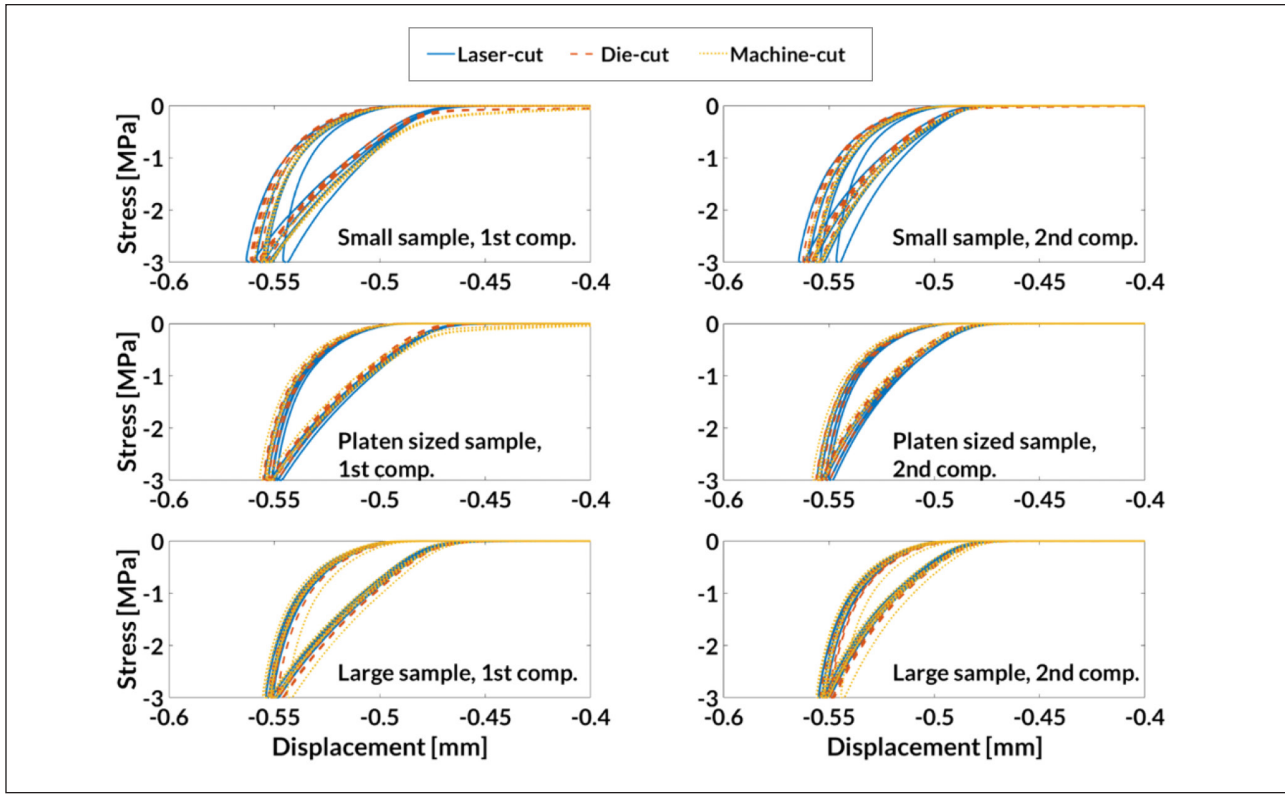
7. Micrographs of the cut surface on compression test samples. Left to right: die cut, machine cut, and laser cut. The die cut edges are slightly crushed. The machine cut edges show roughening of the edge. The light source has the same direction in the images, and the dark edge of the laser cut sample is due to the cutting.

dummy paper and glue. Tensile measurements were performed on the test pieces to provide an indication of the compliance caused by the mounting technique and were found to be linear in the measured range (0–3000 N) for both techniques. The adjusted method was ~40% stiffer than the reference measure; however, in absolute numbers, this difference amounted to an addition to the measured displacement of 0.0005 mm vs. 0.0008 mm at 3000 N. This can be compared with displacement at peak load for the stiffer of the two tested paperboards, which was 0.03 mm at 370 N for the strongest sample.

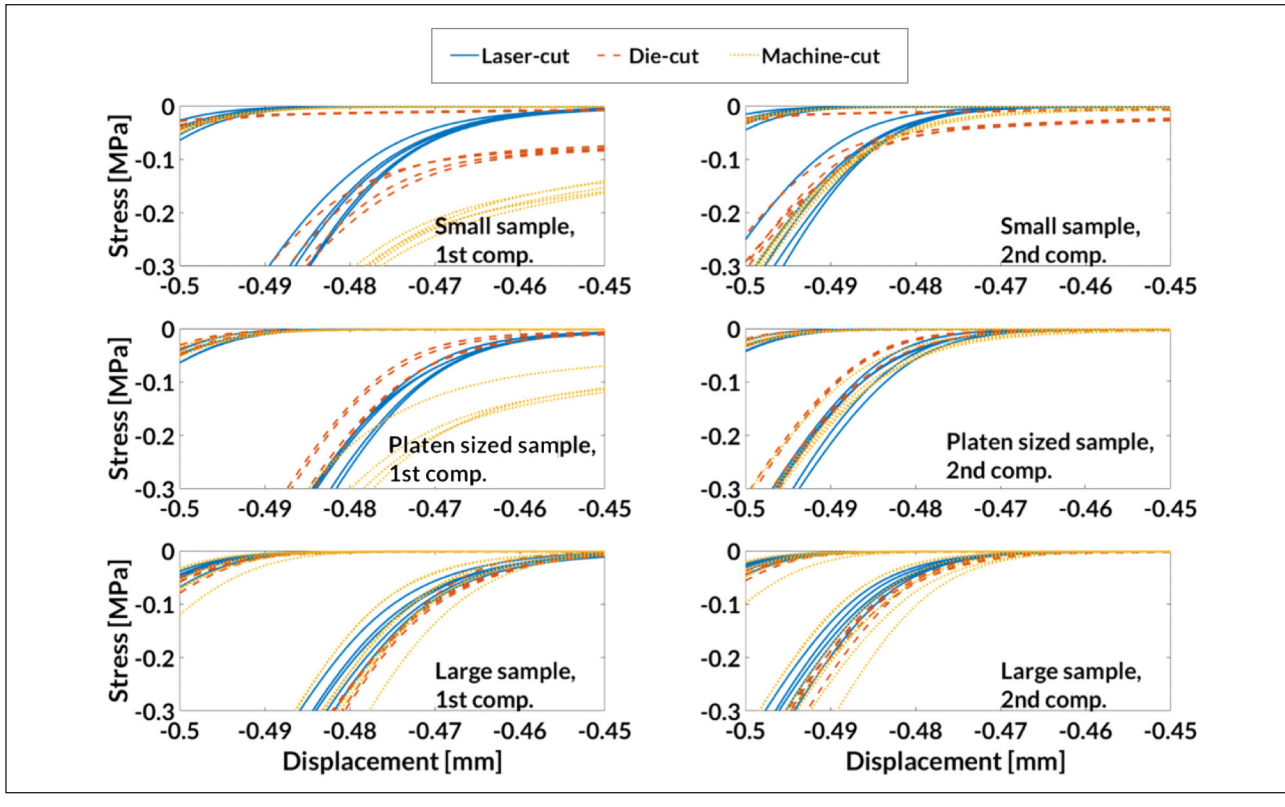
Effect of edges and cutting technique on ZD compression

To minimize edge effects in ZD compression of paperboard, it is common to use a sample size larger than the testing platens. The setup for the combined testing required samples that were smaller than the testing platens, and the effect of different cutting methods was evaluated in ZD compression for the heavy paperboard. The edges of three samples, one for each method, are shown in **Fig. 7**. The micrographs clearly show the damage to the edges due to the cutting method. The die cut edges are slightly crushed. The machine cut edges show roughening of the edge, and the laser cut samples are straight and clean cut, although darkened and a little burned by the method.

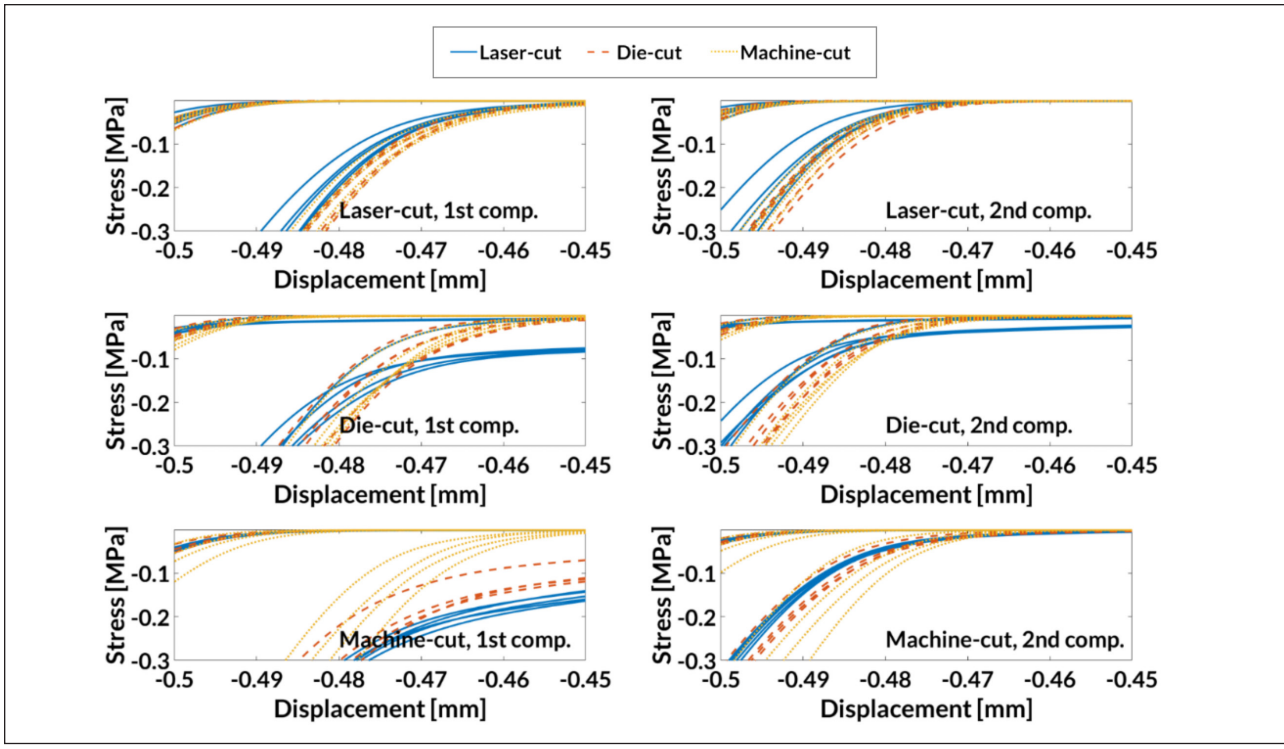
The resulting curves per sample size are shown in **Fig. 8**, with a closeup of the initial region in **Fig. 9**. The same curves sorted by cutting method are shown in **Fig. 10**. When the sample size was larger than the compression clamps, the cutting method did not matter. For die cut samples, this was also true for the same size samples as the compression clamps. When the area of the sample was smaller than the platens, the initial behavior was affected for both the die cut and machine cut samples. The laser cut samples, on the other hand, showed the same initial behavior for all sample sizes. To provide a quantitative measure of the differences between the cutting methods, the initial energy was calculated as the area between the curve and the x-axis between the displacement at 0.01



8. Compression measurements using three sample sizes (top to bottom) and three cutting methods (indicated by color). The compressions are repeated twice (first repeat in the left column and second to the right).



9. Closeup of the curves in Fig. 8. Compression measurements using three sample sizes (top to bottom) and three cutting methods (indicated by color). The compressions are repeated twice (first repeat in the left column and second to the right).



10. Closeup of the compression measurements. Three sample sizes (indicated by color) and three cutting methods (top to bottom) are used. The compressions are repeated twice (first repeat in the left column and second to the right).

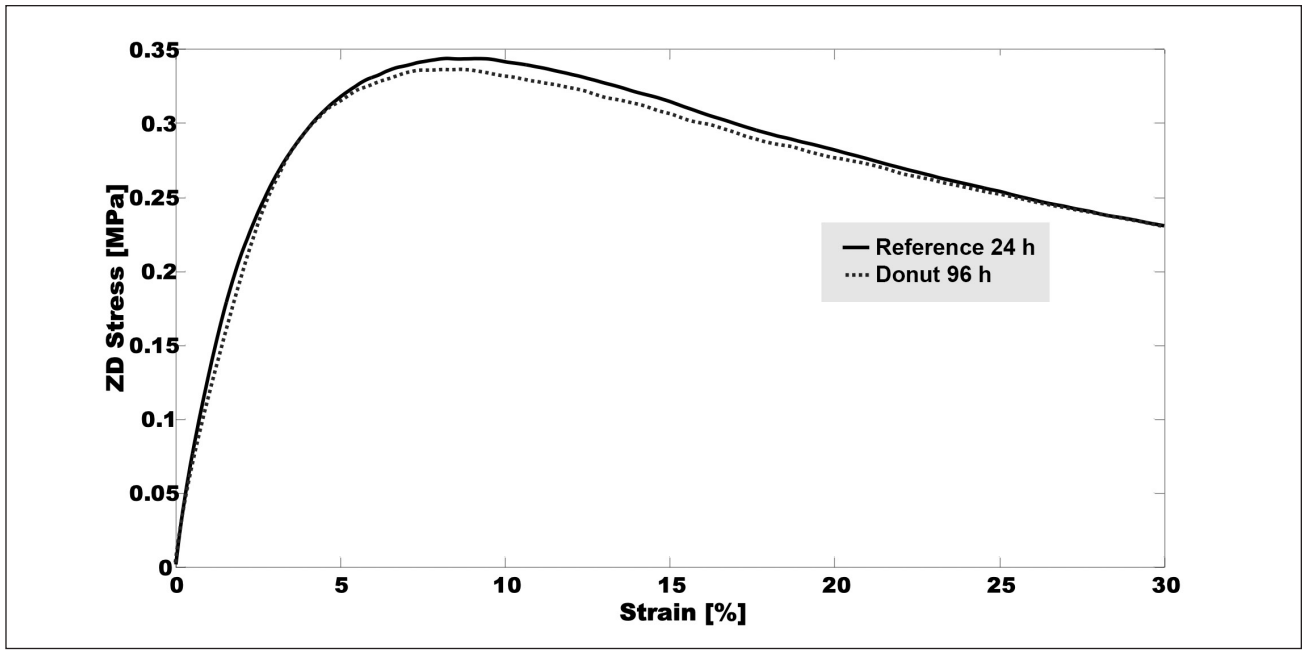
	Heavy Paperboard	Compressive Stiffness, First Compression (1–2 MPa)		Compressive Stiffness, Second Compression (1–2 MPa)		Initial Energy	
Size	Method	Mean, MPa/mm	Std. Dev., MPa/mm	Mean, MPa/mm	Std. Dev., MPa/mm	Mean, N mm	Std. Dev., N mm
Small sample, 30 mm	Laser	49	3.9	65	6.2	3.6	0.1
	Die cut	48	1.6	65	2.2	13.5	1.3
	Machine cut	45	1.6	63	2.6	11.1	0.7
Platen-sized sample, 36 mm	Laser	40	0.8	50	1.3	3.2	0.2
	Die cut	41	0.3	51	0.7	3.3	0.1
	Machine cut	38	1.0	50	0.3	9.9	1.3
Large sample, 40 mm	Laser	40	0.6	50	0.9	3.2	0.1
	Die cut	40	1.0	51	1.3	3.1	0.2
	Machine cut	40	1.1	49	2.0	3.0	0.1

III. The compressive stiffness between 1–2 MPa and the initial energy.

and 0.3 MPa, as presented in **Table III**. The initial energy showed that, for the smallest samples, the die cutting method exhibits worse results than the machine cut samples. The initial energy for the laser cut samples, both platen-sized samples and small samples, remains close to the large sample results.

To verify that the effects caused by the cutting method were limited to the initial behavior, the slope of the curves after the initial compression was also calculated. A linear fit was calculated in the region between the compressive stresses 1 MPa and 2 MPa, and the results are presented in Table III. Only minor differences appear in the slope when comparing samples of the same size, regardless of cutting method.

The low initial energy and similar curves, regardless of sample size, convinced the authors that laser cutting the samples for the combined tensile and compression testing would have negligible edge effects. The difference in initial



11. Mean curves from the different ZD tensile tests. The curves are mean curves aggregated through interpolated approximations of the actual behavior.

behavior of the compression and how sensitive it is to edge (and surface) effects also provides additional motivation for using the combined tensile-compression testing.

ZD tensile testing without glue

The thin material was used to verify that the attachment between paper and laminate in the new ZD tensile method was sufficiently strong, with a clear fiber surface at the break. The strength of the thin material was 2.48 ± 0.140 MPa, and an inspection of the surfaces after break showed fiber surfaces, thereby confirming that the heat-seal laminate had not penetrated through the thin material.

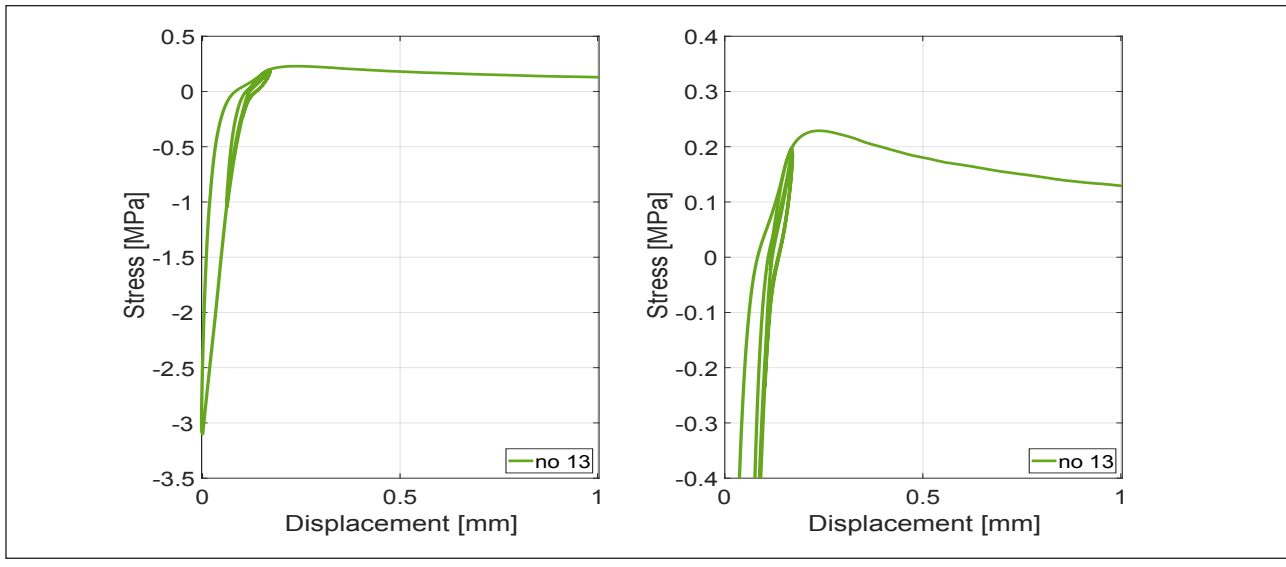
The setup for combined testing requires the donut-shaped samples. Therefore, the ZD tensile behavior of the donut-shaped samples and platen-sized samples was compared. The donut-shaped samples were attached to the platens using only the laminate, while the platen-sized samples were attached using laminate, dummy paper, and glue. The tensile response was found to be similar, as seen in **Fig. 11**. The mean ZD stiffness was 14.1% lower for the donut shaped samples, while the strength was 3.1 % lower, as shown in **Table IV**. It was therefore concluded that the assembly of donut-shaped samples, without dummy paper and glue, would provide insights into the paperboard behavior in combined testing, with negligible influence from the adjusted setup.

RESULTS AND DISCUSSION OF THE COMBINED TENSILE AND COMPRESSION TESTS

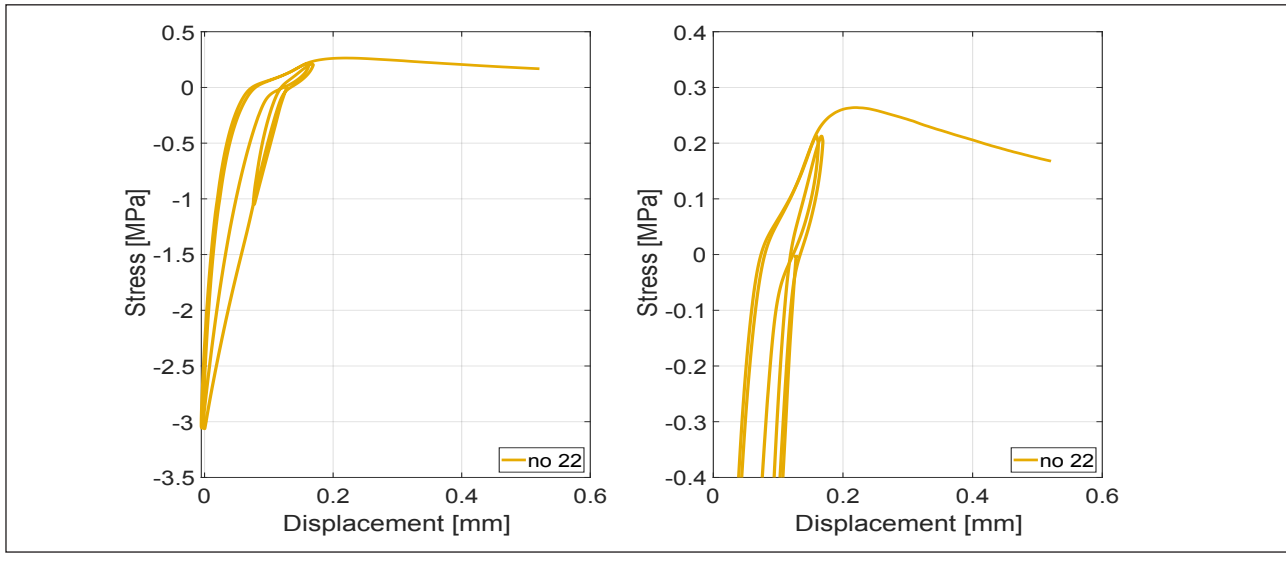
The purpose of the present work is to study the ZD behavior of paperboard in loading cycled between tension and compression. The transition point between tensile and compressive loading is at the turning points of the piston. However, in the present study, we examine the behavior of the material at the transition across 0 stress. The 0-stress transition takes place in either tension or compression, depending on the direction of the piston. Examples of the combined tension-compression curves, with a closeup around the origin, are shown for in **Fig. 12** and **Fig. 13**. The cyclic loading starts in tension in Fig. 12 and in compression in Fig. 13. The 0-stress transition is continuous and smooth, and it is independent of the direction of the

Method	Reconditioning Time, h	*E*, MPa	*E* Std. Dev., MPa	*S*, MPa	*S* Std. Dev., MPa	Number of Samples
Reference method	24	11.0	1.5	0.35	0.020	12
Donut	96	9.4	1.4	0.34	0.10	3
	Diff [%]:	- 14.1		- 3.1		

IV. The ZD strength (S) and stiffness (E) from the reference and adjusted method and the difference between them.



12. Example of combined tension and compression loading, starting in tension. To the left, the full curve. To the right, a closeup of the region around the origin.



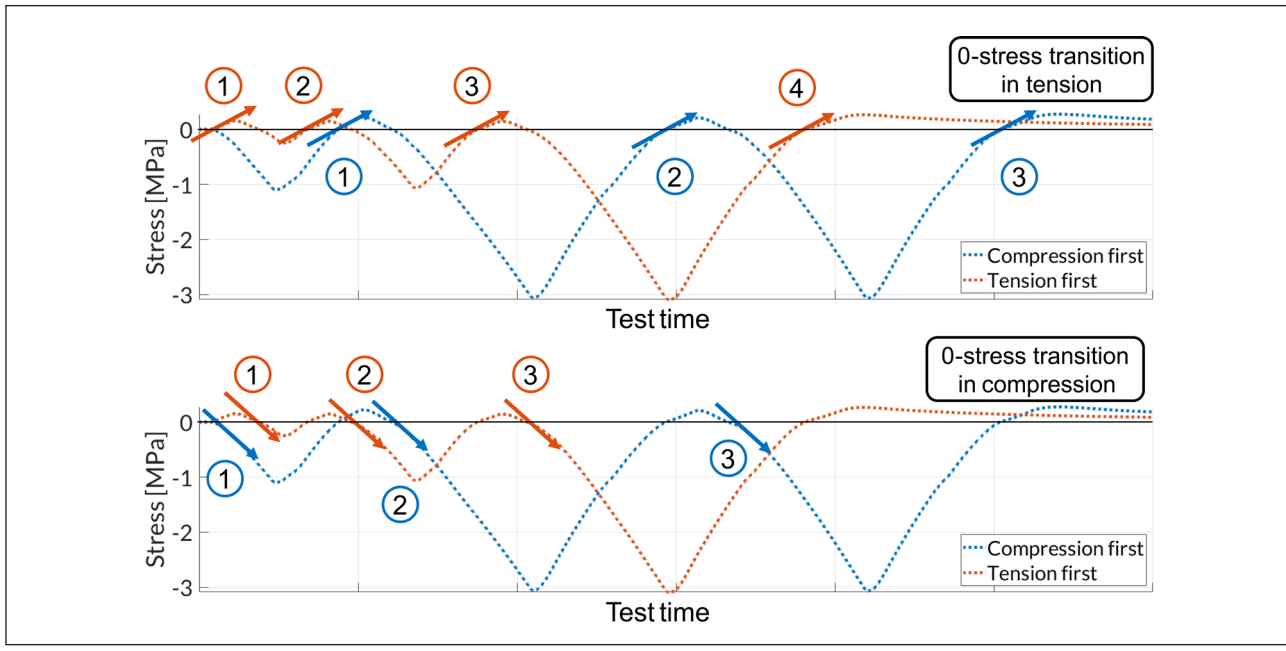
13. Example of combined tension and compression loading, starting in compression. To the left, the full curve. To the right, a closeup of the region around the origin.

transition. This behavior indicates that the initial flat slope seen in referential compression tests (e.g., Fig. 1) is mainly a result of geometric properties of the substrate, e.g., thickness variations, slight curl, or twist.

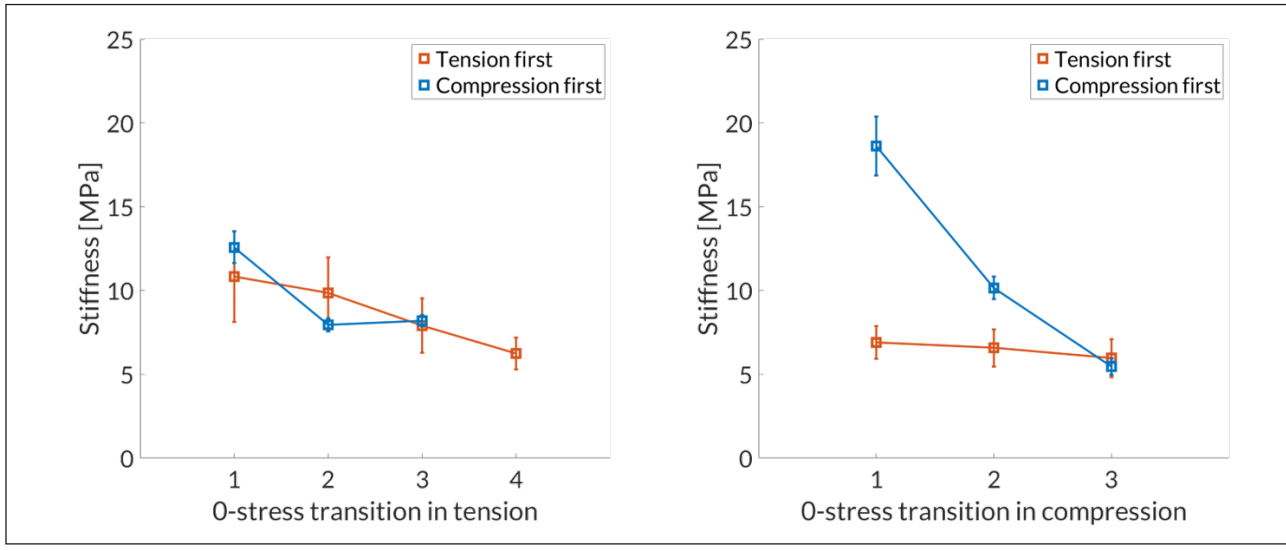
As seen in Fig. 12 and Fig. 13, tensile stress is required to return the sample to zero strain after compression. A larger tensile stress is needed to return to zero strain for the load cases starting in compression. The additional tensile stress needed to return the sample to its original thickness after compression indicates that the network is either permanently compressed (e.g., by fiber plasticity) or temporarily locked in a deformed state (e.g., by means of internal friction) during said compression.

The cycling in both test protocols means that the material experiences several 0-stress transitions in both tension and compression. A schematic description of the 0-stress transitions is shown in **Fig. 14**, where the stress is plotted against test time for clarity. The numbered 0-stress transitions correspond to the number of transitions on the *x*-axis in **Fig. 15** where the stiffness in 0-stress transition is shown. The left graph in Fig. 15 shows 0-stress transitions in tension, i.e., that go from negative to positive stress, whereas the right graph shows 0-stress transitions in compression, i.e., that go from positive to negative stress. Each graph in Fig. 15 contains the 0-stress transition stiffness for both protocols; i.e., tests started in either compression (blue) or tension (red).

The left graph in Fig. 15 shows that regardless of protocol, the samples have a similar behavior at 0-stress transition in tension. At 0-stress transition in tension, the slope of the curve (i.e., the stiffness) becomes less steep with each



14. Schematic description of the transitions between compression and tension for the two load protocols.

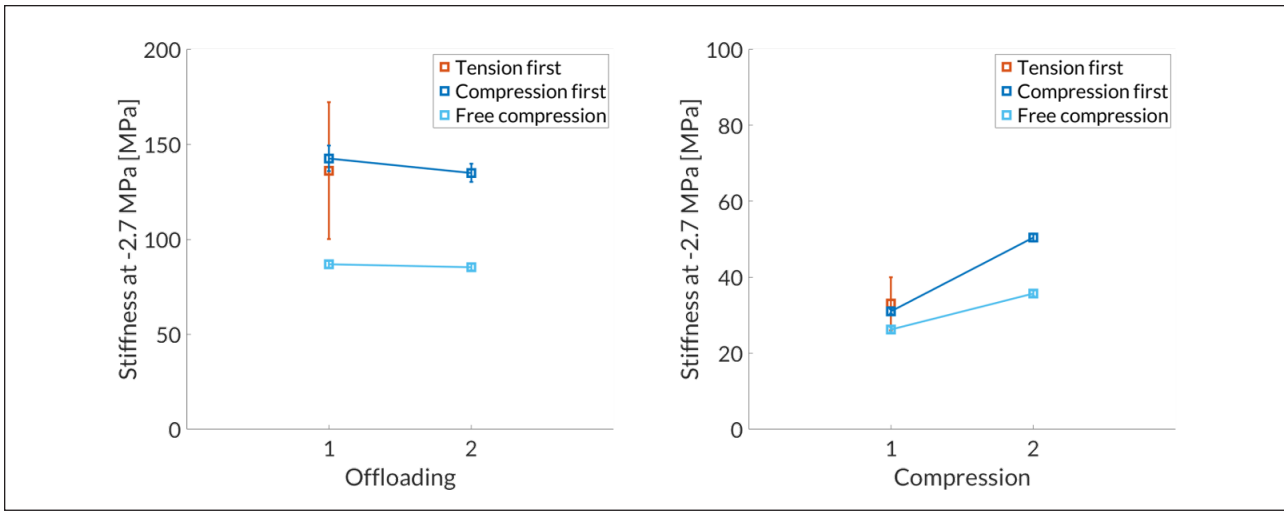


15. The stiffness of the material at 0 MPa. The left graph shows the stiffness at 0-stress transition from compression to tension, as indicated with arrows in the upper part of Fig. 14. The right graph shows the stiffness at 0-stress transition from tension to compression, as indicated with arrows in the lower part of Fig. 14.

transition. The material behavior could be explained by the fiber network being deformed during tension. The tensile load makes the network sparser, and due to fiber plasticity, the new network configuration is locked. The transitions from tension to compression (change of piston direction) take place beyond the elastic part of the curve, but before break. Therefore, each 0-stress transition in tension shows a decrease in stiffness.

On the other hand, the right graph in Fig. 15 shows a difference in the stiffness at 0-stress transition in compression, depending on whether the cycles started in tension or compression. The samples that started in tension show only a small change in stiffness for each transition. In contrast, during the first 0-stress transition, the samples that start in compression have a stiffness more than double that of the samples starting in tension. The stiffness is then reduced with each cycle. At the third transition, the stiffness for samples starting in both tension and compression coincides.

A possible mechanism to explain the initial difference in compressive stiffness is the previously mentioned fiber network deformation during tension. The network acquires a new configuration after each 0-stress transition in tension. For samples starting in tension, the 0-stress transition stiffness in compression remains almost unchanged, indicating



16. The stiffness of the material at -2.7 MPa in both compression and offloading.

that the new configuration is not reset by the compression phase of the cycle.

When starting in compression, the first 0-stress transition in compression is performed on undamaged material exhibiting a high stiffness. As was observed in Fig. 12 and Fig. 13, the tensile stress needed to return the material to its original thickness was higher for the samples starting in compression. The displacement between returning to the original thickness and the switch in piston direction (from tension to compression) is therefore smaller for the samples starting in compression. The second 0-stress transition in compression therefore still shows a higher stiffness for the samples starting in compression than the samples that started in tension. By using displacement control for the end positions instead of force control, the stiffness might have converged sooner.

The compressive stiffness beyond the initial or elastic part of the curve also reveals information about the material. In **Fig. 16**, the stiffness (slope of the curve) at 2.7 MPa compression is presented. The stiffness for samples in combination testing — adhered samples — is compared to samples tested during free compression, i.e., when the sample is not adhered to the testing platens. The protocol that starts with tension only has one compression cycle that reaches 3 MPa.

The left graph in Fig. 16 shows the stiffness after the change in piston direction, i.e., offloading for the free compression, and tension for the samples adhered to the testing platens. The stiffness is the same for both adhered sample sets during the first cycle, regardless of protocol. The adhered samples have roughly twice the stiffness of the free samples. Neither the free samples nor the adhered samples that start in compression show any large change in the stiffness between the first and second compression cycle.

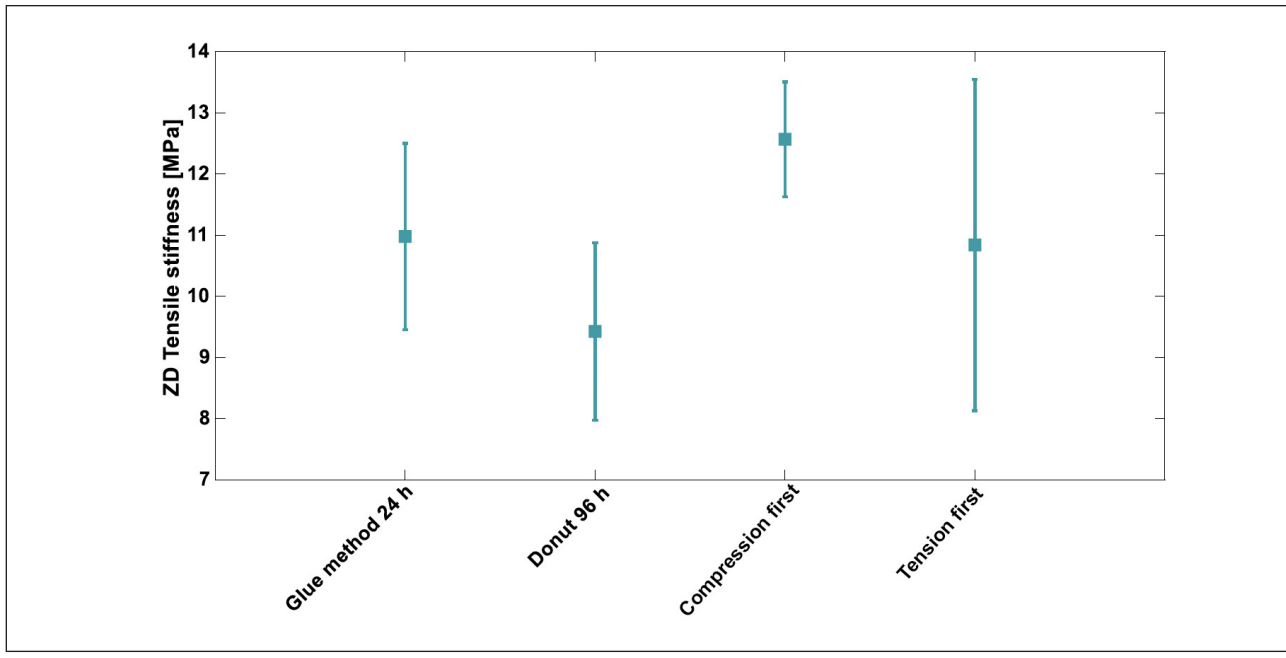
During cyclic compression, the stiffness during offloading is expected to be the same for each cycle as long as the network has been compressed the same amount, and the stiffness is calculated around the same point. This is also what can be observed in the Fig. 16. However, it is apparent that the stiffness is much higher during forced offloading as compared to if the network is allowed to spring back freely.

The right graph in Fig. 16 shows the stiffness during compressive loading. The difference between adhered and free samples is small at the initial compression. Both free samples and adhered samples starting in compression show an increased stiffness during the second compression, with a slightly smaller increase for the free samples.

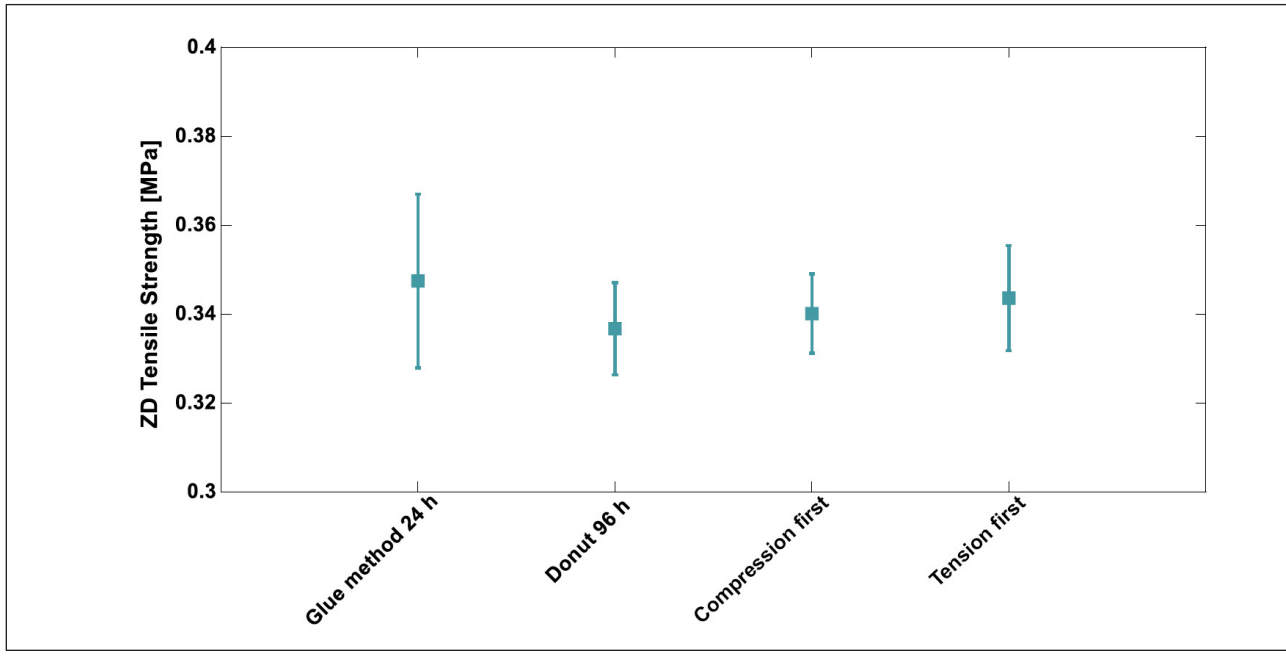
It is expected that the stiffness during compressive loading is different from compressive offloading, as can be seen in the present cases. During compaction of the network, the stiffness should increase for each compression cycle, especially for the free compression where nothing helps the material return to its original thickness. However, as can be seen on the right in Fig. 16, the stiffness increase is larger for the samples that have been pulled back in tension instead of just being offloaded. This might be an indication that the forced offloading allows the network to rearrange itself into a denser configuration.

After cycling the samples through the two protocols, they were pulled to break to investigate the ultimate ZD strength and how it compared to samples that were only tested in tensile. Two sets of tensile tests are used for comparison. One set of samples was prepared with heat seal laminate, dummy paper, and glue. The second set consisted of donut-shaped samples just like the ones in combination testing, which were adhered to the testing platens only by heat seal laminate.

The tensile stiffness is presented in **Fig. 17** and the strength is presented in **Fig. 18**. The tensile stiffness from the combination tests is calculated at the first 0-stress transition in tension. Overall, the stiffness and strength of the samples are similar, especially when accounting for the variation between samples.



17. Tensile stiffness for the ZD tensile samples, with comparison to the initial tensile stiffness of the combined samples.



18. Tensile strength for the ZD tensile samples, with comparison to the tensile strength of the combined samples.

The mechanism observed in the 0-stress transitions in Fig. 14 suggests that the network is either damaged between the cycles or that there is permanent deformation of the network induced by the alternating tensile and compressive loads. Each load direction induces plastic deformation of the fibers and rearrangement of the network. The effects work against each other and cannot be undone by reversing the load direction. An indication that this behavior is due to permanent deformation rather than damage is that the strength remains largely unchanged — even after repeated load cycles — and thus that the fiber-fiber bonds remain untouched in number and strength.

A drawback in the present study is the number of samples and the limitation to only one material in the combination test. A larger sample set and a few more materials would be needed to draw more general conclusions. However, the present study shows the necessity of looking further into the ZD mechanisms to understand paperboard behavior.

SUMMARY AND CONCLUSION

In this study, we set out to investigate the material response of a paperboard in combined out-of-plane compression and tensile load. The following conclusions can be drawn from the combined tensile and compression testing:

- The zero transition between tension and compression (and vice versa) is continuous. This result also indicates that the initial flat slope seen in referential compression tests is mainly a result of geometric properties of the substrate, e.g., thickness variations, slight curl, or twist.
- The stiffness at the 0-stress transition is decreased for each cycle. The effect is larger if the sample is initially loaded in tension.
- The slope is history dependent. The stiffness close to the maximum compression is increased for each compressive cycle. This effect is larger for a material that is subjected to tensile load between compression cycles, which indicates a different effect than the incomplete recovery seen in freely (non-glued) compression samples.
- The stiffness of the return curve right after the maximum compression is stiffer when force is applied to pull the material back than seen in the offloading of free compression.
- The ultimate strength is largely unaffected by the interspersed compression segments of the test cycles.

The tests did prove the initial hypothesis, i.e., that the material response is continuous over the zero crossing, which is expected of a cohesive material without radical phase changes. By combining the ZD compression and tensile testing, it is possible to gain information about the paperboard that cannot be acquired by testing separately. The method to achieve the combined testing shows promise but requires additional work to make it more robust. It is necessary to lock down key properties and expand the sample set to a larger selection of paper qualities. However, the results in the present study show that this work is important in order to expand the knowledge and understanding of the out-of-plane properties of paperboard. **TJ**

ABOUT THIS PAPER

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We studied this topic because z-direction (ZD) tensile and ZD compressive response, as typically measured by separate methods, do not fit together in a manner expected from a material properties perspective. This research complements the understanding of the out-of-plane behavior of paperboard by giving insight into the actual material response of the studied material, which is useful for modeling purposes.

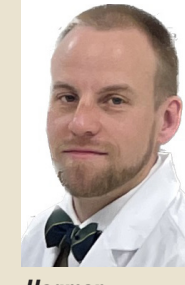
Great care was needed to ensure that the combined tensile-compression test method used in this study did not interfere with the results and provided results that were in line with the currently used separate methods. Additionally, presenting rather complicated data in a suitable way was also a challenge.

This study's results showed the hypothesis that material responses continue across the zero crossing was correct. A particularly interesting finding was how history dependent the different parts of the combined loading curve were.

The ZD behavior of paperboard is important during converting and printing. A combined load case can occur during offset printing, for example. Knowledge of the mechanisms at work is helpful for mills when designing paperboard for such applications.

The next step is to make the combined tensile-compression test method proposed here more robust, to lock down key properties, and to expand the test method to a larger selection of paper qualities.

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