Lappeenranta
University of Technology

Ville Leminen

LEAK-PROOF HEAT SEALING OF PRESS-FORMED PAPERBOARD TRAYS

Thesis for the degree of Doctor of Science (Technology) to be presented with due permission for public examination and criticism in the Auditorium 2310 at Lappeenranta University of Technology, Lappeenranta, Finland on the 27th of May, 2016, at noon.

Acta Universitatis
Lappeenrantaensis 698

Supervisor	Professor Juha Varis LUT School of Energy Systems Lappeenranta University of Technology Finland
Reviewers	Professor Jens-Peter Majschak Department of Mechanical Engineering Technical University of Dresden Germany
	D.Sc. Mika Vähä-Nissi VTT Technical Research Centre of Finland Ltd Finland
Opponent	D. Sc. Mika Vähä-Nissi VTT Technical Research Centre of Finland Ltd Finland

ISBN 978-952-265-954-5
ISBN 978-952-265-955-2 (PDF)
ISSN-L 1456-4491
ISSN 1456-4491

Lappeenrannan teknillinen yliopisto
Yliopistopaino 2016

Abstract

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Leak-proof Heat Sealing of Press-Formed Paperboard Trays
Lappeenranta 2016
73 pages
Acta Universitatis Lappeenrantaensis 698
Diss. Lappeenranta University of Technology
ISBN 978-952-265-954-5, ISBN 978-952-265-955-2 (PDF), ISSN-L 1456-4491, ISSN 1456-4491

Three-dimensional (3D) forming of paperboard and heat sealing of lidding films to trays manufactured by the press forming process are investigated in this thesis. The aim of the work was to investigate and recognize the factors affecting the quality of heat sealing and the leak resistance (tightness) of press-formed, polymer-coated paperboard trays heat-sealed with a multi-layer polymer based lidding film. One target was to achieve a solution that can be used in food packaging using modified atmosphere packaging (MAP). The main challenge in acquiring adequate tightness properties for the use of MAP is creases in the sealing area of the paperboard trays which can act as capillary tubes and prevent leak-proof sealing.

Several experiments were made to investigate the effect of different factors and process parameters in the forming and sealing processes. Also different methods of analysis, such as microscopic analysis and 3D-profilometry were used to investigate the structure of the creases in the sealing area, and to analyse the surface characteristics of the tray flange of the formed trays to define quality that can be sealed with satisfactory tightness for the use of MAP. The main factors and parameters that had an effect on the result of leak-proof sealing and must be adjusted accordingly were the tray geometry and dimensions, blank holding force in press forming, surface roughness of the sealing area, the geometry and depth of the creases, and the sealing pressure.

The results show that the quality of press-formed, polymer-coated paperboard trays and multi-layer polymer lidding films can be satisfactory for the use of modified atmosphere packaging in food solutions. Suitable tools, materials, and process parameters have to be selected and used during the tray manufacturing process and lid sealing process, however. Utilizing these solutions and results makes it possible for a package that is made mostly from renewable and recyclable sources to be a considerable alternative for packages made completely from oil based polymers, and to achieve a greater market share for fibre-based solutions in food packaging using MAP.

Keywords: press forming, paperboard, modified atmosphere packaging, MAP, packaging, heat sealing

Acknowledgements

This work was carried out in the Laboratory of Packaging Technology, Mechanical Engineering at Lappeenranta University of Technology, Finland, between 2012 and 2016. First, I would like to thank my supervisor, Professor Juha Varis, for his guidance and comments during the work. I would also like to thank the preliminary examiners of the dissertation, Professor Jens-Peter Majschak and Dr. Mika Vähä-Nissi for their valuable comments and criticism.

I would like to thank my colleagues and co-authors in the articles for their valuable work in the experiments. Thanks go to Panu Tanninen for the valuable discussions we have had in the office and in the laboratory, sometimes work related, sometimes not. Mika Kainusalmi is thanked for his help, especially with several research projects. Sami Matthews and everybody else in our lab also deserve a big thank you. Professor Henry Lindell's help and valuable comments during joint research projects and other interesting topics are appreciated. Sami-Seppo Ovaska and Katriina Mielonen are acknowledged for their help, especially with answering my material related questions. Many other people from the current and former LUT Mechanical Engineering staff have also helped me during these years, thank you all.

My parents Helmi and Heimo deserve a special acknowledgement; thank you for supporting me through my studies and throughout my life, your support enabled me to achieve my education.

My biggest thank you is reserved for my family. Niina, Miko, Otso, this is dedicated to you. I know it hasn't been always easy when I have been busy, travelling and sometimes in my own thoughts. You have been and always will be there for me. I love you.

Lappeenranta, May 2016

Ville Leminen

Contents

List of publications and the author's contribution

This thesis is based on the papers listed below. The rights have been granted by the publishers to include the papers in the dissertation.

I. Leminen, V., Kainusalmi, M., Tanninen, P., Lohtander, M. and Varis, J. (2012). Effect of Sealing Temperature to Required Sealing Time in Heat Sealing Process of a Paperboard Tray. *Journal of Applied Packaging Research*, 6(2), pp. 67-78.

II. Leminen, V., Tanninen, P., Mäkelä, P. and Varis, J. (2013). Combined effect of paperboard thickness and mould clearance in the press forming process. *Bioresources*, 8(4), pp. 5701-5714.

III. Leminen, V., Tanninen, P., Lindell, H. and Varis, J. (2015). Effect of blank holding force on the gas tightness of paperboard trays manufactured by the press forming process. *Bioresources*, 10(2). pp. 2235-2243.

IV. Leminen, V., Mäkelä, P., Tanninen, P. and Varis, J. (2015). Methods for Analyzing the Structure of Creases in Heat Sealed Paperboard Packages. *Journal of Applied Packaging Research*, 7(1), pp. 49-60.

V. Leminen, V., Mäkelä, P., Tanninen, P. and Varis, J. (2015). The use of chromatic white light 3D-profilometry in the surface quality analysis of press-formed paperboard trays *Proceedings of the 25th Flexible Automation and Intelligent Manufacturing, FAIM2015, Volume II,* pp.74-81. Wolverhampton, UK: The Choir Press.

VI. Tanninen, P., Leminen, V., Kainusalmi, M. and Varis, J. (2016). Effect of Process Parameter Variation on the Dimensions of Paperboard Trays. *Bioresources*, 11(1). pp. 140-158.

VII. Leminen, V., Mäkelä, P., Tanninen, P. and Varis, J. (2015). Leakproof Heat Sealing of Paperboard Trays - Effect of Sealing Pressure and Crease Geometry. *Bioresources*, 10(4). pp. 6906-6916.

Author's contribution

The author was the principal author and investigator in papers I, III-V and VII and responsible for planning and performing the experiments and writing the text. In papers II and VI, the author participated in planning and doing the experiments and was responsible for the writing of the article together with D.Sc. (Tech.) Panu Tanninen.

Supporting publications

1. Leminen V., Kainusalmi M., Tanninen P., Lindell H., Varis J., Ovaska S.-S., Backfolk K., Pitkänen M., Sipiläinen-Malm T., Hartman J., Rusko E., Hakola L., Ihalainen P., Määttänen A., Sarfraz J. and Peltonen J. (2013). Aspects on Packaging Safety and Biomaterials. *26th IAPRI Symposium on Packaging, IAPRI2013,* pp. 483-493. Espoo, Finland: VTT Technical Research Centre of Finland.
2. Leminen V., Kainusalmi M., Tanninen P., Lindell H., Varis J., Ovaska S-S., Backfolk K., Mielonen K., Pitkänen M., Sipiläinen-Malm T., Rusko, E., Hakola L., Sarfraz J., Ihalainen P. and Peltonen J. (2015). Ways for Improving the Safety of Fibre-based Food Packages. *27th IAPRI Symposium on Packaging, IAPRI2015,* pp. 249-266. Valencia, Spain: ITENE – Packaging, Transport and Logistics Research Center
3. Tanninen, P., Leminen, V., Eskelinen, H., Lindell, H., and Varis, J. (2015). Controlling the Folding of the Blank in Paperboard Tray Press Forming. *Bioresources*, 10(3). pp. 5191-5202.
4. Tanninen, P., Kasurinen, M., Eskelinen, H., Varis, J., Lindell, H., Leminen, V., Matthews, S. and Kainusalmi, M. (2014). The effect of tool heating arrangement on fibre material forming. *Journal of Materials Processing Technology*, 214(8). pp. 1576-1582.
5. Leminen, V., Eskelinen, H., Matthews, S. and Varis, J. (2013). Development and utilization of a DFMA-evaluation matrix for comparing the level of modularity and standardization in clamping systems. *Mechanika.* 19(6). pp. 711-715.
6. Tanninen, P., Saukkonen, E., Leminen, V., Lindell, H. and Backfolk, K. (2015). Adjusting the die cutting process and tools for biopolymer dispersion coated paperboards. *Nordic Pulp & Paper Research Journal*, 30(2). pp. 336-343.
7. Leminen, V., Ovaska S-S C., Tanninen, P. and Varis, J. (2015). Convertability and Oil Resistance of Paperboard with Hydroxypropyl-Cellulose-Based Dispersion Barrier Coatings. *Journal of Applied Packaging Research, 7(3), pp. 91-100.*

Abbreviations

2D	two-dimensional
3D	three-dimensional
AKA	also known as
ASTM	American Society for Testing and Materials
BHF	blank holding force
CD	cross-direction
CPP	cast polypropylene
D_{CG}	depth of the creasing groove
F	force (N)
FFS	form-fill-seal
GN	gastronorm
gsm	grams / square meter
LF	lidding film
LLDPE	linear low-density polyethylene
LST	lower sealing tool
MAP	modified atmosphere packaging
MD	machine direction
OPP	oriented polypropylene
OTR	oxygen transmission rate ($cm^3/m^2/24h$)
p	pressure (N/mm^2)
PE	polyethylene
PET	polyethylene terephthalate
R_{CR}	radius of the creasing rule tip
RH	relative humidity
Ra	roughness average
Rp	peak height (roughness)
RT	room temperature
Rv	lowest valley (roughness)
Rz(DIN)	average peak to valley (roughness)
SBS	solid bleached sulphate
SC	sealing chamber
SEM	scanning electron microscope
v	speed (m/s)
W_{CG}	width of the creasing groove
W_{CR}	width of the creasing rule
T	temperature (K)
T_{PB}	thickness of the paperboard
UST	upper sealing tool

1 Introduction

1.1 Background

Packaging is an important part of almost any product that is sold today. Packages have evolved from mere containers to an important part of product design. The basic functions of a food package are to contain and protect the packed product, preserve the food by preventing or inhibiting chemical or biochemical changes and microbiological spoilage, and to inform about the product. The package must also be convenient, presentative, and communicative about the brand, and promote (sell) the product. The package must also be economical and environmentally responsible (in manufacture, use, reuse, or recycling and final disposal). (Coles et al. 2003)

The global packaging industry was worth $690 billion in 2011, having increased by over $120 billion since 2006. Smithers Pira predicts that this market will grow by a further $150 billion between the years 2010 and 2016, by which time it will be almost $820 billion. The single biggest end user for packaging is the food industry, which accounted for 31 % of the demand in 2010, totaling more than $206 billion. The growth in this sector is predicted to be up to $40 billion to $245 billion. The total packaging consumption in 2010 was $1822 million in Finland. (Harrod 2010)

Rigid plastic packages, such as trays were globally the fastest growing market between the years 2006 and 2010. The market for rigid plastic increased by about 6.5 % annually during this period. In 2010 the rigid plastic market was over $144 billion, and this figure is predicted to be $201 billion in 2016 (Harrod 2010). Rigid trays are used to pack various food products, such as cold cuts, cheeses, minced meat, poultry and ready-made meals. These trays are usually manufactured from polymer materials by thermoforming.

Board consumption has also grown steadily since 2006. The total board consumption grew about 6 % between 2006 and 2010, was worth over $216 billion in 2010, and is predicted to be $250 billion in 2016. (Harrod 2010)

There are several advantages which make the use of fibre-based materials more attractive than petroleum-based polymer products. These advantages include recyclability, good printability, better image (“green”), renewability and biodegrability (Vishtal and Retulainen 2012). Also the legislation in the European Union is moving towards a direction that favors fibre-based materials and recyclability.

The converting of fibre-based materials to complex 3D shapes, such as rigid trays by pressing is more challenging than with polymer-based materials. This is caused by many factors, the main ones being worse formability properties, such as low elongation properties, which can cause the formed product to crack and have pinholes, shape inaccuracies or visual defects (Vishtal and Retulainen 2012, Wallmeier et al. 2015).

However, if fibre-based solutions can be developed to be an alternative for rigid plastic packages, the market potential is very significant.

Also the leak-proof sealing of formed paperboard trays is challenging. The main reason for this are capillary tubes or grooves in the sealing area caused by wrinkles (Hauptmann et al. 2013, Leminen et al. 2012, Leminen et al. 2015a). These wrinkles are caused by several things: compressive forces in a transverse direction in the material (Vishtal and Retulainen 2012), the material properties (lower formability, stiffness) of paperboard which force the material to wrinkle. The folding of material and the formation of wrinkles is often controlled by pre-creasing of the tray blank to enable formation of deeper and more complex shapes and to control the location of the wrinkles (folds) (Kunnari et al. 2007, Tanninen 2015c). Pre-creased, formed grooves are often described as wrinkles (Vishtal 2015) while Tanninen (2015c) defines pre-creased grooves which are formed as folds, and wrinkles as folds that are not assisted by creases.

1.2 Objectives of the thesis

The objectives of this thesis are to investigate and identify the factors affecting the heat sealing quality and the leak resistance (tightness) of press-formed, polymer-coated paperboard trays which have been heat-sealed with a multi-layer polymer - based lidding film. Both press forming and heat sealing processes are investigated.

One of the objectives is to achieve a solution that can be used in food packaging by using modified atmosphere packaging (MAP) without any additional process phases or additional material between the press forming and heat sealing of the lidding film processes.

Another objective is to find whether “sealable” quality can be quantified based on the dimensions of the creases or other key properties.

1.3 Hypotheses

The experimental work and the theoretical aspects used in this study are based on the following hypotheses:

- Press-formed, polymer-coated paperboard trays can be sealed with a multi-layer lidding film to achieve satisfactory gas tightness for the use of modified atmosphere packaging (MAP) in food packaging, if the manufacturing process of the tray is of sufficient quality.

- The quality which makes satisfactory, leakproof sealing possible can be quantified by evaluating the crease geometries with microscopic analysis and the surface quality of the sealing area of the tray (the rim area aka tray flange).

1.4 Scope of the thesis

The subtext of the research focus is presented in Figure 1. The author's view of the critical factors in the heat sealing of press-formed trays is presented in Figure 2.

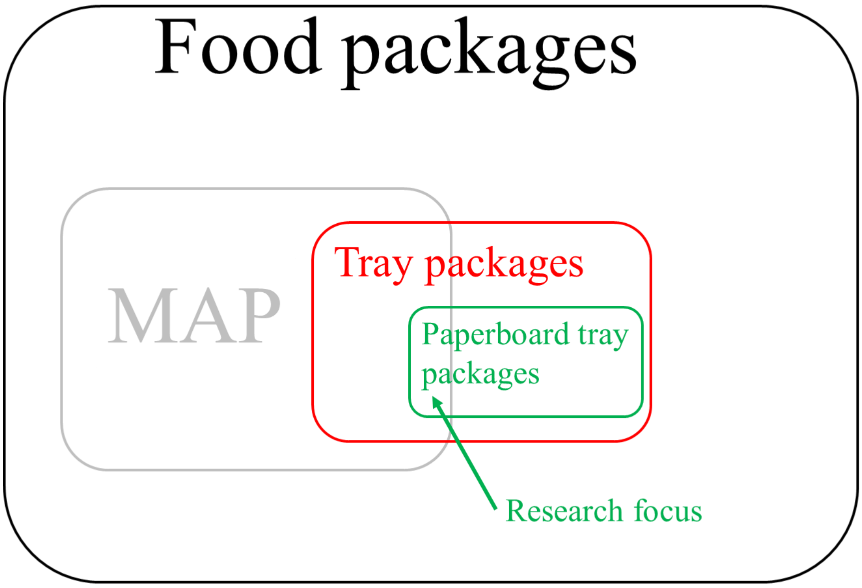

Figure 1. Subtext of the research focus (pointed with the arrow).

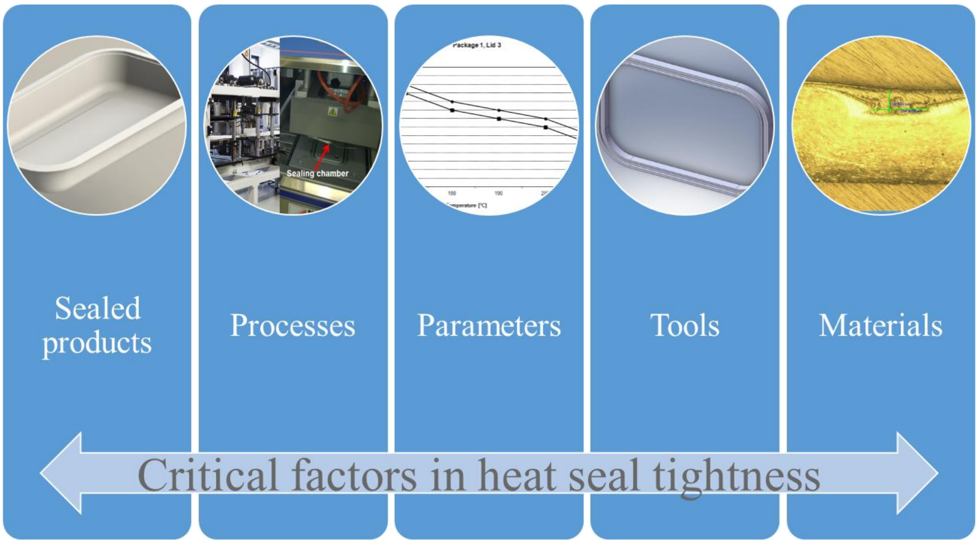

Figure 2. Author's view of the critical factors in the lid heat sealing of press-formed paperboard trays.

The work is divided into seven subcategories, and the synthesis of the thesis is based on seven articles which deal with the following subcategories:

1) Background for heat sealing of paperboard trays and the effect of sealing temperature on the sealing time and liquid tightness in heat sealing of paperboard trays
2) The effect of press forming mould clearance and material thickness on the quality of paperboard trays
3) The effect of blank holding force on the quality and gas tightness of press-formed paperboard trays
4) Methods for microscopical analysis of formed creases in press-formed paperboard trays
5) Surface roughness analysis of formed trays
6) The effect of tray dimensions on the gas flushing and heat sealing of trays
7) The effect of sealing pressure and crease geometry on leak-proof sealing of press-formed paperboard trays

The main goal of the synthesis phase is to determine the impact of different process phases and parameters on the tightness (leak-proof quality) of the heat seal in polymer-coated paperboard trays that have been sealed with a lidding film. Both the effect of press forming of trays and heat sealing of the lidding film processes are investigated. Also different ways to analyse the quality – and to define the actual quality of trays than can be reliably sealed are investigated. The structure of the thesis is presented in a flow-chart form in Figure 3.

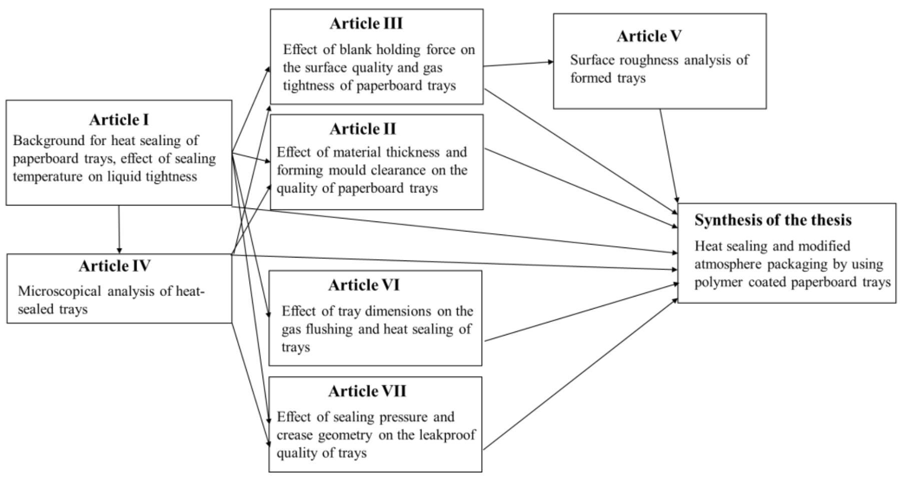

Figure 3. Structure of the thesis.

This thesis focuses on the mechanical aspects of the press forming and heat sealing processes. The focus in the trays used in the experiments was in the tray flange (rim area), and there were no pinholes or other defects outside the sealing area. The experiments were done by using commercially available materials, but some of the forming experiments were done with equipment that is not currently commercially available. Material properties and their investigation were limited to the key properties essential for the scope of the articles.

1.5 Outline

Chapter 2 is an introduction to the different processes that are used in 3-dimensional forming of paperboard.

Chapter 3 is an introduction to the heat sealing and MAP processes.

Chapter 4 presents the main materials and methods used in the experiments.

Chapter 5 contains a review of the results and discussion.

Chapter 6 presents conclusions of the work.

2 Paperboard trays and three-dimensional forming of paperboard

3D forming of paperboard trays, plates and other products can be done with several methods. The 3D forming processes used in forming paperboard-based products include press forming, deep drawing, hydroforming, thermoforming and pulp moulding. Press forming can also be combined with injection moulding. There is some variance in these terms regarding the exact process and what term is used for it. However, the terms that are used here are mentioned several times in the literature. The different processes used for 3D-form paperboard are introduced in this chapter.

According to Östlund et al. (2011), there are two main categories in forming double-curved paper structures. The first is spraying pulp onto a mould, the other is to form paper or paperboard that is produced in a traditional fashion.

Lately there has been increasing interest and more publications regarding the 3D forming of paperboard, but in the past a lot of research behind commercial paperboard-based packages seems to have been done by the industry (Östlund et al. 2011).

Paperboard and other fibre-based materials tend to cause difficulties during 3D forming processes. The quality of 3D-formed paperboard products is uneven, and the formed parts show commonly distinctive wrinkles, abrasion at wrinkles and discoloration. (Hauptmann and Majschak 2011)

Vishtal (2015) divides the forming processes of paper-based materials to two main groups: sliding (such as deep-drawing and stamping aka press forming) and fixing blank processes (such as air/vacuum forming, hydroforming and hot pressing). In the sliding blank processes the forming is caused by the sliding of paper into the mould and lateral contraction of paper. This causes microfolding of the paper. In the fixed blank processes the paper is formed via straining of the paper. This is a generalized view, however, because the blank holding force can be adjusted to form a shape with the best possible appearance, and straining can occur also in the sliding blank processes. Also in fixed blank processes lateral microfolding can occur to some extent (Vishtal 2015).

2.1 Press forming and die cutting of blanks

Press forming (aka tray pressing, press moulding, stamping, sometimes also called deep drawing or thermoforming) of paperboard-based materials is done by using moulding tools which consist of a male mould (punch), female mould (die) and a blank holder (rim tool) (Tanninen 2014). The principle of the process is introduced in Figure 4. The main parameters in press forming are: forming force (F_1), forming speed (v), blank holding force (F_2), temperature of the male mould (T_1), temperature of the female mould (T_2), and the dwell time. Both coated and uncoated materials can be used, depending on the application.

Press-formed paperboard trays and plates are used in the packaging of various food products such as fast food, ready-to-eat meals and frozen food. Press-formed trays are not widely used in modified atmosphere packaging (MAP), however. A major factor in this is the quality of industrially manufactured trays, which does not enable gas-tight sealing of the lidding film (Hauptmann et al. 2013, Leminen et al. 2015a). An example of a press-formed tray is shown in Figure 6b.

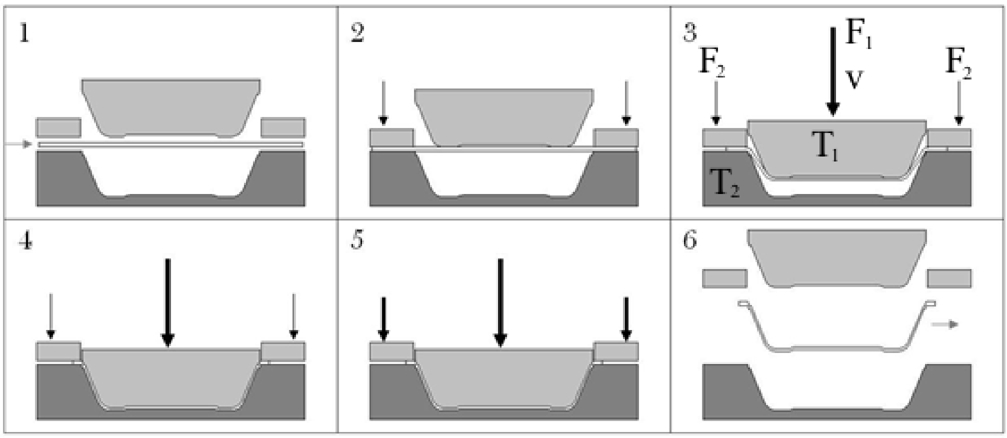

Figure 4. The press forming process. The main forming parameters are visible in the top right corner (modified from Leminen et al. 2013).

Phase 1: The paperboard blank is positioned between the moulding tools.

Phase 2: The blank holding force tightens the blank between the blank holder (rim tool) and the female tool.

Phase 3: The male tool presses the blank into the mould cavity in the heated female tool. The folding of the tray corners is controlled with blank holding force.

Phase 4: The male tool is held at the bottom end of the stroke for a set time (0.5 to 1.0 s).If the tray is coated, the plastic coating softens, and creases in the corners of the tray are sealed together.

Phase 5: The flange of the tray is flattened by the blank holder.

Phase 6: The formed tray is removed, and a new blank can be fed into the tray press. The tray achieves its final rigidity when it cools down.

The typical grammage of paperboards used in press forming varies roughly from 200 to 450 g/m^2, while the used coating grammage varies roughly from 10 to 70 g/m^2. The material properties of paperboards suitable for 3D-forming has been researched by Vishtal (2015), who states that the paperboards suitable for sliding blank processes should have low compressive strain and strength, a low paper-to-metal friction coefficient and low elastic recovery.

Press forming is usually done by using die cut blanks which have been pre-creased to enable better formation of trays. If the blanks are not creased, wrinkling will appear nonetheless, but the formation and location of wrinkles is not as controlled as with pre-creased blanks. Tanninen et al. (2015a) discussed the effect of creasing tools on the quality of press-formed trays. In tray pressing, creases are used to fold excess material in the tray corners, while traditionally creases are used as hinges, for example with paperboard cartons (Tanninen et al. 2015a). This means that the use of creases in the press forming process is much more complex compared to the folding process, as the geometry of the tray does not contain clear faces and the shape of the corner of the tray consists of multiple folds and is rounded (Tanninen et. al 2015b).

The cutting and creasing are done by using a die cutting machine, which can be either rotary or flatbed. In flatbed die cutters, the blanks are cut and creased by a die which consists of cutting knives and creasing rules. The cutting is done by knives with sharp edges, while the creases are made by creasing rules with round edges. These rules are thin strips of metal with rounded edges which indent the surface of the board and push it into a groove on the other side of the paperboard. The groove is formed in a thin, hard material which is called the matrix or the counter die (Kirwan 2008). The principle of making a crease and the main dimensions of the creasing rule and groove are presented in Figure 5 (Tanninen 2015c).

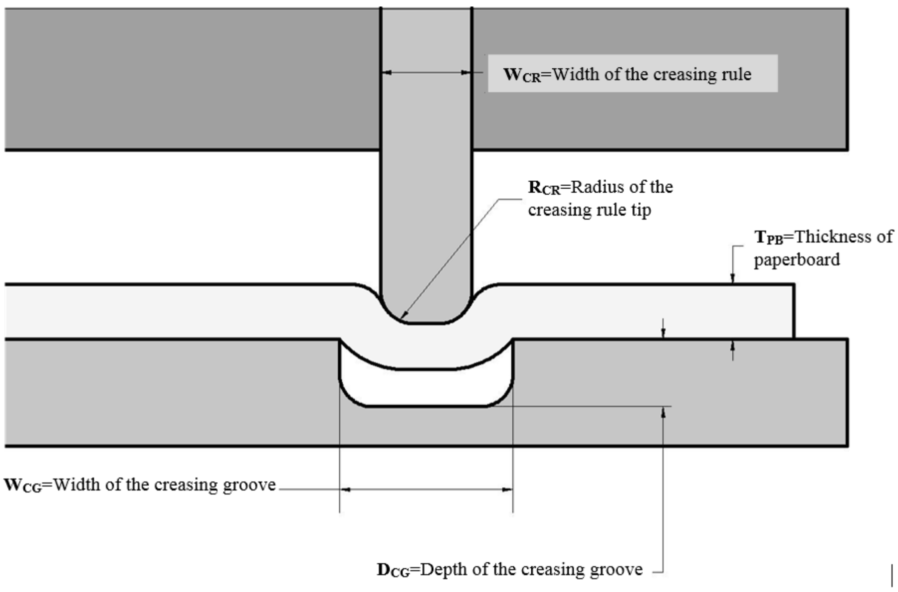

Figure 5. Principle of creasing and the main dimensions of the creasing rule and groove (Tanninen 2015c).

Toolsets with different creasing coefficients and creasing groove profiles were compared by Tanninen et al. (2015a). The creasing coefficient defines the creasing groove width in relation to the substrate thickness, and is normally between 1.2 and 1.7. Toolsets with wider creasing grooves tended to produce wider folds with smaller thicknesses in the tray walls. According to the results, the dimensions of folded creases after tray forming varied by 5-10 % when different creasing rule profiles were compared. It is quite clear that pre-creasing has a major effect on the formability of paperboard trays, but as long as the actual creasing toolset is selected according to material thickness and instructions given by die cutting tool and paperboard manufacturers, the creases should work as planned in tray forming, and the geometry of the creasing tool would have only a minor effect on the folding behaviour of the tray corner (Tanninen et al. 2015a).

The trays manufactured for this thesis and the articles in it concentrate on heat sealing of trays manufactured from polymer coated paperboard, which was pre-creased and cut to blanks and then pressed into tray-shape by the press forming process. Examples of blank geometry and a tray produced by press forming are presented in Figure 6. The main equipment used in the studies are presented in Chapter 4. Press forming was selected because it is a widely used method in paperboard tray and plate manufacturing in the package manufacturing industry.

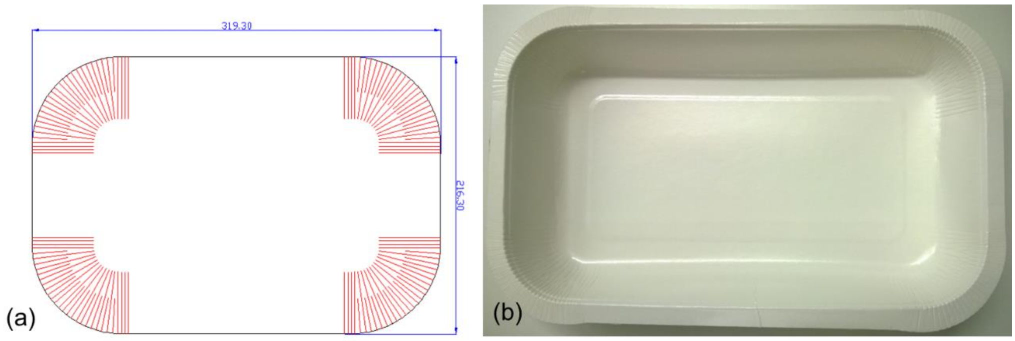

Figure 6. (a) A typical blank geometry and creasing pattern. The creases are presented in red. (b) A rectangular tray produced by press forming.

2.2 Deep drawing

Hauptmann and Majschak (2011, p.420) describe deep drawing of paperboard as being a process in which “a blank is drawn through a shaping cavity into a calibration cavity by using a die.” In addition to this, a blank holder is used by positioning it to a set distance towards the shaping cavity. This is done to avoid the material from standing up during drawing. The process is described in Figure 7. Also a counter holder can be used, but it is not necessary.

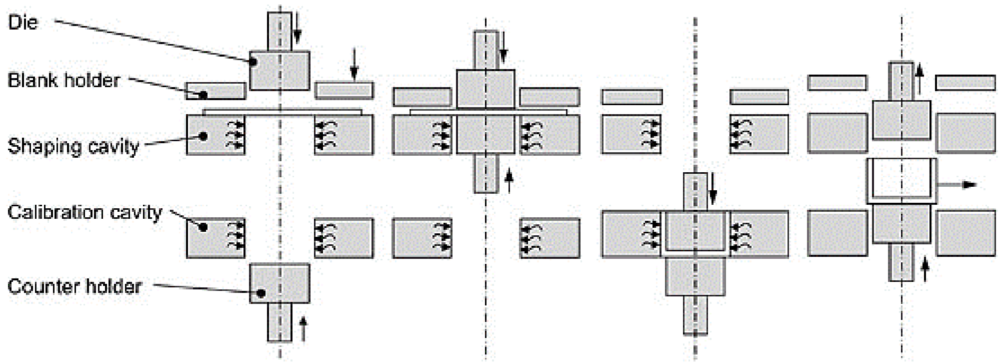

Figure 7. Deep drawing of paperboard (Hauptmann and Majschak, 2011, p. 420).

Deep-drawn paperboard products are common in only few applications, which include low-quality cheese packaging, microwave food cups, egg packaging etc. According to Hauptmann and Majschak (2011) this is due to the difficulties caused by fibre-based materials during the 3D forming processes.

The quality of deep-drawn paperboard cups has been evaluated by a few methods. One strategy is to evaluate fractures and structural damage, and another is to evaluate the shape accuracy, shape stability and visual quality of the packages (Hauptmann and Majschak 2011). Visual quality can be at least partially evaluated by counting and measuring the wrinkles that appear during forming. This has been discussed by Hauptmann and Majschak (2011) and Wallmeier et al. (2015). Basically high wrinkling and uniform distribution are desired, as a low number of wrinkles tends to cause defects and more pressure in the gap between the punch and the die.

2.3 Hydroforming

Hydroforming (as well as deep drawing) are common processes for sheet metal forming. To adapt a hydroforming process for paperboard, the requirements for this kind of process must be clarified. (Groche et al. 2012)

Östlund et al. (2011) discuss a solution for the hydroforming of paperboard which works by applying pressure on a rubber membrane which inflates like a balloon above the paper specimen. The edge of the paper specimen can be restrained by pressure from the ring outside the mould. The process parameters for hydroforming are forming pressure, the flow rate for the pressure application and the length of time the specimen stays in the mould (Östlund et al. 2011). In comparison to press forming, hydroforming uses a flexible membrane, while press forming uses rigid moulds. Even though Vishtal (2015) considers hydroforming as a fixed blank process, according to Groche et al. (2012) and Östlund et al. (2011), the sliding of the blank can be controlled by controlling the blank holding force. This would indicate that hydroforming could be defined as a sliding blank process. The process has been currently applied only in laboratory scale (Vishtal 2015).

2.4 Thermoforming

In thermoforming, heated thermoplastic sheets are formed and shaped with the assistance of mechanical load and/or vacuum/pressure (Pettersen et al. 2004). Thermoforming is widely used with polymer-based materials for the manufacturing of pre-formed packages, and also with so called form-fill-seal (FFS)-lines, but only few applications for fibre-based materials exist. This is probably mainly because the forming appears mostly by straining the material, as in commercial equipment the web is fixed from the sides. Thermoforming can therefore be considered a fixed blank process, even though the forming is usually done when the web is attached without a separate blanking stage.

2.5 Pulp moulding

Moulded pulp is widely used as a packaging material for protective packaging, for food service trays and beverage carriers, and for the packaging of fruits or berries. A well-known example of a product manufactured by pulp molding is the molded fibre egg package. Molded fibre products can withstand grease and fat for a moderate time. (Järvi-Kääriäinen and Ollila 2007)

The manufacturing process consists of mixing water, (recycled) paper and possibly microspheres and starch powder into a pulp and pouring the pulp composite to a mould. The mould is heated with steam, and the pulp is heated to a temperature of about 100 °C. The moulded product is released after the product has dried. The cycle time can be around 90 seconds (Noguchi et al. 1997). Products manufactured by pulp moulding have a rough surface and are limited by a demoulding angle of at least 7° (Hauptmann and Majschak 2011).

2.6 Combined press forming and injection moulding

In combined press forming and injection moulding the tray is formed from pre-creased and cut paperboard blanks by the press forming process, but the rim area (tray flange) is made of injection-moulded plastic. This results in a flat sealing surface and improved rigidity of the manufactured trays. This kind of solution is used by Delight Packaging Oy. The setbacks in this process are slower production speed, increased price and reduced fibre percentage of the package, although in Finland the package can be recycled similarly to paperboard milk cartons. However, due to the flat sealing surface the tray is easier to seal tightly by using MAP, compared to trays manufactured without the injection-moulded rim. An example of an injection-moulded tray flange can be seen in Figure 8.

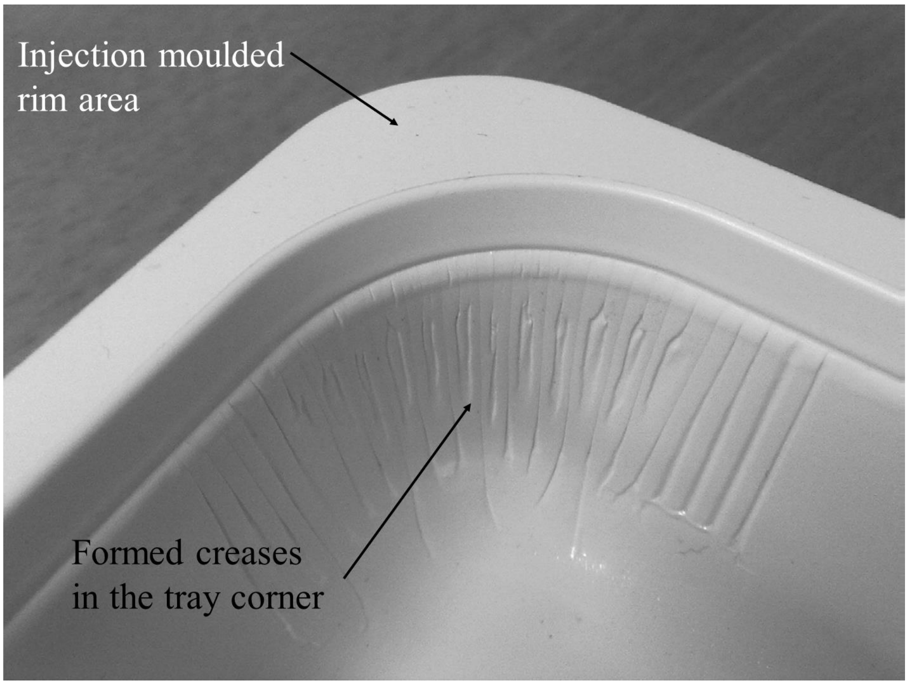

Figure 8. Injection-moulded tray rim.

2.7 Summary and characteristics of forming processes

Table 1 presents a rough comparison of the forming processes described above.

Table 1. Rough comparison of forming processes for fibre-based materials.

Process	Strengths	Weaknesses	Lid sealing
Press forming	Widely used industrially, good production speed, high drawing depths (up to 70 mm) [1*]	Usually requires pre-creasing, the quality of the rim area a challenge [1*-3*]	Possible, MAP challenging [1*-3*, 8*]
Deep drawing	High drawing depths possible without pre-creasing [4*]	Uniform distribution of wrinkling challenging, forming of the tray rim area requires modifying the process [4*, 5*]	Challenging without a separate rim area in the formed product
Hydroforming	Even distribution of load on the material [6*]	Not widely adapted, used only in laboratory scale [6*]	Plausible
Thermoforming	Widely used machinery	Machinery and tooling not suitable for fibre-based materials, requires high stretch from the material, currently achieved drawing depth low [6*]	Possible, sealing area should appear flat
Pulp moulding	Shape diversity, widely used process [7*]	Slower production speed, weak barrier properties, poor appearance [4*,7*]	Requires separate sealing layer to be added
Combined press forming and injection moulding	Improved tray rigidity, flat sealing surface [8*]	Slower process, reduced fibre-% of trays, more expensive [8*]	Possible with MAP [8*]

[1*] Tanninen et al. (2015b); [2*] Leminen et al. (2015a); [3*] Leminen et al. (2015b); [4*] Hauptmann and Majschak (2011); [5*] Wallmeier et al. (2015); [6*] Vishtal (2015); [7*] Noguchi et al. (1997); [8*] Leminen et al. (2012)

3 Heat sealing of paperboard trays and modified atmosphere packaging (MAP)

Heat sealing can be defined as a method for joining two thermoplastic materials. It is typically used for forming bags or sealing packages. (Mueller et al. 1998)

3.1 Principle of heat sealing

The basic idea of heat sealing technology is to attach and heat both sides of two thermoplastic adherents. In the most commonly used thermal press type of heat sealing, heat is conducted from the surface of the thermoplastic films by a heat jaw and the heat is then conducted to the heat sealing zone through the film. The bonded surface is first heated to an appropriate temperature, and then cooled down to complete the bonding. Heat sealing can be used to create airtight closures which can prevent all bacterial incursions. (Hishinuma 2009)

In conventional heat sealing, the actual temperature of the melting surface is not controlled, but the surface temperature of the heat generator is. The appropriate heating temperature range depends on the thermoplastic films that are sealed. (Hishinuma 2009) The main critical control elements for heat sealing are temperature, time and pressure. The most common method to control the heat sealing process has for decades been adjusting the temperature of the heating block (the heating source) (Hishinuma 2009). To achieve a reasonable bond, adequate pressure on the surfaces must be used for a sufficient time so that the polymer chains can diffuse and form bridges across the interface (Mueller et al. 1998). The most common shapes for packaging materials that utilize laminate films are bags or pouches (Tetsuya et al. 2005).

Other methods for heat sealing include ultrasonic welding which uses high-frequency ultrasonic acoustic vibrations under pressure to generate heat to the sealed materials (van Oordt et al. 2014) and induction sealing which uses an electromagnetic field to heat a metal material to heat a polymer based sealing layer (Babini et al. 2003).

3.2 Heat sealing of paperboard trays and the main challenge

The heat sealing of press-formed paperboard trays is more challenging than the heat sealing of polymer-based trays. The main reason for this are the capillary tubes in the sealing area caused by wrinkles (Hauptmann et al. 2013, Leminen et al. 2012, Leminen et al. 2015a). These wrinkles are caused by several things: compressive forces in a transverse direction in the material (Vishtal and Retulainen 2012), the material properties (lower formability, stiffness) of paperboard which force the material to wrinkle. When paperboard is formed 3-dimensionally, wrinkles cannot be completely avoided (Hauptmann and Majschak 2011). Wrinkling can be controlled by pre-creasing the paperboard blanks to control the location where wrinkling appears and to enable the forming of deeper geometries. The rim area (the surface where the lidding film is sealed on) of the paperboard trays is thus more uneven and more challenging to seal as leak-proof than with polymer-based trays, or paperboard trays with injection moulded plastic rim area, which usually have very flat sealing surfaces. This presents a challenge to the leak-proof sealability of paperboard trays and is a major contributing factor when thinking about paperboard trays becoming more common with the use of MAP.

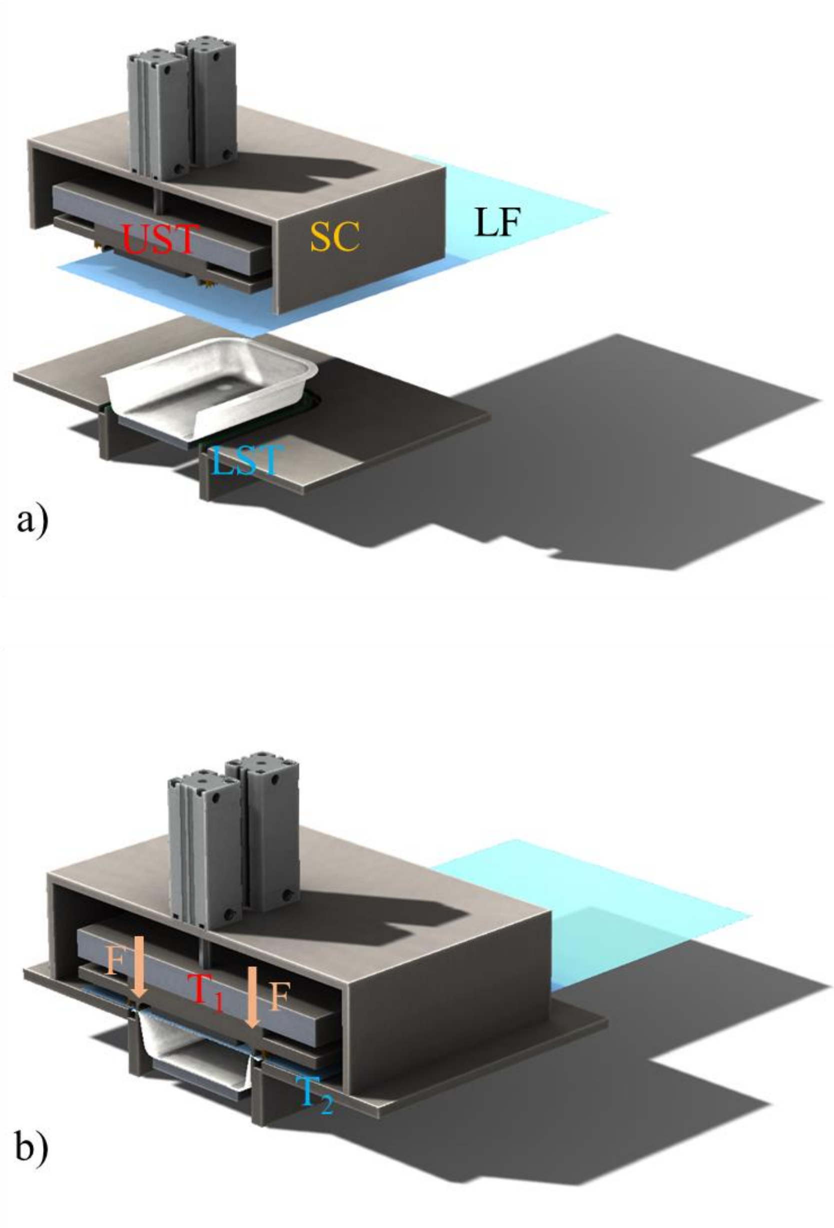

Figure 9. The heat sealing process.

A schematic of the heat sealing of a paperboard tray is shown in Figure 9 a). The paperboard tray is located between the sealing tools. In 9 b) the lower sealing tool (LST) lifts the tray under the tray flange, the sealing chamber (SC) is closed, Then, the sealing tools (UST and LST) are pressed together with a set force (F), the tray and the lidding film (LF) are sealed together for a set time, and the seal is formed. At the same time, a sharp blade cuts the lidding film according to the tray geometry. Usually the upper healing tool is heated (T_1) while the lower sealing tool is at room temperature (T_2). The heat sealing process is often combined with modified atmosphere packaging, in which case a vacuum is generated to the chamber by removing the oxygen from the package. After that the tray is flushed with a protective gas before sealing the lidding film.

The heat sealing of lidding films has not been widely reported for paperboard trays, except for some patents which present different solutions to acquire an adequately tight sealing result. Faller (1982) discusses a solution which combines ultrasonic sealing and heat sealing to improve the bond between the film and the plastic surface of the tray. Seiter and Gould (1984) have presented a solution where a hot melt or a wax is applied to the indentations (creases or wrinkles in the tray). Wilkins (2009) discusses both the manufacturing process of the paperboard tray and the heat sealing of the lidding film to achieve a hermetic sealing. In this solution, crease lines are formed to project out of the inner face of the blank, and two spaced-apart adjacent heating points are applied to the rim area of the tray to form a double seal. The aim of these patents seems to be achieving a gas-tight sealing result. Also Hauptmann et al. (2013) discuss the topic. In their article, paperboard trays manufactured from pre-creased blanks were not able to achieve adequate tightness properties.

The low number of journal articles on the heat sealing of paperboard trays suggests that the research has been mainly done in research and development projects by the industry. However, the patents indicate that there is interest in replacing polymer-based packages in food applications with polymer-coated paperboard trays. The focus of the work in this thesis is to achieve a satisfactory, leak-proof result by using press forming and heat sealing without extra process phases.

3.3 Modified Atmosphere Packaging (MAP)

Modified Atmosphere Packaging (MAP) is defined as “the packaging of a perishable product in an atmosphere which has been modified so that its composition is other than that of air” (Hintlian & Hotchkiss, 1986, p.71).

MAP is used to slow down microbiological growth in food. Air causes many food products to spoil rapidly due to a reaction with oxygen, growth of aerobic microorganisms such as bacteria and moulds, or moisture loss or uptake. Microbiological growth can render food potentially unsafe for human consumption by changing the texture, colour, flavor and nutritional value of the food. (Coles et al. 2003)

The shelf life of food products can be extended and product presentation improved, making the product more attractive to the retail customer when food is packed in a modified atmosphere. (Coles et al. 2003)

Three main gases are used in MAP: oxygen (O_2), carbon dioxide (CO_2) and nitrogen (N_2). The gas should be selected according to the food product being packed. The gases can be used singly or they can be mixed to balance the safe shelf life extension and optimal organoleptic properties of food. (Coles et al. 2003)

Carbon dioxide is often used in gas mixes for fresh meat products due to its antimicrobiological properties (Daniels et al. 1985). It slows down the growth rate of microorganisms and thus increases the shelf life of food. Mullan and Mcdowell (2003) state that the antimicrobial effect is higher when the products are stored under 10^{o} C when compared to products that are stored at temperatures above 15^{o} C.

Nitrogen is often used as a filler gas because packages with high concentrations of CO_2 tend to collapse, as CO_2 has a high solubility in meat tissue. N_2 is used to replace O_2 in the packages to slow down rancidity and stop the growth of aerobic microorganisms. (Arvanitoyannis and Stratakos 2012)

Oxygen is generally used in MAP mixed with N_2 or CO_2 to preserve a desirable cherry red colour of meat (Kropf 2004). However, storage of meat under high-oxygen atmospheres has been found to reduce its quality (Monahan 2003, Lund et al. 2007). Certain types of food can be damaged when exposed to oxygen concentrations of 1-2 %. The level of residual oxygen in the package headspace is a concern for food processors, and should therefore be under 1 % for many products (Coles et al. 2003).

Leakage of MAP can cause reduction in the sensory shelf life and microbiological quality of packed food (Randell et al. 1995). Because leakage can often occur more easily with paperboard trays than with trays manufactured from polymer materials, the factors affecting the leak-proof sealing of paperboard trays are of great interest.

3.3.1 **MAP machinery and compensated vacuum gas flushing**

The basic function of MAP machines is to modify the atmosphere and seal the package while retaining the product, as well as to cut and remove waste in producing the final pack. When pre-formed trays are packed by using MAP, the filled pack is loaded into the machine and the chamber is closed. A vacuum is then pulled in the chamber and the package is flushed with the modified atmosphere. The package is then sealed with heated tools and the chamber opens. After that the pack can be removed and the cycle repeated. (Coles et al. 2003).

Depending on packed product, the tray geometry and the used sealing equipment, it is possible that some residual oxygen is still left in the package. As stated above, the amount of oxygen in the headspace of the package should be as low as possible, and almost always under 1 %. Nowadays, tray packaging using modified atmosphere packaging (MAP) consists mostly of rigid plastics.

3.3.2 Barrier properties, oxygen transmission rate and analysis of package integrity

Barrier properties are necessary to protect the packed product (Kirwan 2008). Barriers separate a system, for example the packed food, from the environment. Barrier polymers limit the movement of substances through the polymer, or in some cases, into the polymer. These substances are called permeants (Delassus 2002). The required protection type must be defined to select the type, amount and thickness (coating weight) of the barrier materials to meet the needs of the required protection (Kirwan 2008).

There are several types of protection requirements for packages. These include barriers to moisture and moisture vapour, gases such as oxygen, carbon dioxide and nitrogen, and to oil, grease and fat. (Kirwan 2008)

The oxygen transmission rate (OTR or O_2TR) is measured as the amount of O_2 gas that passes through a substance over a given time (Yam 2009). OTR values are usually measured as $cm^3/m^2/24h$. OTR is an important value for MAP because it has an effect on the shelf life. Exact values for optimal OTR are hard to define and depend on packed product, but generally if perishable products are packed, a low OTR is desired. Dawson et al. (1995) investigated packaging films with OTR values ranging from 30 to 12,000 $cm^3/m^2/24h$ using ground chicken meat. The growth of aerobic bacteria was significantly reduced when packed with a film with an OTR of 30 $cm^3/m^2/24h$ compared to films with OTR's from 2,000 to 12,000 $cm^3/m^2/24h$ (Dawson et al. 1995).

The package integrity, headspace and gas composition can be analysed by using several methods. One common method is to use the dye penetrant test method according to a standard, e.g. the European standard EN 13676, ASTM F1929-12 or ASTM F3039-13. In industry methods, such as leak detection systems which form a vacuum into a chamber and detect leaks, are used. A good method for following the change in gas headspace over time is to use an optical fluorescence O_2 analyser (EN 13676, ASTM 2012, ASTM 2013a, ASTM 2013b, Witt 2014, Leminen et al. 2015c).

4 Materials and methods

The materials and methods are presented in detail in the articles (I – VII). The general materials and methods for the articles are presented in Table 2.

Table 2. The main materials and methods used in the articles.

Article	Materials	Methods
I	290 g/m^2 paperboard + 40 g/m^2 PET, 290 g/m^2 paperboard + 40g/m^2 PE, various multi-layer lidding films	Literature review, press forming, creasing, heat sealing, detection of leaks with a colouring solution
II	190 g/m^2 paperboard + 40 g/m^2 PET, 230 g/m^2 paperboard + 40 g/m^2 PET, 310 g/m^2 paperboard + 40 g/m^2 PET, 350 g/m^2 paperboard + 40 g/m^2 PET	Press forming, creasing, microscopic analysis, visual grading
III	350 g/m^2 paperboard + 40 g/m^2 PET, multi-layer lidding film	Press forming, creasing, heat sealing, MAP, detection of leaks with a colouring solution, oxygen content analysis
IV	350 g/m^2 paperboard + 40 g/m^2 PET, multi-layer lidding film	Press forming, creasing, heat sealing, microscopic analysis, detection of leaks with a colouring solution
V	350 g/m^2 paperboard + 40 g/m^2 PET	Press forming, creasing, chromatic white light 3D-profilometry
VI	350 g/m^2 paperboard, 350 g/m^2 paperboard + 40 g/m^2 PET, multi-layer lidding film	Press forming, creasing, heat sealing, dimension measurements by machine vision, oxygen content analysis
VII	350 g/m^2 paperboard + 40 g/m^2 PET, multi-layer lidding film	Press forming, creasing, heat sealing, oxygen content and permeation analysis, detection of leaks with a colouring solution

The base board (Stora Enso Trayforma Performance) used in the articles consisted of three solid bleached sulphate (SBS) layers. The lidding film used in Articles II-VII was a multi-layer film (Westpak WestTop 405B PET) with the total thickness of about 115 µm, consisting of a PET-sealable inner layer and several barrier layers.

The main equipment that was used in the experiments consisted of the following (a more detailed description of the specific equipment and processes can be found in Articles I-VII). The LUT Packaging line (aka the Flexible Packaging Line of the Future or the Adjustable Packaging Line of the Future) is a line that is used to produce paperboard trays. It has separate die cutting and press forming units, and the forming parameters can be adjusted very accurately for research purposes. The line also includes a quality control unit which utilizes machine vision and can be used to analyse for example the dimensions of the trays. The line can be seen in Figure 10. The trays manufactured for Articles II-VII were manufactured with this line. The trays manufactured for Article I were produced with a commercial press forming machine (Markhorst VP3-70).

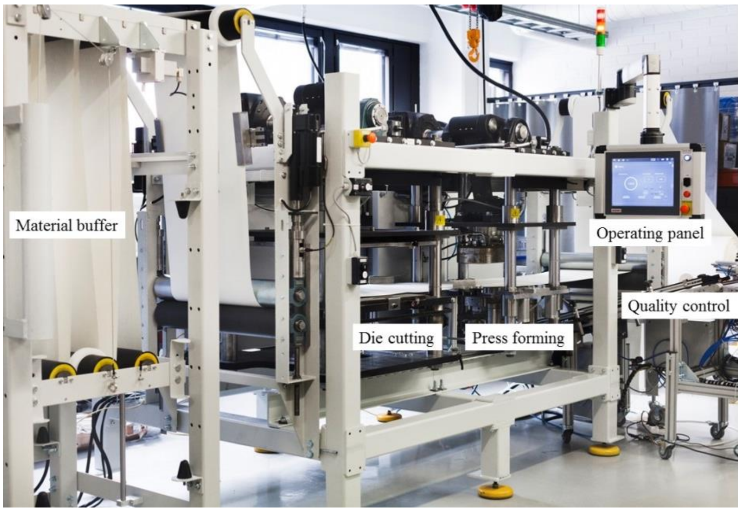

Figure 10. LUT Packaging Line (modified from Laitinen 2012).

The sealing equipment (Ilpra Speedy) used in this thesis is presented in Figure 11 and the sealing process and tooling are clarified in Figure 9. The sealing equipment was modified by adding a precision pressure regulator (Festo LRP-1/4-10), which could be used to adjust the sealing pressure when needed. Also the sealing tools were tailored specifically to be used with paperboard trays. The sealing experiments in Article I were done by using a different sealing machine manufactured by Satmec, while all the other sealing experiments were done with the equipment presented in Figure 11.

The tested paperboard materials were stored in a constant humidity chamber (Weiss) to obtain the desired moisture content, which was verified with a moisture analyser (Adams

Equipment PMB 53) before the converting trials. The oxygen content measurements were made with a Mocon Optech O_2 Platinum analyser.

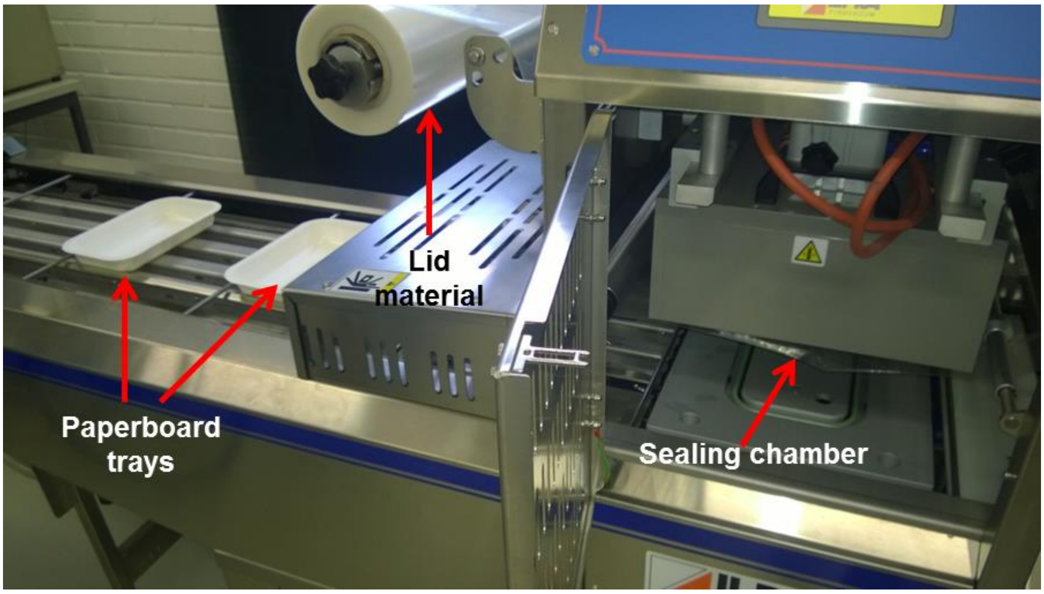

Figure 11. The equipment used in the heat sealing and MAP experiments (modified from Leminen et al. 2015a).

5 Review of the results and discussion

A brief description of the work and the main results are presented in this chapter. More detailed information can be found in each article (I – VII). In addition to the summary of different papers, chapters 5.1 and 5.3 contain some unpublished results.

5.1 Background for heat sealing of paperboard trays, the effect of sealing temperature on liquid tightness

Paper I presents the background and main challenges for leak-proof sealing of a lidding film into a press-formed paperboard tray. Also patents that suggest possible solutions to achieve a tight seal are presented. The experimental part of the article investigates the possibility to achieve a liquid-tight seal, as well as the effect of the sealing temperature on the sealing time.

Food packaging very often requires the use of MAP. Packaging food in paperboard trays by using MAP is challenging because of discontinuity tunnels that are formed by creases or wrinkles in the corner areas of the packages. One step towards using MAP is to make the package liquid-tight.

Several patents have been presented to overcome the leakage caused by wrinkling in press-formed plastic-coated paperboard trays. These solutions include combined ultrasonic bonding and heat sealing, adding a hot melt or wax to the rim area, injection moulding a separate plastic rim to the tray, and high temperature heating ridges. What is common to these methods is that they all require additional process phases or modifying or creating new equipment, which is not a desired procedure.

Six different tray and lid combinations were tested to investigate the tightness of the press-formed trays. The trays were manufactured by using an industrial forming machine. The leak inspection was done by flushing the sealed trays with a colouring solution in accordance with the European standard EN 13676 (2001). The tested trays were manufactured by using a commercial forming press, Markhorst VP3-70, and the trays were sealed by using a Satmec tray sealer, with a flat, heated upper sealing tool, which resulted in even pressure throughout the tray flange area. The tray dimensions were 209 x 139 x 35 mm. The width of the tray flange (and hence the seal width) was 10 mm. The sealing pressure was 6 bar.

A liquid-tight package was acquired with all six combinations. The sealing time and temperature for each material combination was found to vary quite a bit, and optimization of these parameters is crucial when maximum production speeds are desired. Of the tested material combinations, the fastest sealing time that resulted in liquid-tight seals was 1.2 s. As expected, the most decisive physical factor found to affect the tightness in the packages was the creases in the corner area of the trays. The results confirmed the assumption that the leaks always occur first in the creased area. A leak highlighted by the colouring solution is shown in Figure 12. Even though it was known that the sealing time has an effect on the tightness of the seals, an interesting result was that liquid tightness was acquired with all the tested lid and tray combinations.

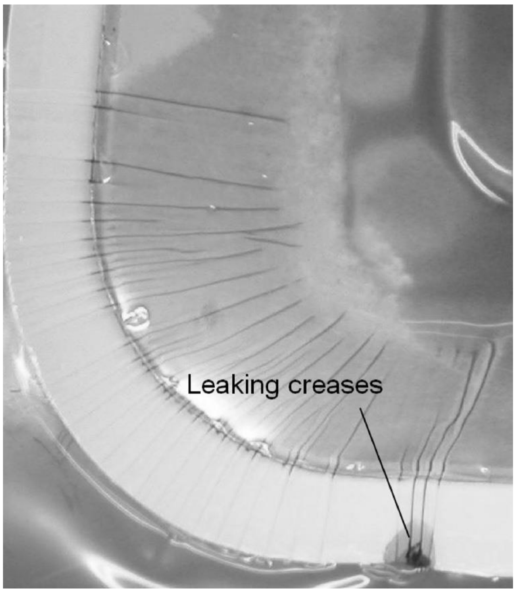

Figure 12. Leaking problem area of sealing. Two creases highlighted by dye penetrant examination.

Additional experiments were done to investigate the gas tightness with all lidding film combinations. The trays were flushed with a gas mix of 70 % CO_2 and 30 % N_2. The results showed that there was significant leakage of MAP in all lid and tray combinations (Figure 14). Figure 13 shows a typical crease geometry with the samples manufactured with the commercial forming press used in Paper 1. The average O_2 content (Figure 14) after 14 days was close to the content in air (20.95 %). This was suspected to be caused by creases that were not sealed properly, were too deep in depth and caused capillary tubes, which subsequently made the gas to leak.

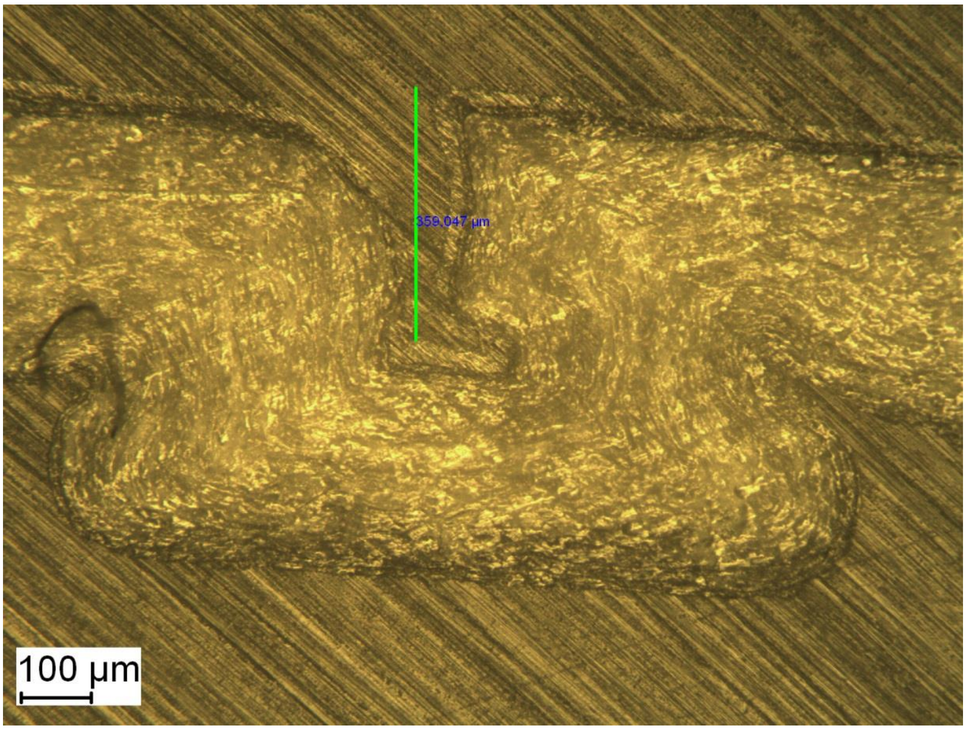

Figure 13. Typical insufficient crease geometry for MAP in the sealing area (tray flange) of a tray. Depth of the crease is approximately 360 µm.

Gas flushing was also found to be incomplete, resulting in residual air in the package (Figure 14). The most likely reason for the incomplete flushing is the dimensional inaccuracy of trays, which is more thoroughly discussed in Article VI.

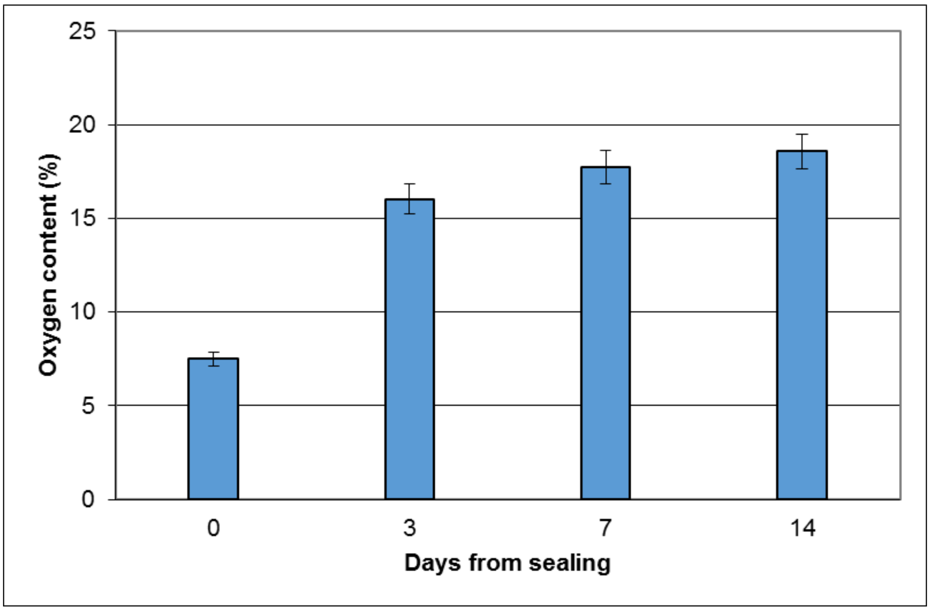

Figure 14. Oxygen measurement averages of trays with insufficient quality in the sealing area.

Microscopic analysis of the corner area showed the crease geometry to be improperly sealed, and the average depth of the formed creases was measured to be 351 µm before sealing, which was too deep to achieve a gas-tight sealing result. The crease formation was similar in all samples, indicating that the creases were not sealed properly in the press forming, therefore forming a tube that caused leakage.

5.2 Effect of material thickness and forming mould clearance on the quality of paperboard trays

In Paper II, the effect of material thickness and subsequently the forming mould clearance on the quality of formed trays was investigated. Clearance is the distance between the forming surfaces of the tray manufacturing tools. Because clearance cannot be adjusted after the forming tools have been manufactured, the suitable clearance must be defined in advance for the used material thickness.

The forming phenomena of the paperboard tray corner were studied by doing a series of converting tests with varying material thicknesses. The forming was studied to obtain data for better forming process control and subsequently better end product quality. The corner is the area where the most severe deformation in the trays occurs, and it is therefore the area most likely to have cracks or other faults that can cause leaks in the package. The quality of the tray flange (rim area) surface in the tray corners is critical for a tight seal when the package is sealed with a lid.

As stated above, the creases in paperboard trays work differently in the paperboard press forming process compared to traditional folding. The folding of the paperboard blank was controlled with a pre-creasing pattern which was done to represent a typical creasing pattern in the tray pressing process. The target was to obtain evenly folded creases, smooth tray walls and flat flanges of the tray.

The forming result was analysed both visually and by microscopic analysis to determine suitable mould clearances. The formed creases were analysed in the machine direction (MD), cross direction (CD) and at a 45-degree angle.

The results showed that the recommended material thickness would be from 95 to 135 % of mould clearance for the tested paperboard types and example trays. It must be noted, however, that mould clearance does not directly adjust the tray flange area. This is because the flattening force in the rim area can be adjusted independently. However, the dimensions of the creases in the rim area changed in relation to the material thickness (as was the case in the whole tray). The width of the press-formed creases decreased when material thickness increased. The results showed that with grammage 190 + 40 gsm (thickness before forming 270 µm) the average length, which correlates with the width of the crease, of a formed crease was about 1000 µm, while with grammage 350 + 40 gsm (thickness before forming 465 µm) the average length was only 800 µm. The measurements were made in the rim area of the trays, and examples of the creases are presented in Figure 15. Another observation was that in the press-formed trays there was no significant difference in the formation of creases in the CD, MD or 45-degree angle after press forming.

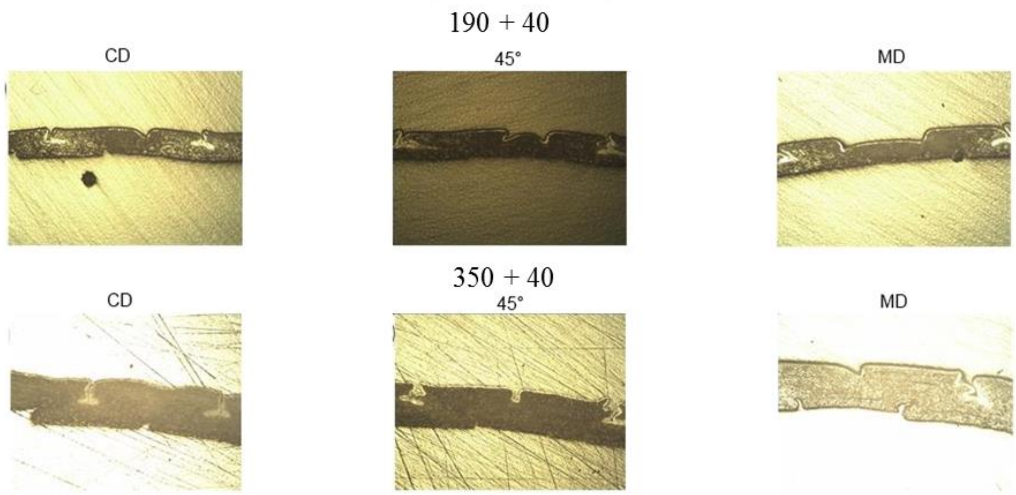

Figure 15. Creases in the cross direction (CD), 45° angle and machine direction (MD) from the rim area (tray flange) of press-formed trays with grammages 190+40 (top row) and 350+40 (bottom row).

With lower thicknesses (larger clearance), such as 190 + 40 gsm and 230 + 40 gsm the paperboard became wrinkled in areas that were not creased, which had an effect on the overall visual quality of the package. These wrinkles are visible in Figure 16. This effect is believed to be caused by the lower in-plane stiffness of the thinner materials. It is possible that this kind of wrinkling would have an effect on the tightness of the heat seal.

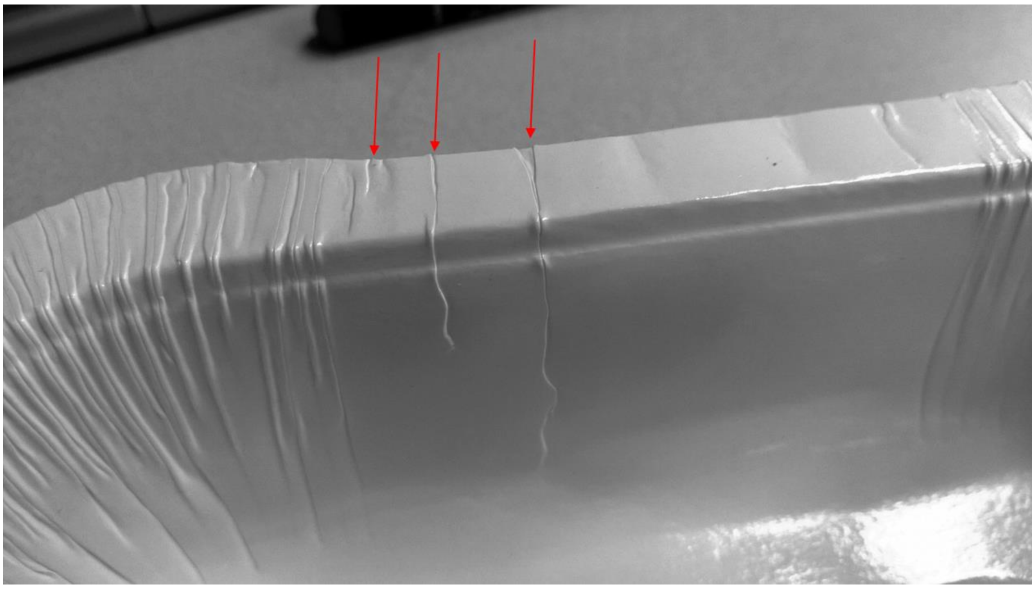

Figure 16. Wrinkles outside the creased area highlighted by arrows.

The formation of creases was similar in each corner of the tray. This observation means that in future studies with symmetrical geometries such as a rectangular tray, the analysis can be limited to just one corner.

5.3 Effect of blank holding force on the surface quality and gas tightness of paperboard trays

In Paper III, the effect of blank holding force (BHF) in the press forming process on the surface quality of the trays and subsequently to the liquid and MAP-tightness of sealed trays was investigated.

The blank holding force (i.e. rim tool force) is the force that controls the folding of the tray corners during the press forming of paperboard trays (Figure 4). The effect of the blank holding force on the quality of the formed products was discussed and known to some extent. However, the effect to the tightness of sealed products was somewhat unclear. The blank holding force was varied to create trays with different rim area surface qualities to observe the effect of the blank holding force on MAP-tight sealability. In previous studies, gas tight sealing resulting from pre-creased blanks was not achieved. The other process parameters were kept constant during the study.

The forming and sealing tests were done by using two differently shaped trays, a rectangular tray and an oval tray. These two geometries were meant to represent the most typical tray shapes used in the food packaging industry. The trays were sealed with the Ilpra speedy tray sealer (Figure 11). Based on earlier findings, two new sealing tool sets were designed and manufactured. To prevent leaks, instead of a flat upper tool, the tools consisted of a shaped upper tool with a flat, heated surface. The tools were shaped according to the tray flange, and the width of the sealing surface was 3 mm for the rectangular geometry and 4 mm for the oval geometry. The bottom tool consisted of an unheated tool with a silicon rubber gasket positioned in a groove on the middle of the tool. The tool widths were designed to achieve the same pressure on the seal regardless on the tray dimensions. The sealing parameters were kept at constant values: sealing temperature 190 °C, sealing dwell time 2.5 s and sealing pressure a typical network pressure 6 bar, which resulted in a pressure of about 2.7 N / mm^2 on the rim area touched by the sealing tools. The trays were flushed with a common gas mix for food applications; 70 % N_2 and 30 % CO_2.

The oxygen content inside the package was analysed with a Mocon Optech O_2 Platinum analyser which utilizes the standard ASTM F-2714-08 (Standard Test Method for Oxygen Headspace Analysis of Packages Using Fluorescent Decay). The analysis occurred over the course of 14 days. The sealed trays were stored in a refrigerator, at a temperature of 6 °C, to simulate realistic storage conditions. After the O_2 measurements, the trays were flushed according to the above mentioned colouring solution test method.

The effect of the blank holding force on the flatness of the tray flange was quite apparent. When the blank holding force is too low, the paperboard blank folds insufficiently and the desired quality of the rim area (flange) is not achieved. The change in the flatness of the rim area in relation to the blank holding force could be evaluated to some extent. However, it was not possible to evaluate visually the exact quality in the tray flange in which a tight seal could be achieved.

Figure 17 shows the corners of rectangular trays manufactured with different blank holding forces. Both the worse surface quality and the subsequent leakage caused in the heat sealing by the poor surface quality can be detected with the colouring solution. If the blank holding force is low enough, the change in the rim area quality is clear even in pure visual inspection. Figure 17 d shows a leak detected with the colouring solution.

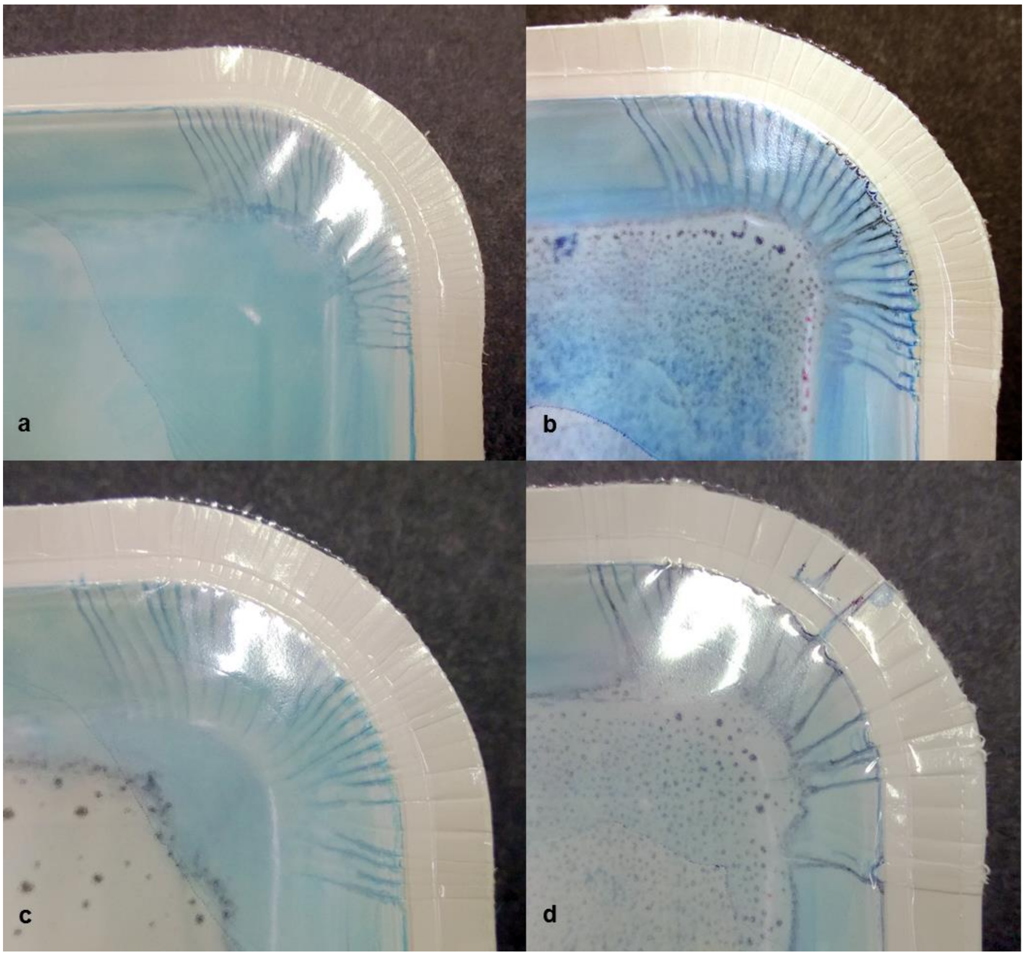

Figure 17. Rectangular tray corners with different blank holding forces: (a) 1.16 kN, (b) 0.77 kN, (c) 0.68 kN, and (d) 0.58 kN.

Figure 18 shows that there is gas leakage in the packages manufactured with blank holding forces of 0.58 kN and 0.68 kN. While the tray in Figure 17c appears to have an intact seal, the MAP composition in the package had changed drastically (Figure 18, 0.68 kN). This shows that even though the results of the colouring solution test would indicate a gas-tight package, the gas tightness of a seal cannot be confirmed solely by the colouring solution test.

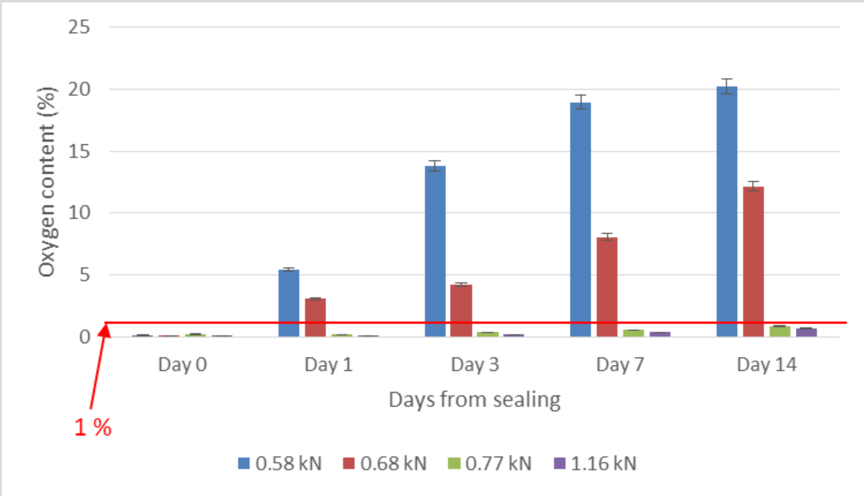

Figure 18. Oxygen measurement averages of rectangular trays manufactured with a varied blank holding force. The red line represents 1 % oxygen level.

The oxygen content in the rectangular packages manufactured with a blank holding force of 0.77 kN and 1.16 kN still registered at less than 1 % two weeks after the initial sealing. The results of the colouring solution test and O_2 measurements indicated that the blank holding force (BHF) has a clear effect on the tightness of the sealed package.

Figure 19 shows the oxygen content of oval trays, which was less than 1 % oxygen after 14 days with all blank holding forces.

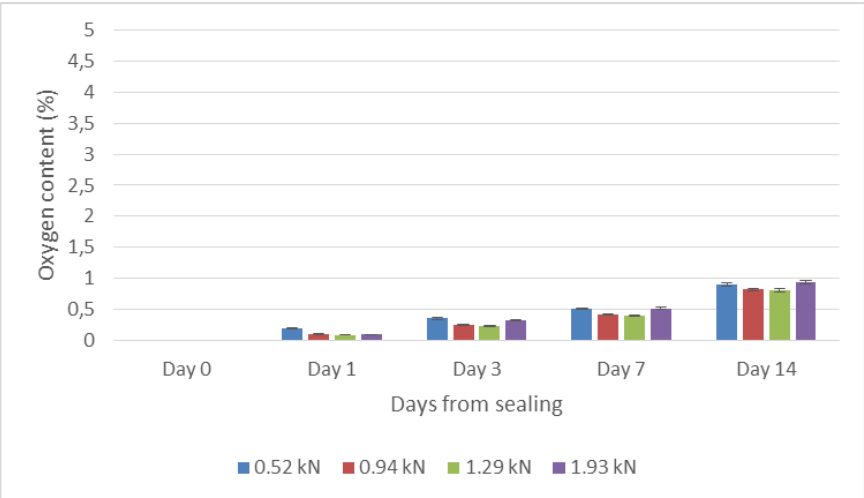

Figure 19. Oxygen measurement averages of oval trays manufactured with a varied blank holding force.

Inadequate blank holding force in the forming results in faults, which respectively result in leaks. The faults include: too deep creases, inadequately sealed or formed creases, and even cracks, which can be seen in Figure 20.

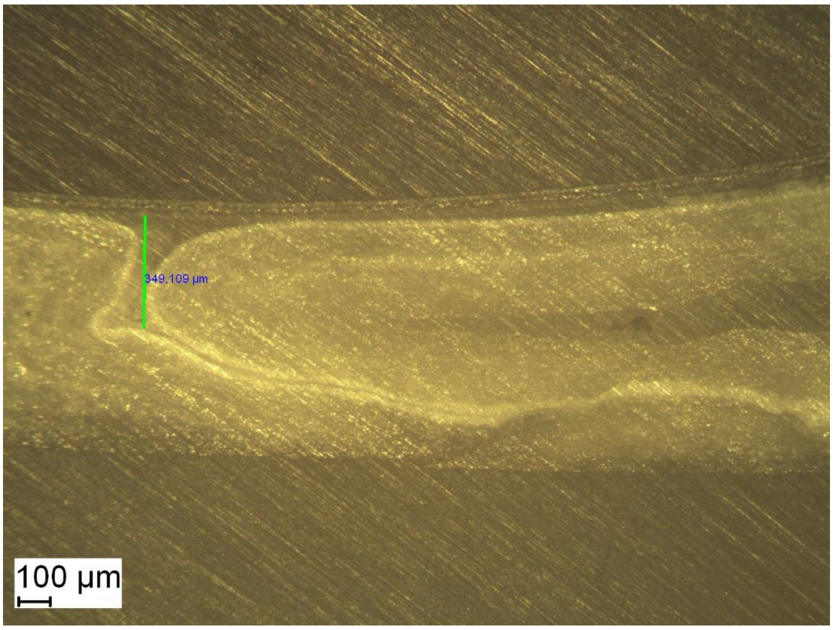

Figure 20. An approximately 350 µm deep crease in a rectangular tray, leading into a crack in the tray, resulting from non-optimal sliding of the blank caused by a low blank holding force (0.58 kN).

The average and peak depths of the creases in the rectangular trays manufactured with different blank holding forces clearly show that the depth of the creases is effected by the blank holding force. Table 3 shows the average and highest depths of creases in the trays manufactured with a blank holding force of 0.58 kN, 0.68 kN and 1.16 kN .

Table 3. Average and peak depths of creases in rectangular trays manufactured with a varied blank holding force.

Blank holding force [kN]	Average depth of creases [µm]	Highest depth of a single crease [µm]
0.58	294	349
0.68	211	238
1.16 kN	109	165

The formation of the oval trays during tray pressing occurred more homogenously than that of the rectangular trays. This could be due to the less demanding geometry of the tray, as the density of creases and the radius of the tray corners are greater. The rectangular geometry is more sensitive to process parameter alterations, and therefore its processing window is smaller. The geometry of the tray is also a greatly affecting factor when a leak-proof seal is desired.

The gas tightness of the trays sealed with a multi-layer polymer film proved to be satisfactory for the use of MAP in food solutions. Due to advancements in the forming process control and converting tooling, a flatter surface in press forming can be produced to enable the basis for a tight seal. Achieving a leak-proof seal requires that suitable tools, materials and process parameters are selected and used during both the tray manufacturing and the lid sealing process.

5.4 Microscopic analysis of heat-sealed trays

In Paper IV, different microscopic imaging methods are investigated and compared to find an optimal imaging method for the formation of creases in the press forming process of paperboard trays. The objective was to find a cost-effective and reliable method for the comparison of creases after press forming and heat sealing of a lidding film. This kind of analysis is needed to get better understanding of the formation of creases and to improve the quality of the trays produced in the press forming process, as traditional methods for testing package and seal integrity do not provide insight into the exact mechanisms causing leaks. This kind of information can be achieved only by microscopic analysis.

Four different imaging methods were used; scanning electron microscopy (SEM), X-ray microtomography, optical light microscopy, and polarized light microscopy. All methods were tested extensively by analyzing leaking creases revealed by the colouring solution and also creases that were found to be sealed properly. Cross-sectional imaging methods were tested in general forming studies and leak analysis. All the four tested methods delivered clear images. However, there were big differences in the usability of the different methods.

Figure 21 shows a sample image taken with X-ray microtomography. While X-ray microtomography offers insightful information of a single crease and its deformation through the sealing surface, the lidding film is not clearly visible in the images. The lack of visibility of the sealing film makes it impossible to use X-ray microtomography in leak detection and analysis. High equipment cost and challenging sample preparation are also significant shortcomings of microtomography.

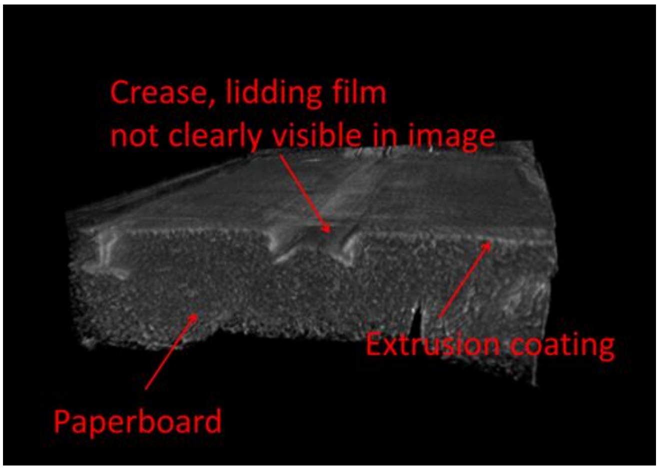

Figure 21. X-ray microtomography image of a single formed crease.

SEM images (example in Figure 22) offer great detail and show the different layers of materials clearly. SEM has far greater magnification and closer details than the other tested methods. The magnification of SEM allows investigating the formation in individual fibres of the paperboard. However, when analyzing the formation of a single crease and the sealing of the lidding film, this kind of accuracy and magnification level is not required. Also, the high cost of the equipment, sample preparation and required gold plating of samples are drawbacks of using SEM.

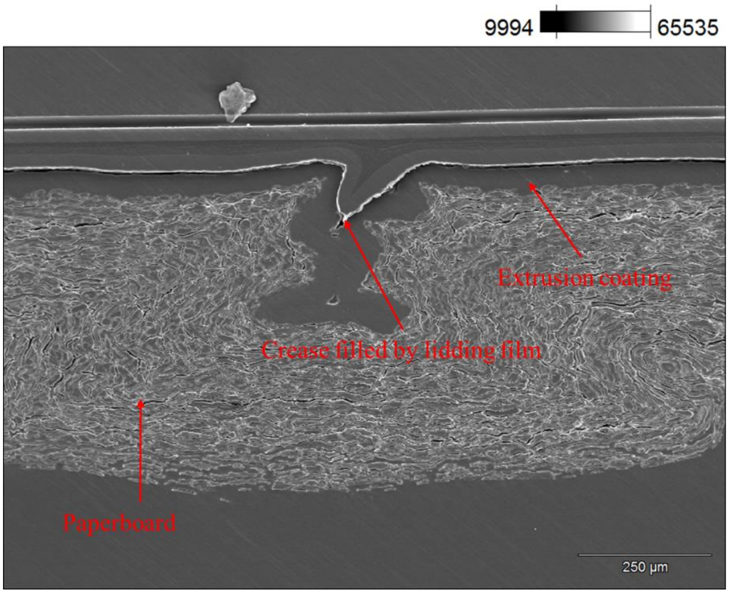

Figure 22. SEM image showing different layers of the structure and a formed crease.

Polarized light microscopy shows clearly the different layers in the materials. A sample of an image is shown in Figure 23. The different layers are recognizable but may be harder to understand than pictures taken with a white light microscope

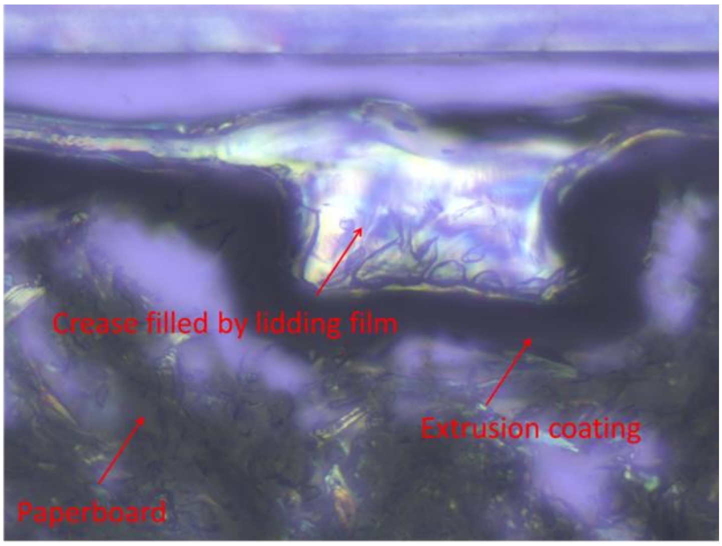

Figure 23. Microscopic image of a tight seal taken with a polarized light microscope.

Casting the samples in an acrylic resin and light microscope imaging was found to be the most suitable method for this kind of analysis. The formation of creases, lidding material and leaks are easy to recognize in the images. Light microscopy is also the fastest and most affordable solution when general material behaviour is studied, as it also allows wider sample areas in a single sample, when compared to for example microtomography. Figures 24a and 24b show a cross section of sealed creases; a tightly sealed crease in figure 24a and a leaking crease in figure 24b.

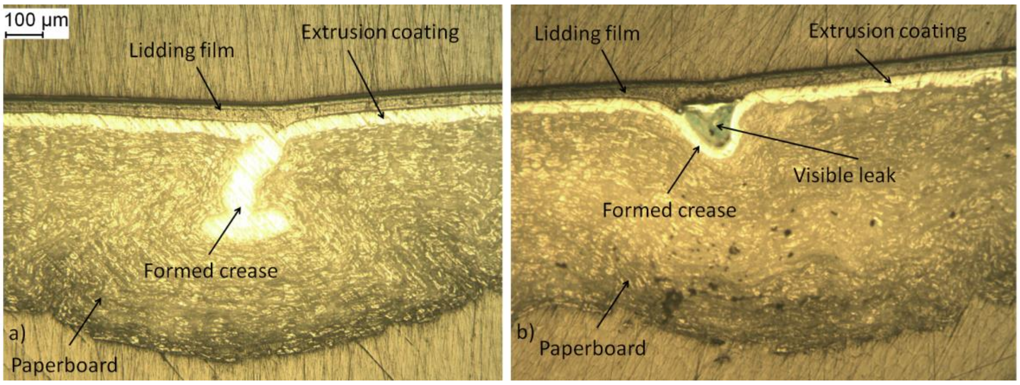

Figure 24. Cross section images taken with an optical light microscope. (a) A tightly sealed crease. (b) a leaking crease. The colouring solution and leaking crease are clearly visible.

All the tested systems can be used in leak analysis, but microtomography, polarized light microscopy and SEM require very precise sample preparation when an individual crease indicated by a colouring solution is studied. Light microscopy is also the only method that allows visual confirmation of leaks, by showing the discolouration caused by the colouring solution. This is an important feature, because leaks under the surface of the rim area of the tray cannot always be detected in visual inspection. These small leaks, which can cause the modified atmosphere to be compromised, can be detected only in microscopic images.

The use of microscopic imaging in the analysis of paperboard trays enables deeper understanding of the material behaviour of polymer-coated paperboard in the press forming process. Structural analysis of a paperboard tray can be done with all the tested methods. However, when large amounts of samples are to be studied, optical light microscopy is the most affordable and efficient method. In addition, if leak detection by a colouring solution and understanding of leak mechanics are to be studied, optical light microscopy is the most practical solution.

5.5 Surface roughness analysis of formed trays

In Paper V, the objective was to assess the feasibility and applicability of using chromatic white light 3D-profilometry to investigate the surface quality of press-formed polymer-coated paperboard trays, and additionally to investigate the correlation between surface quality and tightness of the seal.

Rectangular trays were manufactured by using a varied blank folding force, similarly to the forming parameters in paper III, making it possible to compare the surface roughness results with the previously achieved tightness results.

Surface analysis of the tray rim area was conducted by using a chromatic white light 3D-profilometer to study the sealing surfaces of paperboard trays. The system scans the desired surfaces and calculates 17 different roughness statistics and 17 waviness statistics.

After reviewing the 34 parameters produced by the system, four of them were selected for further analysis. The selected parameters were the roughness average (Ra), peak height (Rp), average peak to valley (Rz(DIN)), and lowest valley (Rv). The parameter selection was based on the physical characteristics and the universal use of the selected parameters. Ra and Rz(DIN) are widely used surface roughness parameters, Rv and Rp were selected because leaks in a sealed paperboard tray often occur in deeper wrinkles that are not filled by the heat-sealed lidding film, and the mean level to which peaks and valleys would be compared could not be established before the measurements.

The 3D-profilometry results were provided in three formats; numerical form, 2D intensity diagram and 3D solids. The 2D and 3D visual representations show the surface in detail and it is relatively easy to distinguish different surface qualities with visual inspection, as Figure 25 shows. Visual identification of the surface quality is, however, not useful in determining the actual sealability of the trays, and numerical values for surface quality would be more practical.

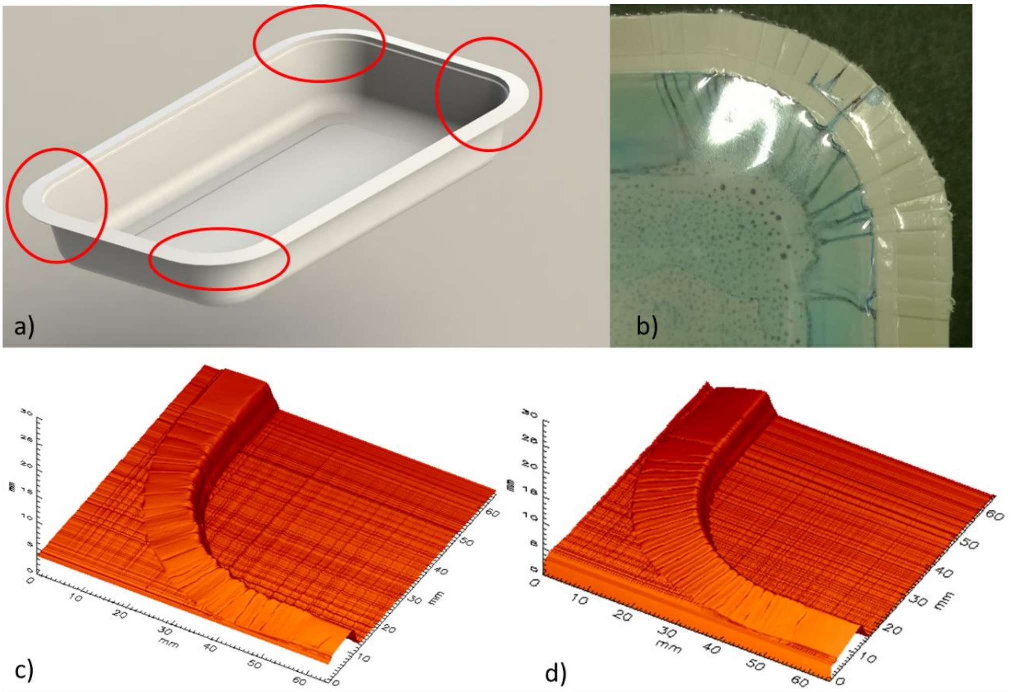

Figure 25. (a) Tray geometry with the location of creased corners highlighted in red.(b) A corner formed with a blank holding force of 0.58 kN and a leak indicated with a penetrant liquid. (c) 3D-presentation of a tray corner formed with a blank holding force of 0.58 kN and (d) with 1.16 kN.

The measurement areas of the surface roughness parameters are shown in Figure 26. The measured values from the tray corner areas were grouped as follows: vertical and horizontal measurements were included, whereas measurements taken in the 45° angle were discarded because the location of the measurements in the 45° direction could not be defined consistently enough. The numerical data received from the measurements was compared to the results of previous studies (Paper III) and compared to leak analysis made on trays manufactured with different blank holding forces (Table 4).

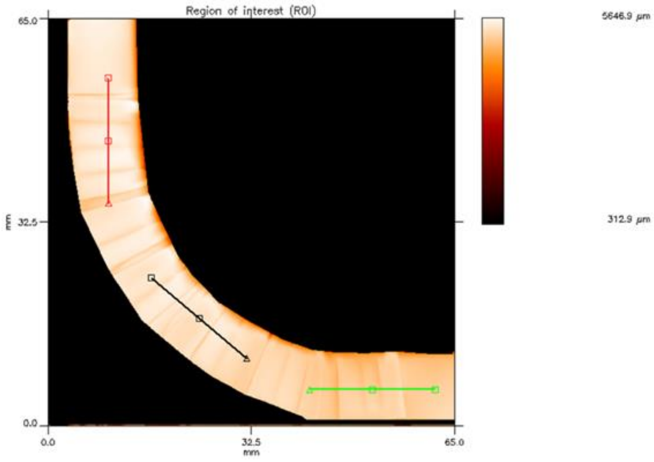

Figure 26. Measurement areas of surface roughness parameters (red, black and green lines).

Table 4: Comparison of leakage and blank holding force.

Blank holding force	Leaks shown by liquid penetrant testing	Leaks shown by gas analysis
0.58 kN	Yes	Yes
0.68 kN	No	Yes
0.77 kN	No	No
1.16 kN	No	No

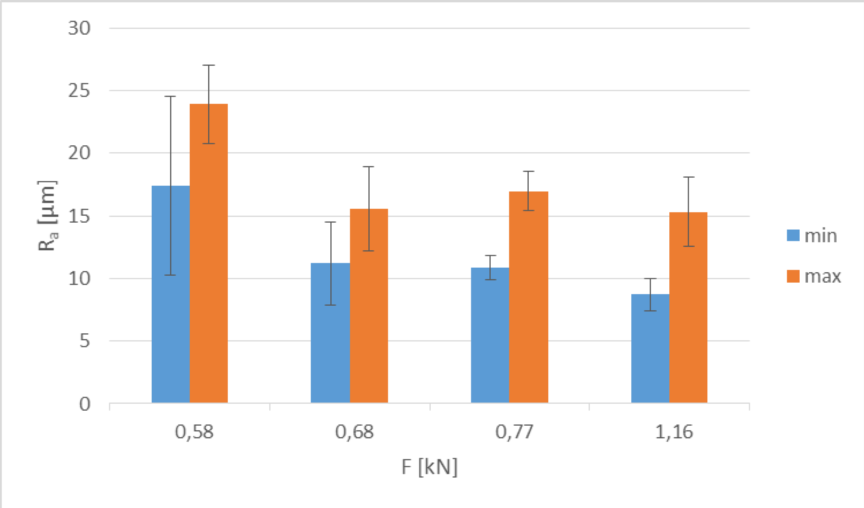

Figure 27. R_a values with different blank holding forces measured by a 3D-profilometer.

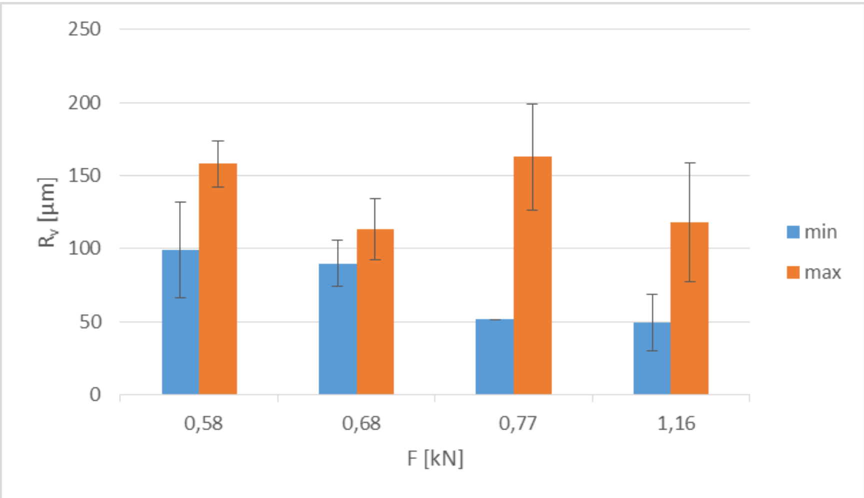

Figure 28. R_v values with different blank holding forces measured by a 3D-profilometer.

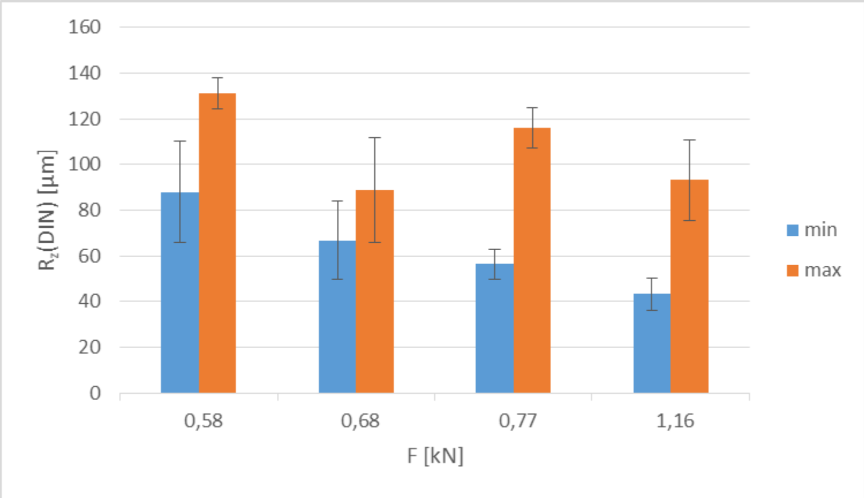

Figure 29. R_z (DIN) values with different blank holding forces measured by a 3D-profilometer.

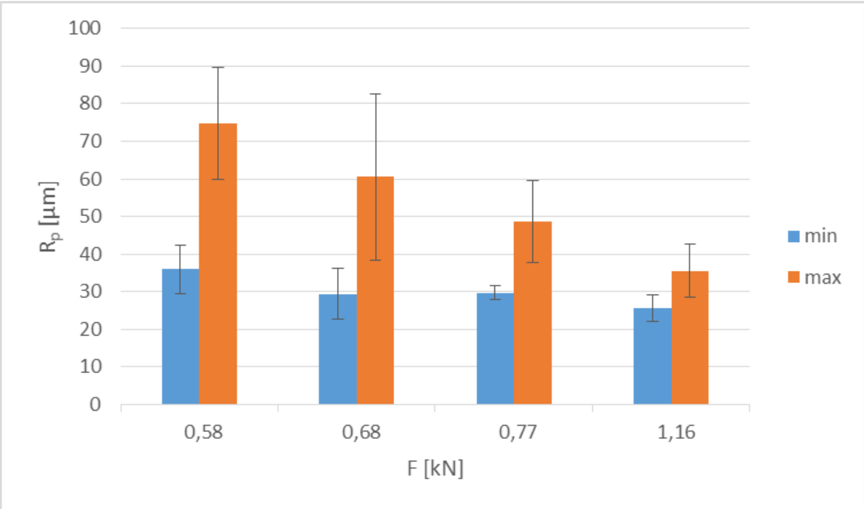

Figure 30. R_p values with different blank holding forces measured by a 3D-profilometer.

The Rp (min) and Rp (max) values showed a strong correlation between surface quality and blank holding force (Figure 30). As was already visually observed, the Rp values correlated with the fact that when the blank holding force is reduced, the surface quality of the rim area deteriorates. The other measured parameters (Ra (Figure 27), Rv (Figure 28) and Rz(DIN) (Figure 29) did not show such correlation. This was assumed to be due to the fact that the white light 3D-profilometer has difficulties in measuring very narrow and deep grooves (Boltryk et al. 2008. When the data acquired in this research is compared to that of Boltryk et al., there is a strong similarity between the grooves (creases). Ra, Rv and Rz values may be distorted because the full surface details were not represented in the data used to calculate the said values, while the Rp discarded all data below the mean surface line.

The results showed that the Rp (max) value of press-formed packages should be below 45 to achieve a good, leak-proof sealing result. However, this value must be treated with some caution because a fairly small sample size was measured and there was some variance in the measurements. When average values are measured it is possible that some local influences are not clearly shown in the results. The results still showed that the system can be used to analyze the surface quality of manufactured trays, while traditional touch-based systems have difficulties in doing this.

5.6 Effect of tray dimensions on the gas flushing and heat sealing of trays

Paper VI focuses on the dimensional accuracy of formed trays. The effects of several forming parameters and material moisture content on the outer dimensions of formed trays are investigated. Subsequently, the effect of the tray dimensions and the mass of the packed product on the lid sealing process are investigated.

When polymer-based packages are used, fill and form thermoforming lines are frequently used. The formed packages are attached to the polymer web and thus positioned correctly during the heat sealing of the lidding material to the trays. After this, the packages are cut in cross-directional and longitudinal directions. This results in dimensionally uniform packages. However, adjustment of the outer dimensions during the production of paperboard trays is a more complex task. In the press forming of paperboard trays, the length and width of the tray can be altered by changing the blank size or by adjusting the forming process parameters.

The effects of all essential press forming parameters on the tray dimensions were studied with a series of tests. Each process parameter was changed separately while the others were kept constant in the following set of values: male mould temperature 50 °C, female mould temperature 160 °C, blank holding force 1.6 kN, pressing force 120 kN, dwell time 600 ms, and pressing speed 150 mm / s. The quality of the trays was also evaluated. Trays with good quality have a smooth sealing area in the tray flange, the creases in the corners are folded evenly, and there are not fractures, wrinkles or other defects in the tray walls.

Figure 31 shows the effects of dwell time and female mould temperature. The mould set has been designed to produce trays of 265 x 162 x 38 mm. As can be seen, a higher heat input results in smaller trays (closer to the designed), but the dimensions are still too large.

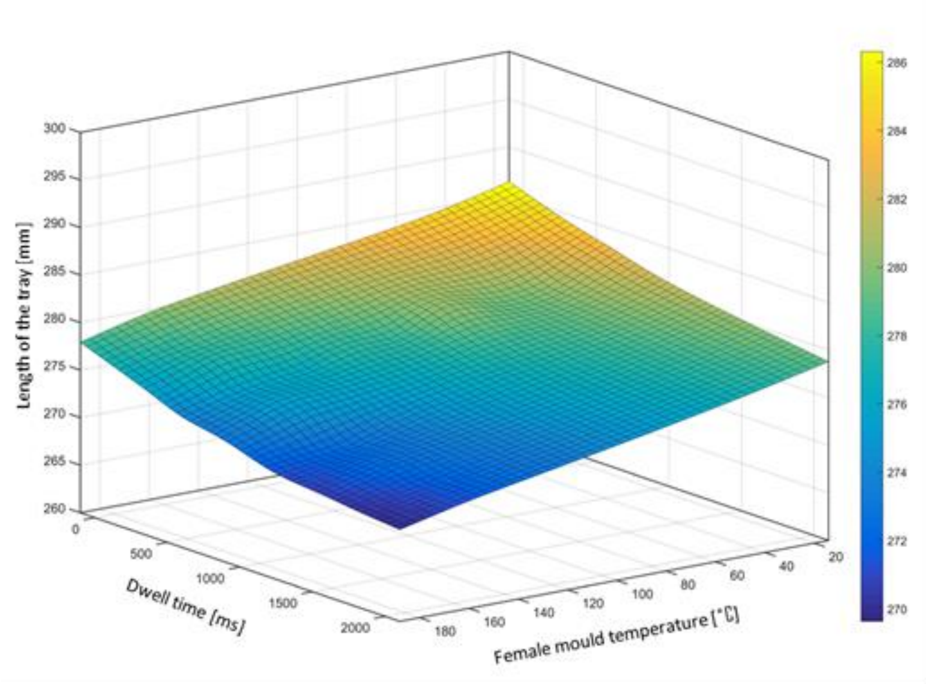

Figure 31. The effect of dwell time and female mould temperature on the length of the tray.

The heat sealing process requires the sealed trays to be dimensionally accurate. It was assumed that if the dimensions of a tray are too small, the tray rim area is not positioned correctly, and leaks can occur. On the other hand, if a tray is too large, it will not necessarily be positioned correctly in the sealing process.

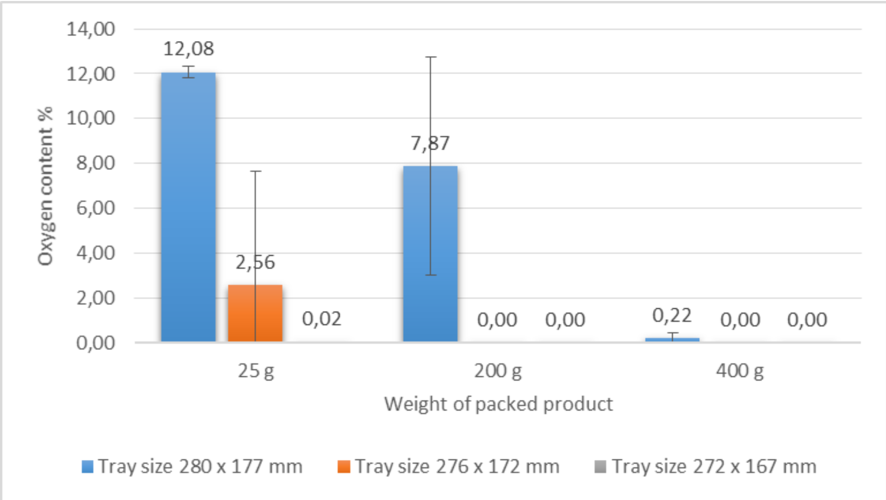

Figure 32. Oxygen content of heat-sealed and gas-flushed packages with varying dimensions and product mass.

Figure 32 shows the average residual oxygen in the sealed trays. With 400 grams of product, the dimensions of the tray did not have a significant effect on the amount of residual oxygen. However, with lighter products (200 g and 25 g) there was significant amount of oxygen in the packages if the tray dimensions were too large. It is clear that the package size has a significant effect on the residual oxygen, and that the weight of the product also affects the amount of residual oxygen in the packages. This is because the tray does not fit between the lower parts of the sealing tools when the vacuum chamber is closed. Normally, the tray is lifted from under the rim area to the sealing position. This effect is clarified in Figures 33 and 34. The packed product is not visualized in the figures.

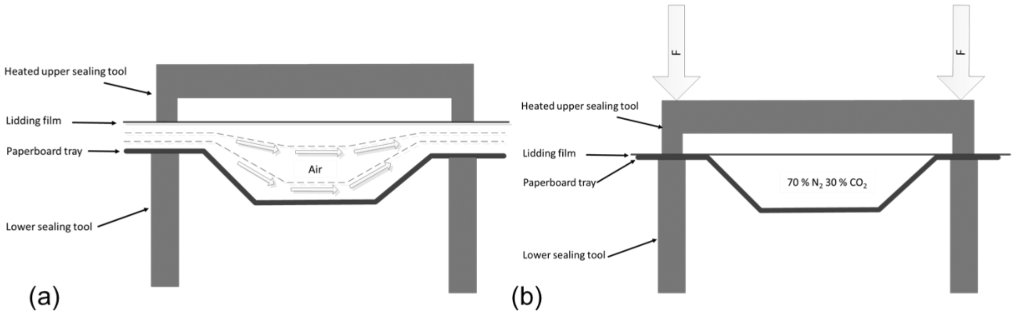

Figure 33. (a) A correctly sized tray is flushed with a vacuum and then with the protective gas, air is removed from the package. (b) The Modified Atmosphere Packaging (MAP) -filled tray is sealed with the lidding film.

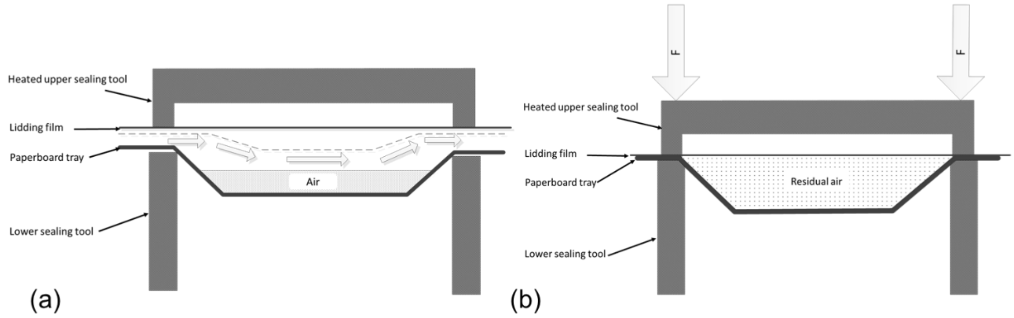

Figure 34. (a) A dimensionally too large-sized tray is flushed, but the flushing is incomplete, because of the positioning of the tray is not correct. (b) The tray is sealed with the lidding film and some air is left in the package.

When the dimensions are too large, the tray walls will touch the sealing tool before the rim area, and the tray will not be in the correct position when the chamber is flushed with the gas. A larger mass of the packed product will cause the package to position properly inside the lower sealing tool, and the gas flushing will not be disturbed. However, if the dimensions of the package are too large, even the mass of the product will not fix the situation and the flushing will be incomplete. With a product mass of 25 g, even an increase of length from 4 mm and width of 5 mm resulted in inadequate flushing of the trays. On the other hand, with a product mass of 400 g, an increase of 8 mm in length and 10 mm in width did not disturb the gas flushing of the tray.

The lid sealing process reduced the sizes of the trays and evened out the size differences substantially. Reduction in tray dimensions happens when the sealing tools force the dimensions of the tray to decrease and the sealed lidding film prevents spring-back.

All the tested parameters had an effect on the dimensions of the formed trays. However, the temperature of the female mould was the only parameter which did not have an effect on the production speed. Therefore, the required amount of heat should be pursued by using as high a mould temperature as possible. The upper limit of the usable mould temperature is usually limited by bubbling or melting of the polymer coating layer.

All the produced trays were measured to be bigger both in length and width compared to the design values of the mould set. The mould set has to be designed undersized to obtain trays with certain outer dimensions. The length of the tray mould should be 4 % and the width 5.5 % smaller than the desired trays with the tested tray design and substrate combination. With this dimensioning, the process window of press forming allows fine-tuning of the dimensions during the production without compromising the other properties of the trays and production speed reduction, and still enabling reliable and tight sealing of the formed trays. This has an effect on the design of sealing tools as well, and if possible, it is recommended to design the sealing tools based on actual manufactured trays, instead of the dimensions of the forming toolset.

5.7 Effect of sealing pressure and crease geometry on the leak-proof quality of trays

In Paper VII, the effect of sealing pressure on the seal tightness of press-formed paperboard trays is investigated. The objective was to determine the surface pressure required for adequate seal tightness and properties. The investigation was done in relation to the sealing temperature. Also the dimensions and shapes of the creases in the trays were measured and analyzed to determine the depth of the creases and wrinkles, so that the tray could be sealed with adequate tightness. The determination of the required sealing pressure is important for the design of new sealing tools for paperboard trays. If the required (optimal) surface pressure is known, then this information can be used to design optimal tooling for the best tightness results. The evaluation of creases can also provide insight into the question of the quality of trays that can be sealed as leak-proof. The sealing was done at temperatures of 170 – 210 °C and sealing pressures of 3 - 6 bar, which resulted in pressures of 1.3 – 2.7 N/mm^2 on the surface of the sealing area. The sealing time was a constant 2.5 s.

The leak inspection was done by several methods. First the sealed packages were inspected by flushing the sealed trays with a colouring solution in accordance with the European standard EN 13676 2001. The packages that had no leaks of the colour solution were then selected to be sealed with the same parameters to investigate the oxygen composition inside these packages. The oxygen composition inside the package was analysed by using a Mocon Optech O_2 Platinum analyser utilizing the standard ASTM F-2714-08, as described above in chapter 5.3 The oxygen transmission rate of trays sealed at 190 °C and 6 bar was also analysed with an Oxygen Transmission Rate (OTR) testing system (Mocon Ox-Tran, Mocon Inc., Minneapolis, USA) according to the standard ASTM D3985-05 ("Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor," 2010) to verify the results of the platinum analyser. The OTR measurements were conducted at 50% relative humidity and 23 °C.

Table 5 shows the average oxygen content in the packages after 14 days of storage. The values are averages of 5 trays. The trays that leaked when flushed with the colouring solution were discarded from the gas tightness test runs. The results showed that the oxygen content averages in the packages were well under 1%. The measured Oxygen Transmission Rate (O_2TR) average of the trays sealed at 190 °C and 6 bar was 4.1 cm^3/package/day.

Table 5. Average oxygen contents in the packages after 14 d of storage.

Sealing temperature (°C)	Sealing pressure (bar)	Resulting surface pressure (N / mm^2)	Average oxygen content and standard deviation after 14 days (%)
190	4	1.8	0.68 (0.24)
190	5	2.2	0.39 (0.01)
190	6	2.7	0.67 (0.33)
210	4	1.8	0.68 (0.18)
210	5	2.2	0.57 (0.28)
210	6	2.7	0.51 (0.15)

In Paper III it was stated that even if the colouring solution exhibited no leaks, there could be significant gas leakage into some of the packages. With the trays used in Paper VII, it was shown that if the colouring solution did not reveal any leaks, the trays were also gas-tight. This was assumed to be caused by the better surface quality of the latter trays. When the surface quality deteriorated, there was more variance between the analysis methods.

Figure 35 shows two samples with sealing pressures 3 and 6 bar. In Figure 35a, a clear leak is visible on the bottom of the crease in the sealing surface. It is clear that the sealing pressure has had an effect on the melting depth of the lidding film. If the sealing pressure is too small, the film could be melted to the bottom of the crease, and leakage occur. This shows that trays with deeper wrinkles and creases could be potentially tightly sealed if the surface pressure were higher.

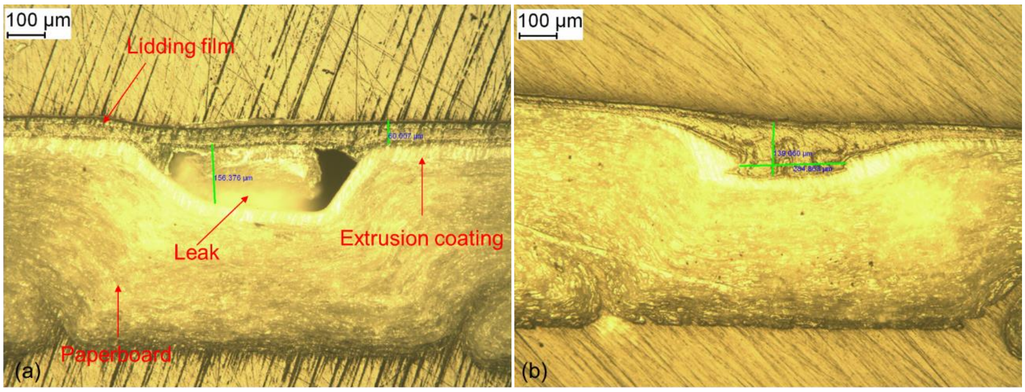

Figure 35. Samples of trays heat sealed at a pressure of (a) 3 bar (1.3 N/mm^2), resulting in inadequate depth in the melting of the lidding film and leaks. (b) 6 bar (2.7 N/mm^2), resulting in a successful, non-leaking seal.

Figure 36 shows three creases sealed with the lidding film. The shape of these creases is typical for a creasing pattern, like those presented in Figure 6a. The longer creases are usually formed as “closed”, like creases 1 and 3, while the shorter creases are formed as “open”, like crease 2. However, this kind of shape variance did not have a noticeable effect on the sealing result, as both geometries could be sealed with a satisfactory leak-proof result when the depth of the creases formed was not too large and the tray was otherwise intact. The results indicate that creases and wrinkles with depths of about 150 µm can be sealed in a leak-proof manner.

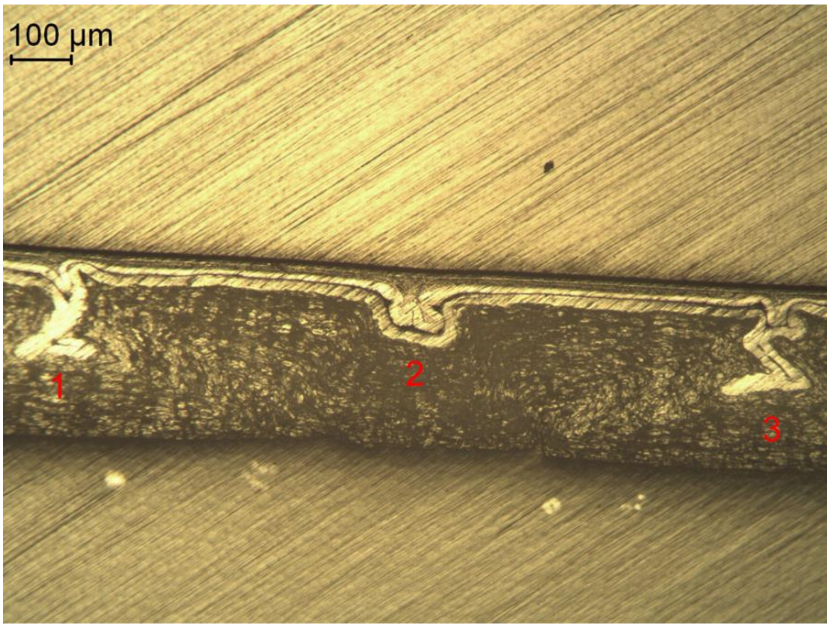

Figure 36. Heat-sealed creases with a sealing pressure of 6 bar, resulting in the lidding film melting to the bottom of the creases. Creases numbers 1 and 3 are so called “closed” creases and crease number 2 is an “open” crease.

Three “industrial-grade” trays were also analysed to investigate the dimensions and shapes of the creases in the sealing surfaces of trays manufactured by commercial equipment. One of these trays was used with MAP for cold-cut ham and the other two samples were not sealed. In the tray used with MAP, the depth of the tray was approximately 16 mm, and the geometry of the tray was designed so that the radius of the creased area was very large (about 110 mm). This generally makes the quality of the rim area flatter and prevents leakage (Leminen *et al.* 2015a).

The depth of the unsealed, industrial-grade trays ranged from 28 to 32 mm and the corner radius was about 50 mm. Figure 37a shows an image of a crease in an industrial-grade tray with an open crease that is approximately 400 µm deep. This kind of shape and dimension prevents the tray from being sealed without leaks. The formation of creases in similar trays, analysed also in Paper I, differs from the trays manufactured in Paper VII. The shape of formed creases in the sealing area in trays with a lower quality differs from the leak-proof creases in several ways; the creases are greater in width and depth and there is no similar pattern in “closed” and “open” creases. Also, the number of “open” creases compared to trays with better quality is greater. This kind of insufficient flattening of the tray flange can be caused by several reasons; insufficient BHF, too big clearance in the forming, or insufficient force in the final flattening phase of the tray flange (Figure 4, phase 5).

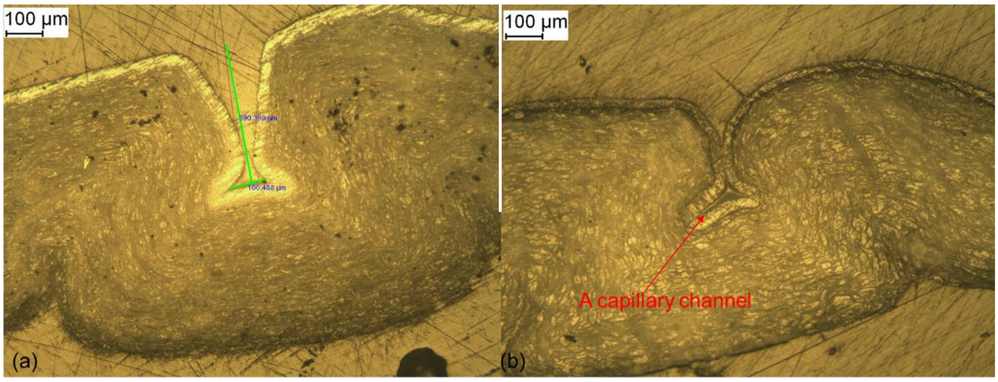

Figure 37. (a) An open, roughly 400-µm-deep crease in an industrial-grade tray, (b) A capillary channel on the sealing surface of an industrial-grade tray (modified from Leminen et al. 2015b).

The depth and width of the creases and the sealing process parameters are not the only important factors when considering whether the tray can be sealed without leaks. When the manufacturing process of the tray is not satisfactory, the tray can have capillary channels that compromise its integrity. An example of an industrial-grade tray with a capillary channel is shown in Fig. 37b. The heat sealing of the lidding film cannot mend this kind of defect in the trays. This kind of effect is also discussed by Hauptmann *et al.* (2013).

A too low sealing pressure results in leaks, which occur first at the bottom of the creases in the sealing surface. The surface pressure which resulted in successful seal tightness with these products ranged from 1.8 to 2.7 N/mm^2. This should be taken into account when sealing tools for press-formed trays are designed. The result also has an effect on the equipment that can be used for heat sealing of press-formed trays, as the force produced by the sealing equipment must be adequate to provide leak-proof seals. It was also proven in the study that creases in the sealing surface with depths of up to 150 µm can be sealed without leaks.

5.8 Synthesis and discussion

When the leak-proof sealability of press-formed, polymer-coated paperboard trays is considered, it is apparent that there are a number of things to take into account. The material must be suitable for forming, the forming process must be done correctly, and the heat sealing of the lid must be done correctly with a suitable lidding film.

Many of the observations made in this thesis apply to tray manufacturing and lid sealing in general, even though for example a MAP-tight product is not desired. Based on the results, it can be noted that it is possible to manufacture a leak-proof end product for the use of MAP. However, it is challenging to measure the exact quality of trays that enables reliable, leak-proof sealing of trays. Several suggestions to measure this, such as the depth and width of creases and the numerical surface quality values have been presented in this thesis.

The effect of the sealing temperature on the required sealing time is apparent, but another interesting observation is that the liquid tightness was acquired with all the tested lid and tray material combinations. This is contrary to the observations of Hauptmann et al. (2013), but is most likely caused by the different rim area quality of the sealed trays, which as stated, has a major impact on the potential for having a leak-proof sealing result. Liquid tightness was acquired with all the tested lidding film and tray material combinations.

The effect of mould clearance in press forming (material thickness) on the quality of the sealing area (tray flange) is twofold; mould clearance has an effect on the dimensions of the creases, but on the other hand, flattening of the tray flange can be done regardless of the material thickness. Visual grading of the formed trays in the sealing area showed that material thickness did not have a great effect on the overall quality of the trays in the tray flange, but the effect was more obvious below the tray flange nearer the bottom of the tray. The only flaws (small wrinkles) located in the tray flange were with thinner materials, and it was assumed to be caused by the weaker stiffness of the material.

The blank holding force had a clear effect on the quality and flatness of the tray flange – which directly affects the sealing result and tightness of the sealed trays. Wallmeier et al. (2015) also describe a similar observation in deep-drawing of paperboard cups, where the surface of the wrinkled flange flattens when the pressure on the flange grows and the number of wrinkles is increased (resulting in better quality). The results of Hauptmann and Majschak (2011) also support the fact that utilizing a higher BHF results in a flatter surface and better visual quality of formed products. The surface quality of press-formed trays affects the gas tightness of the trays directly, and the BHF can be described as the most important parameter to adjust in the press forming process, while other important parameters such as the forming force, dwell time and temperature are usually defined by the material properties and the mechanical aspects of the machinery.

The geometry of the trays is also an important factor in the manufacturing of MAP trays, since the geometry, as well as incorrectly selected forming parameters can cause deterioration in the tray quality. As noted above, the deterioration – if significant enough – cannot be mended during the heat sealing of the lid. The use of MAP with press-formed paperboard trays is possible and the gas tightness is considered satisfactory. This result contradicts some earlier observations by Hauptmann et al. (2013), where trays formed from pre-creased trays were found to be leaking, and those of Vishtal (2015), where the wrinkling in sliding blank processes was said to lead to uneven height on the upper surface of the package and to prevent gas-tight sealing. Some reasons for the improvement in gas-tightness are improvements in the tray forming technology and the optimization of the heat sealing process and tooling, which result in better surface quality of the tray flange area. Improvements in the controlling of the press forming process are thoroughly covered in Tanninen (2015c).

The microscopic analysis of formed and sealed creases by optical light microscopy (stereomicroscopy) is undoubtedly a practical solution to analyze differences in the creases in the tray flange area, since it is quite easy to recognize the main factors that have caused a crease to leak. Microscopic analysis of bent creases has been previously done for unsealed samples with paperboards (Nygårds et al. 2014), but there is no earlier data of such analysis for heat-sealed trays. Other methods to analyze the quality of 3D-formed paperboard packages has been discussed previously, however. Tanninen et al. (2015b) discuss controlling the folding of the blank in press forming of paperboard trays, and focus on the design and investigation of different creasing patterns. The number of creases was found to be the most important variable in the pattern design. This article utilized observation of the quality of folding and the flatness of the tray flange. Wallmeier et al. (2015) present a solution to detect and count the wrinkles by a surface image analysis system to evaluate the quality of formed cups. Both methods are effective solutions for comparing formability, but they do not evaluate the microscopic quality and cannot be directly be applied to the analysis of sealability of trays or the quality of sealed trays.

A similar observation can be made regarding the surface quality measurements of formed trays. Numerical values for a “good” or “sealable” quality can be hard to define. Using 3D-profilometry in the surface roughness evaluation gives some insight into the actual numerical values compared to pure visual evaluation of the sealing surfaces. A possible solution would also be to use a surface image analysis system similar to the one used by Wallmeier et al. (2015). This could enable faster analysis of the tray flange area by modifying the analysis code suitable for the analysis of the rim area of formed trays. Combining these methods (calculating the number of wrinkles and wrinkle distance, and analyzing the surface roughness value Rp), and combining these values with leak analysis results could result in an even more reliable analysis of the leakproof sealability of formed trays.

The shape accuracy and dimensions of the formed trays have a great impact on the heat sealing process and particularly the gas flushing of the trays in MAP, since if the dimensions are too big, the gas flushing can fail, which will result in accelerated spoilage of the packed product. A high heat input in the forming stage was proved to be advantageous, and it should be desired to achieve better dimensional accuracy of formed trays. This result correlates with the observations of Hauptmann and Majschak (2011), where increasing the temperature sum up to a certain limit was found to reduce the spring-back angle and deflexion and to induce a better shape stability. Also Wallmeier et al. (2015) state that a higher die and punch temperature resulted in better shape accuracy and smaller deviation. Also the number of wrinkles was found to be increased with elevated temperature, which is an indication of better quality (Wallmeier et al. 2015). Because the tray dimensions differ from the forming tool dimensions, it is recommended to design and manufacture the sealing tools after the manufacturing of trays. This way the variation in tray size can be taken into account when the sealing tools are designed.

In addition to sealing dwell time and temperature, sealing pressure is a critical parameter in the heat sealing process of paperboard trays. It is also a parameter that is often pre-defined by the sealing equipment and the geometry of the sealing tools and cannot be adjusted during production, hence special attention should be paid to the sealing forces of the tray sealer and tool geometry (area of sealing surface). The area of the sealing surface (mostly defined by the width of the sealing area) is a compromise of achieving an adequate pressure while still maintaining a sufficient width of the seal to prevent leakage. The width cannot be reduced indefinitely, however, because this will eventually cause the tool to turn into an equivalent of a cutting blade. No drawbacks of a high sealing pressure were found, so generally a high pressure on the sealing surface should be desired. Hishinuma (2009) describe the forming of a polyball to be a possible problem in heat sealing, but evidence of such a phenomenon was not found when the paperboard trays were sealed. An interesting topic for future research is the modification of sealing machines to boost the sealing pressure from the standard network pressure 6 bar to 10 bar. This could enable a wider range of sealing equipment to be used with paperboard trays by increasing the resulting sealing pressure.

As stated above, the sealing surface should be as flat as possible. Nonetheless, crease depths of up to 150 µm were sealed without problems when the other process parameters were adjusted appropriately. A flat enough surface is achievable when the formation of creases is homogenous in a way that every second crease is “open” and every second is “closed”. It must be remembered still that the depth of formed creases is not the only factor in tray defining if the seals are leak-proof; defects such as capillary tunnels – as also described by Hauptmann et al. (2013) - can appear if the forming process of the tray is not controlled properly.

Based on the findings of the articles, a summary table (Table 6) has been composed to present the investigated main material and process factors affecting the sealing result.

Table 6. Summary of the investigated factors that have an impact on the leak-proof sealing result, and the main papers that discuss the factors

Parameter or factor	Relates to	Impacts	Indications and recommendations
Material thickness / mould clearance (Paper II)	Press forming	Dimensions of creases, visual quality of trays, possible cause of cracks or pinholes in the trays	Material thickness should be from 95 % to 135 % of the mould clearance for the tested paperboards
Blank holding force (BHF) (Papers III and V)	Press forming	Quality of sealing area and overall quality of trays	A high BHF (until breakage occurs) should be targeted, must be defined individually for each tray geometry
Tray geometry (Papers III and VI)	Press forming and heat sealing	Formation of trays and crease geometry	A higher radius of tray corners is advantageous and should be desired if possible
Crease geometry (Papers III, IV, V and VII)	Press forming and heat sealing	Visual quality of trays, leakage	Homogenous forming of creases should be desired and deep and wide creases should be avoided, the creases should be analysed before sealing, the depth of the creases should be as low as possible (under 150 µm)
Surface quality of rim area (Papers II – V and VII)	Press forming and heat sealing	Visual quality of trays, leakage	Surface should be as flat as possible, capillary tubes should be avoided, Rp (max) should be under 45.
Tray dimensions (Paper VI)	Press forming and heat sealing	Operation in filling and sealing can have a negative effect on the gas flushing of trays	Pressing process parameters should be controlled accurately to achieve correct size trays, high heat input in press forming is recommended, the sealing tool should be designed after example trays have been manufactured
Sealing pressure (Paper VII)	Heat sealing	Leak-proof quality of the seal	High surface pressure should be desired by the means of sealing tool design and equipment adjustment, the minimum surface pressure for tested trays was 1.8 N / mm^2
Sealing temperature (Papers I, III and VII)	Heat sealing	Leak-proof quality of the seal, process speed	Correct sealing temperature should be investigated case by case to prevent leaks and to optimize sealing time and production speed
Sealing time (Paper I and VII)	Heat sealing	Leak-proof quality of the seal, process speed	The sealing time should be high enough to prevent leaks by melting the lidding film adequately

6 Conclusions

The aim of this research was to investigate and recognize the factors affecting the heat sealing quality and the leak resistance (tightness) of press-formed, polymer-coated paperboard trays sealed with a multi-layer polymer-based lidding film. One objective was to achieve a solution that can be used in food packaging by using modified atmosphere packaging (MAP). The work focused on two main processes: press forming of paperboard trays and heat sealing of multi-layer polymer-based lidding films by the heat sealing process.

The first hypothesis was that press-formed, polymer-coated paperboard trays can be sealed with a multi-layer lidding film to achieve satisfactory gas tightness for the use of modified atmosphere packaging (MAP) in food packaging, if the manufacturing process of the tray is of sufficient quality. To achieve a satisfactory gas tightness, the sealed surface (tray flange) must be flat enough and free of deep capillary tubes, to prevent leakage. If the tray quality after press forming is not on a proper level, the heat sealing process cannot mend these defects, and therefore the use of MAP is not possible. Critical factors in press forming regarding the quality of the tray flange are the tray geometry and the blank holding force, which affect the lid heat sealing process. The results of the study show that the quality of press-formed, polymer-coated paperboard trays and multi-layer polymer lidding films can be satisfactory for the use of MAP in food solutions. This requires that suitable tools, materials, and process parameters are selected and used during the tray manufacturing process and lid sealing process.

The second hypothesis was that the quality which makes satisfactory, leakproof sealing possible can be quantified by evaluating the crease geometries with microscopic analysis and the surface quality of the sealing area of the tray. The surface roughness of trays and the depth of creases in the sealing area are critical factors when gas tightness and the use of MAP are targeted. The peak height (Rp) roughness of the tray corners was found to correspond to the leak-proof sealability of lidding film on the formed trays. Of the three main parameters in heat sealing – sealing dwell time, sealing temperature and sealing pressure –sealing pressure usually cannot be adjusted in the production. Hence it should be taken into account when designing new package geometries and sealing tools. The sealability of trays can be quantified and evaluated beforehand by measuring the surface roughness of the tray flange area, evaluating the crease depth and geometry by microscopic analysis, and investigating the resulting sealing pressure in the sealed surface area.

Also the dimensions of the sealed trays are crucial and must be within a certain range to prevent failure in the gas flushing of MAP. Depending on the mass of the packed product, even a small increase in the tray dimensions can result in inadequate flushing of the trays. This should be taken into account when the press forming moulds are designed, as the length and width of the tray mould set should be smaller than the desired tray dimensions. To prevent insufficient gas flushing, it is recommended to design and manufacture the sealing tools after the actual trays have been manufactured.

Utilizing these solutions and results makes it possible to have a food package that is made mostly from renewable and recyclable sources, and can be used with MAP. The trays can be sealed with standard industrial equipment with appropriately designed and manufactured sealing tools. This kind of package can be a considerable alternative for packages made completely from oil-based polymers, to achieve a greater market share for fibre-based solutions in food packaging.

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Publication I

Leminen, V., Kainusalmi, M., Tanninen, P., Lohtander, M. and Varis, J.
Effect of Sealing Temperature to Required Sealing Time in Heat Sealing Process of a Paperboard Tray

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Journal of Applied Packaging Research
Vol. 6, No. 2, 2012

R E S E A R C H

Effect of Sealing Temperature to Required Sealing Time in Heat Sealing Process of a Paperboard Tray

V. LEMINEN*, M. KAINUSALMI, P. TANNINEN, M. LOHTANDER and J. VARIS

LUT Mechanical Engineering, Lappeenranta University of Technology, P.O. Box 20, FI-53851 Lappeenranta, Finland

ABSTRACT: The importance of modified atmosphere packaging (MAP) is significant in fresh food packaging. By using MAP the shelf life of a food product can be significantly lengthened without using preservatives.

MAP requires a strict tightness to the sealing process of the package. The first step to modified atmosphere packaging is to get the package liquid tight.

Unsuccessful sealing causes leaks in the package and even without MAP requirements can cause inconvenience for the consumer. This paper concentrates on the effect of sealing temperature to required sealing time (dwell time) when the used sealing pressure is constant.

Temperature has a clear effect on the required sealing time. However with different material combinations this temperature varies and the optimization of the sealing temperature with every material combination is crucial when maximum production speeds are wanted.

1. INTRODUCTION

Modified atmosphere packaging (MAP) is the removal and/or replacement of the atmosphere surrounding the packaged product before sealing in vapor-barrier materials. Packing foods in a modified atmosphere can offer longer shelf life and improved product presentation in a convenient container, making the product more attractive to the customer [1].

Modified atmosphere packaging with carbon dioxide as an active gas component has been widely reported to inhibit microbial growth on fresh food products such as fish or shrimp by Goulas & Kontominas [2],

*Author to whom correspondence should be addressed.

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Hovda *et al.* [3], Rosnes *et al.* [4] and Laursen *et al.* [5] and also meat products as described by McMillin [6].

Paperboard trays in food packaging are used, but lid heat sealing with paperboard trays has not been widely reported except for some patents [10–12]. This suggests that the research is mainly done in corporation's product development projects.

First step to get the package MAP tight is to get it liquid tight. Liquid tightness also affects the usability of the package in small scale production where cooked meals are manually packed to trays that are sealed with plastic lids. This paper discusses the effect of sealing temperature to required sealing time (dwell time) when the goal is a liquid tight package.

1.1. Heat Sealing

Sealing of the lid is a critical step in modified atmosphere (e.g. MAP) packaging, since the production rate and shelf life can be affected by the sealing process and the quality of the seal. In addition to preventing the package from leaking, the seal must also prohibit air from coming in contact with the food [7].

Sealing conditions are a compromise between dwell time and the temperature and pressure of the sealing tools. The requirement is to apply sufficient energy to cause the sealant to fuse together and become one medium [1].

In the most widely used thermal press type of heat sealing, heat conducted from the surface of the thermoplastic films, the bonded surface is heated to the appropriate temperature, and then it is immediately cooled down to complete the bonding [15].

Heat seal technology is used for packaging pre-heated and sterilized foods, baby and family care products, injectable and oral medicines, snacks, toiletries, and components of electronics and precision machines [15].

Because the process is widely used and the product range is very wide, the suitable machine choice depends on the sealed package and the required production capacity.

Heat sealing machines are almost always used with plastic materials. The use of heat sealing in specific paperboard products has not been widely researched. However fibre based packages are a challenger to plastics in primary food packaging.

Several authors [10–13] have researched different methods to obtain

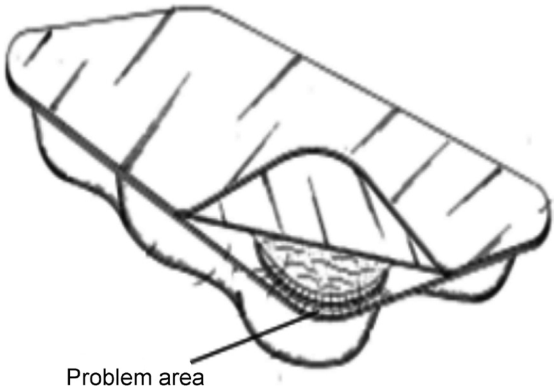

Figure 1. *Problematic area at the tray corner. Modified from [10].*

a tight seal in similar paperboard packages. Some of these solutions are presented next.

1.2. Combined Ultrasonic Bonding and Heat Sealing

Faller [10] presented a possible solution in which arcuately shaped troughs are formed in the face of the flange at each corner of the tray and a plastic cover sheet is filled with food. The cover sheet is first bonded to the tray with ultrasonic bonding. After the ultrasonic bonding heat is applied to the cover sheet to assure complete sealing of the cover sheet to the flange. This solution is presented in Figures 1 and 2.

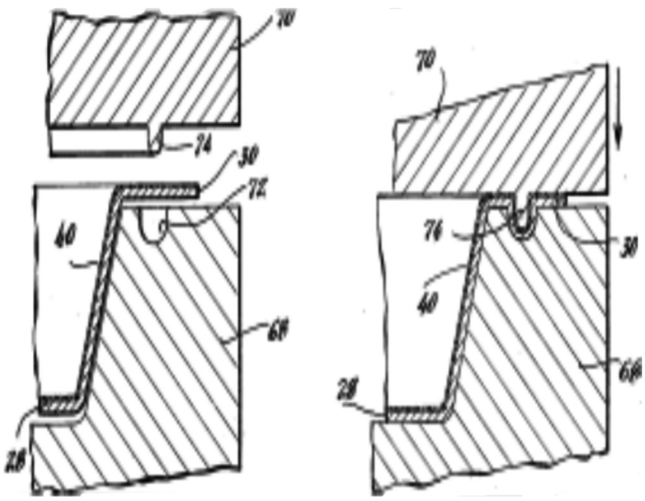

Figure 2. *Close up picture of the sealed area and tool shape [10].*

1.3. Hot Melt or Wax

Seiter *et al.* [11] presented a solution in which a hot melt or wax is applied to the creases in the corners. After this a film cover is adhered to the tray and the hot melt or wax filling should provide a hermetical seal for the interior of the package. This solution is presented in Figure 3.

1.4. Injection Molding

Nylander [12] discusses a technique in which a plastic rim is injection molded to the package. The advantage is that this flat rim should provide a surface in which the lid can be sealed and a gas tight seal should be obtained. Some disadvantages of this technique are the expensiveness of the injection molding tools and machinery, and also the slow speed of the injection molding technology compared to a regular package. A commercial solution of a similar product with injection molded rims is in stores, introduced by Stora Enso [14]. An example of a injection molded rim is presented in Figure 4.

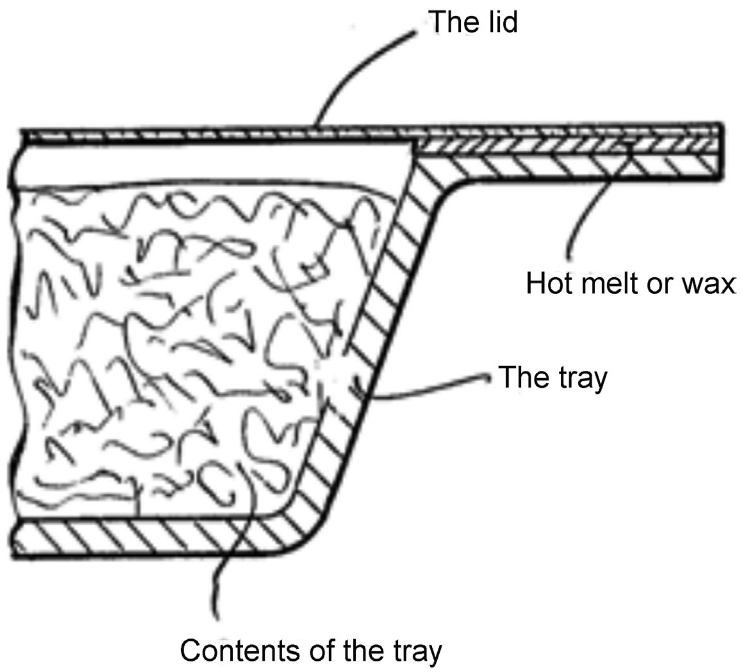

Figure 3. *Hot melt or wax positioning in the corner Modified from [11].*

Figure 4. *An injection molded plastic rim.*

1.5. High Temperature Heating Ridges

Wilkins [13] presents a solution in which the rim area is heated with two heating ridges that come into contact with the underside of the rim. These ridges are heated at a temperature of 500°C. The lid is then lowered to the rim and they are pressed together with a pressure of e.g. 5.5 bar.

According to Wilkins the two adjacent heating ridges provide two adjacent point contacts around the rim of the tray and can conveniently form an air-tight seal with the lid.

The main objective of this study was to research the effect of sealing temperature to required sealing time in the heat sealing process of paperboard tray using constant sealing pressure and to test the liquid tightness of the sealed products. Part of the optimization of the process is to reduce the sealing time as much as possible but still getting a successful sealing. A shorter sealing time is desired because it means faster production in large scale production. In this study the focus was in the sealing temperature and its effect to the sealing time in this process.

Another objective was to examine what kind of methods have been researched to obtain a tight seal in similar kind of paperboard packages. These methods are introduced in Chapter 2.

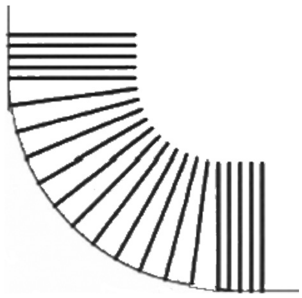

Figure 5. *The shape of the creases in the package corner pictured from above before forming.*

2. MATERIALS AND METHODS

2.1. The Package Format and Materials

The product used in this study was a pressed plastic coated paperboard tray which has creased corners. These creases, which are critical to the package's manufacturing, are clarified in Figures 5, 6 and 7.

It is assumed that the leaks will occur in this area because the creases can form a discontinuity tunnel which causes leaks in the package.

In this work six different tray and lid combinations were tested to research the tightness of the pressed tray. Two base materials were used. Both base materials were paperboard which was coated with plastic. These tested tray and lid combinations and their grammage are presented in Table 1. Trade names of the lids are used in the table.

2.2. Liquid Tightness Tests

Different combinations of tray and lid combinations were tested by

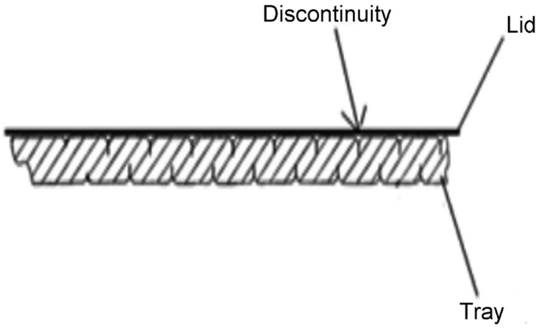

Figure 6. *Shape of the creases (discontinuities) pictured from the side. Modified from [8].*

Table 1. Tray and Lid Combinations.

Tray	Lid	Figure Number
290 g paperboard+40g PET (Package 1)	MSL 65 Bialon (Lid 1)	4
290 g paperboard+40g PET (Package 1)	NFI 208 (Lid 2)	5
290 g paperboard+40g PET (Package 1)	TER EZ-Peel (Lid 3)	6
290 g paperboard+40g PET (Package 1)	TER RC (Lid 4)	7
290 g paperboard+40g PE (Package 2)	NFI 208 PE (Lid 5)	8
290 g paperboard+40g PE (Package 2)	NFI 213 (Lid 6)	9

keeping the sealing pressure at a constant 6 bar which is a standard pneumatic network pressure. The temperature of the upper tool was modified and its effect to the tightness and the required sealing time. A total of six different package and lid combinations were tested. Test tray and lid combinations are presented in Table 1.

At each sealing temperature twenty specimens were sealed and the seals were tested with a colouring solution applying the European standard EN 13676 [9]. The reagents in the colouring solution were dyestuff E131 Blue and Ethanol (C_2H_5OH, 96%). The colour solution consisted of 0.5 g dyestuff in 100 ml ethanol.

The colouring solution was poured into the package and after that the lid was sealed and cooled in room temperature for one minute. After the lid was cooled, the colouring solution was applied to the sealed area for five minutes and the seal was inspected for leaks. An example of a liquid tight seal is presented in Figure 7.

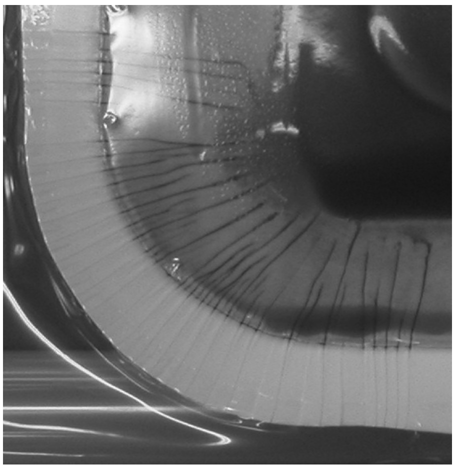

Figure 7. *A liquid tight seal.*

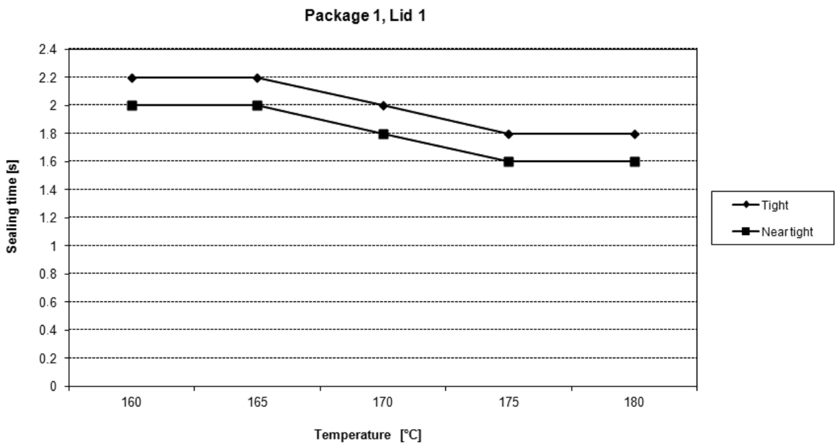

Figure 8. *Effect of temperature to required sealing time for a liquid tight seal with Package 1 and lid 1.*

3. RESULTS AND DISCUSSION

3.1. Liquid Tightness Results

In the liquid tightness tests the value "Tight" was given when all the packages sealed with specific parameters had no leaking seals. The value "Near tight" was given when some of the packages with the parameters were leak proof but some were not. In all package and lid combinations the required sealing time to achieve a liquid tight seal was reduced when the temperature was raised. However with all lid materi-

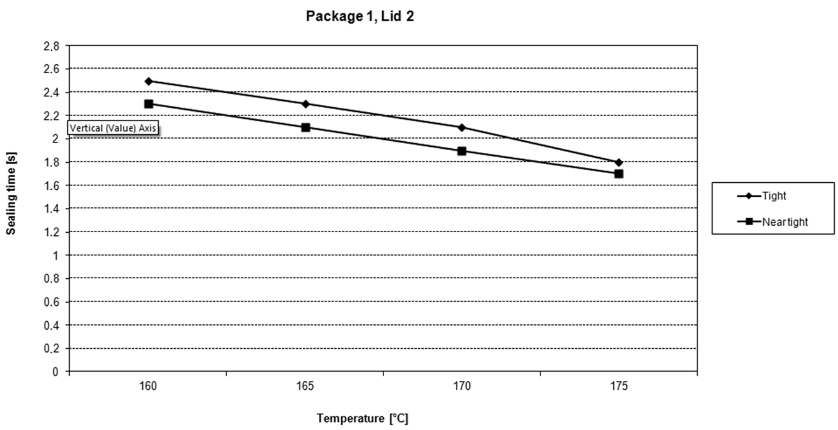

Figure 9. *Effect of temperature to required sealing time for a liquid tight seal with Package 1 and lid 2.*

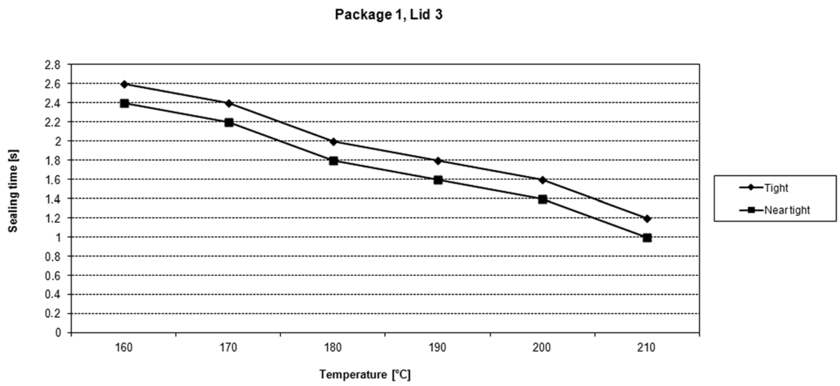

Figure 10. Effect of temperature to required sealing time for a liquid tight seal with Package 1 and lid 3.

als there is a unique temperature point when the lid material fails and is broken by melting. This melting point ranged from 180°C to 215°C depending on the used lid and its material.

Package 1 Liquid Tightness Tests

Package 1 was a tray which's material was PET coated paperboard. It was tested with four different lid combinations which are sealable to PET. Lids 3 and 4 were more heat tolerant than Lids 1 and 2 and the required sealing time was shorter with them. The effect of sealing temperature to reduce the sealing time with Package 1 is visible in Figures 8, 9, 10 and 11.

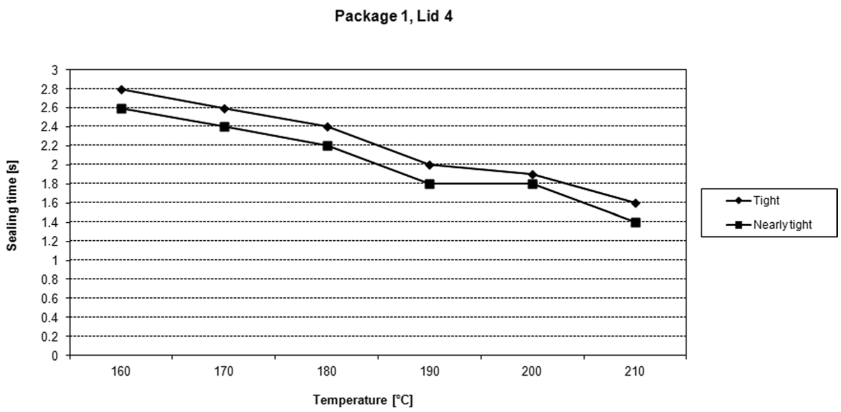

Figure 11. Effect of temperature to required sealing time for a liquid tight seal with Package 1 and lid 4.

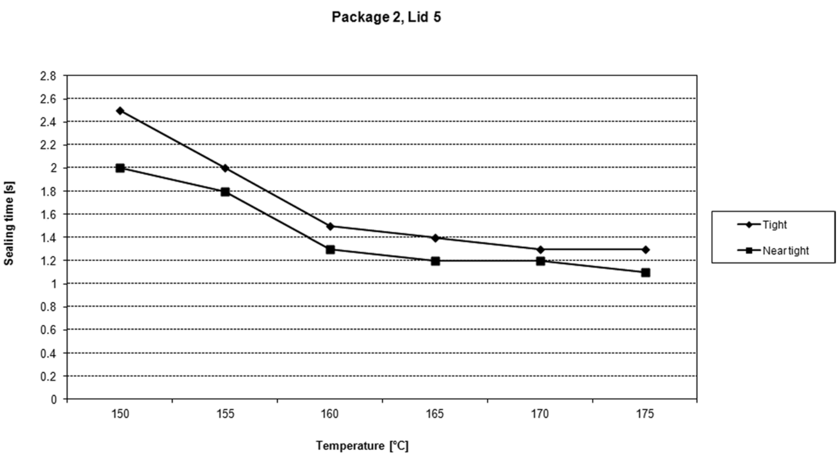

Figure 12. *Effect of temperature to required sealing time for a liquid tight seal with Package 2 and lid 5.*

Package 2 Liquid Tightness Tests

Package 2 was coated which's material was PE coated paperboard. It was tested with two different lid combinations which are sealable to PE. The effect of sealing temperature to reduce the sealing time with Package 2 is visible in Figures 12 and 13.

These creases visible in Figure 14, which are necessary for the pack-

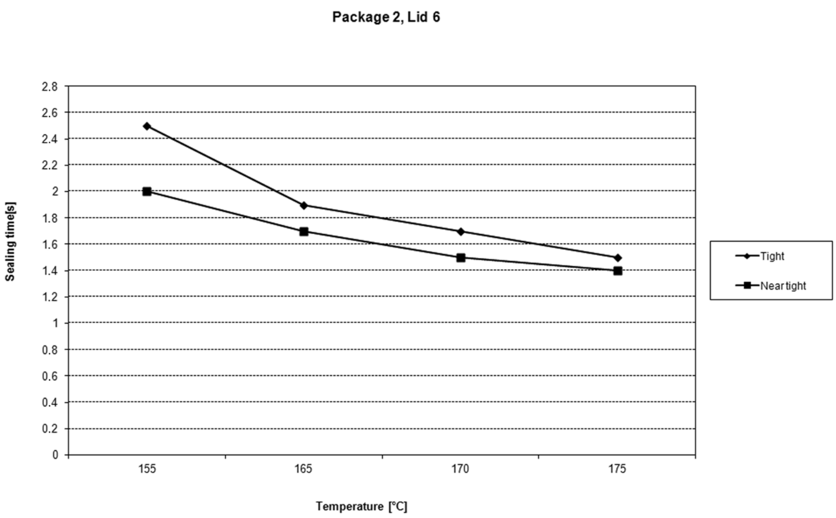

Figure 13. *Effect of temperature to required sealing time for a liquid tight seal with Package 2 and lid 6.*

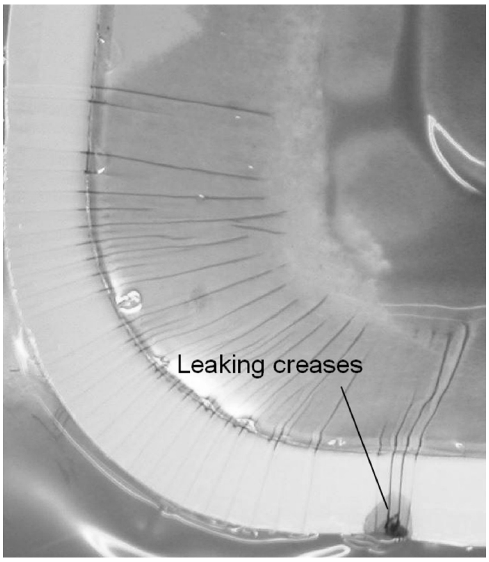

Figure 14. *Problem area of the sealing and two leaking creases highlighted by dye penetrant examination.*

age's manufacturing, cause the seals of the package to leak easily. The critical leaks almost always occur in the last creases of the creased area.

A liquid tight package was achieved with all six lid and package combinations. The best achieved sealing time was 1.2 seconds with the combination Package 1, Lid 3. Sealing temperature has a clear effect on the required sealing time with every material combination.

The most decisive physical factor in the tightness of the package is the creases in the corners. These creases act as a channel which causes the gas to leak from the package.

4. CONCLUSIONS AND SUGGESTIONS FOR FURTHER RESEARCH

It is apparent from all the test combinations that temperature has a clear effect on the required sealing time. However with different material combinations this temperature varies and the optimization of the sealing temperature with every material combination is crucial when maximum production speeds are wanted.

Plastic coated paperboard trays are used in food packaging and liquid tightness of the tray and the sealed lid is acquired. However there are challenges in obtaining a gas tight seal. Several solutions have been presented by different authors but further research is needed to compare and research their performance in production environments.

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Publication II

Leminen, V., Tanninen, P., Mäkelä, P. and Varis, J.

Combined effect of paperboard thickness and mould clearance in the press forming process

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Bioresources
Vol. 8, No. 4 pp. 5701-5714, 2013

Combined Effect of Paperboard Thickness and Mould Clearance in the Press Forming Process

Ville Leminen,* Panu Tanninen, Petri Mäkelä, and Juha Varis

Structural and mechanical aspects of the forming of paperboard have received attention in the literature; however, specific forming phenomena of the tray corner and rim area of paperboard packaging have not been researched widely. In light of the importance of the corner for packaging quality, and to enable improved process control of forming, this study considers the forming phenomena of the corner of a press-formed paperboard tray. Four different thicknesses of extrusion-coated paperboard were studied to research the effect of paperboard thickness and mould clearance on the final product of the press-forming process. Suitable mould clearance, *i.e.*, the percentage of the mould cavity that is filled with paperboard, was found to be from 95% to 135% for the tested paperboard types.

Keywords: Creasing; Paperboard; Forming; Thickness; Clearance; Pressing; Tray; MAP; Modified atmosphere packaging

*Contact information: Department of Mechanical Engineering, Lappeenranta University of Technology, P.O. Box 20, FI-53851 Finland; *Corresponding author: ville.leminen@lut.fi*

INTRODUCTION

The creasing and folding of paperboard and corrugated board and its simulation have been previously studied (Isaksson and Hägglund 2005; Beex and Peerlings 2009; Nygårds *et al.* 2009; Nagasawa *et al.* 2003). Previous work describes the difference in creasing and folding between MD (machine direction) and CD (cross machine direction) (Kim *et al.* 2010). However, previous work and traditional laboratory tests performed for fiber materials do not properly describe the material behavior in press forming, especially when forming occurs in multiple directions.

Some patents and articles have described the forming of paperboard (Määttä *et al.* 2011; Hauptmann and Majschak 2011; Vishtal and Retulainen 2012) and paperboard elongation (Zeng *et al.* 2013), but the forming of the tray corner and rim area has not been researched widely.

This research is needed to better understand the effect of different factors on the end product quality. This quality is important for several reasons, such as the visual appearance and the modified atmosphere packaging tightness and thus the microbiological safety of the packaged product.

In this research, the forming phenomenon of the corner was studied to obtain essential data for better forming process control and therefore better end product quality. The corner area is studied for several reasons. First, the most severe deformation occurs in the corners, and it is therefore the area most likely to have cracks that cause leaks in the package. Second, the corner area surface quality is critical for a tight seal when the package is sealed with a lid.

The forming process involves a combination of material and tool properties. This is why certain parts of tool geometry must be included in this study.

Heat-sealed tray-shaped packages are widely used with modified atmosphere packaging (MAP). However, MAP is not widely used with plastic-coated press-formed paperboard packages in tray form because of the challenges associated with package tightness.

Sealing the lid is a critical step in MAP, as the sealing process and the quality of the seal can affect the production rate and shelf life. In addition to preventing the package from leaking, the seal must also prohibit air from coming in contact with the food (Yeh and Benatar 1997).

A previous paper (Leminen *et al.* 2012) shows that the critical area to the gas tightness of heat-sealed trays with press formed plastic-coated paperboard trays is the corner of the rim area of the tray. This is why the quality of the tray corner should be at a sufficient level and reliably achievable in tray production. Creases are located in this corner area, which are necessary for the package manufacturing process (Fig. 5).

A microscopic figure of a typical press-formed crease is presented in Fig. 1, compared to a traditionally bent crease in Fig. 2. The crease works differently in the paperboard press forming process compared to traditional folding.

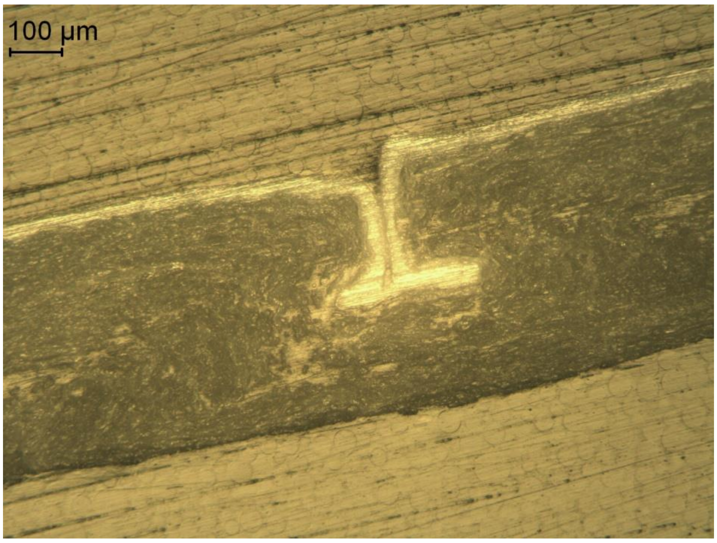

Fig. 1. A microscopic image of a single crease of a press-formed tray of PET-coated paperboard

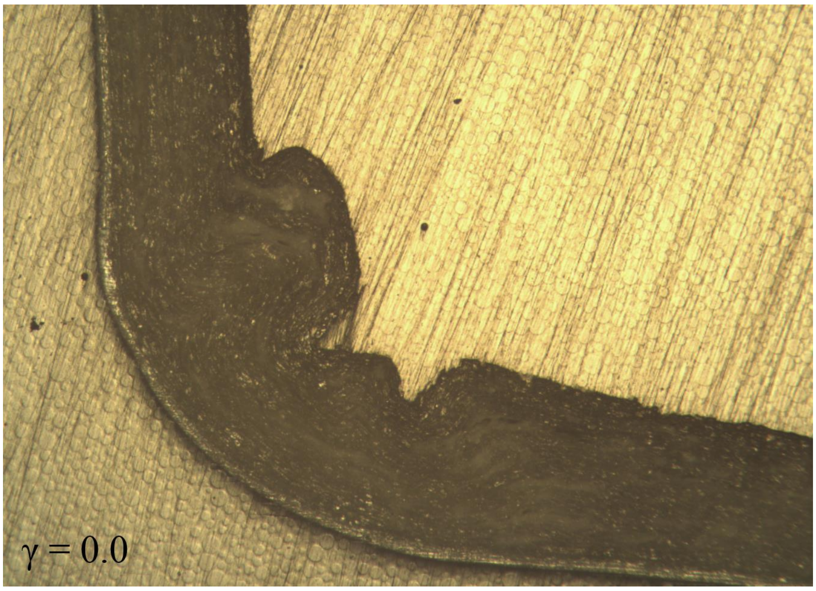

Fig. 2. A microscopic image of bent paperboard. γ is the nominal initial shearing strain of tested samples.

EXPERIMENTAL

Materials

Four different thicknesses of Stora Enso Trayforma Performance polyethylene terephthalate (PET) extrusion-coated paperboard (Stora Enso 2010) were investigated. The materials were stored in a constant humidity chamber at 85% relative humidity to ensure sufficient humidity.

This higher humidity was used to maintain the delivery moisture content of the paperboard. The average humidity of the tested materials was measured using an analysis scale. The tested material thicknesses and their moisture contents are presented in Table 1.

Table 1. Tested Materials

Base board consists of three SBS (Solid Bleached Sulphate) - layers		
Base material grammage	PET-coating grammage	Moisture content
190	40	9.4%
230	40	10.7%
310	40	11.0%
350	40	10.5%

Elongation values for tested materials measured in 23 °C and 50% RH was approximately 5% in cross direction and 2.5% in the machine direction. However according to Kunnari *et al.* 2007, by varying the moisture and / or temperature of paper based materials, it is possible to get gains in elongation of around 2 to 2.5 percentage points, and therefore the elongation values in tray pressing can be higher.

Methods

The press forming of fiber materials was studied with a series of converting trials. The research was limited to the material thickness and its relation to forming mould clearance. The clearance is the distance between forming surfaces of tray manufacturing tools. Content of outer and middle plies of the base board were the same in all the test materials; only the thickness varied in constant scale. Polymer coating was identical in all materials. The process parameters of tray blank preparation and preconditioning of test materials were kept constant to ensure reliable results. Process parameters are shown in Table 2.

Folding of the paperboard blank was controlled with a set of carefully positioned creases (Fig. 5) and process parameters in the press-forming process. The used creasing pattern represents a typical layout in the tray pressing process. The target was to obtain evenly folded creases in smooth tray walls and in flat flanges of the tray. The ratio of mould clearance and thickness of the paperboad also has a significant effect on the appearance of the tray wall. The mould clearance cannot be adjusted during production runs; therefore, the selection of clearance is a critical phase of the mould design process. The behaviour of paperboard blank was observed in different parts of the mould cavity and is presented in Fig. 3.

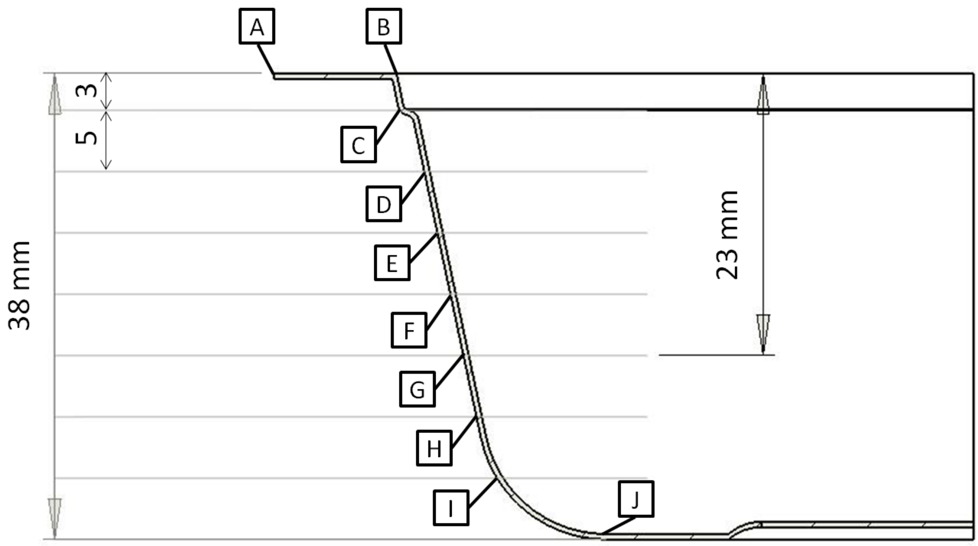

Fig. 3. Observation points of the tray corner; tray walls were analysed from multiple heights with 5-mm intervals.

At each observation point, the length of the tray wall tracing arc was compared to its original length and the ratio of material reduction was calculated. The arc drawn in observation point D can be seen in Fig. 4. The material is folded into 67.63% of its original length. This value is derived from the volume of the mould cavity in a single corner of the tray using the 3D model of the moulding-tool set. At point I, the required material reduction is largest; therefore, the material has to endure the greatest amount of forming. The usage of mould clearance describes the portion of the mould cavity that is filled with paperboard. If the usage is over 100%, then the paperboard has to compress and contract. Because pressing force forms the fibre structure permanently, the irreversible part of the compaction deformation of the paperboard blank in tray pressing can be considered plastic deformation. The surface smoothness of the tray and the quality of folding of the tray wall in the corners was analysed to determine suitable mould clearances. The grading scale of the tray wall quality is presented in Table 3, and sample pictures are shown in Fig. 7.

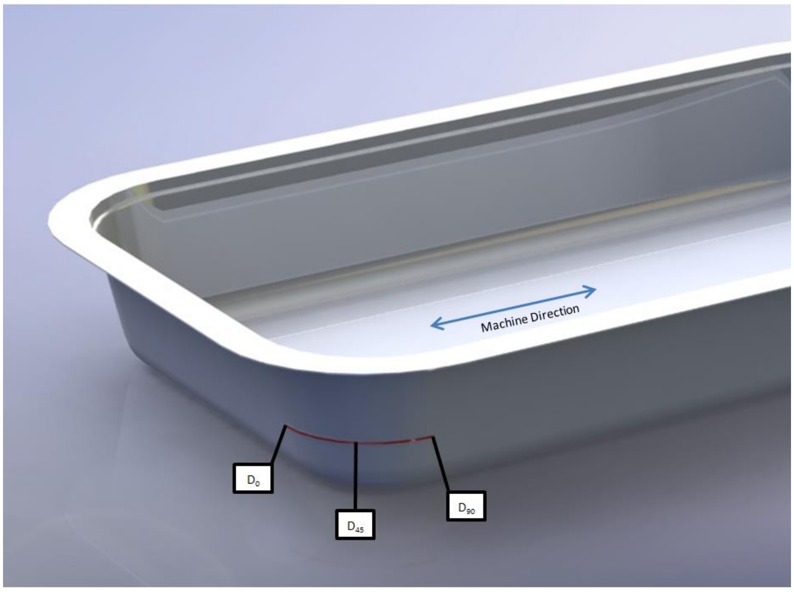

Fig. 4. Observation point D in the tray corner; clearance between the moulds is constant at each selected height.

Blank geometry and preparation

Tray blanks were cut into shape using a die cutting machine according to the geometry presented in Fig. 5. The tray blank area was 651.9 cm^2, and the dimensions were 319.3 mm x 216.3 mm. The creased blank and press formed tray corners are presented in Fig. 6.

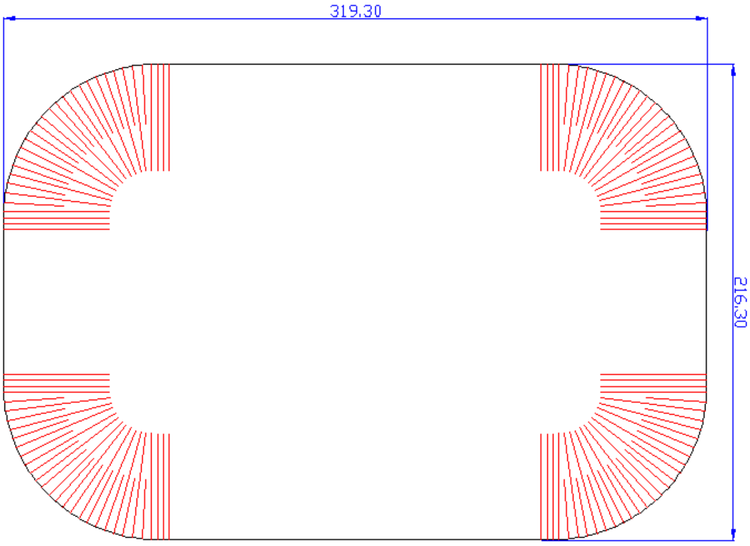

Fig. 5. Tray blank geometry; creases are presented in red

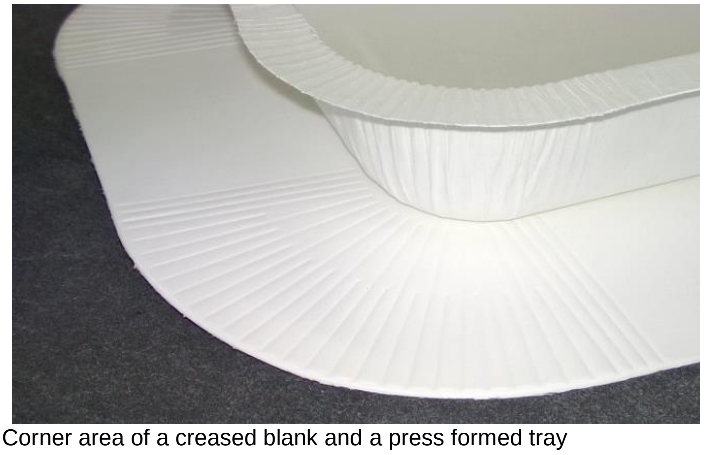

Fig. 6. Corner area of a creased blank and a press formed tray

After blank preparation, the pre-cut and creased blanks were pressed to the tray form. The tray-forming process is described in Fig. 7.

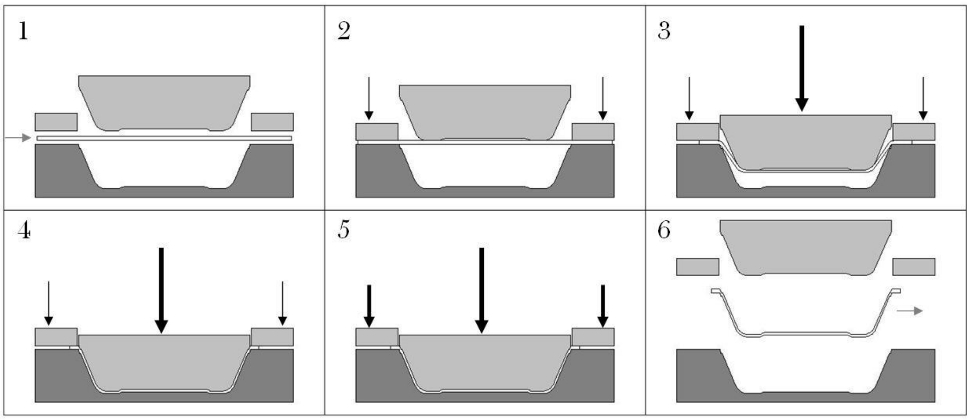

Fig. 7. Tray-forming process

Phase 1:	The paperboard blank is positioned between the moulding tools.
Phase 2:	The blank holding force tightens the blank between the rim tool and the female tool.
Phase 3:	The male tool presses the blank into the mould cavity in the female tool.
	Folding of the tray corners is controlled with blank holding force.
Phase 4:	The male tool is held at the bottom end of the stroke for a set time (0.5 to 1.0 s).
	The plastic coating softens, and creases in the corners of the tray are sealed together.
Phase 5:	The flange of the tray is flattened by the rim tool.
Phase 6:	The formed tray is removed, and a new blank can be fed into the tray press.
	The tray achieves its final rigidity when it cools down.

The process parameters of tray pressing are presented in Table 2. The female tool was heated, while the male tool was kept at room temperature. Heat was applied only to the board size of the material to prevent melting of PET coating. Pressing speed is the speed of the male tool until it is stopped by the female tool. The creases were analysed after press forming. Ten different analysis points were analysed from each corner. The analysis was done by evaluating the structure of the creases after forming. Samples of tray corners were cast in acryl plastic to preserve shape of the creases during processing. Then, the samples were ground, polished, and analysed with an optical microscope. The creases were analysed in the machine direction, cross direction, and at a 45-degree angle (Fig. 4). The samples were then compared to each other to determine if the material thickness had an effect on the crease geometry and the quality of the tray rim. The creases were measured and average dimensions are presented in Table 5. The dimensional measurements of creases were made from microscopy images. Also, a visual evaluation of press formed trays was made. Ten different samples of each analysis point were analysed.

Table 2. Forming Parameters

Female tool temperature	Pressing dwell time	Rim tool holding force	Pressing force	Pressing speed
170 °C	1 s	1.16 kN	135 kN	130 mm/s

The quality of ready-made trays was graded according to the scale presented in Table 3. Examples of corners of the tray evaluated with different grades can be seen in Fig. 8.

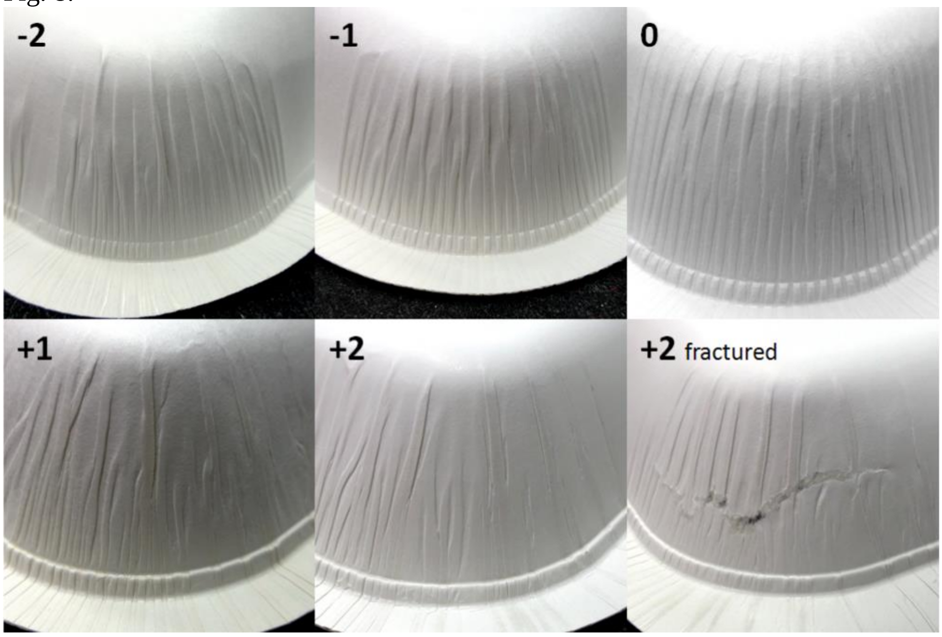

Fig. 8. An example of grades of corner quality at observation point D on the basis of Table 2; Grade 0 is the most desirable

Grade 0 is targeted when process parameters of the tray production are adjusted. Grades -1 and +1 are mainly visual defects; the functionality of the tray is not compromised. However, these grades indicate the direction where the paperboard thickness or the mould clearance should be altered.

Table 3. Grading Scale of the Tray Wall Quality

Grade	Description
-2	Mould clearance too large, creases fold irregularly
-1	Mould clearance a bit too large, unaccomplished crease smoothening
0	Mould clearance ideal, creases perfectly smoothed
+1	Mould clearance a bit too small, tray wall starts to polish too much
+2	Mould clearance too small, compaction of material causes fractures

RESULTS AND DISCUSSION

Measurements and tray wall quality evaluations at observation points A to J are presented in Table 4.

Table 4. Folding and Compaction of Paperboard in Tray Pressing

Observation points	Distance from tray bottom [mm]	Length of compressed paperboard blank compared to original length in one corner	Trayforma 190 + PET 40		Trayforma 230 + PET 40		Trayforma 310 + PET 40		Trayforma 350 + PET 40	
			Usage of mould clearance	Tray wall quality	Usage of mould clearance	Tray wall quality	Usage of mould clearance	Tray wall quality	Usage of mould clearance	Tray wall quality
A	0	84,70 %	49,81 %	-2	57,19 %	-2	80,24 %	-1	85,78 %	-1
B	5	84,58 %	49,88 %	-2	57,27 %	-2	80,36 %	-1	85,90 %	-1
C	10	75,46 %	55,91 %	-2	64,19 %	-2	90,07 %	-1	96,28 %	0
D	15	67,63 %	62,38 %	-2	71,63 %	-1	100,51 %	0	107,44 %	0
E	20	61,76 %	68,31 %	-2	78,43 %	-1	110,06 %	0	117,65 %	0
F	25	58,15 %	72,55 %	-1	83,29 %	-1	116,88 %	0	124,94 %	0
G	30	53,55 %	78,79 %	-1	90,46 %	-1	126,93 %	0	135,69 %	+1
H	35	51,49 %	81,94 %	-1	94,08 %	-1	132,01 %	0	141,11 %	+1
I	38	50,23 %	83,99 %	-1	96,43 %	0	135,31 %	1	144,65 %	+1
J	38	57,37 %	73,53 %	0	84,43 %	0	118,47 %	0	126,64 %	0

Based on these results, the recommended usage of mould clearance is from 95% to 135% for the tested paperboard types when forming selected example trays. This result cannot be applied to other paperboard types without further tests.

Material thickness and its relation to forming mould clearance depend on the strength properties of formed materials and the surface friction between blank and moulds. Mould clearance between male and female moulds does not affect the tray flange area (observation point J). The tray flange is flattened between the rim tool and female mould, and the flattening force can be adjusted independently; therefore, all materials have grade 0 at observation point J.

The formation of creases was similar in each corner of the tray. In future studies with symmetrical geometries such as a rectangular tray, the analysis can be limited to just one corner.

Observation points B, D, G, and I were selected for microscopic analysis. Because the forming forces used in this study were constant for each material, elongation was largest with material of the smallest grammage (Fig. 9). The shape of the creases therefore varied more, and the width of the crease was larger.

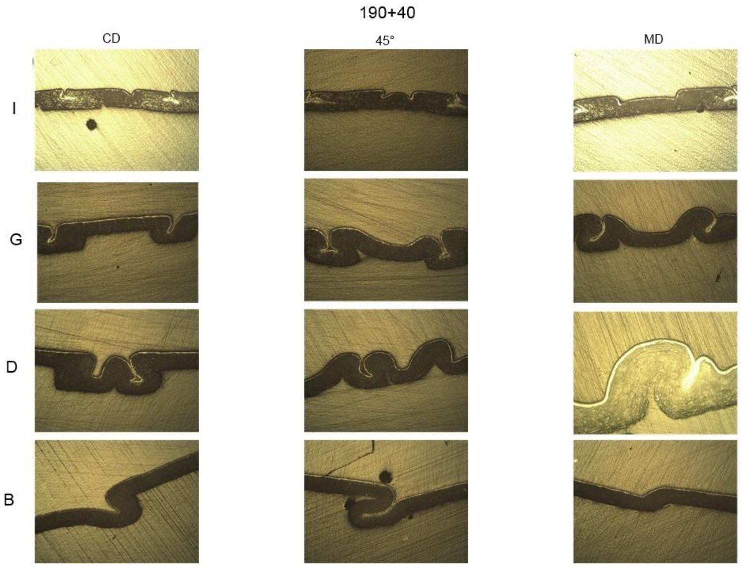

Fig. 9. Sample microscopy images of 190 g paperboard + 40 g PET at different observation points

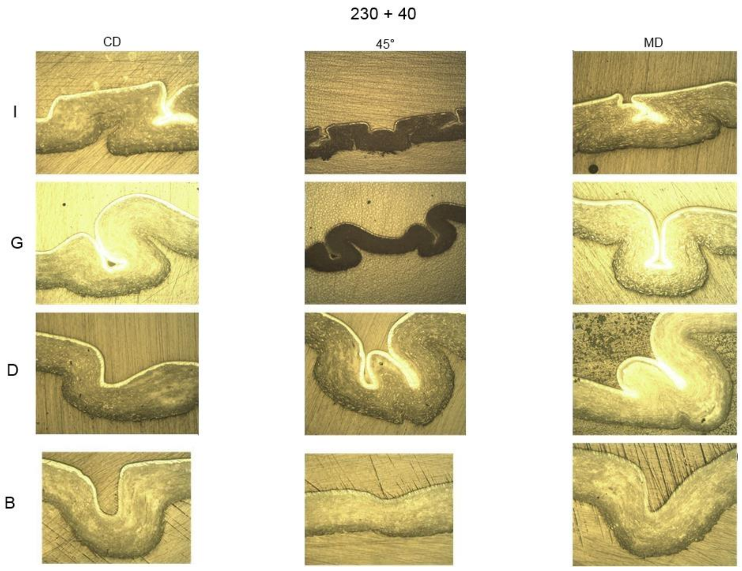

Fig. 10. Sample microscopy images of 230 g paperboard + 40 g PET

Shape of the creases between 230+40 and 190+40 was quite similar, but elongation was significantly higher with thinner material.

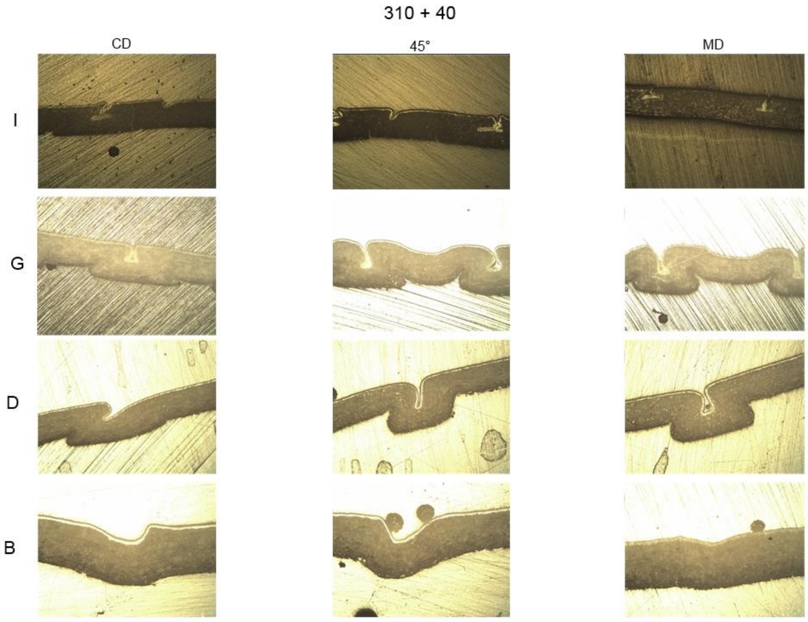

Fig. 11. Sample microscopy images of 310 g paperboard + 40 g PET

The higher strength properties of thicker materials, such as 310 + 40 (Fig. 11) and 350 + 40 (Fig. 12), decrease the variations in crease shape. The width of the crease is more compact.

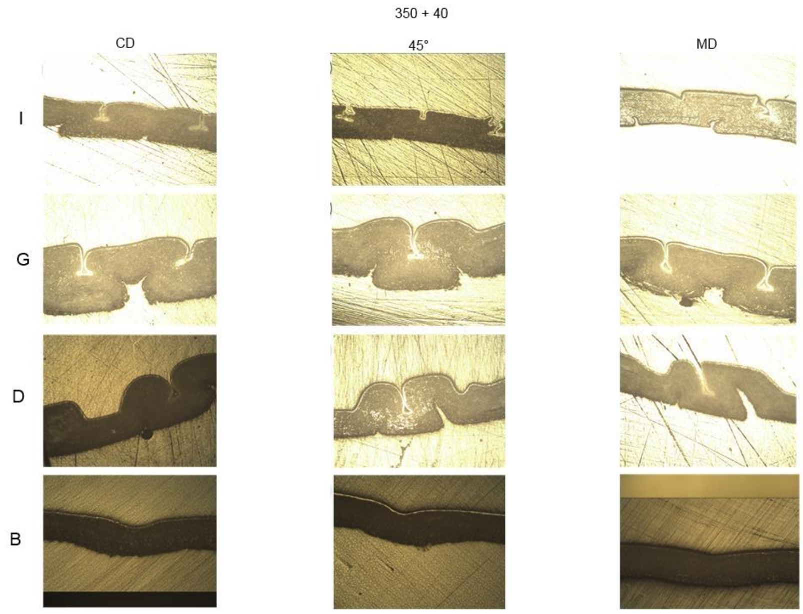

Fig. 12. Sample microscopy images of 350 g paperboard + 40 g PET

The dimensional measurements of creases were made from all material thicknesses. Two adjacent creases (Fig. 13) were measured from each sample and the averages of these measurements are presented in Table 5. The values indicate that the width of the press-formed creases decreases when the material thickness increases.

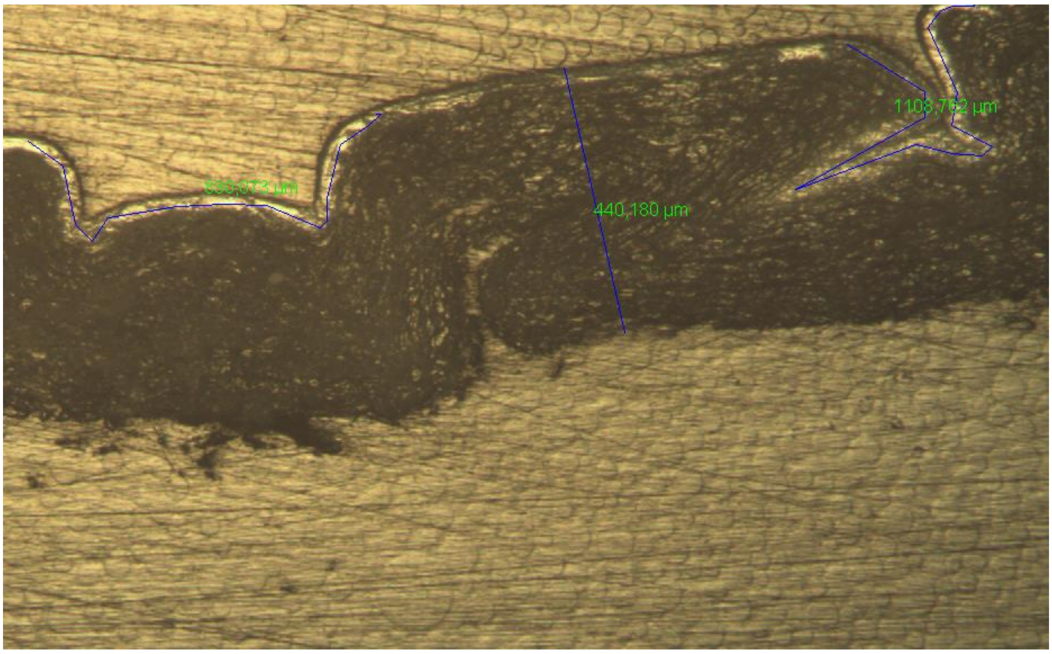

Fig. 13. Sample microscopy image of crease length measurements

Table 5. Crease Length Averages

Grammage [g/m^2]	Thickness before forming [µm]	Crease length average [µm]
190+40	270	1003
230+40	310	901
310+40	435	856
350+40	465	809

Formation of creases varied less and shape of the creases was more homogenous with 350 + 40, which had the best usage of mould clearance.

With every material thickness, there was some variance in crease formation. However, a conclusion can be made regarding the width of press-formed creases, which decreased when the material thickness increased. On average, in press-forming, creases in each tested material seemed to perform similarly in the cross direction, machine direction, and at a 45-degree angle.

With lower material thicknesses, such as the 190 + 40 and 230 + 40, the paperboard also became wrinkled in areas that were not creased, which had an effect on the overall visual quality of the package. These wrinkles are visible in Fig. 14 and are believed to be caused by the lower in-plane stiffness of the thinner materials.

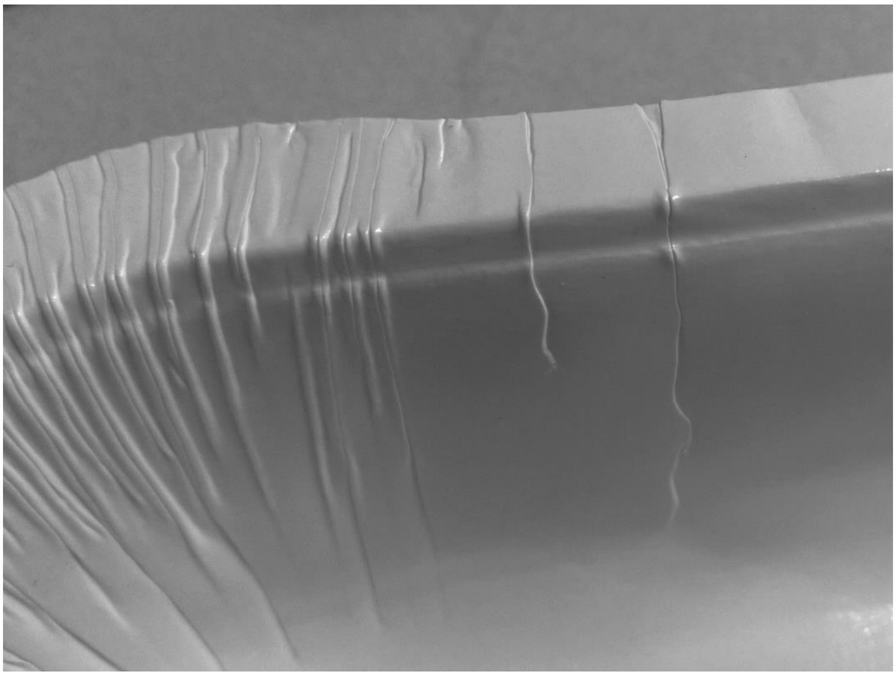

Fig. 14. Wrinkles outside the creased area

In this article, the effects of constant factors such as material thickness and mould clearance were observed. These factors cannot be altered during production runs. However, many forming parameters, such as temperature, speed, and force, can be adjusted to improve the quality of the end product and consequently the suitability for modified atmosphere packaging. The effect of these factors should be further investigated in future work.

CONCLUSIONS

1. Mould clearance usage needs to be sufficient to ensure the correct formation of creases and smoothening of the tray corner surface. Overusage of mould clearance causes fracturing and visual defects on the paperboard surface. The recommended usage of mould clearance is from 95% to 135% for the paperboard types used in this study when forming selected example trays. This result cannot be applied to other paperboard types without further tests.

2. The width of the press-formed creases decreases when the material thickness increases. On average, in press-forming, creases in each tested material seemed to perform similarly in the cross direction, machine direction, and at a 45-degree angle.

ACKNOWLEDGMENTS

The authors would like to thank the European Union's regional funding, project A32103.

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Article submitted: May 17, 2013; Peer review completed: June 22, 2013; Revised version received and accepted: Sept. 13, 2013; Published: September 24, 2013.

Publication III

Leminen, V., Tanninen, P., Lindell, H. and Varis, J.

Effect of blank holding force on the gas tightness of paperboard trays manufactured by the press forming process

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Bioresources
Vol. 10, No. 2 pp. 2235-2243, 2015

Effect of Blank Holding Force on the Gas Tightness of Paperboard Trays Manufactured by the Press Forming Process

Ville Leminen,* Panu Tanninen, Henry Lindell, and Juha Varis

Although several authors have studied 3D forming using the press forming process, the gas tightness of polymer-coated paperboard trays has not been widely researched. In this paper, the effect of blank holding force on the surface quality and tightness of press-formed paperboard trays was researched. The press-formed trays were heat-sealed with a multilayer polymer lid. The tightness of the trays was analyzed by following the oxygen content of the packages over the course of 14 d and by using a penetrant coloring solution to locate possible leaks. The results indicate that the blank holding force had a great effect on the quality and tightness of the trays, especially in the case of a rectangular geometry. The geometry of the formed trays played a significant role in process parameter selection, and more demanding geometries emphasize the importance of parameter optimization. However, with the correctly selected parameters, the use of modified atmospheric packaging (MAP) in polymer coated paperboard trays was shown to be possible. The oxygen content of both analyzed geometries was found to be less than 1% 14 d after sealing. It was also demonstrated that the gas tightness of a seal cannot be confirmed using a penetrant solution test exclusively.

Keywords: Paperboard; Press-forming; MAP; Modified atmosphere packaging; Tightness; Blank holding force

*Contact information: Department of Mechanical Engineering, Lappeenranta University of Technology, P.O. Box 20, FI-53851 Finland; *Corresponding author: ville.leminen@lut.fi*

INTRODUCTION

Both three dimensional (3D) forming and the material behavior of paperboard during press forming have been previously studied by several authors. These articles describe the effect of process parameters, adjustability, and tooling technology (Hauptmann *et al.* 2014; Leminen *et al.* 2013) functionality of materials in forming (Vishtal and Retulainen 2012; Tanninen *et al.* 2014; Zeng *et al.* 2013) and also the gas tightness of trays (Hauptmann and Majschak 2011). However, tray-shaped, paperboard-based, and polymer-coated packages have not yet become a significant competitor to polymer-based packages when the use of modified atmosphere packaging (MAP) is required. The quality of industrially manufactured trays is not often good enough for MAP-usage. One reason for this may be due to insufficiently controlled process and parameters. One reason for polymer-based package dominance is due to the tightness of the packages, as instances where the lid is heat sealed to the package have been problematic. A major cause for this phenomenon is creases in the sealing area. The paperboard blank is creased to control and enable paperboard formation to certain geometric conditions when the blank is press-formed to three dimensional form. Furthermore, the creased wrinkles can act as capillary tubes, which may cause leaks in the package (Leminen *et al.* 2012; Hauptmann *et al.* 2013).

In previous studies, gas tight sealing resulting from pre-creased blanks was not achieved (Hauptmann *et al.* 2013). Poor sealing can critically affect the overall shelf life of food (Yeh and Benatar 1997).

In this study, the press forming of polymer-coated paperboard trays and the heat sealing process of lidding films into these trays with MAP was studied. The study attempted to show that a MAP-tight paperboard based package, manufactured by press forming, can potentially be manufactured. The effect of the blank holding force on the rim area surface quality, and consequently, on the gas tightness of the package with a sealed lid, is presented. The modified atmosphere in the packages was analyzed using an optical fluorescence O_2 analyzer. The purpose of the atmosphere analysis was to investigate the headspace gas and the tightness of the sealed packages. Results of the experiments are presented to support the claims.

EXPERIMENTAL

Materials

The primary material used in the trays was Stora Enso Trayforma Performance 350 + 40 WPET (Stora Enso Imatra Mills, Finland). This material is a polyethylene terephthalate (PET) extrusion-coated paperboard with a base material grammage of 350 g/m^2 and a coating grammage of 40 g/m^2. The base board consists of three solid bleached sulphate (SBS) layers.

The materials were stored in a constant humidity chamber at 85% relative humidity (RH) to ensure sufficient humidity. The high humidity was used to maintain the delivery moisture content of the paperboard, and the average humidity of the tested materials was measured using an analysis scale. The measured moisture content of the material was 10.5%.

Elongation values for tested materials, measured in 23 °C and 50% RH conditions, was approximately 5% in cross direction and 2.5% in the machine direction. However, according to Kunnari *et al.* (2007), by varying the moisture and/or temperature of the paper based materials, it is possible to increase the elongation from around 2 to 2.5 percentage points. Vishtal *et al.* (2014) report a conditioning at 75% RH to increase the maximum drawing limit up to 70% at room temperature and up to 30% at 165 °C. Therefore, the elongation values during tray pressing can be increased. The lidding material used in the heat sealing was a PET-sealable multi-layer film, Westpak WestTop 405B PET (WestPak Oy Ab; Säkylä, Finland).

Methods

Experimental design

The blank holding force (*i.e.*, rim tool force) is the force that controls the folding of the tray corners during the press-forming of paperboard trays. The effect of the blank holding force on the quality of the formed product has been discussed in previous studies (Hauptmann and Majschak 2011; Tanninen *et al.* 2014). In this study, the blank holding force was varied to create trays with different rim surface qualities to observe the effect of blank holding force with respect to the MAP-tight sealability. During the study, the other process parameters were kept constant.

Two differently shaped trays, a rectangular tray and an oval tray, were selected for the forming and sealing tests. These two geometries are meant to represent most typical tray shapes used in the food packing industry. The maximum blank holding force for both geometries was investigated. The blank holding force was found using the highest possible force that did not cause cracks in the formed trays. This was the force that was used as the starting point, from which the force was lowered, in order to investigate the effect of blank holding force. The lowest force used was the force that was required to keep the rim tool in the starting position before the pressing process was initiated.

A detailed description of the press forming process is presented in a previous manuscript (Leminen *et al.* 2013). The forming parameters with both geometries were female tool temperature 170 °C, pressing dwell time 1 s, pressing force 135 kN and pressing speed 130 mm /s. The blank holding forces are presented in Table 1. The tray geometries used in the study are presented in Figs. 1a and 1b.

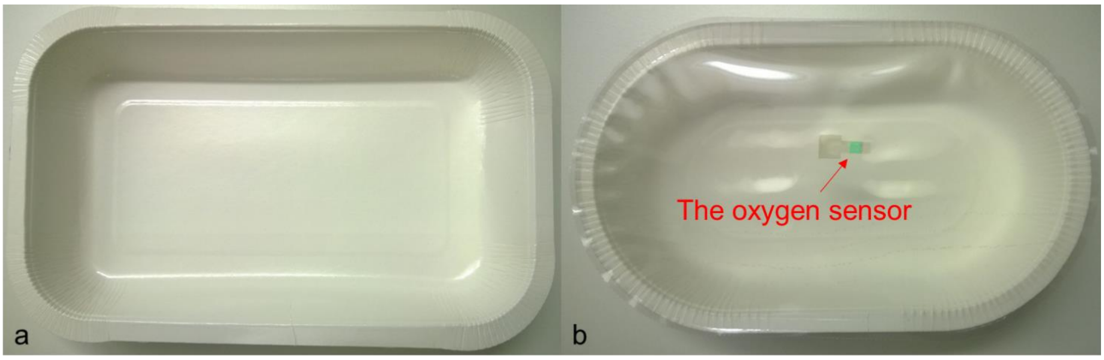

Fig. 1. The tray geometries used in the study: (a) rectangular tray and (b) oval tray with the heat sealed lidding film and O_2 sensor

Table 1. Blank Holding Forces for Oval and Rectangular Geometry

Oval tray	Rectangular tray
1.93 kN	1.16 kN
1.29 kN	0.77 kN
0.94 kN	0.68 kN
0.52 kN	0.58 kN

The manufactured trays were sealed with a lid using an Ilpra Speedy tray sealer (Ilpra S.p.A; Vigevano, Italy (Ilpra 2014). The tray sealer is presented in Fig. 2.

After manufacturing of the trays, a lidding film was sealed on top of the trays' rim area. The heat-sealing parameters used in this were sealing temperature 190 °C and sealing dwell time 2.5 s. Lower sealing temperatures and dwell times were also tested, but they resulted in seal leaks with a high frequency. Higher heat input resulted in the melting, and subsequent leaking of the lid material outside the sealing area. Sealing pressure was kept at a constant 6 bar, which is the normal pneumatic pressure used in industrial scale tray sealers. This resulted in a surface pressure of 2.65 N/mm^2. The trays were flushed with a common gas mix for food applications. The used gas composition was 70% N_2 and 30% CO_2.

The used sealing tool was designed specifically for use with paperboard trays. The tool-set consisted of a heated upper tool with a flat metal surface, and a bottom tool with a silicone surface. The tray rim was placed between the tools and the lid film, and the trays were sealed together by applying pressure and heat.

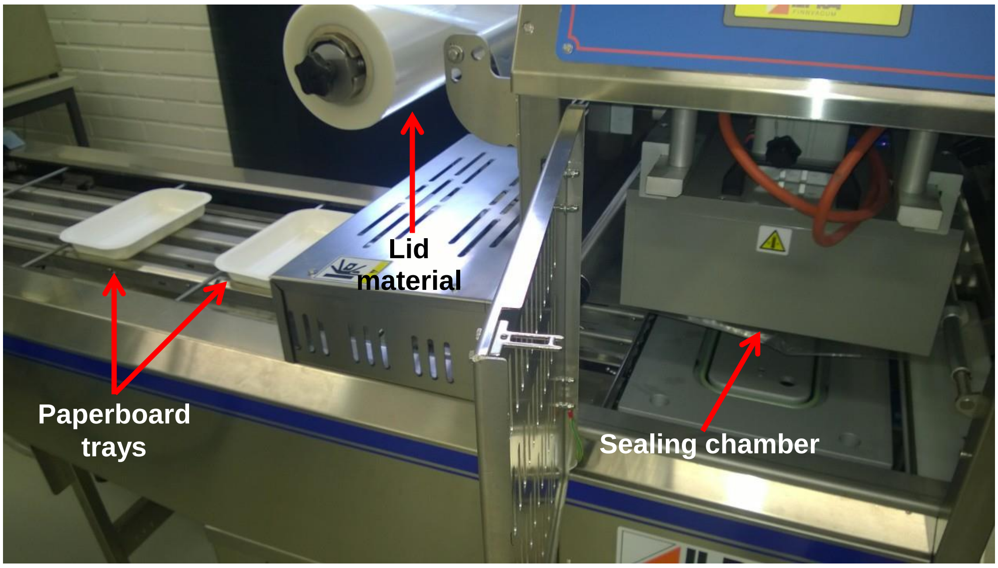

Fig. 2. Sealing equipment used in the study

The oxygen composition inside the package was analyzed using a Mocon Optech O_2 Platinum analyzer (Mocon Inc.; Minneapolis, USA). The analyzer utilizes the standard ASTM F-2714-08 (Standard Test Method for Oxygen Headspace Analysis of Packages Using Fluorescent Decay). The measurement method consisted of inserting an oxygen sensor inside the lidding film before heat sealing the film to the tray. The response of the phosphorescent sensor was analyzed using a hand held light beam device. The analysis occurred over the course of 14 d. The sealed trays were stored in a refrigerator, at a temperature of 6 °C, to simulate realistic storage conditions. Figure 1b depicts a tray with a heat sealed lid and an O_2 sensor inside the package.

After the O_2 measurements, the trays were flushed with a coloring solution in accordance with the European standard (EN 13676 2001). The gas sensor was removed, and a hole was cut into the lid. The coloring solution was applied to the tray and the sealed area for five minutes and the seal was inspected for leaks. The reagents in the coloring solution were dyestuff E131 Blue and ethanol (C_2H_5OH, 96%). The color solution consisted of 0.5 g of dyestuff dissolved in 100 mL of ethanol. Flushing was done to detect leaks in the package and sealing area. This was done to investigate if this commonly used leak detection method can be used to confirm the gas tightness of a package.

RESULTS AND DISCUSSION

The effect of the blank holding force on the flatness of the tray flange was quite apparent. The magnitude of parallel and perpendicular forces enabling the folding process is known to depend on the blank holding force. When blank holding force is too low the paperboard blank folds insufficiently and the desired quality of the rim area (flange) is not achieved. However, it is not possible to visually evaluate the exact quality in which a tight seal can be achieved.

Figures 3 and 8 show tray corners with different rim holding force settings. Figure 3 shows that, if the blank holding force is low enough, the change in rim area quality is clear in trays with rectangular geometry. Both the lower surface quality and the subsequent leakage caused by the heat-sealing can be detected with the coloring solution. Rectangular trays manufactured with a blank holding force of 1.16 kN, 0.77 kN, and 0.68 kN did not have leakage in the coloring solution tests, while the trays manufactured with a blank holding force of 0.58 kN showed leakage in the tray rim area.

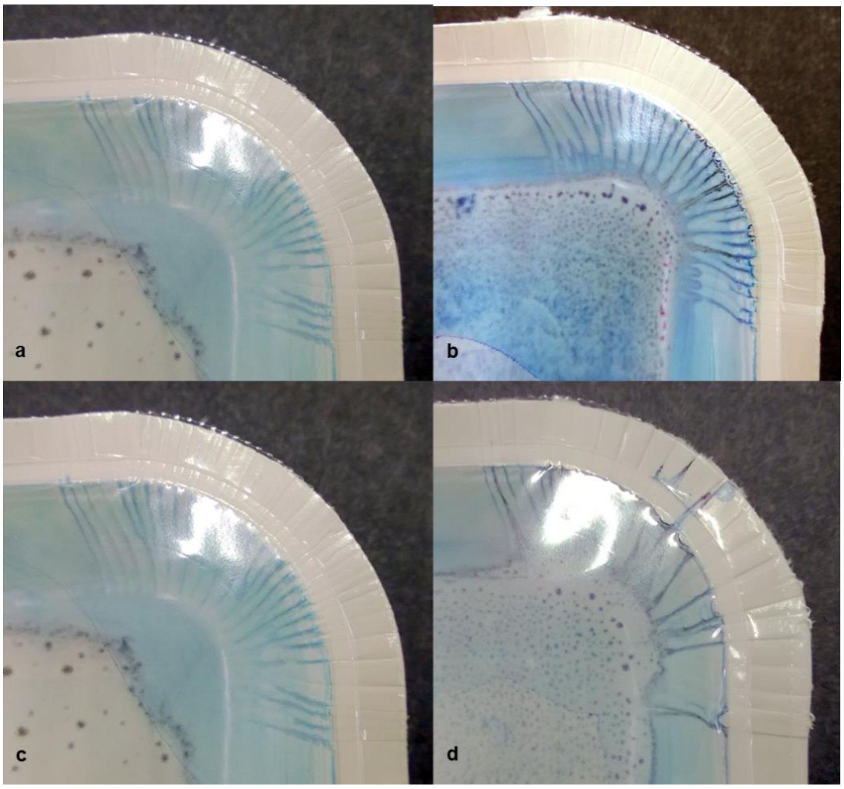

Fig. 3. Rectangular tray corners with different blank holding forces: (a) 1.16 kN, (b) 0.77 kN, (c) 0.68 kN, and (d) 0.58 kN

Figure 4 clearly shows gas leakage in the packages manufactured with a blank holding force of 0.58 kN and 0.68 kN. While the tray shown in Fig. 3b appears to have an intact seal, the MAP composition in the package changed drastically (Fig. 4, 0.68 kN). This shows that the gas tightness of a package cannot be confirmed using the coloring solution, even though the results would indicate a gas-tight package.

The oxygen content in the packages manufactured with a blank holding force of 0.77 kN and 1.16 kN still registered at less than 1% two weeks after the initial sealing. The results of the coloring solution test and O_2 measurements indicate that the blank holding force has a clear effect on the tightness of the sealed package. There is a threshold between blank holding forces of 0.68 kN and 0.77 kN which enables the tray rim area to form more evenly and make possible the gas-tight sealing.

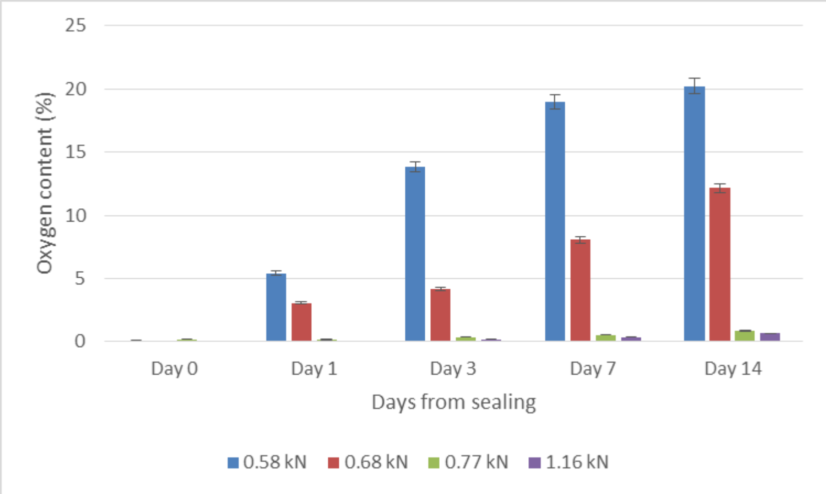

Fig. 4. Oxygen measurement averages of the rectangular trays manufactured with a varied blank holding force. Data provided with ± 3% accuracy of measuring.

Fig. 5. Oval tray corners with different blank holding forces: (a) 1.93 kN, (b) 1.29 kN, (c) 0.94 kN, and (d) 0.52 kN

Figure 5 shows no visible leakage in any of the tested oval trays. It seems that when the trays were subjected to a pressing force of 135 kN and the press-forming process was otherwise controlled correctly, the rim area quality remained tight, withstanding all blank holding forces. Oval trays with all blank holding forces did not have leakage in the coloring solution tests. The coloring solution test also did not reveal any cracks or pinholes in either tray geometry.

Figure 6 shows that the gas composition of the oval trays was less than 1% oxygen after 14 d with all used blank holding forces. The formation of oval shape trays during tray pressing occurred more homogeneously in comparison to the rectangular trays with different blank holding force values. This could be due to the less demanding geometry of the tray, as the radius in the tray corners and the density of creases are larger. The rectangular tray is more sensitive to process parameter alterations; therefore, its processing window is smaller.

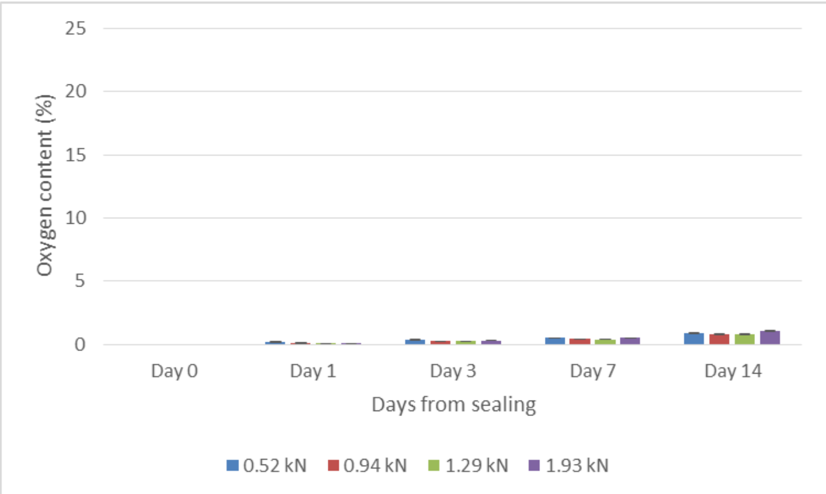

Fig. 6. Oxygen measurement averages of the oval trays manufactured with a varied blank holding force. Data provided with ± 3% accuracy of measuring.

The geometry of the trays affects the manufacturing process of the MAP-trays as a whole, as incorrectly selected process parameters for the press-forming stage can cause deterioration in the tray quality. Typically, deterioration of tray quality cannot be mended during the heat sealing of the lid.

Due to paperboard properties a crimpled appearance cannot be avoided completely if certain geometries are formed. However, when every process step is done correctly, gas-tight seals can be reliably achieved when polymer-coated paperboard trays are formed from pre-creased blanks.

CONCLUSIONS

1. The blank holding force affects the surface quality of the rim area of press-formed polymer coated paperboard trays. The surface quality directly affects the gas tightness of the package after a lid has been sealed to the tray.

2. The change in surface quality is more apparent in trays with a rectangular geometry than those with an oval geometry.
3. The gas tightness of press-formed polymer coated paperboard trays sealed with a multi-layer polymer lidding film is considered satisfactory for the use of modified atmosphere packaging (MAP) in food solutions. This requires that suitable tools, materials, and process parameters are selected and used during the tray manufacturing process and lid sealing process.

ACKNOWLEDGMENTS

The authors would like to thank Stora Enso and Westpak for providing materials, as well as the European Union for providing regional funding, project A32103. Petri Mäkelä's help with the test runs is also appreciated.

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Article submitted: November 24, 2014; Peer review completed: January 22, 2015; Revised version received and accepted: February 6, 2015; Published: February 18, 2015.

Publication IV

Leminen, V., Mäkelä, P., Tanninen, P. and Varis, J.
Methods for Analyzing the Structure of Creases in Heat Sealed Paperboard Packages

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Journal of Applied Packaging Research
Vol. 7, No. 1 pp. 49-60, 2015

Journal of Applied Packaging Research

Volume 7 | Number 1 Article 5

2015

Methods for Analyzing the Structure of Creases in Heat Sealed Paperboard Packages

Ville Leminen
Lappeenranta University of Technology, ville.leminen@lut.fi

Petri Mäkelä
Lappeenranta University of Technology, petri.makela@lut.fi

Panu Tanninen
Lappeenranta University of Technology, panu.tanninen@lut.fi

Juha Varis
Lappeenranta University of Technology, juha.varis@lut.fi

Methods for Analyzing the Structure of Creases in Heat Sealed Paperboard Packages

Cover Page Footnote
ACKNOWLEDGMENTS The authors would like to thank the European Union's regional funding, project A32103 and also Stora Enso and Westpak for providing materials.

Methods for Analyzing the Structure of Creases in Heat Sealed Paperboard Packages

Ville Leminen
Lappeenranta University of Technology
ville.leminen@lut.fi

Petri Mäkelä
Lappeenranta University of Technology
petri.makela@lut.fi

Panu Tanninen
Lappeenranta University of Technology
panu.tanninen@lut.fi

Juha Varis
Lappeenranta University of Technology
juha.varis@lut.fi

ABSTRACT

Press-forming of paperboard has been previously studied by several authors. A point of interest regarding gas tight heat sealing of the packages are the creases in the package. The objective of this article was to study and compare different microscopic imaging methods to research an optimal imaging method for the formation of creases in the press-forming process of polymer coated paperboard trays. The studied methods were: Scanning electrode microscopy (SEM), X-ray Microtomography, Optical light microscopy and Polarized light microscopy. All four tested methods delivered clear images. Casting of the samples in an acrylic resin and light microscope imaging was found to be the most suitable method for the analysis of heat sealed creases and leakage detection.

Key Words: *paperboard, press-forming, heat sealing, microscope, crease*

1.0 INTRODUCTION

Three dimensional forming and material behavior of paperboard in the press forming process has been previously studied by several authors. [1] - [7] However, tray shaped, paperboard based and polymer coated packages have not yet been able to become a significant competitor to polymer based packages in food packaging when the use of modified atmosphere packaging (MAP) is required. This is mainly because the tightness of the packages especially in the area where the lid is heat sealed to the package has been a problem. A major cause for this are creases in the sealing area that are done to control and enable the formation of paperboard to certain geometries. [7],[8] The creases can act as capillary tubes that may cause leaks in the package when a lid is heat sealed to the tray. A poor sealing result can be a critical factor affecting shelf life of food. [9] Faults in sealing surface or other faults such as cracks or pinholes in the package can cause the MAP to leak from the package. Leakage of MAP can cause reduction in sensory shelf life and microbiological quality of packed foods [18].

There are several different methods for testing package and seal integrity. One common method is destructive dye penetrant testing that is usually done according to standards, such as European standard EN 13676 [14], ASTM F1929 [19] or ASTM F3039 [20]. Industrial practices include test methods such as using a leak detection system which forms a vacuum into a chamber and detects possible leaks. [21] One option is to use an Optical Fluorescence O_2 Analyzer which utilizes standard ASTM F-2714-08 [22]. However, these methods do not provide insight to the exact mechanisms which cause the leaks. This kind of information can be achieved only by microscopic analysis.

This paper compares different microscopic imaging methods to research an optimal imaging method for the formation of creases in the press-forming process of polymer coated paperboard trays. The formation of creases is a critical investigation point when a gas tight heat seal is required in food packaging. The object was to find a cost effective and reliable method for the comparison of creases after press forming of polymer coated paperboard trays and after the heat sealing of a polymer based lidding film. Microscopic- and leakage analysis was done after heat sealing of a lidding film to the tray to investigate the visibility of said lidding film and leakage in the images. This kind of analysis is needed in order to get a better understanding of the formation of the creases and to improve the quality of trays produced in the press forming process

2.0 MATERIALS

The material used in the trays was Stora Enso Trayforma Performance 350 + 40 WPET which is a polyethylene terephthalate (PET) extrusion-coated paperboard with a base material grammage of 350 g/m^2 and a coating grammage of 40 g/m^2. The base board consists of three solid bleached sulphate (SBS) layers. [10]

The materials were stored in a constant humidity chamber at 85% relative humidity to ensure sufficient humidity.

This higher humidity was used to maintain the delivery moisture content of the paperboard. The average humidity of the tested materials was measured using an analysis scale. The measured moisture content of the material was 10.2 %.

Elongation values for tested materials measured in 23 °C and 50% RH was approximately 5% in cross direction and 2.5% in the machine direction. However according to [11], by varying the moisture and / or temperature of paper based materials, it is possible to get gains in elongation of around 2 to 2.5 percentage points, and therefore the elongation values in tray pressing can be higher.

The lidding material used in the heat sealing was a PET-sealable multi-layer film, Westpak WestTop 405B PET.

The material used in the sample preparation of light microscopy samples was Struers ClaroCit which is a clear and easily polished acrylic resin which is suitable to be used with paperboard and polymer materials because it hardens without the use of pressure or heat. [12]

3 METHODS

3.1 PRESS-FORMING OF PAPERBOARD TRAYS

Press-forming is a process which is used to create

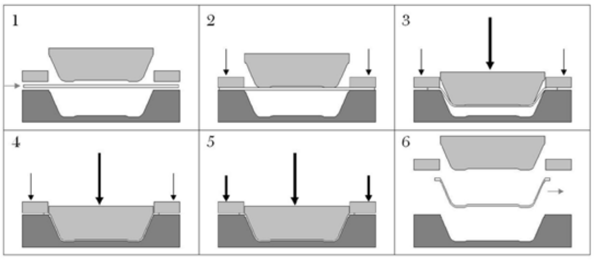

Figure 1: Tray-forming Process

three-dimensional shapes such as plates or trays. The tray forming process is presented in Figure 1.

Phase 1: The paperboard blank is positioned between the moulding tools.
Phase 2: The blank holding force tightens the blank between the rim tool and the female tool.
Phase 3: The male tool presses the blank into the mould cavity in the female tool. Folding of the tray corners is controlled with blank holding force.
Phase 4: The male tool is held at the bottom end of the stroke for a set time (0.5 to 1.0 s). The plastic coating softens, and creases in the corners of the tray are sealed together.
Phase 5: The flange of the tray is flattened by the rim tool.
Phase 6: The formed tray is removed, and a new blank can be fed into the tray press. The tray achieves its final rigidity when it cools down.

Sample trays (example of tray geometry in Figure 2) were manufactured at the Laboratory of Packaging Technology at Lappeenranta University of Technology (LUT). The tray dimensions were 319x216x38 mm. The formation of creases was analyzed using four different imaging methods to compare the suitability of these methods for this kind of analysis. The used press-forming parameters are presented in Table 1.

Female tool temperature	Pressing dwell time	Pressing force	Pressing speed	Rim tool holding force
170 °C	1 s	135 kN	130 mm / s	1.16 kN

Table 1. Forming parameters, rectangular tray

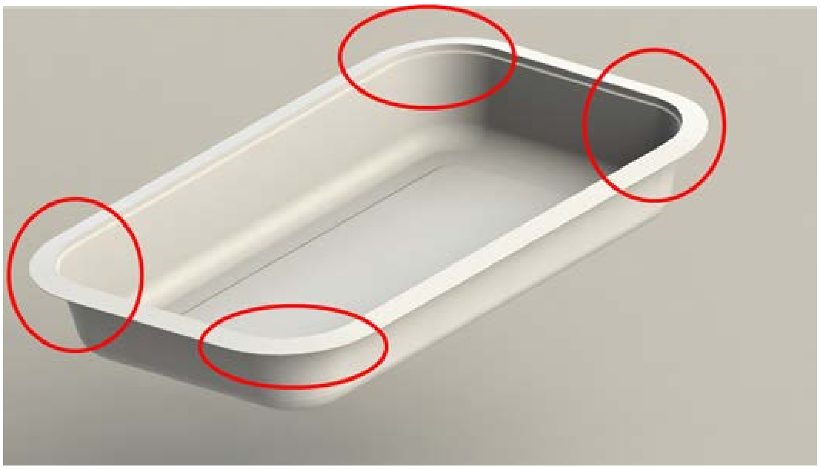

Figure 2. Tray geometry and areas where the inspected samples were extracted from (highlighted by red)

Figure 3. A leak in a tray corner indicated by a coloring solution

3.2 HEAT SEALING AND LEAKAGE INVESTIGATION OF TRAYS

The trays were heat sealed with a PET-sealable multi-layer film using an industrial scale heat sealing device. [13] The seals were tested with a coloring solution applying the European standard EN 13676. [14] The reagents in the coloring solution were dyestuff E131 Blue and Ethanol ($C2H5OH$, 96%). The color solution consisted of 0.5 g dyestuff in 100 ml ethanol. Possible leaks were located (Figure 3.) and after that the leak spot was investigated with different methods to compare the visibility of leaks in the images.

3.3 MICROSCOPIC ANALYSIS METHODS

The heat sealed trays were analyzed using four different imaging methods; scanning electron microscopy (SEM), X-ray microtomography, light microscopy and polarized light microscopy. All methods were tested extensively by analyzing leaking creases revealed by the coloring solution and also creases that were found to be sealed properly. Same samples were not analyzed with every method because the methods require specific sample preparation. In total over 200 trays were analyzed with dye penetrant testing. The amount of samples tested by each of the methods varied from 10 (X-ray microtomography and SEM) to 115 (Optical light microscopy).

Table 2. Number of tested samples by each method

Method	Number of tested samples
Scanning electron microscopy (SEM)	10
X-ray Microtomography	10
Optical light microscopy	115
Polarized light microscopy	14

3.3.1 SCANNING ELECTRON MICROSCOPY (SEM)

Scanning electron microscopy uses focused electron beams to sweep the object and creates a very accurate image of the surface and its topography based on the scattering of the electrons. SEM-images have a nearly three dimensional effect and they make the understanding of the surface structure easier than purely 2D images. Magnification of SEM images varies from 10 times to 500 000 times.

SEM-imaging requires that the surfaces that are studied have to be electrically conductive. This poses a problem with paperboard as samples have to be treated in order for them to conduct electricity. Most common surface treatment for SEM imaging is gold plating. SEM-imaging also requires nearly vacuum conditions, that may affect more delicate samples and it also consumes large amounts of power. [15]

3.3.2 X-RAY MICROTOMOGRAPHY

X-ray microtomography is an adaptation of the tomography method, which is widely used in medical science. It is based on absorption of x-rays within the object that is being imaged. A typical microtomography device has a revolving x-ray source that is opposed by x-ray detectors, that measures the intensity of the radiation passing through the studied object. Process yields a series of projection images that can then be reconstructed into a complete 3D-model of the object of interest by using computer algorithms. [16]

The material preparation was done by cutting a small sample (Figure 4) and attaching it to a stand. The size of the sample is small and only one or two creases can be analyzed at a time.

Figure 4. Prepared sample for X-ray microtomography. Scale numbering is in centimenters.

Figure 5. Prepared samples before (left) and after (right) polishing. The diameter of the samples is 30 mm. One sample contains about 10 creases depending on tray geometry.

3.3.3 OPTICAL LIGHT MICROSCOPY

Light microscopy is the oldest tool in studying microstructure of materials. However, paperboard samples are difficult to study under a traditional microscope without proper sample preparation. A technology used normally to study metallurgical samples was applied into paperboard analysis.

In the method a cross-sectional paperboard sample is cut and then installed into a silicon mold. The mold is then filled with clear acrylic resin. After the resin hardens, the acrylic resin cast sample is removed from the mold. The cast sample is then polished with fine grit sandpapers and aluminum oxide solution, in a same manner as metallurgical samples are polished.

The finished sample (Figure 5) is studied and with a standard metallurgical microscope. The microscope is equipped with a digital camera that allows capturing images from the samples.

3.3.4 POLARIZED LIGHT MICROSCOPY

Polarized light microscopy is a technique involving polarized light. It can be done utilizing a number of optical microscopy methods. Polarized light microscopy can provide information on absorption color and optical path boundaries between different structures of materials. It can be used to identify different materials that are otherwise impossible to separate such as the layers in polymer films. [17]

4 RESULTS AND DISCUSSION

Extensive studies were conducted in order to compare different imaging systems for paperboard samples. Cross-sectional imaging methods were tested in general forming studies and in leak analysis. All four tested methods delivered clear images. However there were large differences in the usability of different methods.

4.1 SCANNING ELECTRON MICROSCOPY (SEM)

SEM images (example in Figure 6) offer great detail and show the different layers of materials clearly. It has far greater magnification and detail than other tested methods. The magnification of SEM allows investigating the formation in individual fibers of the paperboard. However, when analyzing the formation of a single crease and the sealing of the lidding film this kind of accuracy and magnification level is not required. Also, high cost of equipment, sample preparation and required gold plating of samples are drawbacks of using SEM.

4.2 X-RAY MICROTOMOGRAPHY

While X-ray microtomography offers insightful information of a single crease and it’s deformation through the sealing surface, the lidding film is not clearly visible in the images (example in Figure 7). Lack of visibility of the sealing film makes it impossible to use X-ray microtomography in leak detection and analysis. The high equipment cost and challenging sample preparation are also significant shortcomings of microtomography.

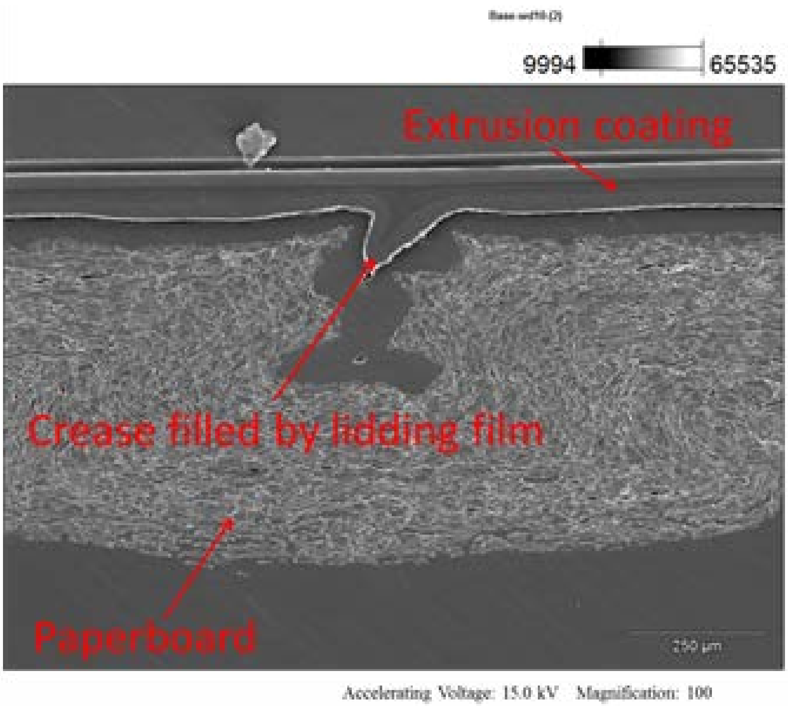

Figure 6. A SEM image

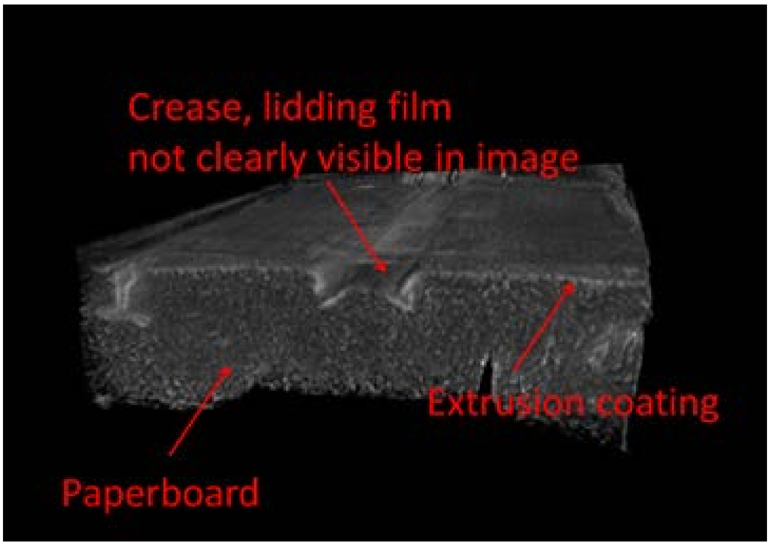

Figure 7. An X-ray microtomography image

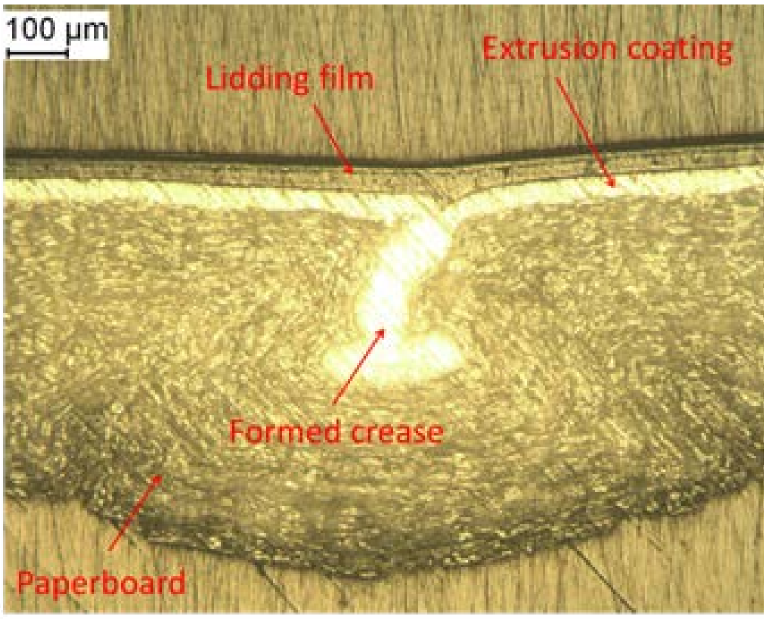

Figure 8. A microscopic image of a tight seal taken with an optical light microscope

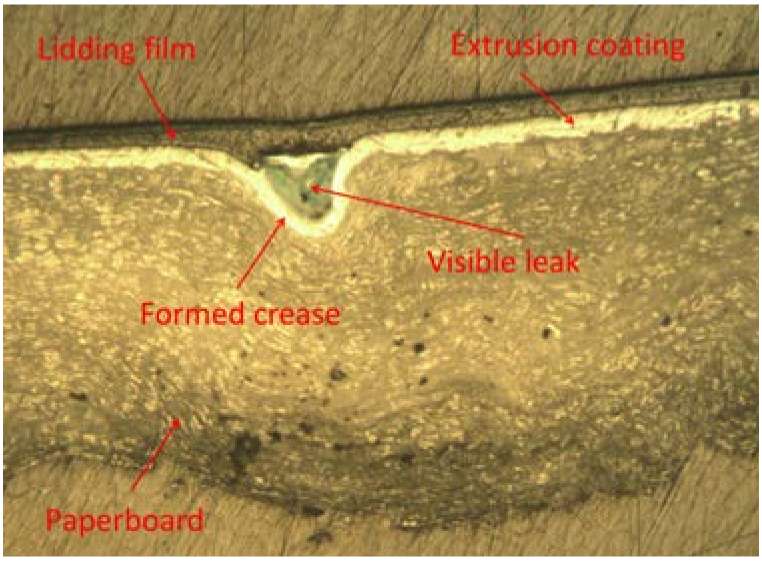

Figure 9. A microscopic image of a leaking seal taken with an optical light microscope. The coloring solution and a leaking crease is clearly visible

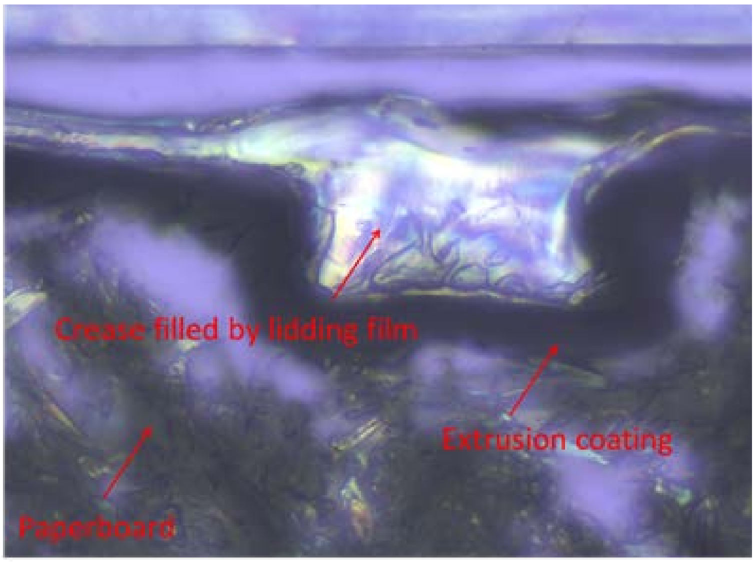

Figure 10. A microscopic image of a tight seal taken with a polarized light microscope

4.3 OPTICAL LIGHT MICROSCOPY

The images (Figures 8 and 9) taken with an optical light microscope show clearly the structure of the different material layers and the formation of the crease. Figure 8 shows a tight seal in which the lidding plastic is properly bonded to the extrusion coating.

Figure 9 shows a leaking seal which has been detected by the coloring solution. The leak spot is easy to spot and investigate the cause of the leak when the coloring solution is visible in the images.

4.4 POLARIZED LIGHT MICROSCOPY

Polarized light microscopy shows clearly the different layers in materials. A sample of an image is in Figure 10. The different layers are recognizable but may be harder to understand than the pictures taken with a white light microscope.

5 CONCLUSIONS

Casting of the samples in an acrylic resin and light microscope imaging was found to be the most suitable method for this kind of analysis. The formation of creases, lidding material and leaks are easy to recognize from the images. Light microscopy is also the fastest and most affordable solution of when general material behavior is studied, as it also allows wider sample areas in a single sample, when compared to for example micro tomography.

All tested systems can be used in the leak analysis, but micro tomography, polarized light microscopy and SEM require very precise sample preparation when an individual crease indicated by coloring solution is studied. Light microscopy is also the only method that allows visual confirmation of leaks by showing the discoloration caused by the coloring solution. This is an important feature because leaks under the surface of the tray's rim area are not always visible in visual inspection. These small leaks, which can cause the modified atmosphere to be compromised, can be detected only in the microscopic images.

The use of microscopic imaging in the analysis of paperboard trays enables deeper understanding of material behavior of polymer coated paperboard in the press-forming process. This kind of information is crucial when new applications for environmentally friendly fiber-based packaging solutions are desired. Structural analysis of a paperboard tray can be done with all of the tested methods. However, when large amounts of samples are to be studied, optical light microscopy is the most affordable and efficient method. In addition, if leak detection by coloring solution and understanding of leak mechanics are to be studied, optical light microscopy is the only practical solution.

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Publication V

Leminen, V., Mäkelä, P., Tanninen, P. and Varis, J.
The use of chromatic white light 3D-profilometry in the surface quality analysis of press-formed paperboard trays

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Proceedings of the 25th Flexible Automation and Intelligent Manufacturing, FAIM2015
Vol. II, pp. 74-81, 2015

The use of chromatic white light 3D-profilometry in the surface quality analysis of press-formed paperboard trays

Ville Leminen, Petri Mäkelä, Panu Tanninen and Juha Varis

Department of Mechanical Engineering
Lappeenranta University of Technology
Lappeenranta, FI-53850, Finland

ABSTRACT

Press-forming of paperboard is a process similar to deep-drawing of sheet metal. When press-formed paperboard trays are used in food packaging, a gas-tight sealing result is often required. A point of interest regarding gas-tight heat sealing is the surface quality of the sealing area of the package. The objective of this study was to assess the feasibility and applicability of using chromatic white light 3D-profilometry to investigate the surface quality of press-formed polymer-coated paperboard trays, and additionally to investigate the correlation between surface quality and gas tightness of the seal. In the experiments, the blank holding force in the press-forming was varied, and the effect of this force on the surface quality of the end product was researched. After reviewing the results given by the measuring system, four parameters were selected for further analysis. The selected surface quality parameters were compared to tightness test results to investigate possible correlations between the parameters and the tightness of the trays when a lid is sealed to the tray. Peak height (Rp) values were seen to be a useful indicator of the surface quality and sealability of paperboard trays, and the surface quality value that enabled gas-tight heat-sealing of the tested samples was found to be Rp (max) < 45. This kind of surface quality analysis can be used to determine if manufactured trays can be sealed tightly, before expensive sealing tools are manufactured. Consequently, it is particularly useful when new products are being developed and new geometries tested.

1. INTRODUCTION

Three dimensional forming and material behavior of paperboard in the press forming process has been previously studied by several authors. [1] - [7] However, tray shaped, paperboard based and polymer coated packages have not yet been able to become a significant competitor to polymer based packages in food packaging when the use of modified atmosphere packaging (MAP) is required. This is mainly because the tightness of the packages especially in the area where the lid is heat sealed to the package has been a problem. A major cause for this are creases in the sealing area that are done to control and enable the formation of paperboard to certain geometries. [7], [8] Poor surface quality of the rim area of the tray may cause leaks in the package when a lid is heat sealed to the tray. A poor sealing result can be a critical factor affecting shelf life of food. [9] Faults in sealing surface or other faults such as cracks or pinholes in the package can cause the MAP to leak from the package. Leakage of MAP can cause reduction in sensory shelf life and microbiological quality of packed foods [10].

The objective of this study was to assess the feasibility and applicability of using chromatic white light 3D-profilometry to investigate the surface quality of press-formed polymer-coated paperboard trays, and additionally to investigate the correlation between surface quality and gas tightness of the seal.

2. MATERIALS AND METHODS

2.1. MATERIALS

The material used in the trays was Stora Enso Trayforma Performance 350 + 40 WPET which is a polyethylene terephthalate (PET) extrusion-coated paperboard with a base material grammage of 350 g/m^2 and a coating grammage of 40 g/m^2. The base board consists of three solid bleached sulphate (SBS) layers. [11]

2.2. Press-Forming of Paperboard Trays

Press-forming is a process similar to deep-drawing of sheet metal. It is used to create three-dimensional shapes such as plates or trays. The tray forming process is presented in Figure 1.

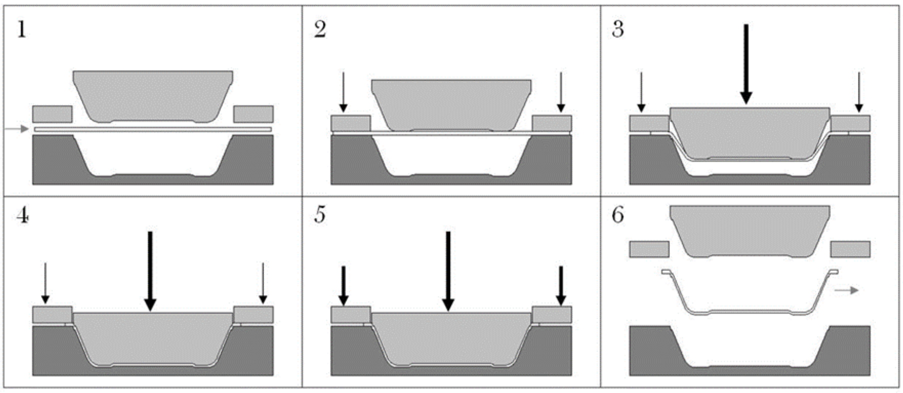


Figure 1: Press-forming process

Phase 1: The paperboard blank is positioned between the moulding tools.

Phase 2: The blank holding force tightens the blank between the rim tool and the female tool.

Phase 3: The male tool presses the blank into the mould cavity in the female tool. Folding of the tray corners is controlled with blank holding force.

Phase 4: The male tool is held at the bottom end of the stroke for a set time (0.5 to 1.0 s). The plastic coating softens, and creases in the corners of the tray are sealed together.

Phase 5: The flange of the tray is flattened by the rim tool.

Phase 6: The formed tray is removed, and a new blank can be fed into the tray press. The tray achieves its final rigidity when it cools down.

Sample trays (example of tray geometry in Figure 2) were manufactured at the Laboratory of Packaging Technology at Lappeenranta University of Technology (LUT). The tray dimensions were 319x216x38 mm.

Test trays were made with four separate blank holding forces, which result in different surface qualities on the sealing surface of the tray. The forming parameters are presented in Table 1.

Table 1: Forming parameters for the trays

Female tool temperature	**Pressing dwell time**	**Pressing force**	**Pressing speed**	**Blank holding force**
170 °C	1 s	135 kN	130 mm/s	1.16 kN
170 °C	1 s	135 kN	130 mm/s	0.77 kN
170 °C	1 s	135 kN	130 mm/s	0.68 kN
170 °C	1 s	135 kN	130 mm/s	0.58 kN

2.3. *CHROMATIC WHITE LIGHT 3D-PROFILOMETRY*

The tray rim area surface analysis was conducted using chromatic white light 3D-profilometer to study sealing surfaces of paperboard trays. Fourteen paperboard trays were analyzed with an FRT MicroProf-profilometer. Chromatic white light 3D-profilometer uses a broad spectrum light that is directed to a test sample through a special lense, which focuses different wavelengths of light to different distances. Returning light is passed on to a spectrometer that separates different intensities. Topography of the test sample can be obtained from the spectrally encoded intensity signal. [12] Typical white light profilometer systems can operate accurately in a very limited measuring range, usually less than 0.5mm. [13] As measuring range is increased the resolution of the topography is degraded. [13], [14]

The system scans the desired surfaces and calculates 17 different roughness statistics and 17 waviness statistics. The different calculated statistics are shown at Table 2.

Table 2: Example of the roughness data produced by the 3D-profilometer, parameters selected for further analysis highlighted with bold text

Roughness average	**Waviness**	**Roughness**
sRa	sWa	**Ra**
sRq	sWq	Rq
sRz(DIN)	sWz(DIN)	Rz(ISO):
sRmax	sWmax	**Rz(DIN):**
sRz25	sWz25	Rmax
sRmax25	sWmax25	**Rp**
sRp	sWp	**Rv**
sRv	sWv	Rt
sRt	sWt	Rsk
sRsk	sWsk	Rku
sRku	sWku	RPc
sRk	sWk	Rk
sRp	sWpk	Rpk
sRvk	sWvk	Rvk

After reviewing the 34 parameters that the system produces four of them were selected for further analysis. Selected parameters were roughness average (Ra), peak height (Rp), average peak to valley (Rz(DIN)) and lowest valley (Rv). Parameter selection was based on the physical characteristics and the universal use of the selected parameters. Ra and Rz(DIN) are widely used surface roughness parameters [14], Rv and Rp were selected because leaks in a sealed paperboard tray often occur in deeper wrinkles that are not filled by heat-sealed lidding film and the mean level which peak and valleys are compared to could not be established before measurements.

3. RESULTS AND ANALYSIS

3D-profilometry results were provided in three formats. Results were in numerical from, in 2D intensity diagram and in 3D solids. 2D and 3D visual representations show the surface in detail and it is relatively easy to distinguish different surface qualities produced with visual inspection, as figure 2 shows. Visual identification of the surface quality is however, not useful in order to determinate the sealability of the trays for quality control or research purposes.

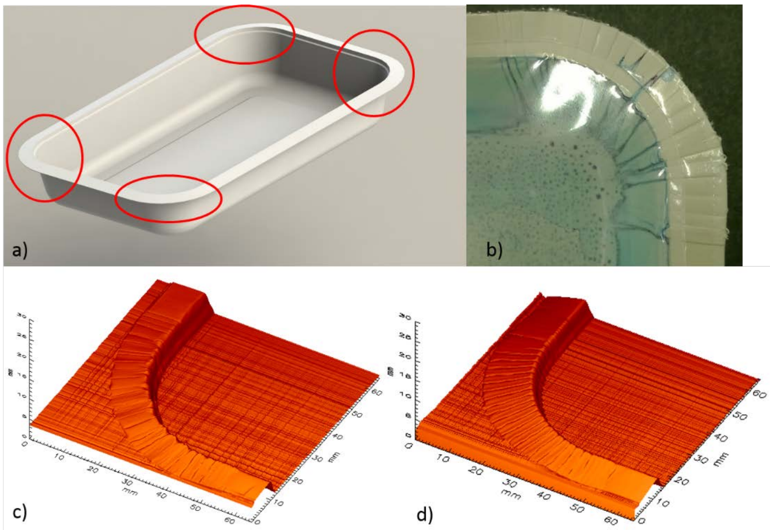


Figure 2: a) Used tray geometry with location of creased corners higlighted in red b) corner formed with a blank holding force of 0.58 kN and a leak indicated with a penetrant liquid c) 3D-presentation of a tray corner formed with a blank holding force of 0.58 kN and d) 1.16 kN

The measurements were taken from three locations of tray corners according to Figure 3 (vertical, 45 degree and horizontal). After that the numerical data of these measurements was compared to tightness results of sealed packages from a previous study [15] shown in Table 3.

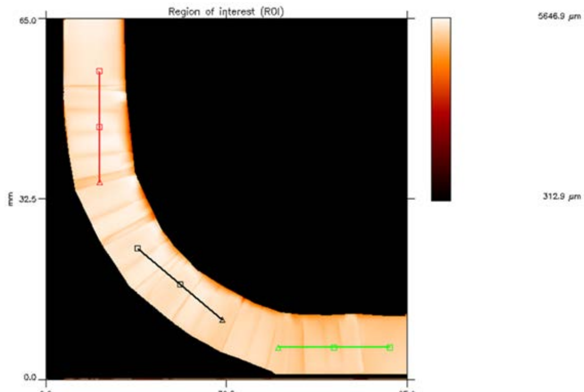


Figure 3: Measurement areas of surface roughness parameters (the red, black and green lines)

Table 3: Comparison of leakage and blank holding force

Blank holding force	Leaks shown by penetrant testing	Leaks shown by gas analysis
0.58 kN	Yes	Yes
0.68 kN	No	Yes
0.77 kN	No	No
1.16 kN	No	No

The measured values were grouped so that those taken in vertical and horizontal direction were taken into account and the ones taken in 45 degree direction were discarded. This was done because the location of measurements in 45 degree direction was not consistent enough for reliable comparison.

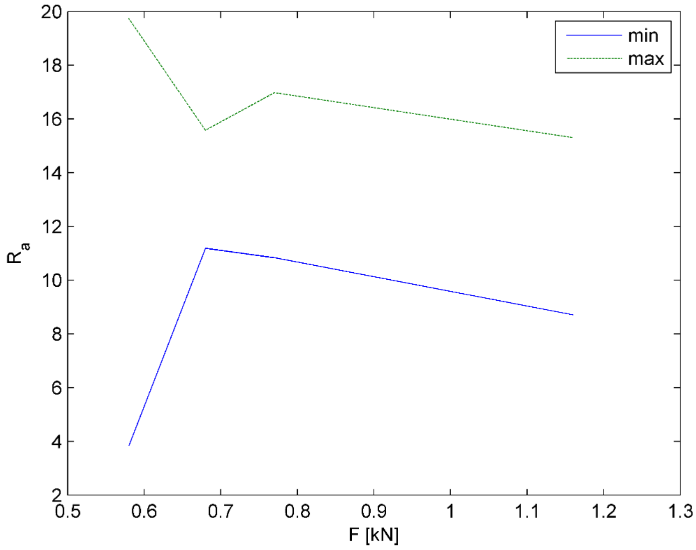
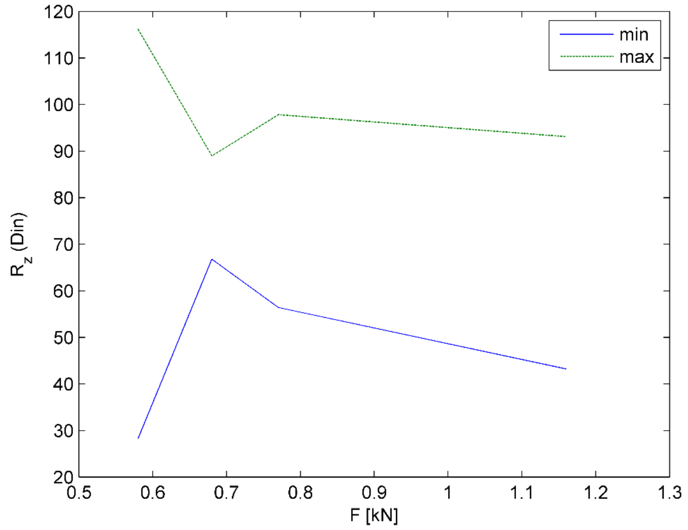


Figure 4: Ra values with different blank holding forces measured by the 3D-profilometer



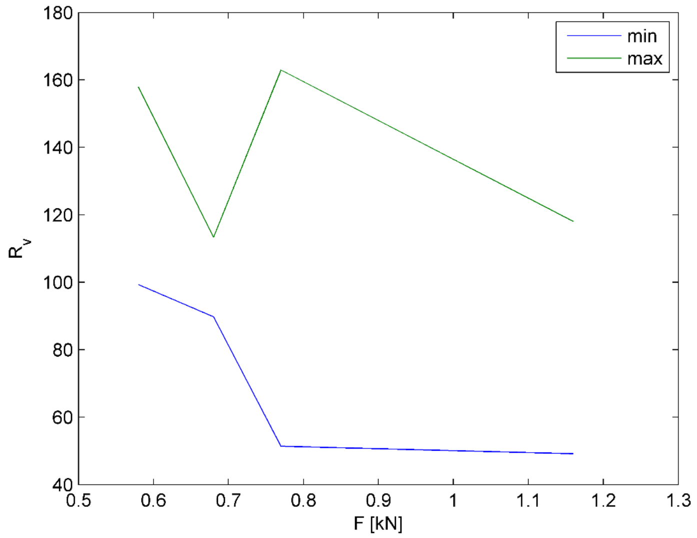


Figure 5: R_z (Din) values with different blank holding forces measured by the 3D-profilometer

Figure 6: R_v values with different blank holding forces measured by the 3D-profilometer

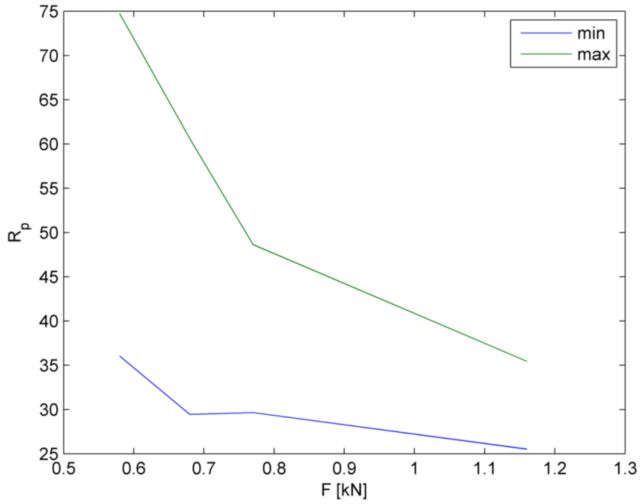


Figure 7: Rp values with different blank holding forces measured by the 3D-profilometer

It was found that the Rp (min) and Rp (max) values showed a strong correlation between surface quality and the blank holding force. Figure 7 shows how surface quality deteriorates as the blank holding force is reduced. No such correlation was found with Ra, Rv and Rz(DIN) values. [13] describes how the white light 3D-profilometer has difficulties in measuring very narrow and deep grooves. When compared to the data acquired from this research to [13] there is a strong similarity between the grooves. Ra, Rv and Rz(DIN) values may be distorted because the full surface details are not represented in the data used to calculate the said values.

Results obtained from the Rp values were compared to the tightness tests (Table 3) which showed leakage between a blank holding force of 0.58 kN and 0.68 kN while blank holding forces above 0.77 kN showed no leakage. These results indicate that the Rp value of press-formed packages should be below 45 to achieve good sealing results.

4. Conclusions

The surface roughness values measured using chromatic white light 3D-profilometry are not possible to achieve from paperboard trays using traditional touch-based measurement devices due to the nature of material hardness of paperboard and polymer-materials used in paperboard coating. The Rp values were shown to be a useful indicator in order to determinate surface quality and sealability of paperboard trays. When the other parameters (Ra, Rz (Din) and Rv) are compared to Rp, the main difference is that the Rp parameter discards all data below the mean surface line.

Rp (max) value seems to be a good representation of the potential to achieve a gas tight seal when a polymer based lidding film is heat sealed to the paperboard tray. For example with the materials and geometry used in this study it seems that the upper limit of Rp (max) must be below 45 if trays are to be used in applications that require tightness of seals because of the use of modified atmosphere.

Main application of surface quality analysis is to determinate if a press-formed tray can be sealed reliably, before expensive sealing tools are ordered. The manufacturing costs of sealing tools can vary from around 5.000 € to 20.000 €. This can mean substantial savings when the cost of 3D-imaging of manufactured trays is only 50 to 500 €. This is particularly useful when new products are developed and new geometries are tested. The main drawback of the system used in these measurements is the time required to study a single sample, which can be up to 45 minutes. However this time is not critical in offline analysis during product development.

To verify the results in the future and to continue research, additional research should be conducted with different materials and tray geometries. Also testing other available surface profilometry methods, including those suitable for online quality control would be beneficial.

ACKNOWLEDGEMENTS

The authors would like to thank Juho Ratava for his help with the graphing the data.

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Publication VI

Tanninen, P., Leminen, V., Kainusalmi, M. and Varis, J.
Effect of Process Parameter Variation on the Dimensions of Paperboard Trays

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Bioresources
Vol. 11, No. 1 pp. 140-158, 2015

Effect of Process Parameter Variation on the Dimensions of Press-Formed Paperboard Trays

Panu Tanninen,* Ville Leminen, Mika Kainusalmi, and Juha Varis

The dimensional accuracy of packages has a great effect on operation of the production and supply chain. In this research, the dimensional accuracy of trays made of polymer-coated paperboard and the effect of all essential press forming process parameters on outer dimensions of the trays were studied to obtain data for the press forming and lid sealing process optimization and for the forming tool design. Paperboard trays were analysed and measured with a quality monitoring system that includes a smart camera and a backlit table. Trays with varying dimensions were sealed to investigate the effect of the package size and the product weight to the residual oxygen in the package's headspace gas. Results showed that all heat related parameters, *i.e.*, mould temperatures, dwell time, and pressing speed can be used to adjust the outer dimensions of the paperboard tray. Lid sealing process was found to reduce size of the trays and even out size differences substantially. All produced trays were measured to be bigger both in length and in width compared to the design values of the mould set. Therefore the mould set has to be designed undersized to obtain trays with certain outer dimensions.

Keywords: Paperboard; Press forming; Package dimensions; Tray; Modified Atmosphere Packaging; Pattern recognition

*Contact information: Department of Mechanical Engineering, Lappeenranta University of Technology, P.O. Box 20, FI-53851 Finland; *Corresponding author: panu.tanninen@lut.fi*

INTRODUCTION

Dimensional accuracy of packages is a prerequisite for operation of the production and supply chain. Package size affects operation of filling and lidding machinery, stacking of packages in transport boxes, and fluency of retail operations. The environmental perspective on packages emphasizes the use of non-oil-based materials, although plastic is the most versatile material for producing three dimensional (3D) packages and providing good barrier properties. From a sustainability and printing point of view, fiber-based materials are fascinating raw materials for the modern packaging industry.

With polymer-based packages, thermoforming is a commonly used method for manufacturing. In these machines, sealing is often in-line with package forming. The formed packages are attached to the polymer web and thus correctly positioned during heat sealing of lidding material to the trays. After this, the packages are cut in cross-directional and longitudinal directions. This results in dimensionally uniform packages. However, adjustment of outer dimensions during the production of paperboard trays is a more complex task compared to thermoformed plastic packages, which are cut to the desired outer dimensions after forming. In press forming of paperboard trays, the length and the width of the tray can be altered by changing the blank size or by adjusting the forming process parameters.

Regarding consumer packages, a change in policy might generate the highest impact in the food sector. To utilize the benefits of both plastic and paper materials, composite structures are frequently used to reduce the amount of plastic needed and to provide the needed barrier properties for the packages. In this research, dimensional accuracy of trays made of polymer extrusion-coated paperboard and the effect of press forming process parameters on outer dimensions of the trays was studied to obtain essential data for the process optimization and the forming tool design.

Heat sealing is a process commonly used for sealing of packaging materials. Rigid packages such as trays made from polymer based materials are widely used in food applications. The process requires the sealed trays to be dimensionally accurate. It is assumed that if a tray's dimensions are too small, the tray rim area is not positioned correctly and leaks can occur. On the other hand, if a tray is too large it will not necessarily be positioned correctly in the sealing process. This can result in insufficient results such as leaks or residual oxygen in the headspace of the package when a modified atmosphere is used.

The conversion of a fiber-based material into a 3D package is a complex process. Thus, there is an urgent need to understand the means by which the forming of 3D packages from fiber-based materials can be improved. Both 3D-forming and the material behavior of paperboard during press forming have been previously studied by several authors. These articles describe the effect of process parameters, adjustability, and tooling technology (Hauptmann *et al.* 2014; Tanninen *et al.* 2015) functionality of materials in forming (Vishtal and Retulainen 2012; Zeng *et al.* 2013) and also the leakproof heat sealing of trays (Hauptmann *et al.* 2013; Leminen *et al.* 2015a).

EXPERIMENTAL

Materials

The substrate used in the die cutting tests and tray pressing was Stora Enso (Finland) Trayforma Performance 350 + 40 WPET, a polyethylene terephthalate (PET) extrusion-coated paperboard with a base material grammage of 350 g/m^2 and a coating grammage of 40 g/m^2. The base board consists of three solid bleached sulfate (SBS) layers, and the total thickness of the substrate is 460 µm. The main component of the substrate is hardwood fiber with alkyl ketene dimer (AKD) hydrophobic sizing additive. The materials were stored in a humidity-controlled chamber at 80% relative humidity to maintain the delivery moisture content of the paperboard. The moisture content was verified before converting tests were performed with an Adams Equipment PMB 53 Moisture Analyzer. The measured moisture content of the material was 9.1%.

Methods

The trays for the experiments were manufactured by using the press forming process. The quality monitoring and dimensional analysis were done using a quality monitoring system of the LUT Packaging Line, which is based on a smart camera, and the trays were sealed by using the heat sealing process.

Press forming process

The basic principle of the press forming of trays is to place a pre-cut and creased, possibly polymer coated, paperboard blank between molds that are pressed together to form a tray of a desired shape, as shown in Fig. 1. The blank is die cut so that the longer side of the blank is parallel to the MD (machine direction) of the paperboard to give ready-made trays more rigidity. The folding of the tray corners is controlled with the blank holding force (max. 5 kN, values in parenthesis apply to the test equipment used in this study), applied by a rim tool. The male mould is held at the bottom end of the stroke for a set time (dwell time) while the polymer coating softens, and creases in the corners of the tray are sealed together. Simultaneously the flange of the tray is flattened by the larger force (max. 150 kN) also applied by the rim tool. Finally the formed tray is removed, and a new blank can be fed into the tray press. The ready-made tray achieves its final rigidity when it cools down. The female mould is heated (max 200 ºC), while the male mould is kept at lower temperature (max. 50 ºC). Greater heat is applied only to the uncoated side of the material to prevent melting of the polymer coating. Pressing speed (max. 200 mm/sec) is the velocity of the male mould until it is stopped by the female mould. All tray manufacturing tests were carried out using the Adjustable Packaging Line developed by the Lappeenranta University of Technology for research work related to packaging and packaging material development (ERDF 2008).

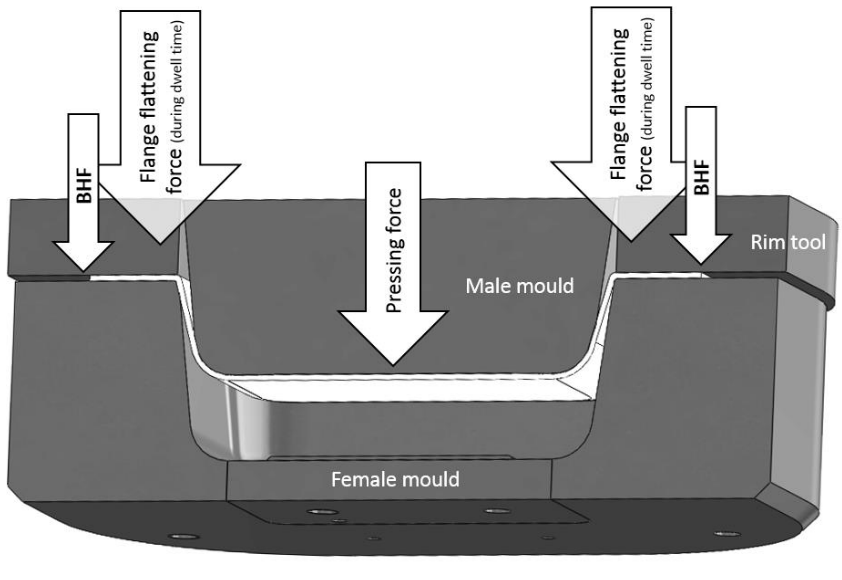

Fig 1. Press forming of paperboard trays. BHF denotes the blank holding force.

The test tray

Gastronorm sizes are standard sizes of containers used in the catering industry specified in the EN 631 standards (SFS-EN 2006). The mould set used in tray production is designed according to the GN1/4-standard size: 265 × 162 x 38 mm. Filled trays are typically transported in boxes with a size of 600 x 400 x 255 mm. Boxes are designed to fit standardized flat pallets used throughout supply chains (ISO 6780:2003). Preliminary stacking tests with the paperboard trays indicated that the maximum tray size that is suitable in the transport boxes is 272 x 168.5 x 38 mm, for 24 (2x2x6) trays in one transport box.

The quality monitoring system of LUT Packaging Line

Ready-made paperboard trays were analysed with the quality monitoring system which is part of the LUT Packaging line. The system transfers inverted trays with a manipulator on a backlit table after press forming phase. The background light brings out defects from the trays and enables accurate measuring of dimensions. Each tray was photographed with the Cognex IS5605-11 smart camera 20 seconds after press forming. In that period of time trays cooled down to the room temperature and settled in the final outer dimensions. Temperature of the cooling room was 23 ºC and humidity 50% RH. Images were taken 650 mm above the tray bottom to prevent image distortion. The smart camera takes black and white images with a resolution of 2456 x 2048. A single pixel is equivalent to 0.017 mm x 0.017 mm area on the monitored surface.

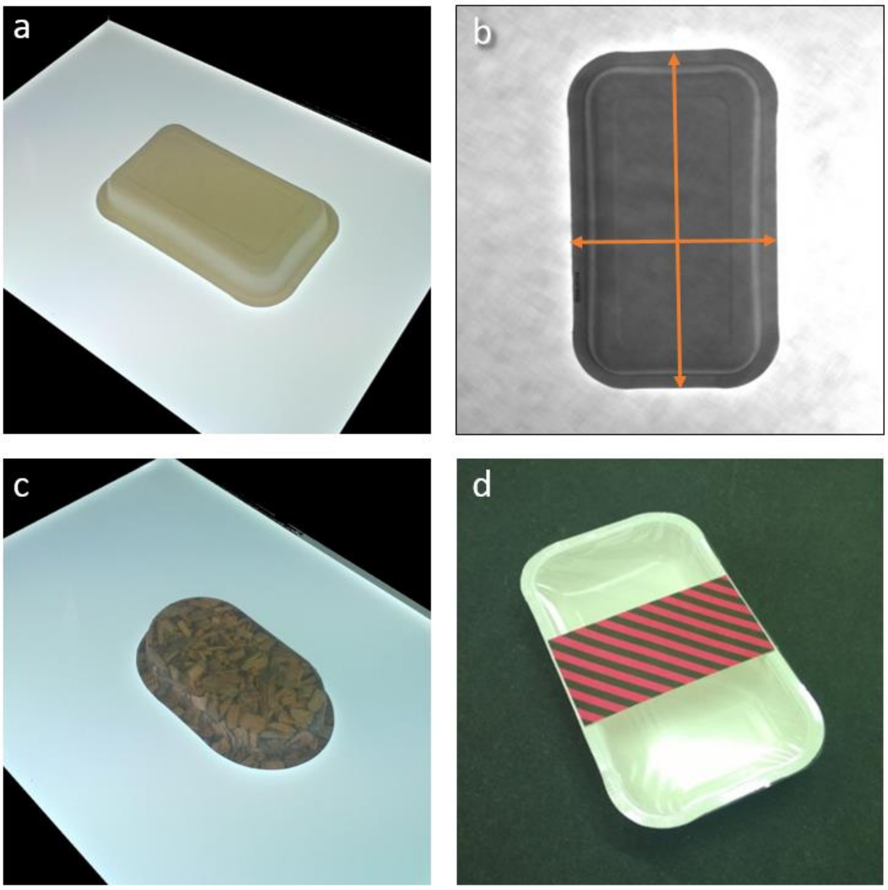

Fig 2. (a) Test tray on the backlit table of the monitoring system, (b) Image of the test tray taken with the smart camera and outer dimensions measured with the pattern recognition, (c) Printed oval shaped tray on the backlit table and (d) Sealed tray (4 mm too wide) with the labeled lid film.

The vision software recognizes patterns and automatically calculates dimensions of the measured object. Accuracy of the monitoring method was tested by measuring the same sample ten times using slightly different orientations. Standard deviation of the measurements was 0.05 mm. Trays were measured also under a compression load caused by 4 kg weight placed on a bottom of the inverted tray. An example of an image processed by the monitoring system can be seen in Fig. 2b.

In this study, only the outer dimensions of the trays were measured, but the monitoring system is suitable for more versatile analysis. Pattern recognition can be used to detect ruptures, fractures, and wrinkles in the paperboard surface. Wrinkles and the smallest ruptures require a light source in the foreground to enable detection. In industrial press forming, this is a poorly applicable method since multicolored and complex images are usually printed on the outer surface of the tray. This makes pattern recognition extremely demanding (Fig. 2c). Therefore, detection methods should utilize mainly background lightning. Bigger fractures can be detected in visible light through the opening and smaller fractures in the tray walls in the contour shape of the tray.

Lid sealing of trays

The press-formed trays were heat-sealed using an industrial scale sealing machine (Ilpra 2014). Sealing parameters were sealing time 2.5 s, sealing temperature 190 °C, and sealing pressure 6 bar, which resulted in a surface pressure of about 2.7 N / mm^2. Trays with varying dimensions were sealed to investigate the effect of the package size and the product mass to the residual oxygen in the package's headspace gas. Three different set of weights were used to simulate packed products: 25 g, for light products such as snacks, 200 g, and 400 g, for other, heavier foods.

The sealed trays were flushed with a commonly used gas for food applications consisting of 70 % N_2 and 30 % CO_2. One minute after sealing the oxygen content in the packages was analyzed using a Mocon Optech O_2 platinum analyser, which utilizes the standard ASTM F-2714-08 ("Standard Test Method for Oxygen Headspace Analysis of Packages using Fluorescent Decay"). The analysis was done to investigate the amount of residual oxygen in packages with different dimensions.

RESULTS AND DISCUSSION

The effect of all essential press forming process parameters on tray dimensions was studied with a series of tests. All presented values are the average of five measured sample trays. The variance of measured dimension values was at the same level for all tests in which only one process parameter was changed. The standard deviation was 0.25 to 0.35 mm. The quality of the trays was evaluated according to method presented in a previous study (Tanninen *et al.* 2014a). Trays with good quality have smooth sealing area in the tray flange, creases in the tray corners are folded evenly, and there are no fractures, wrinkles, or other defects in the tray walls. When press forming production is within the process window, wrinkles and fractures are not probable defects. Dimensional inaccuracy and wear in the print surface are more likely to cause actions. Mould length and width of the tray, which are marked in the following figures, represent the dimensions that the tray has when the forming moulds are pressed against each other.

After the ready-made tray is released from the forming unit, a varying amount of spring-back occurs during the cool down and outer dimensions of the tray increase.

Residual stress within the substrate causes spring-back when the tray is not clamped between moulds any more. Spring-back is a well-known phenomenon in various forming processes that use wood-based materials.

Also, several studies indicate that spring-back is a heat-dependent phenomena. Östlund *et al.* (2011) states that spring-back is the shape change associated with re-establishing equilibrium after external forces are removed. Results of the study show that specimens formed, with a laboratory apparatus for forming paper sheets into double-curved structures, at room temperature and without added moisture exhibited some spring-back, whereas heat, or moisture and then heat, limited the spring-back sufficiently. This may be indicative of a small elastic region of deformation for the paperboard under such conditions, or relaxation-enhancing properties of the heat and/or moisture. The effect of temperature on the spring-back of cellulose-based sheets in hot pressing has also been studied (Golzar and Ghaderi 2009). The results indicated that increasing the process temperature reduces the spring-back of the part. Shape accuracy in deep drawing of paperboard was studied by Hauptmann and Majschak (2011). Spring-back angle was found to have been reduced significantly with increase of temperature sum (the temperature sum of the die and the shaping cavity).

Each process parameter was changed separately to study their effect on the tray dimensions. Other parameters were kept constant in the following set values: male mould temperature 50 ºC, female mould temperature 160 ºC, blank holding force 1.6 kN, pressing force 120 kN, dwell time 600 ms, and pressing speed 150 mm/s.

Previous studies (Leminen *et al.* 2013; Tanninen *et al.* 2014b, Leminen *et al.* 2015b) indicate that blank holding force is one of the most important process parameters in press forming. However, it has only a minor effect on outer dimensions of the tray, as can be seen in Fig. 3. Blank holding forces under 1.2 kN caused wrinkles in the tray walls, making them visually unacceptable, and forces above 2.3 kN caused ruptures in the tray corners.

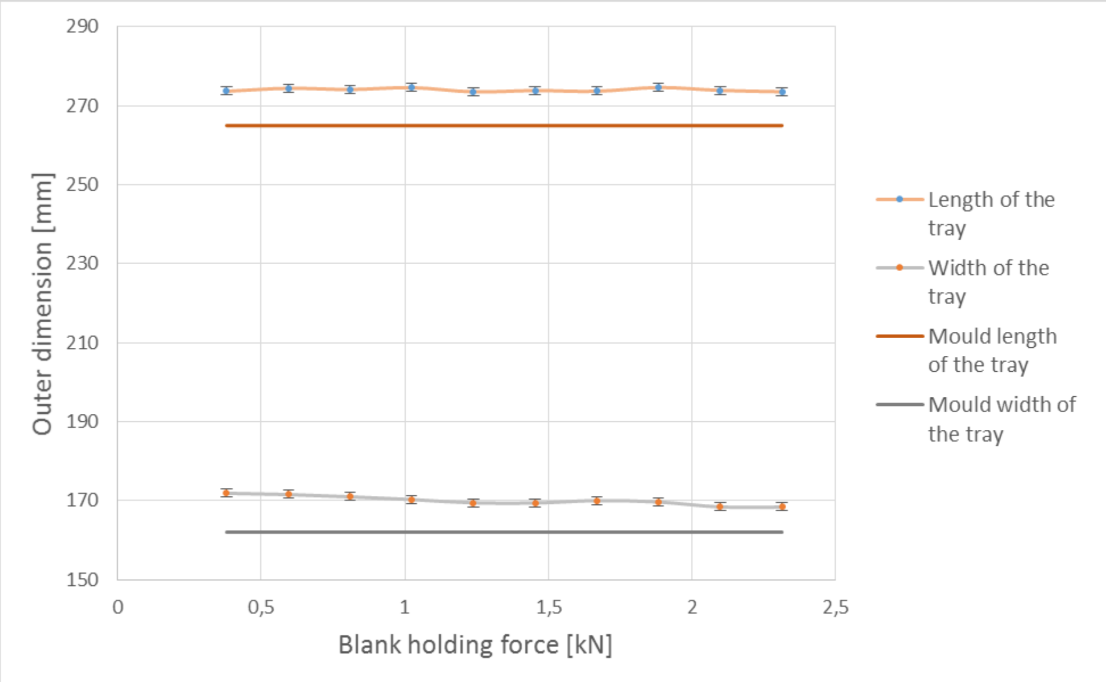

Fig 3. The effect of blank holding force on the outer dimensions

When different substrates are evaluated, *e.g.*, in production start-up, this force where rupturing starts and/or length of ruptures effectively describe formability of materials when otherwise same process parameters are used (Tanninen *et al*. 2014b). The press forming process is usually optimized with quality of folding and flatness of the tray flange in mind which limits the adjustment range of blank holding force significantly. Therefore, it cannot be used to adjust tray dimensions.

Pressing force also had only a minor effect on tray dimensions. Most of the force is applicable only in the flange flattening stage and the amount of the force causing the folding and sliding of the blank can be expected to be invariant. This is due to the fact that the pressing force, which is applied by the male mould, cannot increase until the male mould and the female mould meet each other at the end of the stroke. Clearance between the male mould and the female mould is selected on the basis of the substrate thickness, resulting in the even distribution of the pressing force to the wall and bottom surfaces of the tray. The use of greater pressing force produces slightly smaller trays, as is presented in Fig. 4. Tray quality was insufficient, especially smoothness of the tray flange, when pressing force was under 120 kN, which is why this parameter cannot be used to adjust tray dimensions either.

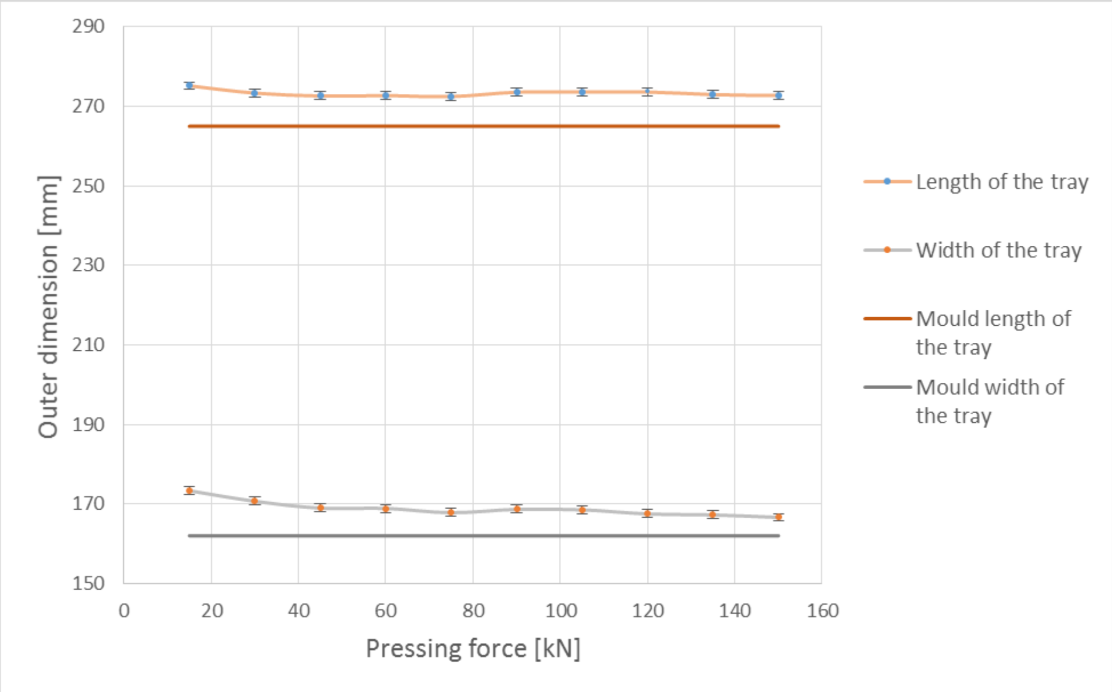

Fig. 4. The effect of pressing force on the outer dimensions

Temperature of the female mould had a significant effect on tray dimensions, as is presented in Fig. 5. The length of the tray was reduced 8.0 mm and the width 10.7 mm when the mould was heated from 22 ºC to 180 ºC. Mould temperatures above 180 ºC caused bubbling of the polymer coating layer and sticking between coating layer and the steel surface of the mould. Tray quality was sufficient in the mould temperature range from 140 ºC to 180 ºC, where the length of the tray changed 3.4 mm and width 6.8 mm. Dimensional change was significantly greater in the cross direction (CD) than in the machine direction (MD).

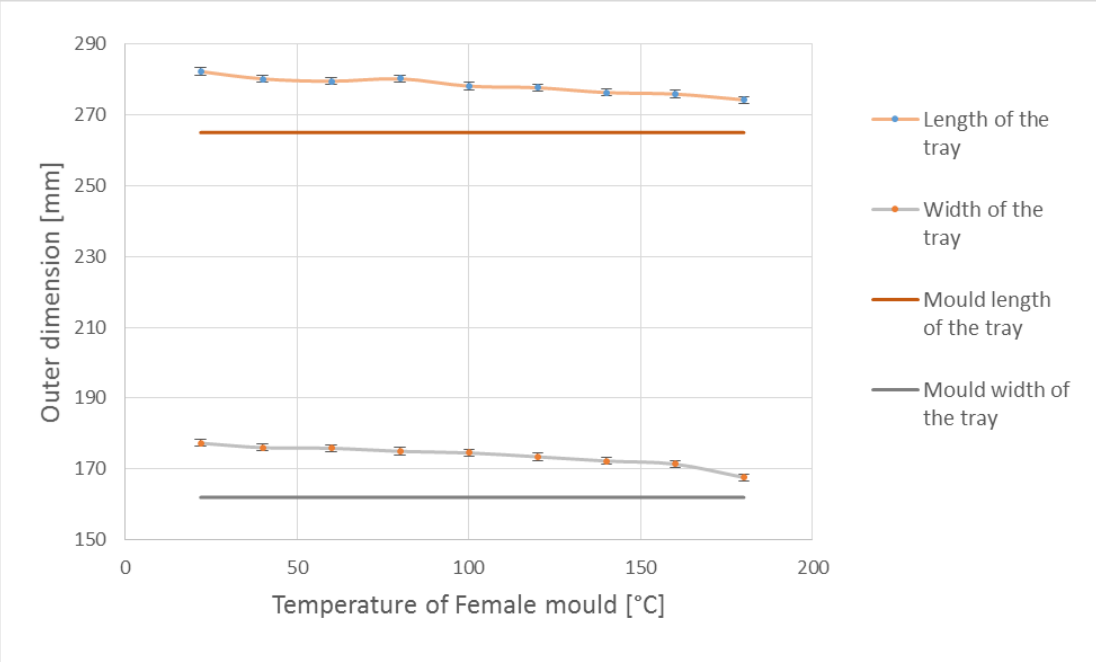

Fig. 5. The effect of female mould temperature on the outer dimensions

The amount of heat transmitted to the tray depends on dwell time along with the mould temperatures. Increase of the dwell time has an effect on the temperature reached in the bulk of the complex, making it higher. This affects the water evaporation also. The use of longer dwell time results in smaller trays. Dwell time longer than 600 ms caused bubbling of the polymer coating layer with the mould temperature of 180 ºC. The whole dwell time range could be used with the female mould temperature of 160 ºC.

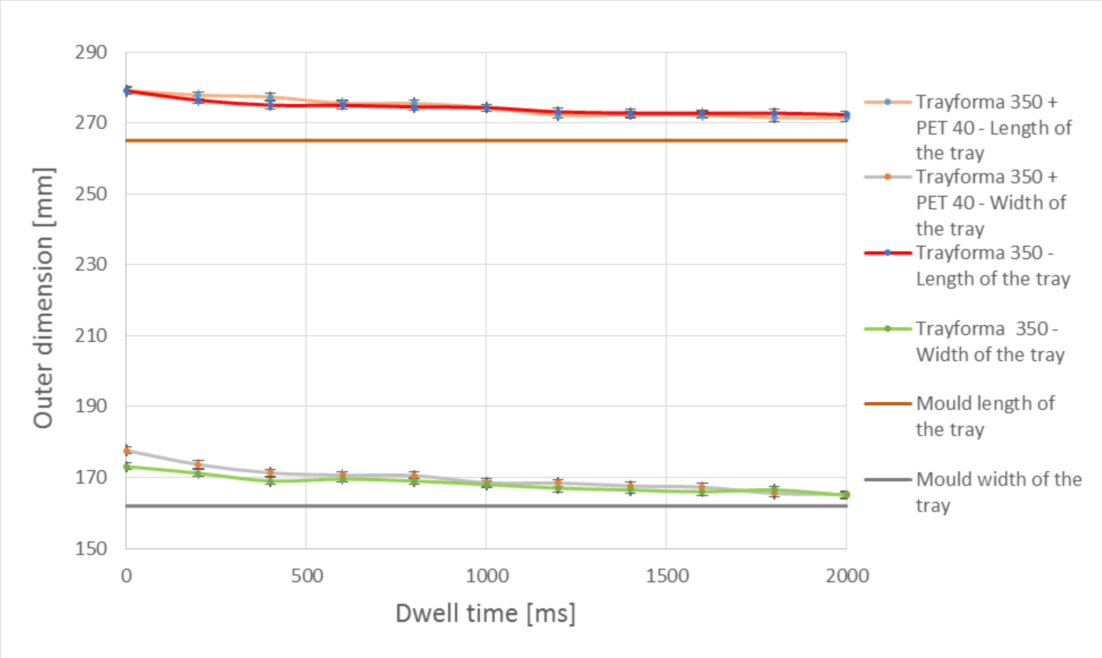

Fig. 6. Effect of dwell time on the tray dimensions with the female mould temperature of 160 °C

The length of the tray was reduced 7.7 mm and the width 11.4 mm when the dwell time was extended from 0 ms to 2000 ms, as is presented in Fig. 6. It should be noted that the actual dwell time with the adjustment value of 0 ms dwell time is approx. 50 ms due to the inertia of tool weight. The main disadvantage of longer dwell time use is the reduction of production speed. When dwell time is increased from 400 ms to 1000 ms, the production speed decreases from 31 trays/min to 24 trays/min.

Test substrate Trayforma 350 was also tested without the polymer coating. From a dimensional point of view, the substrate performed similarly with and without the PET-coating, and consequently the spring back phenomena can be attributed to the material properties of the baseboard.

Increasing the pressing speed caused an increase to the tray dimensions, which is presented in Fig. 7. This can be explained based on the amount of heat being transmitted to the formed substrate. With greater speeds, the blank spends a shorter period of time in contact with the heated mould. An advantage of greater pressing speed is the increase of production speed, but this advantage may be lost if dimensions of the tray have to be altered with a longer dwell time.

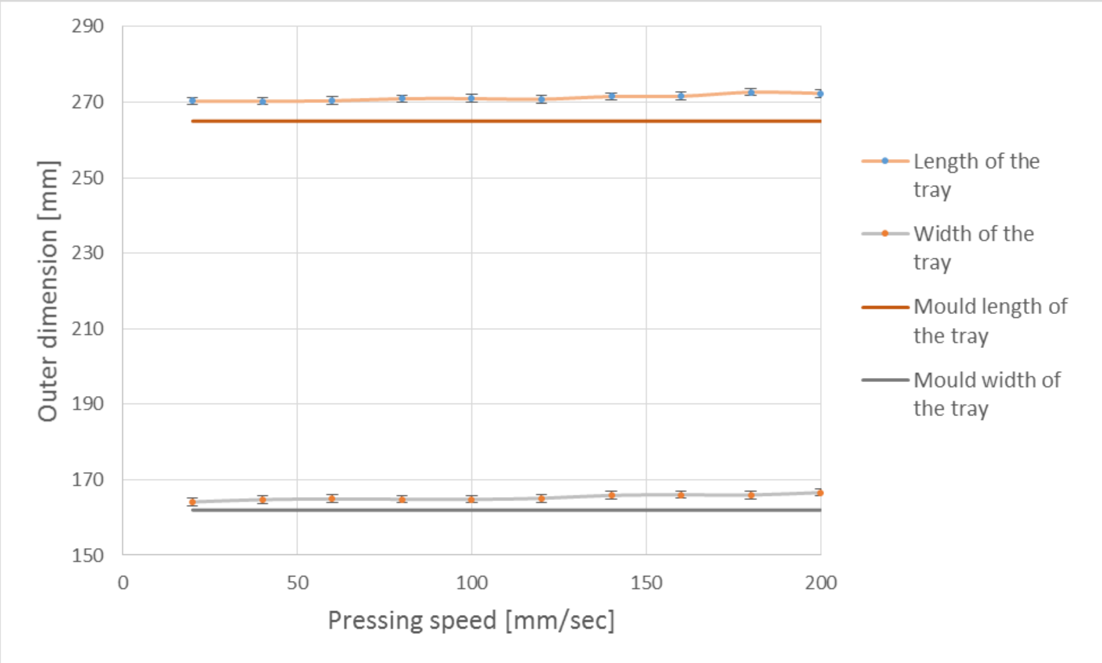

Fig. 7. The effect of pressing speed on the outer dimensions

Interactions between process parameters were also studied. Changes in outer dimensions of the trays were consistent and formed material behave as expected on the basis of Fig. 3 to 7. The effect of dwell time and female mould temperature on tray length is presented in Fig. 8 as an example.

Moisture content of the substrate was 9.1% in the blank stage, which is the initial moisture content of the paperboard and thus conditions correspond to a typical production run. Change in moisture content was measured from ready-made trays which have been produced using different dwell times. Standard deviation in the moisture measurements was 0.2 percentage units.

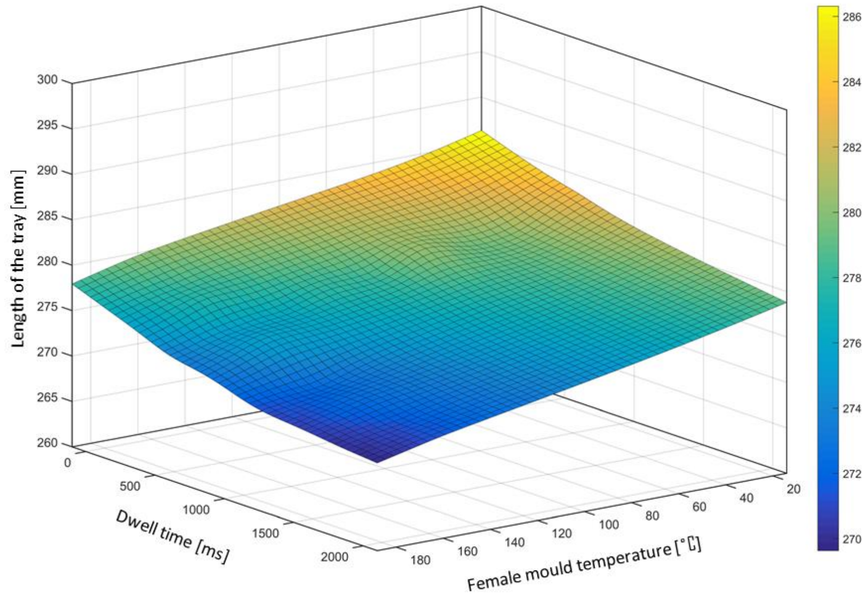

Fig. 8. The effect of dwell time and female mould temperature on the length of the tray

The moisture content was reduced by 1.45 percentage points during press forming process from polymer coated Trayforma 350 + 40 PET. Same base substrate without polymer coating dried up by 2.7 percentage points which is due to the fact that the moisture can evaporate the material on both sides. Reduction in moisture content is presented in Fig. 9.

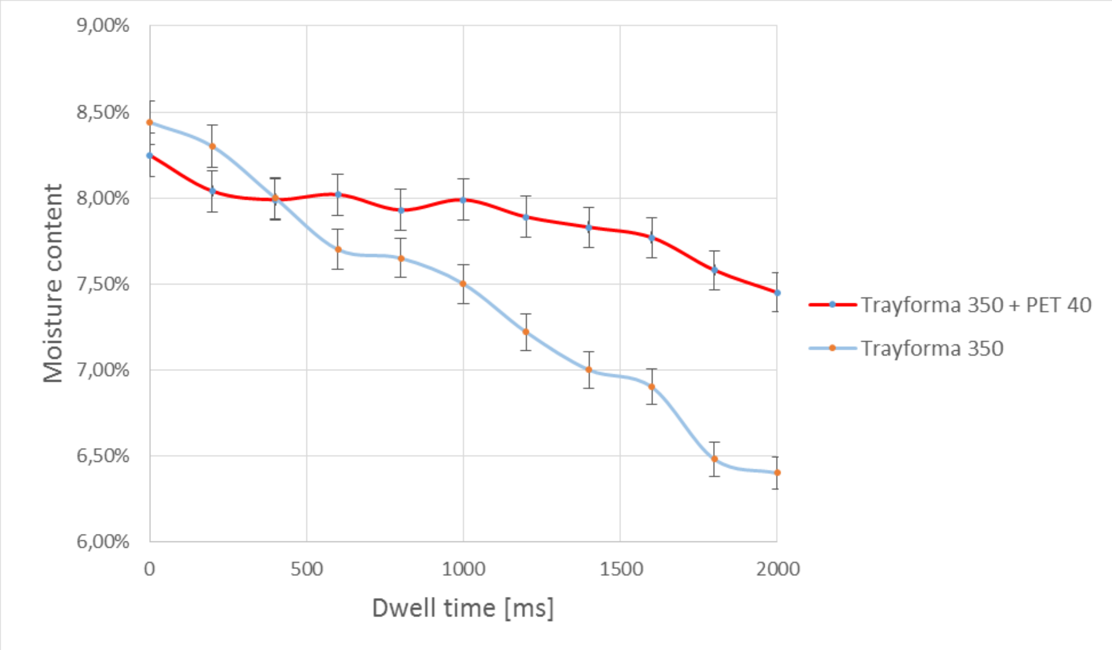

Fig. 9. The effect of dwell time on the moisture content of ready-made trays

Greater drying of the uncoated base board compared to the PET-coated one did not have an effect on the amount of spring-back, which can be seen in Fig. 6.

Ready-made trays were stored under standard laboratory conditions (23 ºC, 50% RH) unstacked and measured again after one week. Equilibrium moisture of trays was measured to be 6.1%, and any additional dimensional changes was not discovered.

Dimensional change describes how much the outer dimensions of the tray increase when 4 kg weight compresses the tray. In Fig. 10, dimensional change is presented in relation to dimensions of the trays at initial conditions. The amount of dimensional change indicates that strength properties of the ready-made tray vary as a function of dwell time. Tested trays were expected to be more rigid in MD, which was confirmed by the fact that length of the tray did not change substantially when a dwell time over 200 ms was used. In CD (width of the tray) the effect of compression load was reduced almost linearly when the dwell time was increased. Improvement of the tray rigidity slowed down when the dwell time was prolonged over 500 ms, which therefore can be recommended as the lower limit for dwell time.

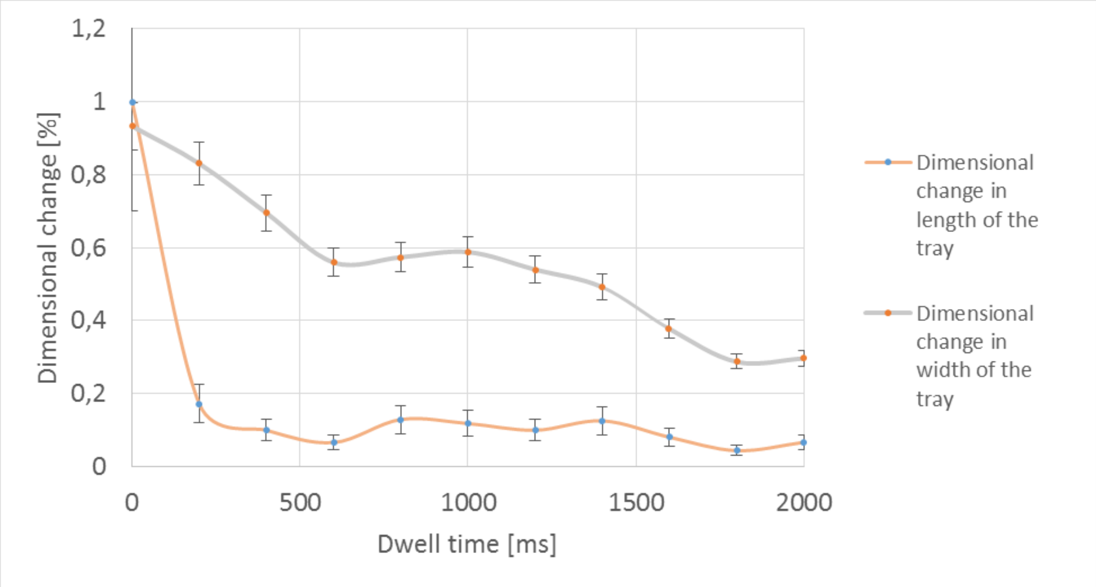

Fig. 10. The effect of dwell time on dimensional changes in tray under load

All measured trays were bigger both in length and in width compared to the design values of the mould set. When optimized process parameters (female mould temperature 160 ºC, blank holding force 2.0 kN, dwell time 600 ms) were used, produced trays were 275.9 mm long and 171.4 mm wide. The target length (265 mm) of the tray mould is 4% and the target width (162 mm) 5.5% smaller. The difference between design values of dimensions and tray dimensions is most likely material-dependent, and further studies with different materials are needed.

Effect of tray dimensions on lid sealing process

Figure 11 shows the average residual oxygen in the sealed trays. With 400 grams of product the dimensions of the tray did not have a significant effect on the amount of residual oxygen. However, with lighter packages (200 g and 25 g) there was significant amount of oxygen in the packages if the tray dimensions were too large.

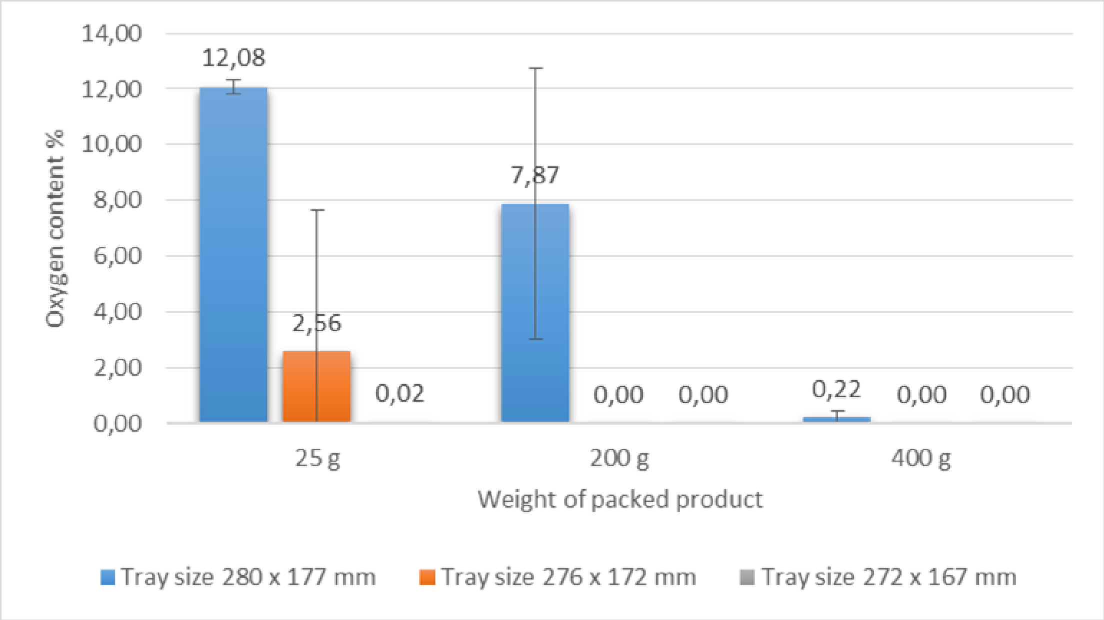

Fig. 11. Oxygen content of the heat sealed and gas flushed packages with varying dimensions and product mass

It is clear that the package size had a great effect on the residual oxygen and that the mass of the product also affected the amount of residual oxygen in the packages. This is because the tray does not fit between the lower tools of the sealing tools when the vacuum chamber is closed. Normally, the tray is lifted from under the rim area to the sealing position. When the dimensions are too large, the tray walls will touch the sealing tool before the rim area, and the tray will not be in the correct position when the chamber is flushed with the gas. The larger mass of the packed product will cause the package to position properly inside the lower sealing tool, and the gas flushing will not be disturbed. However if the packages' dimensions are too large, even the mass of the product will not fix the situation, and the flushing will be incomplete. This effect is clarified in Fig. 12 and 13. The packed product is not visualized in the figures.

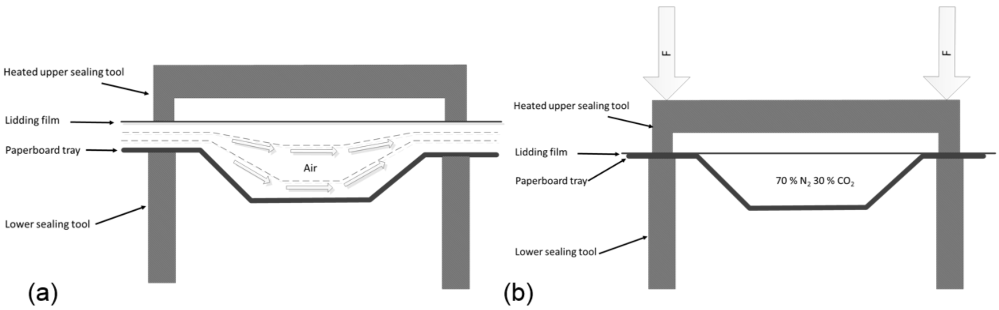

Fig. 12. (a) A correctly sized tray is flushed first with a vacuum and then with the protective gas, air is removed from the package (b) the Modified Atmosphere Packaging (MAP)-gas filled tray is sealed with the lidding film

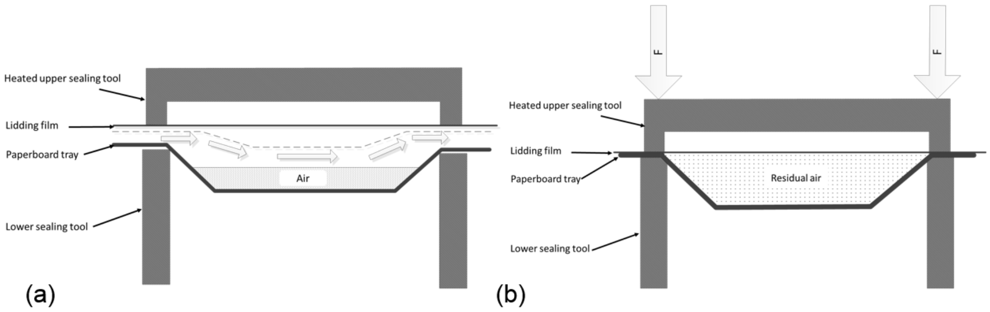

Fig. 13. (a) A dimensionally too large sized tray is flushed but the flushing is incomplete because of the positioning of the tray is not correct (b) the tray is sealed with the lidding film and some air is left in the package

The lid sealing process reduced the size of the trays and evened out size differences substantially. The greatest difference in outer dimensions of the trays was 8 (length) – 10 (width) mm before sealing and only 4.8 – 6.3 mm afterwards. Size changes were the smallest in more compact trays with the largest product mass. This situation is caused when the sealing tools force the dimensions of the tray to decrease and the sealed lidding film prevents spring-back. The change in the dimensions of the trays after the lid sealing phase is presented in Fig. 14.

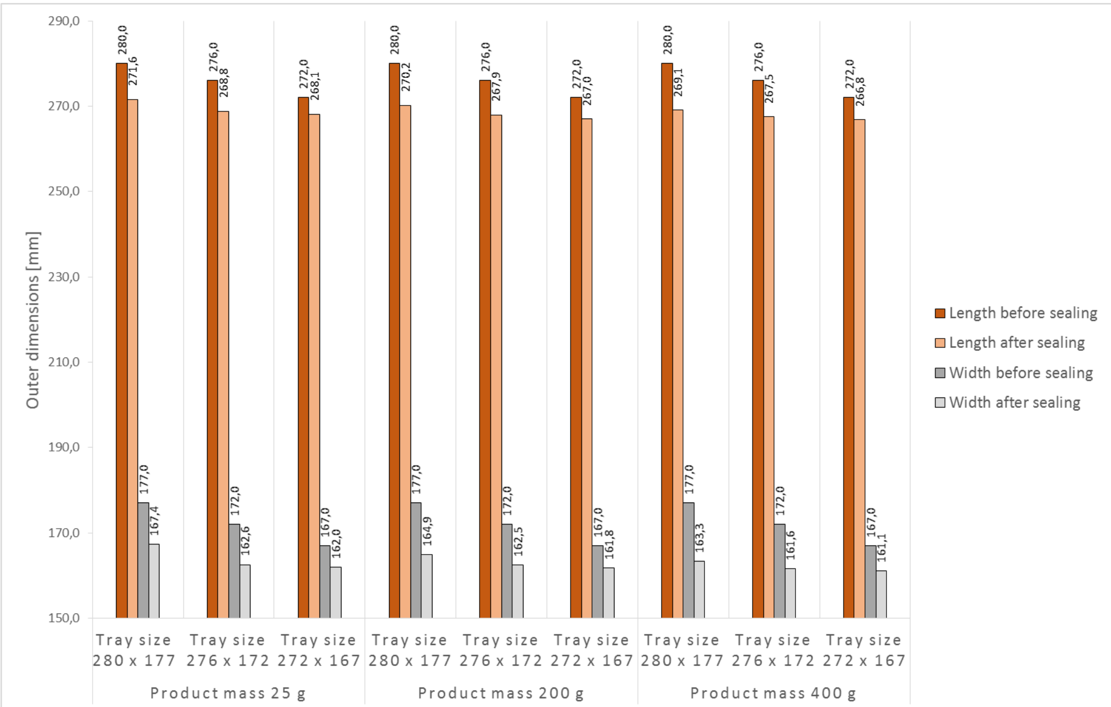

Fig. 14. Outer dimensions of the trays before and after the lid sealing phase

CONCLUSIONS

1. All heat related parameters, *i.e.*, mould temperatures, dwell time, and pressing speed, can be used to adjust the outer dimensions of the paperboard tray. The only process parameter that does not have an effect on the production speed is the temperature of the female mould.
2. The required amount of heat should be pursued using as high a mould temperature as possible. The upper limit of the usable mould temperature is usually limited by bubbling or melting of the polymer coating layer. When mould temperature is set high, the rest of the required amount of heat can be achieved by adjusting the dwell time correctly, simultaneously optimizing the production speed. The effect of pressing speed on tray dimensions is relatively small compared to effect on production speed, and it therefore should not be used on dimensional adjustment.
3. Produced trays with dimensions closest to the mould length and width caused no problems in the sealing machine either in the package transfer or in the lid sealing phases. With alteration of the blank size, sealed trays can be modified exactly to GN-standard dimensions. However, the functionality of trays in the lid sealing process or in transport box packaging does not require it. Most likely, the first defect caused by a small oversize is a visual one – the printing in the tray lid may not fit into the shape of the tray (Fig. 2d).
4. The mass of the packed product has an effect on the required dimensional tolerances when the trays are heat sealed and flushed with MAP. With a product mass of 25 g, even an increase of length from 4 mm and width of 5 mm resulted in inadequate flushing of the trays. On the other hand, with a product mass of 400 g, an increase of 8 mm in length and 10 mm in width did not disturb the gas flushing of the tray.
5. Dimensional inaccuracy of the trays is mended to some extent when the lidding film is sealed to the tray. This is caused when the sealing tools force the dimensions of the tray to decrease and the sealed lidding film prevents the spring-back. The size reduction after sealing was found to be up to between 3 and 4 mm in both length and width.
6. All produced trays were measured to be bigger both in length and in width compared to the design values of the mould set. Therefore the mould set has to be designed undersized to obtain trays with certain outer dimensions. The length of the tray mould should be 4% and width 5.5% smaller than the desired trays with the tested tray design and substrate combination. Using this dimensioning, the process window of press forming allows for fine tuning of dimensions during the production without compromising other properties of the trays and production speed reduction.

ACKNOWLEDGMENTS

The authors would like to thank Petri Mäkelä and Sami Matthews for participating in the practical work.

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Article submitted: July 7, 2015; Peer review completed: September 14, 2015; Revised version received and accepted: October 21, 2015; Published: November 10, 2015.
DOI: 10.15376/biores.11.1.140-158

Publication VII

Leminen, V., Mäkelä, P., Tanninen, P. and Varis, J.
Leakproof Heat Sealing of Paperboard Trays - Effect of Sealing Pressure and Crease Geometry

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Bioresources
Vol. 10, No. 4 pp. 6906-6916, 2015

Leakproof Heat Sealing of Paperboard Trays - Effect of Sealing Pressure and Crease Geometry

Ville Leminen,* Petri Mäkelä, Panu Tanninen, and Juha Varis

The leakproof sealing of paperboard trays depends on factors such as the quality of the sealed tray and the parameters of the sealing process. Leakproof sealing is critical when food products are packed, as poor sealing can result in leakage and cause a reduction in the microbiological quality and sensory shelf life of packed food products. In this paper, factors affecting the leakproof sealing of paperboard trays, such as sealing pressure and the geometry of creases in the trays, were investigated. Trays were sealed with varied sealing pressure and temperature, and the sealed trays were inspected using a coloring solution test, oxygen content measurements, and microscopic analysis. The results show that the sealing pressure is a critical parameter in the sealing process. The minimum sealing pressure that resulted in leakproof within the materials investigated was 1.8 N/mm^2. The depth of crease that can be sealed in a leakproof manner was found to be up to 150 µm.

Keywords: Paperboard; Tray; MAP; Modified atmosphere packaging; Tightness; Sealing pressure; Heat sealing; Leakproof

*Contact information: Department of Mechanical Engineering, Packaging technology, Lappeenranta University of Technology, P.O. Box 20, FI-53851 Finland; *Corresponding author: ville.leminen@lut.fi*

INTRODUCTION

The heat sealing of 3-dimensionally-formed, polymer-coated paperboard trays has been previously researched. Traditionally, the so-called “industrial grade” trays manufactured by the press forming process have not allowed for adequate tightness properties (Leminen *et al.* 2012; Hauptmann *et al.* 2013). However, it has been shown that press formed trays can be also sealed to achieve both liquid tightness and satisfactory modified atmosphere packaging (MAP) tightness (Leminen *et al.* 2015a). The process of sealing paperboard trays is more challenging than sealing polymer trays because of creases and/or wrinkles in the sealing surface caused by the manufacturing process and the material properties of fiber-based materials. The creases and wrinkles can act as capillary channels that may cause leaks in the package (Leminen *et al.* 2012, 2015a; Hauptmann *et al.* 2013). Poor sealing can result in leaks and can reduce the microbiological quality and sensory shelf life of packed food products (Randell *et al.* 1995).

The effect of the resulting sealing pressure on the quality and failure of the heat seals of laminates has been discussed previously. With thin laminates, it has been stated that if too high a pressure (over 0.3 N/mm^2) is used, the sealant of the laminate can form a polyball, which causes the sealant of the laminate film to form along the edge of the heat sealed portion. This can lead to weaker seal strength and a thinner bonding layer (Hishinuma 2009). However, this was observed when two laminates (Al-deposited

CPP/OPP) were used and the heat sealing jaws heated the film from both sides. When a polyethylene terephthalate (PET)-coated paperboard tray was sealed with a multi-layer, PET-sealable film and the heat was applied only from the top of the lidding film, a resulting sealing pressure of about 2.7 N/mm^2 was found to be effective (Leminen *et al.* 2015a). This suggests that the uneven sealing surface and one-sided heating of paperboard trays requires significantly larger surface pressure than thin laminate films. One reason for this might be that the lidding film must fill the wrinkles in the sealing surface, which requires larger surface pressure.

In this study, the effect of the sealing pressure on the seal tightness of press formed paperboard trays was investigated to determine the surface pressure required for adequate seal tightness and properties. The investigation was done in relation to the sealing temperature. Also, the dimensions and shapes of the creases in the trays were measured and analyzed to determine the depth of the creases and wrinkles such that the tray can be sealed with adequate tightness.

The effect of heat sealing variables (temperature and dwell time) has been discussed, for example, with linear low-density polyethylene (LLDPE) (Mueller *et al.* 1998) and paperboard trays (Leminen *et al.* 2012), but the effect of the sealing pressure and crease geometry on paperboard trays has not been investigated. This information is important for the design of new sealing tools for paperboard trays. If the required (optimal) surface pressure is known, then this information can be used to design optimal tooling for the best tightness results. Also, the evaluation of creases can provide insight as to the question of the quality of trays that can be sealed as leakproof.

The modified atmosphere in the packages was analyzed using an optical fluorescence O_2 analyzer and an oxygen transmission rate testing system. The purpose of the atmosphere analysis was to investigate the headspace gas and the tightness of the sealed packages.

EXPERIMENTAL

Materials

The primary material used in the trays was Stora Enso Trayforma Performance 350 + 40 WPET (Stora Enso Imatra Mills, Finland). This material is a PET extrusion-coated paperboard with a base material grammage of 350 g/m^2 and a coating grammage of 40 g/m^2. The base board consists of three solid bleached sulphate (SBS) layers.

The lidding material used in the heat sealing was a PET-sealable multi-layer film, Westpak WestTop 405B PET (WestPak Oy Ab; Säkylä, Finland).

Methods

Experimental design

A detailed description of the press forming process was presented in previous manuscripts (Leminen *et al.* 2013; Tanninen *et al.* 2014). The trays were formed from pre-cut and creased blanks. The forming parameters included a female tool temperature of 170 °C, pressing dwell time of 1 s, pressing force of 135 kN, blank holding force of 1.2 kN, and pressing speed of 130 mm/s.

The tray size used was approximately 265 x 162 x 38 mm. The blank and tray geometry is shown in Fig 1.

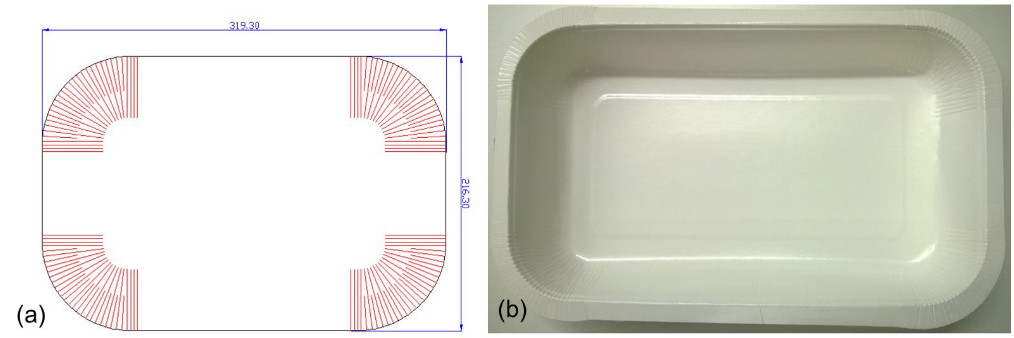

Fig. 1. The (a) blank used and (b) tray geometry. The creasing lines are presented in red.

The manufactured trays were sealed with a lid using an Ilpra Speedy tray sealer (Ilpra S.p.A; Vigevano, Italy (Ilpra 2014)). The tray sealer is shown in Fig. 2. The machine is a standard industrial sealer that was modified by adding a precision pressure regulator Festo LRP-1/4-10 (Festo, Italy) to adjust the sealing pressure.

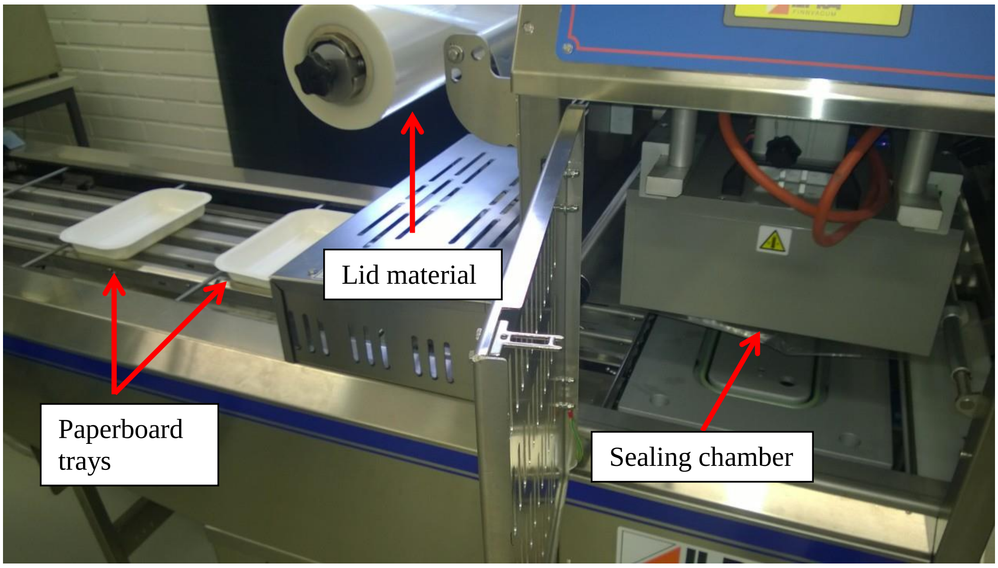

Fig. 2. Sealing equipment used in the study

The sealing process is described in Fig. 3. The sealing time was constant at 2.5 s, and the other heat sealing parameters used are presented in Table 1. The trays were flushed with a common gas mix for food applications. The gas composition was 70% N_2 and 30% CO_2. The accuracy of set temperature in the used equipment was ±4 K.

The sealing tool used was designed specifically for use with paperboard trays. The tool-set consisted of a heated upper tool with a flat metal surface and a bottom tool with a metal surface with silicone rubber. The tray rim and lidding film were placed between the tools and the trays were sealed together by applying pressure and heat. The width of the heated upper tool was 3 mm.

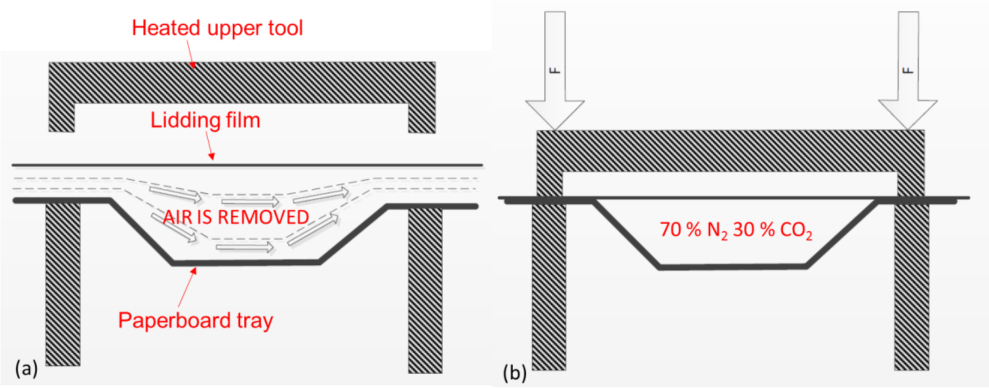

Fig. 3. The heat sealing and MAP process. (a) The tray is flushed with a protective gas and air is removed from the package, and (b) the tray and lidding film are sealed together for a set time and a seal is formed.

Table 1. Heat Sealing Parameters

Sealing Temperature (°C)	Sealing Pressure (bar)	Resulting Surface Pressure (N/mm^2)
170	6	2.7
170	5	2.2
170	4	1.8
190	6	2.7
190	5	2.2
190	4	1.8
190	3	1.3
210	6	2.7
210	5	2.2
210	4	1.8
210	3	1.3

After the sealing of the lid, the trays were flushed with a coloring solution in accordance with the European standard (EN 13676 2001). The coloring solution was applied to the tray and the sealed area for 5 min, and the seal was inspected for leaks. The reagents in the coloring solution were dyestuff E131 Blue and ethanol (C_2H_5OH, 96%). The color solution consisted of 0.5 g of dyestuff dissolved in 100 mL of ethanol. Flushing was done to detect leaks in the package and sealing area. The packages that had no leaks of the color solution were then selected to be sealed with the same parameters to investigate the oxygen composition inside these packages.

The oxygen composition inside the package was analyzed using a Mocon Optech O_2 Platinum analyzer (Mocon Inc., Minneapolis, USA). The analyzer utilizes the standard ASTM F-2714-08 (Standard Test Method for Oxygen Headspace Analysis of Packages Using Fluorescent Decay). The measurement method consisted of inserting an oxygen sensor inside the lidding film before heat sealing the film to the tray. The response of the phosphorescent sensor was analyzed using a handheld light beam device. The analysis occurred over the course of 14 d. The sealed trays were stored in a refrigerator, at 6 °C, to simulate realistic storage conditions.

The oxygen transmission rate of trays sealed at 190 °C and 6 bar was also analyzed with an Oxygen Transmission Rate (OTR) testing system (Mocon Ox-Tran, Mocon Inc., Minneapolis, USA) according to the standard ASTM D3985-05 ("Standard Test Method for Oxygen Gas Transmission Rate Through Plastic Film and Sheeting Using a Coulometric Sensor," 2010) to verify the results of the platinum analyzer. OTR measurements were conducted at 50% Relative Humidity (RH) and 23 °C.

The rim areas of the trays were studied with a stereomicroscope (Olympus Tokyo) to investigate the geometry and dimensions of the creases and the sealing results. The quality of the commercial, industrial grade, press-formed trays was also analyzed. Three different commercial trays were analyzed, the dimensions of the creases were measured, and the shape of the creases in the sealing surface was analyzed. The measured surface roughness parameters of manufactured trays were reported by Leminen *et al.* (2015b). The roughness parameter peak height (R_p) was found to be a useful indicator of the surface quality of paperboard trays. The average R_p value of the creased area of the trays sealed in this study was R_p (max) = 36.

RESULTS AND DISCUSSION

Table 2 shows the results of the color solution flushing. Five trays for each test point were used. The sealing pressure influenced the sealing result significantly, as expected. The temperature also had an effect on the required sealing pressure. However, when the temperature was at a proper level (190 to 210 °C) and sealing pressures of 4 bar or more were used, the seals appeared leakproof. When the temperature was too low (170 °C), the seals exhibited significant leakage with all pressures used. When the pressure was too low, regardless of the temperature used, the lidding film did not melt deep enough to the bottom of the creases, resulting in leaks.

Table 2. Heat Sealing Parameters

Sealing Temperature (°C)	Sealing Pressure (bar)	Leaks Shown by the Coloring Solution
170	6	Yes
170	5	Yes
170	4	Yes
190	6	No
190	5	No
190	4	No
190	3	Yes
210	6	No
210	5	No
210	4	No
210	3	Yes

Table 3 shows the average oxygen content in the packages after 14 d of storage. The values are averages of 5 trays. The trays that leaked when flushed with the coloring solution were discarded from the gas tightness test runs. The results show that the oxygen content averages in the packages were well under 1%. The measured Oxygen

Transmission Rate (O_2TR) average of the trays sealed at 190 °C and 6 bar was 4.1 cm^3/package·d. The area of the tray is approximately 0.053 m^2 and the area of the lidding film is approximately 0.034 m^2. The O_2TR value for the paperboard used is listed at 80 $cm^3/m^2 \cdot d$ (Ipack 2011). The permeation through the material used matches the measured values for the sealed packages. This means that the only permeation was caused by permeation through the tray, and that the lidding film materials and the seals were not leaking.

Table 3. Average Oxygen Contents in the Packages after 14 d of Storage

Sealing Temperature (°C)	Sealing Pressure (bar)	Average Oxygen Content after 14 days (%)
190	4	0.68
190	5	0.39
190	6	0.67
210	4	0.68
210	5	0.57
210	6	0.51

In the work of Leminen *et al.* (2015a), it was stated that even if the coloring solution exhibited no leaks, there could be significant gas leakage into some of the packages. With the trays used in this study, it was shown that if the coloring solution did not leak, the trays were also gas-tight. This was assumed to be caused by the better surface quality of the trays. When the surface quality deteriorates, there is more variance between these analysis methods.

It was noted that a seal that appears intact and properly sealed when inspected visually from the surface can be leaking under the surface on the bottom of the crease. This kind of effect is shown in Fig. 4.

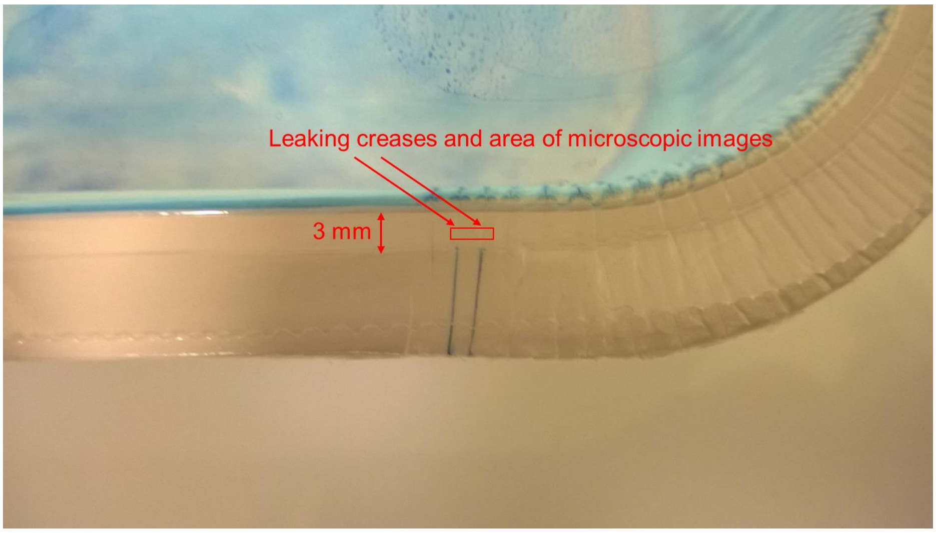

Fig. 4. Two leaking creases as revealed by the coloring solution. The leaks on the bottom of the crease are not visually apparent in the sealed area (the area with a width of 3 mm). The approximate area where samples were cut from the samples is indicated by the red box.

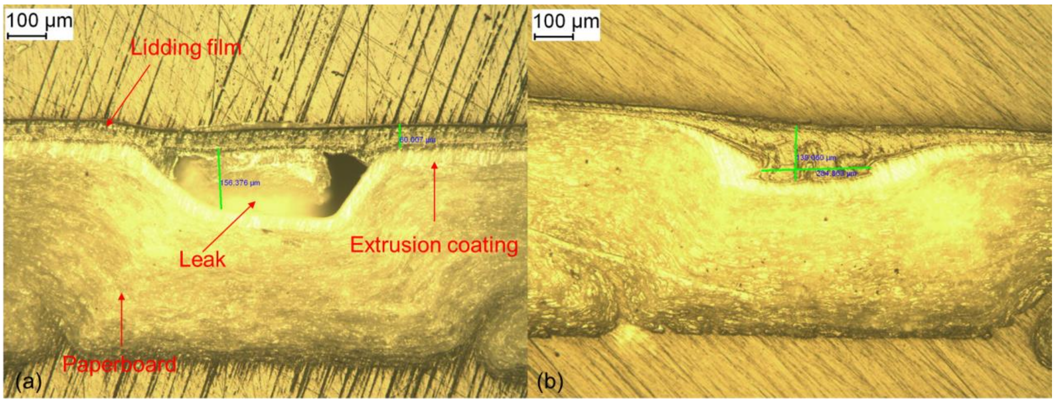

Fig. 5. Samples heat sealed with a pressure of (a) 3 bar, resulting in inadequate depth in the melting of the lidding film and leaks; and (b) 6 bar, resulting in a successful, non-leaking seal

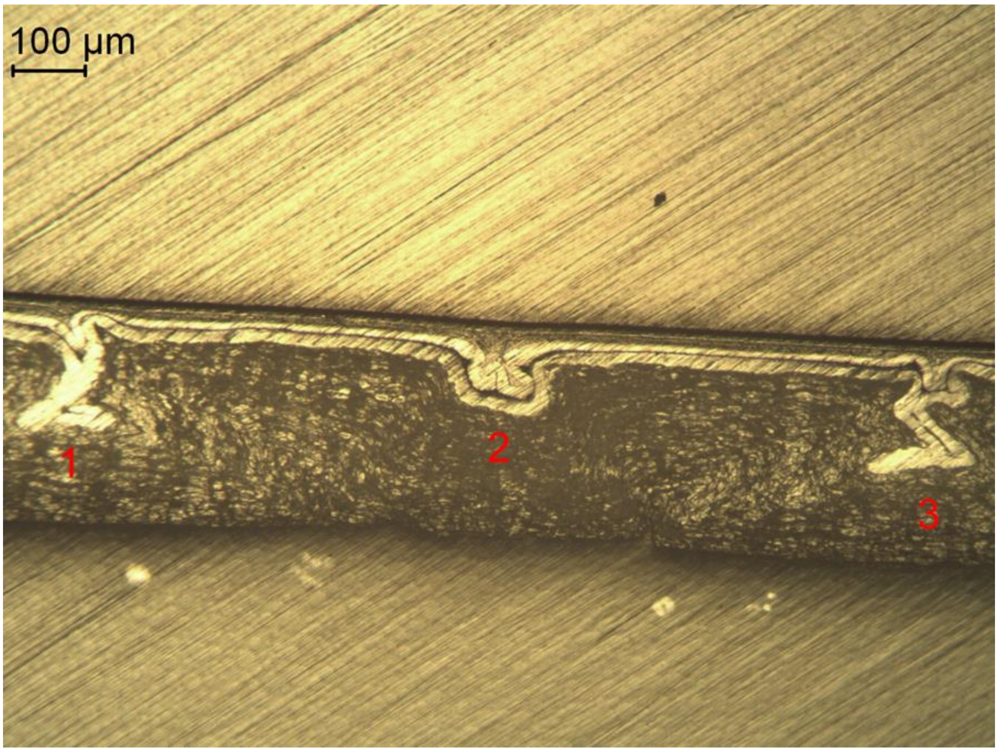

Fig. 6. Heat sealed creases with a sealing pressure of 6 bar, resulting in the lidding film melting to the bottom of the creases. Creases numbers 1 and 3 are so called "closed" creases and crease number 2 is an "open" crease.

Figure 5 shows two samples with sealing pressures 3 and 6 bar. In Fig. 5a, a clear leak is visible on the bottom of the crease in the sealing surface. It is clear that the sealing pressure had an effect on the melting depth of the lidding film. If the sealing pressure is too small, the film could be melted to the bottom of the crease, and leakage occurred. This shows that trays with deeper wrinkles and creases could potentially be tightly sealed if the surface pressure were higher. However, because the sealing tool width (3 mm in this case) cannot be reduced infinitely, the only way to increase the surface pressure would be to increase the pressure in the cylinders that produce the sealing force. Raising this pressure to above around 6 bar would require a pressure booster regulator, which could lift the system pressure to 10 bar. This is an interesting topic for further study. With polymer based trays, which have flatter sealing surfaces, the process window is much larger and there has not yet been a need for a higher surface pressure. This is also apparent in Fig. 5, where the flat surfaces around the wrinkles were successfully sealed even with the smaller surface pressure.

Figure 6 shows three creases which were sealed with the lidding film. The shape of these creases was typical for a creasing pattern, like those presented in Fig. 1a. The longer creases are usually formed “closed”, like creases 1 and 3, while the shorter creases are formed “open”, like crease 2. However, this kind of shape variance did not have a noticeable effect on the sealing result, as both geometries could be sealed with satisfactory leakproofness when the depth of the creases formed is not too large and the tray is otherwise intact. The results indicate that creases and wrinkles with depths of about 150 µm can be sealed in a leakproof manner.

Three industrial-grade trays were also analyzed to investigate the dimensions and shapes of the creases in the sealing surfaces of trays manufactured by commercial equipment. One of these trays was used with MAP for cold-cut ham and the other two samples were not sealed. In the tray used with MAP, the depth of the tray was approximately 16 mm, and the geometry of the tray was designed such that that the radius of the creased area was very large (about 110 mm). This generally makes the quality of the rim area flatter and prevents leakage (Leminen *et al.* 2015a). It was found that the coating film of the tray could not be clearly distinguished from the lidding film. This indicates that the different layers became melted together because of the high heat input and pressure in the sealing process. An example of this tray is presented in Fig. 7a.

The depth of the unsealed, industrial-grade trays ranged from 28 to 32 mm and the corner radius was about 50 mm. Figure 7b shows an image of a crease from an industrial-grade tray with an open crease that was approximately 400 µm deep. This kind of shape and dimension prevents the tray from being sealed without leaks.

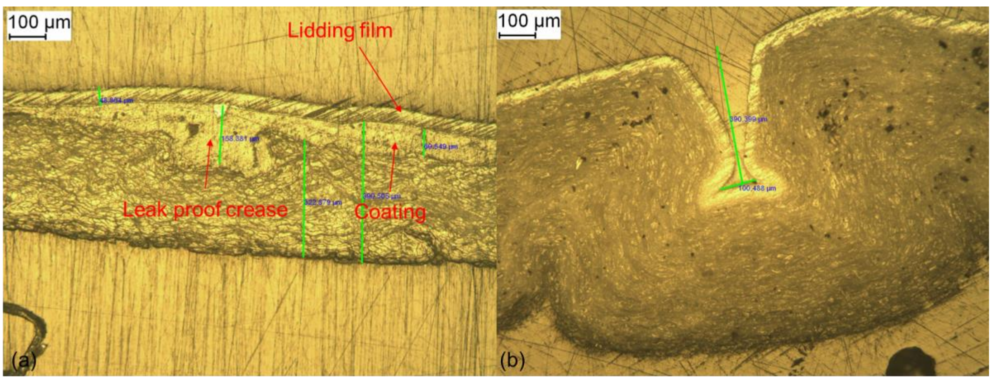

Fig. 7. (a) A heat sealed, leakproof, industrial-grade sample with a crease depth of approximately 160 µm; (b) An industrial-grade tray with an open, roughly 400-µm-deep crease.

The depth and width of the creases and the sealing process parameters are not the only factors important when considering if the tray can be sealed without leaks. When the manufacturing process of the tray is not satisfactory, the tray can have capillary channels that compromise its integrity. An example of an industrial-grade tray with a capillary channel is shown in Fig. 8. The heat sealing of the lidding film cannot mend this kind of defect in the trays. This kind of effect was also discussed in the work of Hauptmann *et al.* (2013).

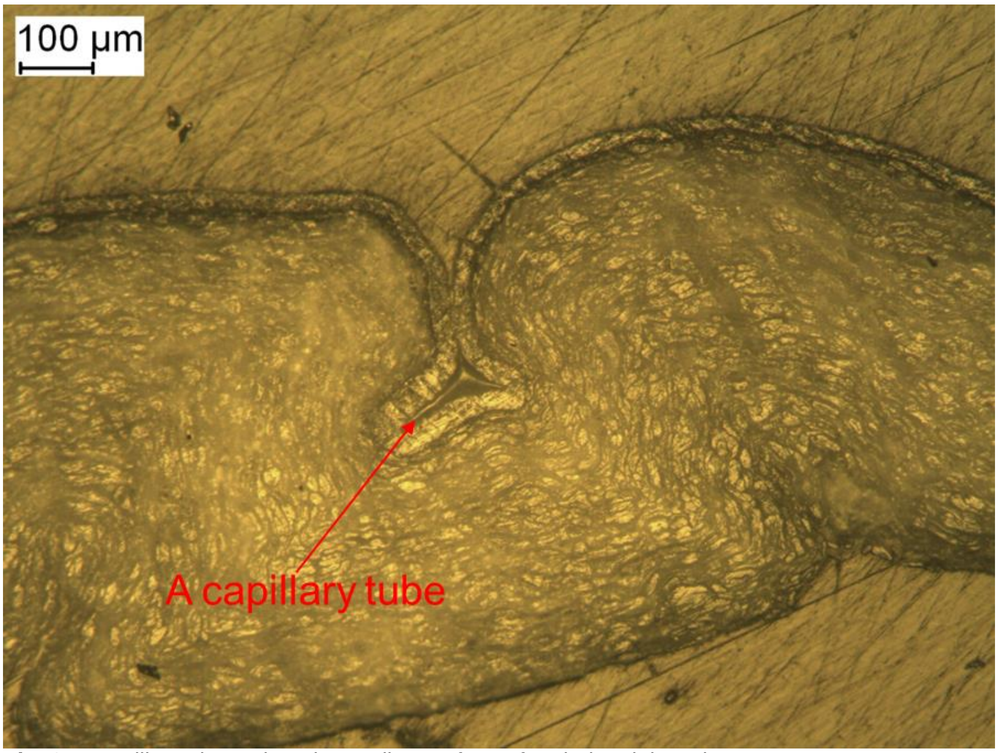

Fig. 8. A capillary channel on the sealing surface of an industrial grade tray

CONCLUSIONS

1. Sealing pressure has a great effect on the tightness of heat seals when press formed, polymer-coated paperboard trays are heat sealed with a lidding film. Too low a pressure results in leaks, which first occur at the bottom of the creases in the sealing surface.
2. The resulting surface pressure which resulted in successful seal tightness with these products ranged from 1.8 to 2.7 N/mm^2. This should be taken into account when sealing tools for press-formed trays are designed.
3. The O_2TR values and oxygen contents of the trays show that press-formed paperboard trays can be sealed without leaks such that the only O_2 permeation is through the sealed materials, not from the seal.

4. Creases in the sealing surface of depths of up to 150 μm can be sealed without leaks.
5. The depth of the creases is not the only factor determining if the seals are leakproof; defects such as capillary channels can appear if the tray manufacturing process is not controlled properly.

ACKNOWLEDGMENTS

The authors would like to thank Stora Enso and Westpak for providing materials and the European Union's regional funding, projects A32103 and A70197. Sami Matthews is thanked for his help with figures.

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Article submitted: May 26, 2015; Peer review completed: July 20, 2015; Revised version received: August 11, 2015; Accepted: August 14, 2015; Published: August 28, 2015.
DOI: 10.15376/biores.10.4.6906-6916

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