Master thesis

Creating a framework to assist heat sealing technology decision-making, with a focus on conduction and ultrasonic sealing

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#### **Master thesis**

'Creating a framework to assist heat sealing technology decision-making, with a focus on conduction and ultrasonic sealing'

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# Abstract

Mono-materials have the potential to have a high recyclability performance [13-17]. The main reason for this is that mono-materials do not require material separation of the packaging system during the sorting, as multi-material alternatives require [14, 18]. Sealing mono-materials however, can form a challenge. Conventional heat sealing technology reaches its limitations when trying to seal mono-materials, due to the small sealing window and the large temperature fluctuations associated with conduction sealing. Ultrasonic sealing is more suitable for sealing mono-materials since it heats the materials at the seal interface. A new and successful combination is ultrasonic sealing implemented in an HFFS system. This raises the question of whether other sealing technologies might be applied in HFFS system and when. To answer this question, sealing technologies need to be compared.

This thesis proposes a framework that compares different types of heat sealing technologies with a HFFS system application. This framework can be used to select the proper type of sealing for a certain application. The sealing technologies are compared based on different parameters. These parameters together will indicate where the main differences between the sealing technologies lie and will form a base for a well-founded choice. The focus is on conduction and ultrasonic sealing. These two types of sealing are most used in practice [19]. Especially conduction sealing is used extensively and has a large amount of scientific papers written about it. Ultrasonic sealing is newer and tests have been performed to fill in the framework for this sealing technology.

# List of abbreviations

HFFS	Horizontal Form Fill and Seal system
US	Ultrasonic Sealing
CS	Conduction sealing
SS	Seal Strength
DoE	Design of Experiment
ANOVA	Analysis of Variance
MAP	Modified Atmosphere Packaging

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# 1. Introduction

## Importance of packaging

One of the global targets of the United Nations is to reduce food waste [20]. Packaging plays a major role in reaching this target since packaging reduces food waste [21]. So packaging is an interesting topic to investigate, especially since almost 90% of plastic packaging is used to package food [22]. By reducing food waste, less impact on the environment is exerted. At the same time, it should be noted that packaging itself also impacts the environment and depletes finite resources [20].

The main function of a packaging system is to protect the product inside against environmental factors like light, oxygen, other gases, microorganisms and moisture. In some applications, the packaging system should also contain the aromatic flavors. The product inside should be protected to extend the shelf life and to ensure the quality and safety of the product [8, 14, 23-25]. Next to protecting the product, the packaging system also has other functions like increasing the transportability, storage and communication to the user [14, 18].

### Importance of sealing

There is a trend towards the use of more flexible plastic packages (thickness between  $13\mu$ m and  $75\mu$ m) instead of rigid ones. This is due to their light weight, flexibility, ability to seal and ability to run on high-speed production lines [14, 24, 25]. To create a closed flexible plastic package, a film of material must be formed and closed. This is usually done through sealing. The film is bonded together at the seal. The packaging system can only protect the product inside if the seal is properly closed and has sufficient strength. Therefore, the consistent performance of the sealing process is critical. [8, 9, 26-28]. The seal strength must be sufficient to not open during for example transportation, but at the same time, the opening of a package containing strong seals can form a challenge by reducing user-friendliness [29, 30].

The film that is used to create the flexible packaging should have enough barrier properties to successfully fulfill its function. This is often achieved by incorporating several different materials into a single film [18, 24]. European legislation development however discourages the use of these multi-material films, because of their incompatibility with recycling [17].

Seals can be created by using heat sealing or cold sealing technologies. Cold sealing connects materials with adhesives [27, 31]. This thesis focuses on heat sealing. There are many different types of heat sealing, but they all somehow soften the inner layers of the films by adding energy and thereby fusing the inner layers [31]. Sealing performance is influenced by many factors, like the material and the application [9, 27].

### Seal performance

As mentioned before, a seal is critical for a package to properly protect the food inside, but what properties does a good seal have? For a seal to have a high seal performance, the seal integrity and the seal strength should be sufficiently high [28]. Seal integrity refers to the leak tightness that a package has [8]. This can be evaluated with methods like dye penetration [27, 32, 33] or the bubble test [27, 33-35]. The seal strength refers to the force it takes to open a certain seal. This can be measured immediately after sealing (hot tack strength) or after cooling. The seal strength referred to in this thesis is the seal strength after cooling unless stated otherwise. This seal strength is relevant in for example transportation and opening behavior for the consumer [8].

A seal can be realized with heat sealing. There are different types of heat sealing with each having its application. To create a greater understanding of when to use what type of heat sealing this thesis is created. This will help the company Omori to make a proper heat sealing technology choice. Usually, conduction sealing is employed in the Omori flow packs. Using the framework, the opportunity to use other types of heat sealing technologies can be recognized.

# Company

The thesis is executed at the company Omori Europe. This company that is located in Oldenzaal sells packaging machines. One of their machines is the horizontal flow packer. This horizontal form fill and seal (HFFS) system wraps a film of material around a product. The film of material is guided from the roll towards the product (mostly from above) and then folded into a tube form using a forming shoulder. This tube is closed by a longitudinal seal. The product is placed in this tube. The product is trapped in the packaging material by two transverse seals (one on each side of the product). This thesis will help them to give adequate and well-founded advice to their clients on what sealing technique is preferred for what application.

# This thesis

This thesis proposes a framework that compares different types of heat sealing technologies. This framework can be used to select the proper type of sealing for a certain application. The sealing technologies are compared based on different parameters. These parameters together will indicate where the main differences between the sealing technologies lie and will form a solid base for a well-founded choice. The focus is on conduction and ultrasonic sealing. These two types of sealing are most used in practice [19]. Especially conduction sealing is used extensively and has a large amount of scientific papers written about it. Ultrasonic sealing is newer and tests have been performed to fill in the framework for this sealing technology.

# Contribution to the academic world

This research will contribute to the academic world by presenting a framework that compares different types of heat sealing. Certain tests have been executed to be able to fill in the framework properly. These tests are mainly performed to get more insights into the ultrasonic sealing process and will increase the academic knowledge of the ultrasonic sealing process. An example of this is that the combination of ultrasonic sealing and mono-materials is not described in public literature, while it is used in practice [36]. This research tries to close the gap between practice and literature by performing tests with mono-materials in combination with conduction and ultrasonic sealing.

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# 2. Background

There are some specific terms and concepts associated with flexible plastic packaging systems. Before the research can start, these terms and concepts should be clear. That is why this chapter about the theoretical background of flexible plastic packaging is there. The elements that will be discussed, are:

- 1. General film composition
- 2. Heat mechanisms
- 3. Materials
- 4. Mono-materials
- 5. Reclosable package
- 6. Film production
- 7. Recycling
- 8. Easy peel
- 9. Conduction sealing
- 10. Ultrasonic sealing

After this chapter, the introduced concepts can be used as a theoretical basis on which can be built further. The goal of the thesis and how this will be accomplished can be properly explained after the background is clear. So the goal and methodology of the thesis will be introduced after this chapter.

# 2.1.General film composition

Within the packaging industry, the so-called 'flexible packaging' is commonly used. These packages are non-rigid and are made of material that is no thicker than  $250\mu m$  [14]. The main application of this kind of packaging is the packaging of food [8].

A flexible package can be realized using materials like paper, plastic and composites [8]. The flexible material that is used to create such a package is called a film. Packaging films usually consist of multiple layers with each layer having its specific functions. Which film material composition is optimal heavily depends on the application and the requirements the packaging must meet [14, 15, 37].



Figure 1. General layout of a flexible packaging film [23]

The general composition of a film that is used for packaging with conduction sealing is shown in **Figure 1**. So in general, a film consists of an outer layer (print layer), a barrier layer and a sealant layer [23]. These layers are often combined using an additional adhesive layer [37]. Combining different layers of material is more costly than using a one-layer film. To make it worth the investment, each layer should add something to the overall film performance. Therefore, each layer should be optimized. Examples of what the multilayer films are optimized for are their sealing, opening, processability and puncture resistance performance [10, 27]. What the properties of each layer should be, is discussed in the following sections.

# **Outer layer**

If the film is sealed with conduction sealing, the outer layer must be heat resistant to prevent contamination of the sealing jaws and to prevent sticking [8]. In case a print is desired on the package, printing ink should adhere to the outer layer. Some materials can hold high-definition multicolor images, while others cannot hold any ink. Ink can also migrate through some materials. This should be prevented by the outer layer [27]. Ink is usually applied on the inside of the outer layer. This way, the ink is protected against environmental factors [15, 37]. An element that should be taken into account when selecting a material for the outer layer, is the coefficient of friction. It is observed in practice that this parameter can heavily influence how well the film moves through the machine.

# **Barrier layer**

Attached to the outer layer is often the barrier layer. This layer must make sure the film has sufficient barrier properties against moisture, gasses and light to ensure an appropriate shelf life [20, 27]. This can be accomplished by using one or multiple layers [37]. The outer and sealant layers also contribute to the overall barrier properties of the film. For example, LDPE is commonly used in food packaging films as a sealant layer but also has high moisture barrier properties [38].

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## Sealant layer

The inner layer, called the sealant layer, has two functions: it should be able to be sealed to another polymer and it should protect the barrier layer [17]. This sealant layer is typically a thermoplastic polymer with a low melting point. A thermoplastic material becomes moldable at higher temperatures and solidifies at lower temperatures [8]. These thermoplastic materials require a low melting point so that the sealant layer can melt and thus seal at lower temperatures without melting the outer layer. Examples of commonly used sealant materials include ethylene copolymers (e.g. EVA), polyethylene elastomers and ionomers with a low melting point [10]. Next to the melting temperature, the surface free energy, surface roughness, chain diffusion rate, melt strength and crystallization rate also influence the sealant material choice [23].

The thickness of the sealant layer should be considered when composing a multi-layer film. Some research shows that the thicker this layer, the higher the seal strength. The thickness of this layer also influences the amount of squeeze-out and the seal initiation temperature. The sealant layer should be thick enough to fill tiny gaps and wrinkles in the seal [23].

It is possible to alter the sealant layer to achieve different functionalities, like a seal that can be opened with a low force. A seal with such functionality is called an easy peel seal [19]. Easy peel seals are more elaborately described in chapter 2.8 Easy peel.

# 2.2.Heat mechanisms

To seal thermoplastic material heat is required. What the introduction of heat does to the thermoplastic materials and what the effects for the different film layers are, is explained in this chapter. This is followed by a description of how the sealing is initiated in general.

## Heat mechanism steps

The steps that happen during the fusion of two polymer films according to the literature are: the application of heat and pressure, then the sealant layer melts or softens, whereafter the diffusion of polymer chains happens at the sealing interface. This is followed by intermolecular entanglement and the cooling of the film when re-crystallization happens [8, 25, 27-29, 39-41]. This process goes quicker at higher sealing interface temperatures, although the temperature should not exceed a certain maximum value. If the temperature goes past this maximum, the seal strength might decrease due to the decomposition of the material or squeeze-out [8, 10].

There is some terminology involved that is important to understand to comprehend the heat mechanisms of heat-based sealing. First, the term glass transition temperature (Tg) will be introduced. At this temperature, the amorphous fraction of the material changes from a solid to a rubbery state [8, 42]. The material behavior changes from solid to somewhat malleable at this temperature [43]. Secondly, the melting temperature (Tm) is an important material property that influences the sealing behavior. At the melting temperature, also the crystalline parts of the material start to melt and the material changes from a solid into a viscous state. The material will start to flow at this temperature. This is only a property for polymers containing a crystalline fraction. So Tm is not applicable for amorphous polymers, but instead uses the Tg [8, 44]. The highest seal strengths are obtained when the crystalline fraction of the polymers are melted completely at the seal interface [39].

Some examples of these temperatures are presented by Dudbridge [27]. They state that the Tm of PET is 250°C, PP 164°C and that of HDPE is 135°C. The Tg of these materials is, respectively, 80°C, -20°C and -30°C. These temperatures also explain why PET is great to use as a heat-resistant outer layer (high Tm), whereas especially PE can function as a great sealant layer (low Tm).

# **Material parameters**

Seal performance depends on the crystallinity of the polymer and its ability to diffuse after the seal interphase reaches a specific temperature [28]. To be more specific, it is dependent on the: sealant layer, film thickness, density, film material composition, molecular weight and its distribution and thermal conductivity [8, 14, 25]. A change in material supplier might even influence the performance [27].

### Heat and pressure

Pressure and heat are required to realize a seal. The pressure is necessary to bring the two films into intimate contact. The introduction of heat is necessary to increase the mobility of the polymers. Polymer mobility can refer to different concepts [43]. In this case, the polymer mobility is related to the viscosity of the material. The higher the temperature, the higher the mobility. Hydration and temperature are the main factors influencing polymer mobility [43]. Next to increased polymer mobility, the addition of heat to the system also increases the amorphous fraction of semi-crystalline polymers. If the amorphous fraction is high enough, sealing can occur and the seal initiation temperature is reached [8].

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## Diffusion

During sealing, the sealant layers must have a high mobility, so that diffusion is possible at the seal interface. This way, new polymer chains can be formed. The type of polymer chains that can melt and diffuse are of great importance for the resulting seal strength. Especially the molecular weight of the chains and the branching morphology is important. A polymer with low molecular weight chains and a high fraction of short chains will easily diffuse at the seal interface but will result in a low seal strength and the other way around. These parameters are even more important than the amorphous fraction of the material [8, 39].

# 2.3. Materials

Flexible packaging films can generally be divided into two categories based on their material composition: mono-material and multi-material. Mono-materials consist either of only one layer or multiple layers with mostly one type of polymer. Within the multi-material category, a distinction is made between films that consist of polymers and films that also contain other materials, like paper and aluminum [15].

Usually, the materials that are included in a film are thermoplastic polymers. These polymers can be softened when they are exposed to heat. There are two general types of thermoplastic polymers: amorphous and semi-crystalline polymers. Crystalline polymers solidify and melt quicker than the other types, which can result in brittle welds [45]. Requirements that may impact the material choice include: stiffness, how well the material can be sealed, transparency, processability, thickness and barrier properties [13, 46]

As mentioned before, each material included in a film should be carefully selected based on the specific functions that the specific layer needs to fulfill. Therefore, a basic understanding of the materials that can be used is required. The selection of materials that will be described are materials that are regularly used for flexible food packaging. Especially the materials that are commonly included in flexible monomaterial films will be described.

# Sealants and outer layer

The first type of material that will be discussed is the polyolefin type. Polyolefins are often used in flexible food packaging because of their low cost, processability and recyclability [47]. Limitations of polyolefins include adhesion, printability, dyeability and compatibility with other polymers. These limitations can be overcome by functionalizing the polyolefins. Functionalizing a polymer is introducing polar functional groups into the material [47]. Three of those will be discussed: polyethylene (PE), polypropylene (PP) and isotactic poly(1-butene) (PB). By far the most used materials for flexible packaging applications are PE and PP [14, 24].

The following part is mainly based on the paper from Bamps, et al. [8] in which they review seal materials used in flexible plastic food packaging.

### ΡΕ

Polyethylene (PE) is the simplest polyolefin. The base chemical formula of PE is  $(-CH_2-CH_2-)_n$ . The ethylene group (C<sub>2</sub>H<sub>4</sub>) is repeated along a chain for 'n' times [48]. These chains can be oriented in different ways, influencing the material properties. PE is a semicrystalline material, so it contains amorphous and crystalline parts in the molecular morphology. The sealing of PE starts when the amorphous fraction increases up to a certain percentage due to heating. For example for low-density polyethylene, sealing starts when the amorphous fraction is 77% [8].

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There are different types of PE. These types differ in main polymer chain orientation and on what is attached to this chain. The so-called 'branches' that are attached to the main chain most dominantly determine the density of the PE. The density influences the crystallinity. The denser the PE, the bigger the crystalline fraction, the stiffer and stronger the material and the lower the permeability [49]. Different types of PE are [8]:

- Low-density polyethylene (LDPE)
- High-density polyethylene (HDPE)
- Linear low-density polyethylene (LLDPE)
- Metallocene linear low-density polyethylene (mLLDPE)

The simplified chemical composition of these different types of PE is visualized in **Figure 2**. The closer the molecules are packed together, the denser the material. It can be observed that the chains with the lower-density versions indeed cannot be packed closely together, while the high-density version chains can.

#### LDPE

From the graphical representation shown in **Figure 2** can be observed that LDPE has long chain branches compared to the other types. This results in relatively low hot seal strengths, because the diffusion is slower and no additional forces are in play (like hydrogen bonds or ionic interactions). However, LDPE is commonly used as a sealant layer because of its good processability [8]. It is very suitable for heat sealing [14].







*Figure 2. Simplified chemical composition of polyethylene types* [8]

#### HDPE

This high-density version of PE is highly crystalline and therefore quite rigid. It has a higher melting point than the other PE versions, therefore this version is rarely used as a sealant layer [8].

#### LLDPE

Linear low-density polyethylene is produced using a catalyst. The type of catalyst that is used (e.g. Philips or Ziegler-Natta) influences the branching on the main chain. LLDPE has a similar density as LDPE, but its composition is more linear. This is due to the shorter branches that are connected to the main chain. This causes the melting temperature to decrease. This is why generally LLDPE is seen as a superior sealant layer than LDPE, although both materials are frequently used as a sealant layer in practice [8].

#### mLLDPE

A special type of LLDPE is metallocene LLDPE. This type is made using a different kind of catalyst than conventional LLDPE types. The catalysts used for mLLDPE, are metallocene based. Metallocene materials are structures that contain positively charged metal ions (e.g. cations of titanium) between two cyclopentadienyl derivatives. This results in a material that has a narrower molecular weight distribution. This causes medium to long chains to participate in the entanglement during the sealing process, which is not as much the case for conventional LLDPE. This is the reason that mLLDPE is considered to have better sealing performance than conventional LLDPE. Without long chain branches attached to the main chain, mLLDPE is difficult to include in a blown extrusion process [8].

#### PP

The second most used polymer in packaging applications is polypropylene (PP). This polymer also consists of chains, just like PE. The difference is that here the chains consist of propylene ( $C_3H_6$ ) groups

instead of ethylene groups ( $C_2H_4$ ). In industry, PP is used for rigid applications, like trays and cups, but also as lidding to seal to the rigid applications. In general, PP has a higher melting temperature than PE. This might not seem like an advantage in sealing applications, but it can be beneficial for certain applications, like pasteurization [8].

There are different types of PP used in practice. A random comonomer may be added to the main chain. It is also possible that the PP is processed in a certain way to alter its properties [8].

Examples of comonomers that can be added to the main chains of PP are ethylene and butene. The comonomers can increase material properties like transparency and flexibility [8].

PP can be processed in different ways. The main distinction that is made for the processing, is between oriented and unoriented PP. Unoriented PP has a higher heat stability, puncture resistance, impact strength, gas permeability and a lower moisture permeability than the oriented versions [22]. Different PP types that are processed differently will be presented below. Starting with the unoriented and finishing with the oriented types.

#### СРР

Unoriented PP is called cast polypropylene (CPP) if it is made with the cast extrusion process. See the section Cast extrusion for more information about this process. If PP is realized with this process, the result is a film with relatively low cost, good moisture barrier, high transparency and relatively low sealing temperature [22, 50]. CPP can be used as a sealant layer [8, 14, 17]. In general, films that are cast are considered to be tough, so they rather stretch instead of break if they are torn [21].

#### OPP

Oriented PP (OPP) is the other type of PP. Films made from OPP don't seal well using conduction sealing. Some material properties are altered due to the orientation process. OPP is considered to be a low-cost packaging material [21].

Within the orientation process, two concepts are of interest: the orientation draw direction and the stretching process. A film can be oriented in two ways: transversely and longitudinally. The stretching processes are described in the section Orientation processes. For OPP films, the tear initiation is difficult in the oriented direction. However, once the tear is initiated, the resistance to further tearing is low [22].

It is possible to only orient the films in one direction (machine and transverse direction) and in both (called biaxially orientation). For PP that gives three options of an OPP: machine direction oriented PP (MDOPP), transverse direction oriented PP (TDOPP) and biaxially oriented PP (BOPP) [51].

The relaxation behavior of oriented molecules, when the temperature increases, is a risk for oriented films. This phenomenon can be observed as shrinkage of the film. Relaxation happens if the temperature increases and the oriented molecule fraction of the material drops. It therefore is important to ensure that during the sealing of oriented films, the oriented molecule fraction is kept below the relaxation temperature. If that is the case, no relaxation is occurring [52].

#### MDOPP

Machine direction-oriented PP (MDOPP) is a PP film that is only stretched in the machine (also called longitudinal) direction. This for example makes it more likely that tears will occur in the transverse direction than the machine direction. This is for example useful in tape applications, where MDOPP is typically applied [51].

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#### TDOPP

Perpendicular to the machine direction is the transverse direction. If a PP film is oriented in that direction, it is called transverse direction-oriented PP (TDOPP). A typical application of TDOPP is shrink sleeves. These sleeves are placed around for example a bottle and shrink only in the transverse direction to tightly surround the bottle [51].

#### BOPP

If the PP film is stretched in both the machine and the transverse direction, the film is called biaxially oriented PP (BOPP). This can be realized using several processes, as explained in the part Orientation processes [22]. BOPP can either be created by a sequential or a simultaneous orientation process [51].

PP is biaxially oriented to increase the toughness, stiffness, transparency, oil resistance, moisture permeability and gas permeability [37]. This material can be realized at a relatively low cost, but BOPP cannot effectively function as a sealant layer [22]. The cause of this is the high seal initiation temperature. This same property also forms a possibility for BOPP to become a heat-resistant outer layer within a film [14, 28].

#### ΡВ

Polybutene consists of a chain of 1-butene groups ( $C_4H_8$ ). The morphology of a 1-butene molecule consists of two groups with each two carbon atoms. In the first group, the C atoms are connected with a double bond. The second group is connected to one carbon atom of the first group. In poly(1-butene) (PB). PB is created by opening the double bond and connecting it to another 1-butene group with opened double bonds.

There are three types of poly(1-butene) depending on the morphology of the branches of the polymer. If all branches point in the same direction, the polymer is called isotactic. If all even-numbered branches face one way and the uneven-numbered branches face the other way, it is called syndiotactic. If there is no logical order in the direction the branches are pointing, it is called atactic [8]. See **Figure 3** for a graphical representation of what these different morphology types look like.



Figure 3. Simplified visualization of isotactic, syndiotactic and atactic [8]

PB is usually used as an addition in the sealant layer to form an easy peelable seal. This is also done in some mono-material films. To be more specific, PB is used as contamination in the sealant layer to create a controlled contamination easy peel film [8]. Sängerlaub, et al. [29] mention that this is for example possible in combination with many types of PE sealant layers.

#### Ethylene copolymers

Previously, it has already been mentioned that PP can have copolymers added to the main chain. This is also possible for PE to create polyethylene-co-vinyl acetate (EVA), polyethylene-co-acrylic acid (EAA) and polyethylene-co-methacrylic acid (EMA). Each of these copolymers can be used as a sealant layer.

EVA is a copolymer that contains ethylene groups and vinyl acetate groups in its chains. It is usually used as a blend with PE in the sealant layer. This has advantages like a lower seal initiation temperature and a wider seal plateau window [8].

The copolymer that consists of ethylene and acrylic acid groups is referred to as EAA and EMA combines ethylene and methacrylic acid groups into one polymer. Both EAA and EMA are used in sealant layers and as adhesives in laminated films. They adhere well to polar materials like PET, aluminum and paper. These materials can create hydrogen bonds, which can for example increase the hot tack strength. An example of what these materials are used for in practice is to create a peelable lid with these materials included in the sealant layer [8].

It is also possible to add ions to the methacrylic acid groups in EMA polymers. The material that is created is called an ionomer. Examples of ions that are used in practice within the packaging world include sodium and zinc. The resulting material has an increased strength potential [8].

#### PLA

Polylactic acid (PLA) is a material made from renewable organic materials (for example starch from corn) and is designed to be compostable [8, 53]. On its own, PLA is a brittle material. This can be adjusted when blended with other polymers. PLA blended with other polymers is a material that is expected to be more and more applied in industry, according to Bamps, et al. [8]. It is a bioplastic that is suitable for flexible packaging applications and is used in industry in for example sweet wrapper applications [54].

The intended end-of-life scenario for PLA is industrial composting. However, it is unlikely that the PLA packages will end up in composting facilities. They are more likely to be disposed of together with general waste [53]. This partly compromises the advantages PLA has in practice.

#### PET

The heat-resistant outer layer that will be discussed is poly(ethylene terephthalate) (PET). It consists of chains of repeating ethylene and terephthalate groups. This material is regularly used in packaging applications because of its lightweight, appearance and barrier properties. PET is typically applied in rigid plastics, like bottles. However, it is also possible to include PET in a film as a heat-resistant outer layer. It is not suitable as a sealant layer due to its low flow behavior and high melting temperature [8, 21]. PET can be processed to alter its properties. An example of PET processing is the biaxial orientation of the material, which results in BOPET.

In the flexible plastics context, PET is considered to be more difficult to recycle than PE and PP. That is due to the recycling process. PE and PP are separated, while PET ends up in a mixed stream with other types of plastics during the sorting phase [15], although recycling of PET is possible [18, 55]. It is even possible to create a film that can be classified as a 'mono-material' and that contains recycled PET [56]. PET is commonly used as material for a rigid tray with flexible lidding.

#### Barrier

### EVOH

An oxygen barrier is often required within a film since many polymers have low oxygen barrier properties [38]. A material that can be incorporated into the film to realize the required barrier property is ethylene-vinyl alcohol (EVOH). This material has excellent oxygen barrier properties and is therefore often incorporated in food packaging films. EVOH is used so regularly, due to its recyclability, transparency and its high processability performance [38, 57]. The main drawback of EVOH is that it tends to absorb moisture and then lose its barrier properties. Therefore, EVOH can be effectively used

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in the barrier layer, but it requires layers on both sides that have high moisture barrier properties [38, 58]. EVOH also requires a layer attached to it to be sealed, since EVOH is not suitable for heat sealing [14].

EVOH poorly adheres to most polymers except nylons. That is why often a tie layer is required around the EVOH layer within a film [38]. This tie layer can be realized by combining acid anhydride molecules with polyolefins through grafting [59]. Grafting is defined as modifying the properties of a backbone polymer by copolymerization [60]. This is also the case for the LDPE and EVOH film. The different layers of this film will thus be LDPE-tie-EVOH-tie-LDPE [38]. This can be realized using coextrusion or lamination, see the section Film production for more information about these processes. [57]. The tie layer is usually a few micrometers thick [15, 44]. Maes, et al. [57] show in their research that it is also possible to create a blend of PP and EVOH. This way the EVOH can be incorporated in a film without the need for additional tie layers.

Ge, et al. [38] illustrate an example of a film with EVOH in it. They give the example of low-density polyethylene (LDPE) as a sealant and an outer layer with EVOH on the inside. This film seems suitable for applications like food packaging since LDPE slows down the diffusion of water molecules into the EVOH and EVOH blocks the diffusion of oxygen molecules to the inner LDPE layer.

Alternatives for EVOH that also have high oxygen barrier properties include polyvinylidene dichloride (PVDC), metalized aluminum and aluminum foil [27].

# 2.4. Mono-materials

Packaging solutions should comply with more and more requirements nowadays. Especially requirements concerning the impact of a package on the environment throughout its entire lifecycle are increasing. This is also strongly encouraged by regulations, as described in the chapter Recycling classifications [13, 20].

There are different ways one can try to fulfill these requirements. One approach is to look into bioplastics. The demand for bioplastics has been rising [8, 61]. Two concepts are associated with bioplastics: biodegradable plastics and biobased plastics. Biobased plastic is not made from fossil resources, but from biomass (e.g. from crops) which can almost reach the same properties as fossil-based plastics [62]. Biodegradable refers to the end-of-life of the plastic. Biodegradable plastic is designed to decompose and convert mainly into carbon dioxide, water, new microbial biomass, mineral salts and methane [63].

Although the use of bioplastics sounds like a great alternative to conventionally used plastics, Escobar and Britz [61] question the impact of the shift towards biobased plastics. They conclude that promoting biobased plastic production instead of conventional fossil-based production is not effective when looking at the economic, social and environmental impacts. Carullo, et al. [13] also mention some limitations; like poor processability, a lack of sorting options and weak composting infrastructure; for biobased plastics. For biodegradable plastics, the Directorate-General for Environment [63] advises that these plastics should only be used when reduction, reuse and recycling are no options. Therefore, this does not seem to be the ideal solution when trying to comply with increasing sustainability requirements for packaging solutions.

Another way arising requirements concerning environmental impact can be fulfilled, is by using monoinstead of multi-materials. Carullo, et al. [13] state that these mono-materials seem to be a valid replacement for multi-materials and offer some examples of successful replacements. They also show in the life cycle assessment (LCA) that they performed, that mono-materials consistently show lower impact on the environment than multi-materials. This is confirmed by research from TNO [56]. This is only the case if a large enough portion is indeed collected, sorted and recycled. One research mentions that the benefits of recycling can be seen if at least 69% is collected and sorted [56].

An ideal scenario for minimal environmental impact when looking at recycling, would be to have a recyclable mono-material monolayer film as packaging material [15, 20]. However, when trying to decrease the impact of a consumer good on the environment, not only the suitability for recycling but also the functionality of the packaging should also be considered. A switch to a mono-material monolayer packaging might for example have negative consequences for the environmental impact of the product. This might be the case if the material switch means a shorter shelf life and thus more food spillage.

So the ideal combination when looking at the environmental impact of a consumer good, is sufficient functionality combined with material that can effectively be recycled. To achieve this, it is good to know that a flexible food packaging mono-material does not necessarily need to consist of only one material. So it is possible to configure a multi-layer film that is still considered to be 'optimal' suitability for recycling (e.g. doesn't cause disruptions in the recycling process) [15]. As shown by Pettersen, et al. [20] it is possible to create a multi-layered mono-material film that shows a similar shelf life as the multi-material alternative. In chapter 2.7 Recycling, a more elaborate description of what makes a material suitable for recycling can be found.

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Another challenge that arises from mainly using one type of material, is the sealing. Usually, a packaging film that is sealed with conduction sealing has a heat-resistant outer layer and a sealant layer with a low seal initiation temperature as described before. The difference in melting temperature between these two layers should be high enough. The sealant layer should be sealed at this temperature without melting the outer layer. Since the material type of both layers is probably the same for mono-materials, the melting temperature does not lie as far apart as dissimilar materials, as is also described by Hauptmann, et al. [17].

Next to the challenge that the melting points of the inner and outer layer should be far enough apart within a mono-material, another challenge is described in the literature regarding this topic. Koo Sin Lin, et al. [64] mention that replacing PET with BOPP as a heat-resistant outer layer will result in less heat penetration to the sealant layer. This might cause problems when trying to replace a multi-material film containing a PET layer on the outside with a mono-PP film.

As already mentioned, successful implementations of mono-material alternatives for multi-material films are reported. Most of these films consist of a two-layer structure with two different types of PE or PP with a barrier layer in between. The mono-PE structure that they mention shows promising properties, like low water vapor and oxygen permeability. Another example that is given in the literature is a mono-PP structure with CPP as a sealant layer and BOPP as heat resistant outer layer [17].

# 2.5.Reclosable package

Some flexible food packages can be reclosed once the original seal is opened. This might prevent food waste. There are different ways to make such a packaging reclosable and one of them is by adding a sticker. A fold is created in the flexible film and it is kept closed by a sticker. The sticker sticks to the film through an adhesive and is peelable. It is good to note that as soon as the seal is opened, the best-before date is usually compromised [27]. A reclosable package with a sticker is especially useful in combination with an easy peel seal. The seal can be opened without destroying the package and can then be reclosed using the sticker. Note that the sticker also has some requirements regarding recycling, see chapter 2.7 Recycling for details about that topic. Examples of reclosable packages can be seen in **Figure 4**.



Figure 4. Examples of reclosable packaging systems [65]

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# 2.6.Film production

To understand what the differences between certain films are and why they behave differently, not only the materials used are important. Also, the production process of these films influences how the films behave. Usually, a packaging film consists of multiple layers, to comply with different packaging requirements [49]. Multilayer films can be realized using thermal lamination, coatings or coextrusion technologies [49]. Lamination and coextrusion techniques will be briefly described in this chapter. The effect each process has on the film is also elaborated on.

The choice between the lamination process and the coextrusion process is dependent on several factors. It is for example dependent on the product needs, how it is processed, the presence of a print, the amount of required layers and film thickness [21].

### Lamination

The process that combines two or more films into one by rolling, is called lamination. Usually, this is done by adding an adhesive in between the different layers, but it is possible to do so without.

This film production process has the risk that the different tensions of the layers may cause problems. It is common for laminated films to curl for example. This might cause problems when the cut edge needs to be flat to be processed. Another limitation of this technique is that if water-based adhesives are used, long drying times are required. It is also possible to add solid adhesives [21]. As an example, the adhesive used to combine EVOH and LDPE is based on graft polymers like PlexarTM (Equistar Plastics) [38].

When two materials that need to be combined both have heat-sealing properties, they can be joined through thermal lamination. In that case, an adhesive might not be required, since the films are diffused in one another. The downside of this process is that the film may slightly shrink or stretch because of the combination of heat and tension. This technique is for example often employed in book cover production [21].

# Coextrusion

Another way to produce multilayer films is by coextrusion. There are two main coextrusion processes: cast film extrusion and blown film extrusion. For both processes, the resins are heated and subjected to pressure whereby the molten resin is forced through a narrow slit. At this point, different layers of material are combined. The film that comes out either has the form of a bubble or is flat. If the film is a bubble, it can be used for blown film extrusion. If it is flat, the process is called cast film [21, 66]. It is possible to create a coextruded film that consists of different materials [66]. Coextrusion with polymers can be complicated since the compatibility needs to be taken into account [49].

There are some differences between films made with cast extrusion and blown extrusion. The main ones are that the clarity of the film, investment cost of the machinery and output efficiency of a film made with cast extrusion are higher, while the transverse strength and the width flexibility are lower than a film realized with blown extrusion [21, 66].

A problem that might occur during the coextrusion process, is interfacial instability. This significantly decreases the clarity of the film. It is influenced by the layer thickness, viscosity, elasticity and interfacial tension of the films [49].

Films produced with both techniques might be stretched in the transverse and the machine direction orientation to increase the film performance (e.g. increased strength) [21].

#### Blown extrusion

For blown extrusion, molten polymers are forced through a circular die under high pressure and temperature. The circular tube that comes out of the die is blown up by the takeoff system to create a bubble. Usually, the diameter of the die is much smaller than that of the blown bubble. The air pressure within the blown bubble is maintained to make sure the bubble does not collapse. After the takeoff system, the blown film goes through the blown extrusion tower and is rolled up at the end to create a flat film from the bubble [21, 49, 66].

By varying the air pressure, winder speed and screw speed of the blown extrusion process, a certain degree of orientation (as well in transverse as in machine direction) can be accomplished [21, 67].

#### Cast extrusion

The cast extrusion process starts at the feedblock. This part layers certain resins in the right order and feeds the molten materials to the cast extrusion die. Here, the molten material is pushed through the die which creates a film of material. This hot film is rolled over a cooled cylinder. Attention must be paid that all layers are evenly spread over the entire film [66]. An example of a cast film application in practice is thermoformed trays [21].

### **Orientation processes**

Another process that might be included in the film production, is an orientation step. As already mentioned, a film can be oriented in the transverse and the machine direction. A film can be oriented in one of these ways or both. If a film is oriented in both, this can be done simultaneously or separately [51].

A film is oriented in the machine direction by different rolls that move faster and faster thereby stretching the film in the machine direction [21, 51].

If a film is stretched in the transverse direction, this is usually done by fixating a film on both ends and letting it pass through an oven with various temperatures. This is usually done using a tenter frame. A tenter frame is a frame that grabs the two sides of a film and takes it along the frame. The frame gets wider and wider, thereby stretching the film in the transverse direction [21, 51].

In case the film is oriented in both ways simultaneously, this can be done with a tenter frame, a tubular process or with the blown extrusion process, which has already been briefly described in the previous parts [22, 51].

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# 2.7.Recycling

One of the actions taken up in the Circular Economy Action Plan from the European Union is the (mandatory) uptake of recycled plastics in new products, like packaging systems. The same action plan also states that waste reduction is one of the priorities [68].

The European Commission has even set a goal to ensure that all plastic packaging in 2030 is either reusable or can be recycled cost-effectively [69]. Therefore, they encourage economic growth by minimizing environmental impact [14].

When trying to minimize the environmental impact of a product, certain actions have more impact than others. One source states the order of actions is as follows: reuse, reduce and recycle, recover and dispose with reuse having the most impact and dispose the least [16]. The source referred to here is The Circular Economy for Flexible Packaging (CEFLEX) which is a European collaboration of organizations, associations and companies invested in flexible packaging. This source is introduced since it will be referred to more often throughout this thesis. Another source states the actions should be refuse, rethink, reduce, reuse, repair, refurbish, remanufacture, repurpose, recycle, recover, reconcentrate and redistribute with refuse having the highest impact and redistribute the least [70]. What they agree on and what is good to note, is that several steps can have more impact than the recycling action.

So when trying to minimize the environmental impact of a packaging system there different possible actions need to be considered. Multiple actions can be taken simultaneously, but it is good to consider what actions can have the most impact. The validity of the environmental hierarchy of different actions mentioned before is illustrated by Nonclercq [14], who states that if a flexible packaging system is disposed and a non-flexible one is recycled, the flexible system still often has a lower environmental impact.

The above-mentioned order is also the reason why for example government regulation mainly focuses on waste reduction [71], instead of only focusing on promoting recycling. This chapter will investigate the recycling of plastic flexible packaging films for which recycling has the least environmental impact. Some legislation within the European Union will be covered, that explains the demand for monomaterial packaging films with high barrier properties.

The end-of-life of a packaging system consists of a few phases. For multi-material flexible plastic packaging systems, Kaiser, et al. [18] present three stages that together form the end-of-life of a product. Starting with the 'collection' and the 'sorting' phase and finishing with the 'reprocessing' phase. Note that this is only valid if the package is properly disposed of, otherwise it is possible that the packaging ends up in a landfill. Each stage will be briefly introduced, but the focus will be on the reprocessing phase, which includes recycling.

# Collection

The collection of post-consumer plastic is the start of the recycling process. This can for example be done by using containers, bags or bring systems [18]. If the post-consumer plastic is brought to a proper site, the sorting can begin. To avoid the plastics ending up in a landfill or incineration sites, plastics should be collected separately from other post-consumer materials [14].

# Sorting

Within the sorting phase, different kinds of materials are separated and cleaned. This phase is necessary to end up with high-quality material after the recycling process. If done properly, recycled

PE can for example almost reach the quality of virgin PE (although food contact is not allowed with post-consumer recycled PE [72]) [18]. Therefore, the output of the sorting phase must be material that has sufficient quality and volume. This is required to also make recycling economically feasible [14].

The different materials of a multi-material film can be separated before recycling. The separation can be realized with dissolution and reprecipitation or with delamination. With the first separation technique, the material is solved in a liquid and later dried [73]. The delamination of a film consists of separating one layer from the other. This can be performed physically, chemically and mechanically. It is also possible that a film is recycled without separating the different materials. Recycling techniques that work with this input often require additives [18].

## Reprocessing

Reprocessing of sorted polymers can be done by energetic utilization and recycling. The latter option will be discussed in the next part. Energetic utilization means that the polymer is incinerated and during this process returns some energy.

### Recycling

In this part, some general statistics about recycling are presented to understand the current recycling context. The scope is within Europe. In 2020, close to 40% of the used plastics are used in packaging applications. Only 40% of the packaging materials are recycled [20, 74]. In 2020 42% of the plastic was recycled in the Netherlands [75]. In 2010 flexible plastics were responsible for 21% of package sales [14].

A part of the explanation why such a small fraction of the packaging materials are recycled is the recycling of multi-material packaging systems is difficult. Therefore, these systems often end up at a landfill or incineration sites [20].

The focus of this thesis is on flexible plastics. It should be noted that most recycling processes focus on rigid plastics. This sometimes means that the flexible plastics are neglected and do not end up in a stream that is ready for recycling [14].

### **Recycling mechanisms**

There are two general ways a material can recycled; mechanical or chemical recycling. The input is sorted post-consumer plastic and the output is recycled raw material [18]. During recycling, degradation of the material quality is inevitable but can be limited [14]. How this material goes from sorted post-consumer waste to recycled raw material, is described in this part.

In mechanical recycling, the polymer chains are not entirely destroyed. The material is processed with physical methods, like shredding or melting which causes some degradation of the material. Packaging materials are generally quite complex, which forms a challenge for mechanical recycling, although some strategies are described in the literature [18, 57].

The recycling processes that are referred to as chemical, bring back the material to a monomer level or an oil. These substances can be used to create new polymers.

### Practice

### Sorting in practice

A description of what the sorting and recycling process of plastic packaging looks like is given in this section. It is mainly based on the research from Nonclercq [14] and the report from CEFLEX [16].

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When post-consumer plastics are collected, they first need to be sorted. The input for this process is shredded into particles of around 6,5cm. In the sorting processes, the material stream will be separated. This is done to end up with material that is suitable for recycling. Some examples of often employed separation steps include: drum sorting, ballistic separation, magnetic sorting, Eddy Current sorting, air classification and near-infrared (NIR) technology [14, 16]. After these separation steps, the material should be sorted and ready to enter the recycling stream.

#### Drum sorting

The drum sorting system consists of a rotating cylinder. This separates too-large and too-small packages from the waste stream that are suitable for sorting [16].

#### **Ballistic separation**

The packaging properties: shape and ability to bounce will be exploited to separate them at the ballistic separation step. This will separate flexible and flat packaging systems from rigid ones [16].

#### Magnetic sorting

This step will take out the magnetic metal parts from the waste stream using magnets. Together with the Eddy Current separation step, most of the metals will be separated from the plastic stream [14, 16].

#### Eddy Current sorting

This separation step exploits the electrical conductivity of the different materials in the waste stream to separate the metallic from the non-metallic materials [76, 77].

If flexible packaging contains an aluminum layer of more than 15 microns, the Eddy Current is likely to extract this package, while this would not be excluded at the magnetic sorting step [16]. A limitation of this step is that if a flexible packaging system for example only contains aluminum foil with a thickness of a few microns, this will not be extracted by the Eddy Current separation step [14]

#### Air classification

In the air classification step, lightweight materials are separated from the heavier material. Materials that are separated are for example films and paper [14].

#### Near-infrared (NIR)

Near-infrared (NIR) can see which package contains what material(s) using a camera and can separate them accordingly. An output of this step is a stream of single plastic resin [14, 16].

NIR technology is not suitable for the separation of films, unlike rigid plastics. The main reasons for this are that it is hard to eject lightweight materials, like films, and because most films consist of PE [14].

#### Recycling in practice

Recycling in practice also includes sorting steps. The output of the sorting process can be used as an input for the recycling process. The first step in the recycling of sorted material is grinding. The material is reduced in size. Sometimes flexible and rigid plastic fractions are combined in this step. Afterward, the grinded material is washed to remove contaminants. The washing also achieves a second goal, since the water bath also separates certain plastics based on their density. PP and PE are separated from the rest of the plastics since their density is below 1,00g/cm<sup>3</sup>. The other materials have a density higher than water. The last step of the recycling process is the extrusion of the dried material [14, 16].

An example of a promising way to chemically recycle multi-material packaging systems is by pyrolytic decomposition [18]. During the pyrolysis process, a sample is heated which causes the polymers to

decompose [78]. Although the environmental impact of this recycling method might compromise the opportunity.

#### Recycling of mono-materials

In chapter 2.4 Mono-materials, it has been explained what a mono-material is. Mono-materials have the potential to have a high recyclability performance [13-17]. This part will briefly describe why this is the case.

The main reason for this is that mono-materials do not require material separation of the packaging system during the sorting. This simplifies the recycling process compared to multi-material alternatives. These alternatives are more difficult or even impossible to recycle into quality recycled material as mentioned before [14, 18].

As has been stated in chapter 2.4 Mono-materials, mono-material films do not have to consist of only one material. For example, often a barrier layer is included in the film that is a different type of material than the sealant and outer layer. The material EVOH is often included in these films (maximum 5% of the weight of the package). It has a high oxygen barrier and is more suitable for recycling than alternatives like aluminum foil and metalized films since it dissolves in the water [14].

A limitation of mono-materials mentioned by Golkaram and Heemskerk [56], is that only when 69% of the mono-materials is collected and sorted a difference in environmental impact can be seen between mono-and multi-material films. Another limitation is that the use of mono-materials brings new challenges with it, for example regarding recycling infrastructure [14, 17, 56]. The recycling infrastructure is for example currently not suitable to retrieve a mono PP stream from the recycling process, while this is desired to create high-quality recycled PP. Despite these limitations, the use of mono-materials is stimulated by regulations in the form of for example by giving discounts on material processing prices [79].

#### Limitations of recycling

Some limitations should be considered when looking at recycling. The main challenge when looking at the sorting step of recycling is to achieve sufficient quality and volume. This is required to make the recycling economically attractive. The challenges are mainly caused by the difficulty of separating multi-material packaging and adequate collection and management systems [14, 61].

Another limitation has already been mentioned implicitly a few times. To make this limitation explicit: most multi-material packaging systems are not recycled but incinerated or landfilled [18]. This is caused by their poor recyclability and inadequate collection and sorting.

A mono-material packaging system ideally would be recycled into a new packaging solution to form a closed loop. This is however not realistic because of the food safety concerns associated with recycled content, as will be elaborated on in the next part. A viable way however to recycle flexible plastics is to apply the recycled content to other applications [14].

#### Legislation related to recycling of plastics

There are also some limitations of recycling connected to the legislation. These will be discussed in this part. In Europe, recycled plastic is only allowed on the market if it comes from a recycling process that is authorized by the European Union. The output of this system is checked by a system to guarantee the quality of the recycled material [80].

The last limitation that will be mentioned in this part, is that the input material of the recycling process must meet a few (strict) requirements for the output to be suitable for food contact. First, the input material must be plastic that is produced according to legislation on plastic food contact material.

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Secondly, the material must either come from a closed and controlled chain or it must be demonstrated that the recycling process can reduce the contamination concentration to such an amount that it does not pose a risk to human health [72, 80]. Note that both sources are regulations from the European Union. For example, the outcome of mechanical recycling of post-consumer PET is mentioned by the European Union as being suitable for food contact [72].

#### Recycling infrastructure

Carullo, et al. [13] suggest that when trying to realize packaging with low environmental impact, more efficient and readily available options should be considered. This means that the context of a certain packaging is of importance and should be included in the end-of-life scenario. An example of this is the recycling infrastructure that is already present in a country. When for example, trying to realize a certain ideal scenario, but this scenario requires a different way of waste collection, sorting and recycling, the overall environmental impact of the realization of this scenario will probably be negative.

That the recycling infrastructure influences the environmental impact of packaging is illustrated by an example of packaging made from PET. This material is currently not seen as 'optimal' for recycling, while a paper suggests that it is [15, 56]. During the sorting phase, PET cannot be separated from other plastics, while PE and PP can. The difference that explains the difference in conclusion, is that one takes into account the recycling infrastructure and the other doesn't.

To conclude, it is important to understand what the recycling context looks like. For that reason, recycling in the Netherlands, Germany and Europe will be described. This will be described by looking at when a packaging is classified as 'optimal' for recycling. The focus is on flexible plastic packaging.

#### **Recycling classifications**

Different classifications exist to evaluate the compatibility of a packaging system for recycling. This differs slightly per country, although these differences are rather small within Europe. The classifications for the Netherlands, Germany and Europe will be described. This is done for flexible plastic packaging systems.

#### Europe

The classifications within Europe are based on the report from [16]. In this report, it is stated what requirements a packaging system should have to be compatible with mechanical recycling of PE, PP and polyolefins. A flexible package is compatible with mechanical PE recycling if at least 90% of the material consists of a type of PE. No more than 5% may be one of the following materials: EVOH, PVOH, AlOx, SiOx or Acrylic. For the rest, no aluminum foil or paper may be included in the film, the package should be bigger than 20 by 20mm, the color should be clear or with natural pigments and the use of adhesive should be less than 5%. The same 5% rule counts for the used inks. These color requirements are there, because transparent material can be recycled into transparent material, but colored material cannot be recycled into transparent material [15]. The material of the label should also be PE. Lastly, the density should be below 1g/cm<sup>3</sup>. This can be explained by the density separation step after the washing. The PE should float to end up in the right material stream. The percentages mentioned in this section are all percentages of the total weight of the packaging system [16].

The same requirements count for the compatibility for mechanical recycling of PP and polyolefins, but instead of PE read PP or polyolefins, respectively.

The waste legislation within Europe usually consists of the party that brings virgin material to market paying a fee (so-called polluter-pays principle). This fee finances the recycling infrastructure [81].

In their report, CEFLEX [16] also mentions what type of recycling is preferred when trying to achieve a circular economy. Mono-PE and mono-PP streams are preferred over the mixed polyolefin stream. The mechanical recycling of PE and polyolefins is already in use in some European countries, whereas the recycling of PP is being developed. It is expected that the recycling of mono-PE will be developed further to include more streams, like PE colored and PE natural. Furthermore, it is mentioned that mono-PP recycling will be developed further for certain market applications.

#### Netherlands

In the Netherlands, KIDV [15] created a guide checklist to see how well a certain package can be recycled. This foundation distinguishes four categories of recyclability: optimal, reasonable, limited and not recyclable. The recyclability category a certain package gets is based on how well it can be recycled by the recycling sites within the Netherlands.

As already described, before recycling can happen, the material should be sorted. In the Netherlands, the material is first separated based on what type of packaging it is (e.g. flexible plastics, board or metal). From the flexible plastics, PE and PP are separated from other plastics based on density differences using a water bath. The PE will end up in a mono stream, so a stream with only PE packages. Sometimes this is also done for PP. The rest of the plastics are recycled in a mixed stream, including for example PET [15].

There are some requirements for a packaging system to be evaluated as 'optimal' for their recycling compatibility. It is considered to be 'optimal' for recycling when the package is bigger than 30mm by 30mm. Furthermore, at least 90% of the packaging material should be PE, it should not contain an aluminum layer with a thickness larger than 1 micrometer and the average density of the packaging system should be less than 1g/cm<sup>3</sup>. Next to this, the color of the system should not be black, the label should consist of the same material as the packaging system and the system should not contain a barrier layer. If the packaging system does include a barrier layer, like EVOH, SiOx or AlOx, the compatibility for recycling is seen as 'reasonable'. The same counts if the main material is PP. [15].

For the labels and sleeves, also some requirements should be met. A label or sleeve is suitable for recycling in a flexible packaging application if the material is the same as the main component of the packaging system. Furthermore, no more than 30% of the surface of the package should be covered by a label or sleeve. No black and metalized layer should be visible. Direct printing is allowed if no more than 30% of the surface is covered [79].

Note that this is different than the guidelines offered by CEFLEX [16]. Especially the observation that only PE and not PP is classified as 'optimal' for recycling. The same counts for the barrier layer EVOH, which is considered compatible for recycling in Europe, but not 'optimal' for recycling in the Netherlands.

#### Financial stimulation

The Dutch government body tries to stimulate the use of more recyclable materials. This is for example done through financial incentives. There is for example a possibility to get subsidies for changing the production process to allow for materials that are easier to recycle. There are two possibilities to apply for these funds. One is to change the current packaging equipment to allow the creation of packages that fall into the category 'optimal' for recycling, where the current packages fall into another category. The rules of the other option are the same, with the only difference being that the equipment can be changed to create packages that can be 'reasonably' recycled [15].

Next to this, the Packaging Waste Foundation (Afvalfonds verpakkingen [79]) gives discounts to stimulate the use of packages that are easier to recycle. If a company brings a product to market with

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virgin packaging material in it, it has to pay a certain material rate. For flexible packaging, this is  $\leq 1,32$  per kilogram. If the packaging fulfills a few requirements, this price can go down. These requirements for flexible packaging systems are that it consists of PE or PP for at least 90%, that the color is transparent, white or natural, that the label is small in size and of the same material as the main package, that post-consumer recycled content is used and that the recyclability is qualified as 'suitable'. Per requirement, a 10 cent discount is given. So in total, a discount of 50 cents per kilogram can be given [79].

#### Germany

In Germany, the packages that are brought into circulation should be reported by the producer. This can be done through the so-called 'dual system'. By signing a contract there, a producer is no longer obliged to take back the packages that he brought in circulation. The fee that one has to pay is determined by the kind and the total weight of packaging material brought into circulation [82]. This is similar to the Dutch regulation [79].

In Germany, the foundation 'Zentrale Stelle Verpackungsregister' (ZSVR) or in English 'Central Agency Packaging Register', oversees that companies register their packages. This foundation also published a document in which minimum requirements about the suitability for recycling are stated. This will be used as the main source to see what classifies a product as suitable for recycling in Germany.

The packaging system should fulfill a few requirements before it can be labeled as suitable for recycling. For flexible packaging, the main materials that can be recycled are PP and PE. For those materials, a collection, sorting and recycling infrastructure is in place. The higher the PE and PP fraction in a package, the more can be recycled. The plastics may not be aluminized and the overall density should be below 0,995g/cm<sup>3</sup>. Lastly, films are incompatible and thus not suitable for efficient recycling in combination with the following materials: fiber-based labels (that cannot be removed by cold washing), PA layers, PE-X, PVDC layers, other layers that are not PE, PP, EVA, EVOH, SiOx, AlOx or metallization. Furthermore, polyolefin (e.g. PE and PP) material should not be used in combination with silicone [83].

These requirements are all very similar to the Dutch legislation, with one remarkable exception. German rules state a few minimal requirements for a package to be suitable for recycling. One of these rules is that a package can consist of 90% of a combination of PE and PP. In the Netherlands, this would only be the case if it consists of more than 90% of either PE or PP, not the combination. Another minor difference is that flexible plastics containing aluminum are assigned to the aluminum fraction, unlike other European countries [18].

#### Future (of European) legislation

As already mentioned, general regulations are presented by the European Union and each country has its specific legislation. To see what future legislation will look like, the documents from the European Commission are interesting to look at. They presented some legislation for the coming years in Europe.

A few interesting pieces of legislation from the European Commission are listed here. First, from 2030 at least 30% of the weight of a plastic packaging system must be recycled post-consumer waste if the package mainly consists of PET and is contact sensitive. The same counts for other plastics than PET, but then a minimum of 10% recycled post-consumer waste should be included in the packaging. Secondly, in 2040, the minimum fraction of a packaging system that should consist of recycled post-consumer waste is 50% for contact-sensitive plastic packaging systems. For non-contact sensitive plastic packaging systems and single-use plastic beverage bottles, the minimum fraction that should
consist of recycled post-consumer waste is 65%. Both regulations do not apply to compostable plastic packaging systems [69].

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### 2.8.Easy peel

Seal strength is the force required to pull apart a seal [27]. Based on this seal strength, there are two types of seals: a lock seal and a (easy) peel seal [23]. The difference between the two types is that the easy peel seal is easy to open for consumers and the lock seal is not. The easy peel seal is seen as a major advantage for the consumer [27] and therefore is applied in a broad range of applications [84]. If a seal is designed to be easy to open, the seal will show lower seal strengths [8, 31]. It should be noticed that a seal should have a certain seal strength to survive throughout the supply chain. For most packaging applications there is a window in which there is a right balance between the seal strength and openability [27].

An easy peel seal is a seal that can be opened manually and does not require any tools, like scissors or a knife. The seal must also be able to be opened with constant force and without destroying the package [29, 85]. To make the easy peel quantifiable, Sängerlaub, et al. [29] defined a seal as easy peel if the peeling force is less than 15 to 20 N/15mm. They differentiate different kinds of (easy) peel seals in their research. They classify a seal as soft peel (1-6 N/15mm), easy peel (6-10 N/15mm), peel (10-20 N/15mm) and strong peel (>20 N/15mm). The strong seal is not seen as an easy peel, since the packaging material is destroyed when the seal is opened.

It is good to note that one requirement for an easy peel seal is that the user should be able to open the package manually, so without using any tools. This requirement is ambiguous since this is different depending on the user. Two examples of parameters that influence this are age and gender [29].

#### Creation

Generally, easy peel seals can be created using one of the following techniques: controlled contamination, dissimilar resins and controlled delamination [23, 46]. Controlled contamination adds an incompatible polymer to the sealant layer. These polymers will create so-called 'islands' in the sealant area, preventing a strong seal from being formed. Dissimilar resins seals two different materials that together cannot create a seal that is too strong. Controlled delamination seals the sealant layers together initially form a lock seal. However, the sealant layer can be detached (delaminated) from the next inner layer [27, 29, 46]. The advantage of this technique is that sealing conditions don't affect the peeling force [29].

#### Observation

When observing easy peel seals that have been peeled, three types are differentiated: adhesive, cohesive and burst peel. In Figure 5 a simplified representation of the different peeling types can be observed. The cohesive peel is usually created through the controlled contamination technique. The peel takes place at a random spot within the sealant layers, which is visible as some residue on the peeled interface. The adhesive peel is peeled apart at the sealing interface. No material is left behind on the peeled interface, which gives this



*Figure 5.* Three of the seal failure modes presented by ASTM F88 [3]

type a clean look. The last type, the burst peel, can be observed after delamination [29].

#### Easy peel through controlled contamination

Usually, an easy peel seal is created using the controlled contamination technique. An incompatible polymer is introduced in the sealant layer, which leads to weak intermolecular bonds. Due to the difference in crystallization temperatures and surface energy, small islands are formed within the sealant layer while cooling. This decreases the peeling force, thereby making the seal easier to open [29]. An example of a contaminant that can be used in an LDPE sealant layer to make it easy to peel is iPB-1 [84].

There are, however, also some limitations to creating an easy peel through controlled contamination. To start, a film with a controlled contamination peel mechanism is more expensive than the same film without these contaminants [27, 84]. Thus, the sealant layer (which includes the peel components) must be kept as thin as possible [84]. Next to this, a film with controlled contamination needs to be thicker than a film without [27]. These films are also more sensitive during processing because the sealing window is smaller. If the temperature is too high for example, a lock seal will be created [27].

Lastly, a film created with controlled contamination has some negative implications for its environmental effects.

### Recycling

According to KIDV [15] a package must consist of PE for more than 90% for it to be considered 'optimal' for recycling. The same counts for PP, but then it is considered 'reasonably' suitable for recycling. The contamination that is introduced into the sealant layer inherently decreases the percentage that is one material. Compared to a film that does not have added contamination, the same film with added contamination will result in less pure recycled content. However, both are seen as optimally suitable for recycling in the Netherlands.

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## 2.9.Conduction sealing

An introduction to the conduction sealing technology is presented in this section. The focus of this thesis is on conduction and ultrasonic sealing. That is why both technologies are extensively introduced. Other heat-sealing technologies are introduced more briefly in the chapter 6.2 Introduction of other heat sealing mechanisms.

Conduction sealing is a relatively simple process and is the most common sealing method [23, 24, 39, 40]. Two heated sealing jaws apply pressure and heat to two films. The heat and pressure will make the polymer chains diffuse and entangle, which will bond the two films at the seal interface [8, 10]. A simplified representation of this process can be found in **Figure 6**.

The parameters that mainly influence this process, are sealing temperature, pressure and dwell time (sealing time) [19, 24, 28]. Inherent to this process is that the seal interface has a lower temperature than the outside of the film. The longer the dwell time, the lower this temperature difference [9, 10, 40]. This should be taken into account when selecting materials for the film, as the sealant layer should melt and the outer layer shouldn't during sealing [64].



*Figure 6.* Simplified representation of the conduction sealing process

Conduction sealing has many advantages compared to other heat sealing types, like low power consumption, high speed and less stress on the film [41].

There are different types of conduction sealing, like heated jaw, band and roller conduction sealing. The focus of this thesis and the process described above is on heated jaw conduction sealing.

### 2.10. Ultrasonic sealing

The heat sealing technology ultrasonic sealing will be described in this chapter. Within this sealing process, electrical energy is converted into heat energy through ultrasonic mechanical waves. The heat is generated at the seal interface and causes the sealant layers to merge [86]. Ultrasonic sealing generates the heat that is necessary for sealing using friction [42]. Intermolecular and interfacial friction are responsible for the sealing [9, 45, 84, 87, 88].

The mechanical waves have a certain frequency and amplitude which together with the dwell time and sealing force (pressure), form the main parameters influencing this process [9, 45, 89]. To find the optimum seal settings, usually, the amplitude and the sealing force are variated since the frequency and dwell time are usually set.

The process starts with electrical energy from the generator that is used as an input. From this input, a high-frequency electrical oscillation is created. A *converter* can convert these electrical oscillations into mechanical ones. This converter typically uses piezoelectric transducers to do so. Piezoelectric transducers use the inverse piezoelectric effect to convert a high-frequency electrical signal into a high-frequency mechanical vibration [90]. At the converter, a mechanical oscillation is created with a certain frequency (oscillations per second) and a certain amplitude (the peak height compared with a reference



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*Figure 7.* Simplified representation of the ultrasonic sealing process

value). This amplitude is transformed by a mechanical *booster*. Connected to the booster is the *horn*. This horn transforms the amplitude and passes the oscillations onto the substrate. Behind the substrate (the to-be-sealed films) there is an *anvil* [9, 91]. A simplified representation of this process can be seen in **Figure 7**. Ultrasonic sealing is associated with high investment costs [92].

This process can be applied to most thermoplastics and some nonferrous metals, such as aluminum, nickel, brass and copper [89]. It can effectively seal mono-materials due to its heating principle [36].

#### Current industry state

Ultrasonic sealing is a subject that has been getting more and more attention in literature in the past years [8]. It can be observed that also in industry this technique is widely available. According to a report from Precision reports [93], ultrasonic sealing will grow worldwide. They base this conclusion on the given information that key players in the market are adopting this technology in their strategy and the market is expected to follow.

Some of the HFFS systems available in the industry already incorporate ultrasonic seal methods. Some seal the longitudinal seal with ultrasonic sealing techniques and the transverse seal with a conduction sealing method. Others use ultrasonic sealing techniques for both the longitudinal and the transverse seal [94-96].

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# 3. Methodology

As already mentioned in the introduction, this thesis will compare different types of heat-sealing technologies based on a few parameters. In this chapter, it will be explained what the goal of this framework exactly is and what is included in it. When that is clear, the route that is taken in this thesis to create the framework will be explained. This will illustrate what the structure of the thesis is and how each chapter correlates.

# 3.1. The goal of the framework

The framework that will be created will compare different types of heat sealing. The goal of the framework is to function as design guidelines for choosing an appropriate sealing technology for a plastic packaging system. The focus of the framework lies on sealing flexible plastics in the horizontal form fill and seal (HFFS) systems, but can also be employed for other applications, like vertical form fill and seal (VFFS) systems of flexible plastic packaging.

During the packaging design process, the following elements should be determined: product needs; distribution needs and wants; packaging materials, machinery and production processes; consumer needs and wants; market needs and wants and environmental performance [21].

The presented framework will act as a tool to facilitate the machinery and production processes step, based on the input from the other elements. The framework makes the differences between several heat-sealing technologies explicit. This results in a tool that can be employed to choose an appropriate heat-sealing technology.

Colvin [97] evaluates different types of sealing technologies according to different parameters. Each sealing technology gets a certain score for each parameter. Weighing factors for the parameters are introduced. The sum of all weighted parameter scores forms the score for a sealing technology. The sealing technology that scores highest is the most suitable. While this seems like a proper method to select a sealing technology, the goal of the framework in this thesis is not to be specified to a specific application but rather to make the differences explicit. Therefore, the concept of the parameters will be incorporated into the framework, but the weighing factors will not (since the weighing factor heavily depends on the application).

## 3.2. The parameters

To effectively be employed as a tool to facilitate the heat sealing technology decision process, relevant parameters should be selected. The parameters selected by Colvin [97] are implementation cost, energy consumption, compatibility for rigid and flexible, production speed, retrofittability, durability and conformability. These parameters are selected based on brainstorming.

For this thesis, a few parameters are also interesting to incorporate in the framework, but retrofittability and conformability do not seem relevant for this purpose. Furthermore, the framework focuses on flexible packaging systems, so the parameter 'compatible for rigid and flexible' is also not relevant.

The parameters that are selected for the framework in this thesis are heat mechanism, mono-material sealing window, energy consumption, implementation costs, production speed, maintenance time, close layer jumps, squeeze-out, contamination and intermitted or continuous. The heat mechanism is there to get a simple understanding of how the sealing technology works. The 'intermitted or continuous' parameter indicates whether a technology is suitable for intermitted sealing, continuous

sealing or both. All selected sealing technologies are evaluated on how well they score on the other parameters.

# 3.3. The heat sealing technologies

The focus of this thesis lies on the heat sealing technologies conduction and ultrasonic sealing. These two types of sealing are most used in practice [19]. Other sealing types are also included: hot gas sealing, impulse sealing, induction sealing, dielectric sealing and digital sealing. The sealing technology choice will be further clarified by reviewing the theory in the chapter 6.3 Framework.

# 3.4.Data collection

There is data required to properly fill in the framework. This data is gathered by looking at public literature, by contacting suppliers and by performing tests. Tests were required to fill the literature gaps. As mentioned, the focus of this thesis lies on conduction and ultrasonic sealing. Limited research has been performed on the latter sealing technology. So the tests are mainly performed to fill gaps in theory for ultrasonic sealing.

The background from the theoretical basis for further research helps to understand the different mechanisms in play when looking at the sealing of flexible packaging systems. Building further on this background theory, the parameters are introduced with their theoretical background. This will function as a start to fill in the framework for conduction and ultrasonic sealing. Tests are performed to complete the framework for these two types of heat sealing. Other types of heat sealing are selected. The technologies will be explained and added to the framework. Both the testing and the chapter framework form the basis for future research. In the end, the framework can thus be seen as a conclusion of the thesis. How the different chapters relate to each other, is visualized in **Figure 8**.



Figure 8. Visualization of thesis chapters connections

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# 4. Parameters

Some principles/parameters that are useful to implement into the framework will be introduced in this chapter. If useful, an introduction of the mechanism in combination with conduction sealing and ultrasonic sealing will be offered.

The principles that will be described, are:

- 1. Heat mechanism
- 2. Energy efficiency
- 3. Start costs
- 4. Production speed
- 5. Maintenance time
- 6. Layer jump
- 7. Squeeze-out
- 8. Contamination
- 9. Life span
- 10. Safety ultrasonic sealing

### 4.1.Heat mechanism

How a film is generally sealed is already explained in chapter 2.2 Heat mechanisms. This section specifies this heat mechanism for conduction (CS) and ultrasonic sealing (US).

### **Heat mechanism CS**

The process of conduction sealing two films is relatively simple. Two heated sealing jaws apply pressure and heat to two films (see **Figure 9**). The heat and pressure will make the polymer chains diffuse and entangle, which will bond the two films at the seal interface [8, 10].

The jaws are heated by the heating cartridges. How hot these are, is referred to as the sealing temperature. This is one of the key process parameters of conduction sealing [8-10].



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*Figure 9.* Heat mechanism of conduction sealing [9]

Inherent to this process is that the seal interface has a lower

temperature than the seal interface. The longer the jaws stay closed, the lower this temperature difference, as can be seen in **Figure 10** where the temperature variation throughout the seal cross-section can be observed. It can also be observed that the difference in temperature between the outside and inside decreases with increasing time (t) [9, 10, 40]. Ponnambalam, et al. [98] observed a linear relation between the jaw temperature and the temperature at the sealing interface, so the difference in temperature does not decrease with higher sealing temperatures.



**Figure 10.** Heat distribution in two films during conduction sealing where t1 < t2 < t3 < t4 < t5,  $\vartheta a$  is the temperature at the seal interface and  $\vartheta s$  is the sealing temperature [9]

### Heat mechanism US

Ultrasonic sealing generates the heat that is necessary for sealing using friction [42]. To understand where the heat is generated, it is important to distinguish two mechanisms: intermolecular and interfacial friction. These two mechanisms are responsible for the sealing [9, 45, 84, 87, 88]. The friction occurs at the sealing interface, so the heat is generated there. So this is substantially different than for example conduction sealing, where the film is heated from the outside. The ultrasonic sealing system is not heated, which reduces the overall thermal input to the film significantly [99]. The way the temperature is distributed throughout the film is presented in **Figure 11**. This figure clearly shows that the material is indeed heated at the interface of the two films. The figure also shows that the difference in temperature between the inside and outside of the film decreases when sealing time is increased.

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**Figure 11.** Heat distribution in two films during ultrasonic sealing where t1 < t2 < t3 < t4 < t5,  $\vartheta a$  is the temperature at the seal interface and  $\vartheta s$  is the sealing temperature [9]

As mentioned in the chapter on ultrasonic sealing, the main parameters influencing the seal strength of the ultrasonic sealing process, are the sealing pressure, the amplitude and the sealing time [9]. Interfacial friction is almost independent of the pressure and results from the asymmetric construction of the contact surfaces of the films. The heat generated from this interfacial friction is shown in **Figure 12** on the left. It can be observed that the heat is mainly generated at the point where the two surfaces make contact. In the research from Bach, et al. [9], only significant interfacial friction of stiff materials is reported. This type of friction is only observed in the first milliseconds of the process and does not significantly speed up the heating [9].



Figure 12. Heat profile dominated by interfacial friction (left) and by intermolecular friction (right) [9]

The heat that is generated from intermolecular friction occurs when the molecules are brought into motion (oscillated). This type of friction is dependent on the pressure and the amplitude. This type of heat always occurs if there is intermolecular diffusion due to ultrasonic sealing [9]. Intermolecular friction dominates when the Tg has been reached [42]. Liu, et al. [100] even state that energy is converted into heat only through intermolecular friction.

Not both processes of heat generation are equally present during the ultrasonic sealing process. For example, softer sealing layers show significant intermolecular friction and a neglectable amount of interfacial friction [9].

The mechanical oscillation generates friction and thus heat at the seal interface. This heat generation is mainly influenced by the sealing force (pressure) and the amplitude [9]. So, within the ultrasonic sealing process, the temperature cannot be directly controlled [99], which makes the control of process parameters less intuitive than for example conduction sealing.

Additionally, to visualize the heat involved in the ultrasonic sealing process and compare this to the conduction sealing process, pictures are taken of these processes with a heat camera (see **Figure 12**). It is visible in these pictures that less heat is involved in the ultrasonic sealing process than in the conduction sealing process.



**Figure 13.** Pictures of running conduction sealing (left) and ultrasonic sealing (right) with a heat camera

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## 4.2. Energy efficiency

According to Coles, et al. [21], when trying to minimize the impact of a packaging system, not only the materials should be considered, but also the energy use should also be minimized. Some energy is used during the sealing. What effect the sealing technology has on energy efficiency will be described in this chapter.

Due to the different heat mechanisms, the energy use will probably also be different. It seems logical that conduction sealing is less energy efficient since it heats the material from the outside and the sealing jaws constantly need to be up to temperature. On the other hand, ultrasonic sealing heats the material from the inside and the equipment hardly warms up. This is clearly illustrated in the previously depicted picture in **Figure 13**, where the heat distribution of a running system with both sealing technologies can be seen. Whether it is indeed true that ultrasonic sealing is more energy efficient than conduction sealing will be discussed and proven in this section.

The powers consumed during the sealing operations will be compared. The energy consumed during the heat-up of the sealing jaws is neglected. It would not be an equal comparison to include heat-up energy in the energy consumption. If this would be included, estimations need to be made of for example how long the machines are operational, how often the machine is shut off and thus required heat up of the sealing jaws. This would add substantially to the bias of the comparison, so it is chosen to neglect the heat-up time for the comparison for this application. So it is judged that comparing the energy consumed during the sealing operation is enough information for the framework.

#### **Conduction sealing**

Conduction sealing heats the material from the outside using heated sealing jaws. These jaws are kept on temperature while the machine is running. This means that while the jaws are not sealing, the heat energy emitted by convection and radiation is wasted. Therefore, the energy required to seal a package is dependent on the machine's speed. The faster the speed, the less waste energy and thus the more energy efficient [9].

To quantify the energy efficiency, the amount of energy required to keep the sealing jaw at a certain temperature is calculated. First, it should be noted that the sealing jaws within the HFFS system used to perform tests in this thesis consist of four separate heat cartridges of 500 W each. The amount of energy required to keep the sealing jaws at a constant temperature can be estimated by looking at the energy it loses. The temperature of the sealing jaws stays constant if the loss is equal to the gain in energy.

The loss of energy is assumed to mainly be the transfer of heat from the profile blocks of the sealing jaws to the air.



This loss is calculated with the following formula [101]:

$$\frac{dQ}{dt} = hA(T_w - T_{air})$$

Where  $h\approx 20 \text{ Wm}^{-2}\text{K}^{-1}$  for solid-to-air transfer [101]. This number means that 20 W is required to keep one square meter of solid warm with the solid being one degree warmer than the surrounding.

There are some assumptions necessary to fill in this formula. These assumptions are:

1. The area of the sealing jaw profiles is estimated to be [11]:  $0,32 \times 0,023 = 0,0074 \text{ m}^2$  for one part of the profile block (see **Figure 15**),  $0,32 \times 0,024 = 0,0077 \text{ m}^2$  for another side and  $0,024 \times 0,023 = 0,00056 \text{ m}^2$ . Each surface is present twice in the profile block, so the entire block has an area of around  $0,031 \text{ m}^2$ .



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*Figure 15. Geometry of sealing jaw* profiles [11]

- 2. There are two heaters with profile blocks on a sealing jaw (see **Figure 14**) and there are two sealing jaws. In total, the area where the heat can transfer to the air is estimated to be (0,031 x 4 =) 0,12 m<sup>2</sup>.
- 3. The temperature difference is estimated to be 100°C. This estimation seems valid when looking at data gathered during testing (see appendix testing Vergeer) and by estimating that the room temperature to be around 20°C.

The total loss of energy to the air is then  $20 \times 0,12 \times 100 = 240$  W. This is the estimated required energy to keep the temperature the same. To warm up the sealing jaws takes a lot more power. This is not constantly happening but it should be noted that this will increase energy consumption substantially. Only the power consumed during the actual sealing is compared, so 240 W seems valid for that purpose.

### **Ultrasonic sealing**

The energy efficiency of the ultrasonic sealing system seems to be very high. This is inherent to the heat mechanism. This hypothesis is confirmed for other applications than sealing (linear friction welding and ultrasonic welding), where efficiencies close to 100% are reported [102, 103]. It is also confirmed by Bach, et al. [9] that ultrasonic sealing is more energy efficient than conduction sealing.

The energy use is dependent on the pressure, frequency, amplitude and time [42]. So it is (almost) independent of the machine speed since the system mainly requires energy when it is sealing [9, 88].

Ultrasonic sealing especially uses less energy compared to conduction sealing with low machine speeds, thick materials and materials with an aluminum layer [9].

To make a comparison between ultrasonic sealing and conduction sealing, among others, the document from Mediana [88] is used. In this paper, it is claimed that by using ultrasonic sealing instead of conduction sealing an energy saving of 25% is an extremely conservative estimation [88].

The power consumed by the ultrasonic sealing system is given in chapter 5.9 Test 5. Energy use US results Energy. This is around **110 W**. This is therefore taken as the estimated power consumption of the ultrasonic sealing system.

### Conclusion

Conduction sealing is inherently less energy efficient than ultrasonic sealing. Taking the estimated powers to seal a normal package, ultrasonic sealing only uses around (110/240=) 46% of the power

compared to conduction sealing. This is used as an indication of the energy efficiency difference between both sealing technologies.

### 4.3.Start costs

An important parameter when selecting a sealing system is the required investment. These costs, together with the costs it takes to run the sealing system together determine the economical perspective of the sealing system choice. What the differences between the systems are, is described below.

### Difference between conduction and ultrasonic sealing

Scientific literature sources all agree that the investment costs of an ultrasonic sealing system are higher than that of a conduction sealing system [9, 14, 64, 99, 103, 104], although none of them quantify this difference. Conduction sealing is associated with low investment costs [105], more economical than other heat-sealing technologies [106].

A packaging news website does mention an investment price *difference* of £45.000 ( $\approx$ €53.000) between conduction and ultrasonic sealing implemented in a VFFS system. This is taken as a rough estimation [107]. This price difference is huge if the price of a heat sealer is found to not even be €600 [108]. Note that this price only includes the purchase of heat-sealing jaws, whereas the aforementioned price difference includes the installation costs as well.

During its lifetime, an ultrasonic sealing system can be cheaper than a conduction sealing system. Although the part costs and investment costs are higher, there are some ways the ultrasonic system is less costly than the conduction system. Elements where ultrasonic is less costly than conduction with material usage (uses less material), lower energy usage, less maintenance and production speed. Typically the production speed of the ultrasonic sealing is higher. Next to this, less complex and thus more affordable materials can be used with the ultrasonic application [89]. It is confirmed by the news article that the payback time can be as short as 12 months, depending on the application [107].

It can be concluded that the investment price difference is huge between ultrasonic and conduction sealing and that conduction sealing is the most economical option. A price difference of  $\xi$ 53.000 is taken as an estimation to quantify the price difference.

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# 4.4.Production speed

When selecting a sealing technology, the production speed is one of the interesting parameters to include in the framework. There is a huge variation in line speeds between flexible packaging, this can go from 100 to 1000 packages per minute [25]. The minimal and maximal production speed that the sealing technology must be able to handle can be compared with the abilities of the different sealing technologies.

### **Conduction sealing**

The production speed is determined by the sealing time. Seal time is one of the parameters determining the seal strength. An increasing sealing time results in an increased seal strength [8, 28, 39, 84]. The lower the required sealing time, the higher the possible production speed will be. To realize greater production speeds (lower sealing times), higher temperatures are required [10]. These higher temperatures should not be applied to lower line speeds, since the risk of squeeze-out and material degradation arises [39]. A machine supplier states on their website that flow wrap machines are generally built for high-speed operations, thereby being suitable for high-volume production [109].

To estimate the production speed, conduction sealing suppliers are used as sources. Average conduction sealing machines can achieve production speeds of up to 80 packs per minute [110]. While also speeds up to 100 and 125 packs per minute have also been reported [111, 112]. 80 packs per minute will be used since this is also used for the testing at Omori (see Appendix 12. Testing at Omori).

### **Ultrasonic sealing**

Short sealing times can be realized with ultrasonic sealing, but high sealing forces (pressure) are required [9]. In general the rule the higher the sealing time, the higher the seal strength is valid [9, 45]. If however, the sealing time becomes too long, the seal strength might decrease again, due to squeeze-out [45].

Bhudolia, et al. [42] state that for ultrasonic welding the optimum seal strength is more dependent on the energy input, than on the welding time. Another research states that increasing the frequency, amplitude or welding force will require less welding time [103]. So higher production speeds are associated with higher seal settings.

Dun, et al. [99] state that ultrasonic sealing requires less time than conduction sealing. They state that ultrasonic sealing times of 20ms have been reported. Mediana [88] agrees with the statement that ultrasonic sealing can achieve higher production speeds. Also, the tools do not have to heat up and cool down, as the conduction sealing tools do require. He states that typical production speeds of 110 packs per minute can be achieved with sealing times as low as 150ms. These sealing times are way higher than reported by Dun, et al. [99] earlier in this part, this can be explained by the notion that the latter research focuses more on what is commonly seen in industry.

### Conclusion

Higher production speeds can be achieved with ultrasonic sealing compared to conduction sealing. Comparing the typical production speeds of 80 (conduction sealing) and 110 (ultrasonic sealing) packs per minute respectively indicates the order of magnitude of their difference. Still, conduction sealing is suitable for high-speed applications, as indicated by industry.

A combination of both technologies is also possible as demonstrated by a company that has a machine in its portfolio that employs an ultrasonic sealing system at the longitudinal seal and a conduction

sealing system at the transverse seal. They claim to be able to realize production speeds of up to 170 packs per minute [113].



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### 4.5. Maintenance time

Sealing systems need to be maintained to function properly. Even a bolder formulation is found in a book, that states that maintenance is crucial to achieve an efficient and effective seal [27]. What is included in the maintenance and what the differences are between the different sealing technologies, will be explained in the following part.

### **Conduction sealing**

For conduction sealing systems, the focus of the maintenance operations lies on the sealing jaws. These moving parts for example determine whether a seal can be considered to be integer. Because of wearout, the profile of the sealing jaws sometimes needs to be replaced. Maintenance can thus be caused by wear out, but many other reasons can cause the necessity for maintenance. For example the breakage of a cable because of external factors.

The maintenance operations for conduction sealing are based on a book that describes the maintenance of a system that seals a lid on a tray [27]. This research makes a distinction between the maintenance of the top tool and the bottom tool. This is not necessary for HFFS systems with conduction sealing since the top and the bottom tools are similar.

Maintenance consists of engineering and hygienic operations. The product inside the package (often food) can come into contact with the heated sealing jaws. This will burn the product and cause carbon deposit build-up on the sealing jaw surface if not cleaned in time. Carbon deposits can be hard to clean and can compromise the seal's integrity. Product rests can be cleaned from the jaw surface in a few seconds, but once it has turned into carbon deposits, it takes at least 15 minutes to clean [27]. The sealing jaws are heated during sealing and take a long time to cool down. This increases downtime significantly if the jaws require maintenance. The fact that the jaws are hot also creates possible complications for maintenance. As an example, cheese is placed in a flexible package and this package is closed with conduction sealing. The cheese sticks out of the packages and is put through the seal. The sealing jaw will melt the cheese and cause a mess in the machine which takes time to clean.

Next to the surface of the sealing jaws, the springs should also be checked. For example, metal fatigue is a risk for these parts. Another important part of maintenance is the calibration of the sensors [27].

There are safety risks involved in the maintenance of conduction sealing systems. The sealing jaws that need to be maintained are hot. Furthermore, the jaws are often combined with sharp knives [27]. This inherently includes some safety risks.

In general, conduction sealing is associated with low maintenance costs [105].

To get an indication of what maintenance operations are associated with conduction sealing, a manual for a conduction sealing system is investigated. The recommended maintenance operations with their corresponding time intervals are checked. The sealers (center and end seal), Teflon tape and connections (wires) should be checked for wear at the start of work. The film scraps should be removed from the sealers at the end of each work and each unit should be cleaned. The cleaning should be done with a nylon brush, a cloth and if necessary some silicone grease. The start and end of each work means that it is at least checked daily. Every month, the tightness of the screws of the seal units should be checked. The surface of the sealers and the springs should also be checked monthly [114].

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### **Ultrasonic sealing**

Ultrasonic sealing has some advantages compared to conduction sealing regarding maintenance. There are fewer moving parts and a reduced part count, which has positive consequences for the reliability which results in less need for maintenance [103, 115].

Wearout might be problematic if no film is placed between the horn and the anvil. This will result in hard metal-to-metal contact, which can cause mechanical seal failures [116].

Less cycle time is required since the equipment is always considered to be cold (not too hot to touch). It might be the case that to be able to seal a different material application, a difference in the ultrasonic sealing system is required. This can be done relatively efficiently and rapidly by replacing whole elements (e.g. anvil or converter) [88, 103].

It is confirmed by an ultrasonic sealing expert that not much maintenance is required. The horn and the anvil include a coating to minimize wear out. After a certain time (around two to four years), the converter should be replaced to ensure acceptable amplitude deviations [117]. That the required maintenance for ultrasonic sealing is only a fraction of conduction sealing is illustrated in a document from a supplier of ultrasonic sealing technology. This document claims that the maintenance costs for ultrasonic sealing are 5% of the conduction sealing maintenance costs [118].

### Conclusion

Maintenance is necessary to ensure the quality of the seals. It can also be done to prevent downtime. This can be done by using planned preventative maintenance. Although this is not extensively employed in practice, there is evidence that preventive maintenance results in better-performing packaging lines [27].

Regarding maintenance, ultrasonic sealing seems to be the preferred option compared to conduction sealing. Mainly due to the sealing jaws being hot during sealing and taking a long time to cool down. Especially the daily required cleaning and inspection of the sealing equipment adds to the maintenance time for conduction sealing.

# 4.6.Layer jump

A certain flexible plastic packaging system design may include a transition of thickness within the seal area. The thickness transition is called a 'layer jump' since the increase or decrease in the amount of film layers causes a transition in thickness [21]. These layer jumps form a critical point within the seal of a package [119]. Layer jumps usually can occur from two to three or two to four layers [52].

When the layer jump from two to four layers occurs, the seal area with two layers will experience a low pressure and more pressure will be exerted on the four-layer side of the layer jump. This pressure deviation might cause problems [52, 99].

Possible failures might occur due to the layer jump in the seal. One of these failures is the emergence of microleakages at the point of the jump in the seal. To make sure this does not occur, the sealant layer should flow into the gap at the layer jump [17]. Another failure that might occur is squeeze-out, but this time due to too high pressures [52]. These seal settings should therefore be carefully selected, to close the created gap and to minimize excessive squeeze-out.

### **Conduction sealing**

For conduction sealing, layer jumps can form a problem. The pressure of the sealing jaws needs to be sufficient to fill the gap at the layer jump and low enough to make sure no squeeze-out occurs [52].

If this results in a too narrow sealing window for a certain material, a possible solution is presented by Hauptmann, et al. [17]. They present results in their paper that a sealing profile can contribute significantly to reducing the gap height at the layer jump. Furthermore, their research shows that adding one flexible membrane to a sealing jaw has positive effects regarding the seal integrity at the layer jump. Even lower sealing temperatures are required to close the gaps, which is especially useful when trying to seal mono-materials. A combination of the flexible membrane and the jaw profile seems to even have a more positive effect [17].

### **Ultrasonic sealing**

Sealing through a layer jump with ultrasonic sealing can form a challenge. It can only be done with materials that reach relatively high seal strengths with relatively low energy input from the ultrasonic sealing system. Still with these materials, the maximum seal strength obtained from a package with a layer jump compared to a package without is significantly lower [99]. The seal strength alone however does not give a complete picture of the seal performance, since the microleakages might be present in a high seal strength seal [52].

Just like described above, a flexible membrane can also be implemented for ultrasonic sealing. In theory, this could also increase the ultrasonic sealing performance at the layer jump. Colvin [97] performed research on this topic. First, a normal ultrasonic sealing system is evaluated on the ability to seal layer jumps. The conclusion is that regular ultrasonic sealing cannot be used in combination with layer jumps. After this first test, another test is performed with an ultrasonic sealing system with a silicone rubber clamped to the anvil. The result of this test seemed to be successful. The horn was now able to make contact with the two-layered fractions as well as the four-layered ones. Next to this, less pressure is required to seal, so less squeeze-out will occur. However, in general, sealing complex shapes is a limitation of ultrasonic sealing [97].

### Conclusion

To conclude, layer jumps can be problematic for both conduction and ultrasonic sealing. The gap that is created at the layer jump needs to be sealed while exerting enough pressure at this area is difficult.

The risk of this forming a problem can be minimized by adding a flexible silicone layer on the sealing jaws. Layer jumps can cause microleakages or excessive squeeze-out. More testing is necessary to determine which layer jumps are problematic for ultrasonic sealing. Therefore this is going to be examined further within chapter 5.10 Test 6. Layer jumps.

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### 4.7.Squeeze-out

In **Figure 16** the seal strength for different temperatures is given for conduction sealing of conventional packaging films. It can be observed that a certain plateau seal strength is reached. If more heat is introduced into the seal, the seal strength will decrease again. This is due to the so-called squeeze-out of the sealant layer [8, 97]. This chapter will focus on this failure that might occur while sealing. First, squeeze-out is defined. Then, squeeze-out for conduction sealing is explained. Next, squeeze-out for ultrasonic sealing and different kinds of films is explained and this chapter will conclude with a way to measure squeeze-out.



Figure 16. Seal strength behavior for different interface temperatures in conduction sealing [8]

So squeeze-out is a possible failure that can occur during sealing. If this failure occurs, some material is squeezed out of the sealing area, so away from its original location. Squeeze-out can be observed at the edges of the seal and it only occurs if the material can flow [10, 52]. The molten polymer moves away from an area of high pressure, so away from the seal area. This causes the sealant layer thickness to decrease or even disappear. If the next inner layer has a lower bonding capacity and there is enough squeeze-out, this will result in a lower seal strength [31]. Squeeze-out can also disturb the seal integrity by causing wrinkles and thus possible microleakages [52]. Another possible consequence of squeeze-out is that molecular misalignment might occur at the sealing interface and the formation of voids [97].

As stated above, this failure only occurs when the material can flow. When reading this, it could seem logical to minimize the flow, to prevent squeeze-out from occurring. This conclusion is however too short-sighted. To fill up possible leakages, like microchannels, high flow rates are desired [8, 10]. So a balance should be found between the seal integrity (leak tightness) and seal strength. While looking for this optimal balance, the packaging film properties (especially of the sealant layer) and the process parameters should both be optimized, since both influence squeeze-out [9, 64].

In **Figure 17** a simplified graphical representation of squeeze-out formation is presented. This illustrates that the sealant layer moves from the inside (seal interface) to the outside, so away from the seal area.



Figure 17. Squeeze-out formation principle according to a model [10]

Colvin [97] describes in his literature review that there are different kinds of squeeze-out mechanisms. It is called squeeze-out if the entire sealant layer is pushed out, but also when the sealant layer only slightly decreases in volume at the seal area.

### Squeeze-out and easy peel

The statement that the seal strength decreases after a local maximum due to squeeze-out, counts for materials that do not have controlled contamination in the sealant layer and monolayer films.

For films that include controlled contamination in the sealant layer to achieve easy peel properties, squeeze-out can cause a seal to be nonpeelable. So for these films, it is possible that the seal strength will not decrease with more squeeze-out. This happens if the sealant layer (with controlled contamination) is squeezed out of the seal area and the next inner layers are sealed together [84]. So if there is controlled contamination in the sealant layer, the seal strength does not show maximum with varying seal forces. This difference between a sealant layer with and without controlled contamination (6, 15 and 30% of the weight) can also be observed in the difference between **Figure 16** and **Figure 18**. The latter figure shows that the seal strength only increases with more sealing power. It might even be valid to state that the relationship between seal force and seal strength is increasing exponentially. It is assumed that this is caused by the squeeze-out phenomenon. This hypothesis will be evaluated during the test phase of this research.



*Figure 18.* Seal strength for different ultrasonic sealing forces with material that includes a controlled contamination easy peel mechanism (PP, LLDPE, LDPE & iPB-1) [84]

Nase, et al. [84] and Bach, et al. [9], whose research forms the foundation of this part, both mostly refer to the sealing force(s) as being the driving parameter that influences the squeeze-out. It is however explained that the amplitude also influences squeeze-out for the ultrasonic sealing process. Furthermore, it is expected that the temperature generated at the sealing interface influences the melt flow and thus the squeeze-out. This means that parameters that influence the temperature at the sealing interface also influence the squeeze-out. This for example means that it is expected that the frequency influences the squeeze-out.

The above text about squeeze-out occurring at the ultrasonic sealing process is mainly based on two researches [9, 84], since that is the only public literature found that discusses the combination of ultrasonic sealing and squeeze-out. To gain a more robust theoretical foundation, more research needs to be done.

The influence of temperature on seal strength in the conduction seal has been extensively investigated. The relationship between the seal strength and the temperature shows certain trends. It is expected

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that the seal strength increases with increasing temperature until a plateau is reached. If the temperature is further increased, it is expected that the seal strength will decrease again. This is only the case if the sealant layer is squeezed out of the sealing area and the next inner layer has less bonding capacity than the sealant layer.

This implies that it is also possible to see an increase in seal strength after the plateau if the bonding capacity of the next inner layer is bigger than that of the sealant layer. This is for example described by Nase, et al. [84] where an easy peel sealant layer (PP, LLDPE, LDPE & iPB-1) is squeezed out of the seal area.

### **Conduction sealing**

Squeeze-out applied on conduction sealing will be briefly reviewed in this part. It is reviewed because squeeze-out can form a risk for conduction sealing. The sealing jaws can push away the sealant layer from the seal area, creating a seal with less seal strength [8]. Squeeze-out at conduction sealing is for example observed in the tests performed by Ilhan, et al. [52].

The process parameters that influence the squeeze-out for conduction sealing are: pressure, temperature, time, sealant thickness and seal bar width [10]. For each parameter, it counts that the higher this parameter, the more squeeze-out will occur, except for the seal bar width. This is the other way around, the smaller the seal bar width, the more squeeze-out will occur. The relationship between the pressure and squeeze-out is linear, while the relationship between temperature and squeeze-out is exponential [10]. Therefore, only a small increase in temperature might severely influence the squeeze-out [52].

Morris and Scherer [10] state in their article that usually, the risk of squeeze-out is not substantial for conduction sealing. So, if the parameters are not set extremely high, the squeeze-out should not be an issue. It can however become problematic in combination with layer jumps. If there are layer jumps in a seal, these can cause pressure variations within a seal [52].

### **Ultrasonic sealing**

In this part of the research, the combination between ultrasonic sealing and squeeze-out will be reviewed. Whether this failure has already been reported for ultrasonic sealing will be checked. Next to this, the parameters that influence the possible squeeze-out at ultrasonic sealing will be investigated.

To start with the first aspect, squeeze-out can cause problems in ultrasonic sealing. The risk of this failure occurring during the ultrasonic sealing process is even bigger than for conduction sealing. This is caused by the heat mechanism. Since ultrasonic sealing heats the material from within, the material will tend to flow more intensely, generating more squeeze-out [9].

As stated before, sometimes squeeze-out is desired during sealing. This is also the case for ultrasonic sealing. For example, Bach, et al. [9] report that a 30-38% decrease in the sealant layer (due to squeeze-out) resulted in the highest seal strength. Furthermore, squeeze-out can push possible contamination out of the seal area within the ultrasonic sealing process [9].

Ultrasonic sealing requires a certain pressure and a mechanical oscillation on the films that need to be sealed. The combination of both results in a mechanical load that varies according to a certain frequency. This varying mechanical load heats the films at the interface (from the inside) and leads to a melt-flow to the border of the seal area [9, 84]. This can result in squeeze-out if the material is pushed

out of the original seal area. So both the pressure and the mechanical oscillations influence the squeeze-out. The next parts will explain these parameters in combination with squeeze-out further.

#### Pressure

First, the combination of pressure and squeeze-out within the ultrasonic sealing process will be evaluated. Nase, et al. [84] show in their research that the higher the pressure, the larger the seal area. The border of the seal area moves to the outside if the pressure is increased. The material that was present in the original seal area is squeezed out towards the edges of the seal, enlarging the seal area. The squeezed-out material can be observed with a light microscope [84]. Note that the seal area includes the squeeze-out in this case.

Bach, et al. [9] show in their research that the width of the seal is decreased when the seal force is increased. Material from the inside of the seal is squeezed out towards the outside, making the inside of the seal thinner. This can also be observed in **Figure 19**, where pictures (c) and (d) show very thin sealant layers at the seal that can later detach and lead to seal integrity issues. It can therefore be concluded that the sealing force (=pressure) influences the squeeze-out.



*Figure 19.* Changes in (ultrasonic) seal width for different sealing forces: 2N/mm (a), 2,75N/mm (b), 5N/mm (c) and 6 N/mm (d) [9]

### Mechanical oscillation

Second, the combination of mechanical oscillations and squeeze-out will be evaluated. These oscillations have a certain frequency and amplitude. The frequency is usually a constant (inherent to the type of ultrasonic sealer used). The amplitude can however be varied and has a certain influence on the squeeze-out. The maximum horn displacement from the origin is the amplitude. The higher this horn displacement (and thus the amplitude), the more squeeze-out will occur. The horn displacement can even approach twice the thickness of the sealant layer. This displacement causes the sealant layer to be almost entirely squeezed out of the seal area [9]. Therefore, the mechanical oscillations influence squeeze-out.

The observation that squeeze-out is happening at ultrasonic sealing is supported by the trend shown by Bach, et al. [9] that the seal strength increases with increasing sealing force until a maximum is reached. After this maximum, the seal strength decreases again if more sealing force is introduced into the seal. This is caused by squeeze-out [9]. Material from the sealant layer is squeezed out of the seal area, making the sealable volume lower, which can result in lower seal strength, as has already been described.

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#### Welding

To achieve this more robust theoretical foundation, there will be looked at literature beyond the field of sealing. The field that will be included in this research is welding. The main difference between welding and sealing is that welding fuses general parts while sealing fuses two thin films [9]. In the welding terminology, squeeze-out is called flash formation and is defined as "the overflow of molten plastic from the joint area" [120]. Flash formation is undesired for aesthetic as well as functional reasons. Flash can be visually observed at the side of the weld and flash indicates that the weld might have fracture initiations at discontinuities [121].

Two types of welding mechanisms will be briefly investigated: ultrasonic and linear friction welding. Both mechanisms exploit the same generation of heat as ultrasonic sealing, this mechanism being friction. Ultrasonic welding resembles ultrasonic sealing most, but also linear friction welding comes close.

#### Ultrasonic welding

The ultrasonic sealing technology has been known for other applications than films for a long time already [87]. If this technology is used for other applications than sealing films, it is called ultrasonic welding. This is a fast and clean process that usually creates welds with minimum or no flash [45, 87]. This process also fuses two elements by applying mechanical oscillations with a certain pressure. The frequency of these oscillations is usually constant and the amplitude can change, with variations in the range of tens of micrometers [87].

The flash formation can occur as a result of having high flow rates [121]. High flow rates can for example be created if the welding forces are too high. If flash formation is observed, the bonding strength might be compromised. A possible way to get rid of flash is by encapsulating the bonding region [120].

Grewell [121] states in his research that the amplitude is a critical variable for determining the squeeze flow rate. So next to the pressure, the amplitude also influences the flash formation of ultrasonic welding. Both the pressure and the amplitude should be balanced to achieve proper welds, with a minimum amount of flash [121].

#### Linear friction welding

Linear friction welding exploits the same heating mechanism as ultrasonic welding. Heat is generated at the welding interface by a combination of applying oscillations with a certain frequency and amplitude with a certain pressure [102]. The difference between linear friction welding and ultrasonic welding is that linear friction welding creates oscillations parallel to the substrate, whereas ultrasonic welding generates oscillations perpendicular to the substrate [87, 102]. So although this type of welding is not used to seal packages, it might result in some interesting insights because of the similar heat generation principle.

Turner, et al. [102] describe in their research that flash formation is a risk in linear friction welding. For this type of welding, flash formation is also caused by the oscillations and the pressure. The effect of the amplitude is visible in the flash. The pressure causes viscous hot material at the weld interface to be steadily extruded. The oscillations drag material away from the weld interface at every oscillation. So both the pressure and the oscillations cause flash formation, which parameter is dominant depends on the parameter settings. If for example the pressure is high and the amplitude of the oscillations low, the pressure will be the driving parameter causing flash formation [102].

#### Insights for sealing

It can be concluded from the literature about welding that for welding the amplitude and pressure influence flash formation. This seems to confirm that squeeze-out for ultrasonic sealing is influenced by the amplitude and the pressure. The higher the amplitude and pressure, the more squeeze-out. Encapsulating the weld can function as a solution for welding, but this is not practical for continuous ultrasonic sealing.

### Conclusion

Squeeze-out for conduction sealing is already well understood. It does occur but does not form problems as long as no excessively high sealing settings are used. Literature offers some hypotheses for the squeeze-out behavior for ultrasonic sealing. It is concluded from the theory that the amplitude and the pressure will probably positively influence the squeeze-out. However, to compare conduction and ultrasonic sealing on the parameter squeeze-out, testing is required.

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### 4.8.Contamination

When a packaging system is closed through sealing, the seal should be integer. This means that for example even little folds in the seal area should be filled with sealant material, so no channels should be observed. Contamination (e.g. food particles) within the seal area can cause channels [10]. Research found that the main reason for seals to fail (more than 50%) is contamination by food. Working on this issue will increase the efficiency of the packaging machine considerably [27]. This is the reason that contamination in the seal area will be investigated for different sealing technologies. There will be looked at how well a certain sealing technology can seal through contamination. This performance can be a parameter to take into account when selecting a suitable sealing process if contamination can form a risk for an application.

The sealing parameters should be optimized depending on what type of contamination can enter the seal area. The seal parameters should be optimized to allow sufficient flow of the sealant layer. This can either cause the material to fill the channels that have been created by the contamination or cause (acceptable) squeeze-out that takes away the contamination from the seal area [10, 27]. Literature shows cases where leak-tight seals can be created with contamination in the seal area, but this often decreases the seal strength [8]. However, it is also stated in the literature that contamination can make it impossible for an integer seal to be created [27].

The ability to seal through contamination is not only dependent on the sealing technology but also on the material of the sealant layer [9, 37]. For example, LLDPE shows a better performance in combination with contamination than LDPE [8].

### **Conduction sealing**

Hettiarachchi [37] illustrates that contamination can indeed form a risk in combination with conduction sealing. She states that no matter how well the operations are controlled, the product can end up between the sealing jaws. When this is the case, the heated jaws will melt the sealant layers thereby encapsulating the contamination within the seal. This can cause seal integrity problems [88].

Research performed by Bach, et al. [9] shows the seal performance of conduction and ultrasonic sealing in combination with contamination. They compare the seal strength of a seal with contamination in the seal area to the seal strength of a seal without. Conduction sealing can reach up to 80% of the original seal strength for grated cheese, olive oil and salad dressing contamination. The performance in combination with solid materials (wheat flour and coffee powder) is poor and thus not suitable. It is illustrated by another research that the thermal resistance in the seal area is locally increased if solid contamination is present there, thereby possibly decreasing the flow of the sealant layer [98]. So contamination can be handled by conduction sealing, although it decreases the seal performance.

### **Ultrasonic sealing**

An inherent advantage of ultrasonic sealing technology is the ability to seal through certain contaminants. This is due to the form and the mechanical oscillations of the horn-anvil combination. First, the expulsion of the contamination caused by the sealing force is visible. Secondly, expulsion caused by the mechanical oscillations is observed. Lastly, the contamination is removed from the seal area by squeeze-out [9, 88, 99]. The three principles occur simultaneously at continuous sealing, but mainly expulsion because of vibration occurs. This effect even increases with higher sealing forces [9].

The same research as referred to before also examined the effect of contamination on the seal strength of seals created with an ultrasonic sealing system. Around 50% of the original seal strength can be

reached for grated cheese and wheat flour contamination. Around 30% can be reached with coffee powder contamination, although the observation is made that some powder is left in the seal area. These low percentages are not in line with other statements that claim that contamination hardly affects the seal strength for ultrasonic sealing [9]. Fluids do not perform very well in this research. This is probably caused by the energy absorption of the fluids. Higher sealing forces would partly counteract this disadvantage [9].

A limitation that should be mentioned is that Bach, et al. [9] did not optimize the sealing parameters for the contaminants. With higher sealing forces, the seal strength would increase thereby decreasing the influence of the contaminants. Next to this, the created seals are only tested on seal strength and not on seal integrity.

Another advantage of ultrasonic sealing is that the contaminant is not exposed to heat in the same way this applies to conduction sealing. For example, cheese does not melt if it is present in the seal area. This is an advantage compared to conduction sealing. If cheese is included in the seal area, the conduction sealing system will melt the cheese, causing a mess in the machine. This is associated with high downtime periods for cleaning.

Based on the information stated above, it can be concluded that ultrasonic sealing mainly performs well with loose material contaminants, such as powders.

### Conclusion

Conduction sealing can reach up to 80% of the original seal strength for grated cheese, olive oil and salad dressing contamination. The performance in combination with solid materials (wheat flour and coffee powder) is very low and thus not suitable.

Ultrasonic sealing can successfully seal through some contamination. It especially performs well in combination with powder contamination. An indirect advantage of ultrasonic sealing is that it does not melt the contaminants.

The differences between conduction and ultrasonic sealing are quite small, except in the case of loose materials (like powders) [9]. It is therefore concluded that ultrasonic sealing slightly performs better, depending on the type of contaminant. Although the aforementioned limitations should be considered.

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### 4.9.Life span

From an investor's point of view, the life span of the sealing technology is a parameter that is highly interesting to consider. The life span and the investment costs together show how much the technology itself costs per period.

However, the life span of a sealing system is difficult to quantify exactly, since it is dependent on many factors, like the application and the maintenance. In addition, no publicly available scientific references have been found that describe the life span of sealing tools. Only general information is found, like that in general flow wrap machines are robust and if properly maintained can function for a long time [109].

The average life span values stated by sealing tool suppliers vary a lot. It is stated by a packaging machine company that the average life span of a carton packaging machine is 10 to 15 years [122]. Although another source states that a flow wrap machine has been running for more than 60 years [123] This example illustrates the huge variations among life spans of sealing systems that cause the difficulty to estimate the life span. An interview with an ultrasonic sealing expert gave the insight that if properly maintained, an ultrasonic sealing system can have a long life span [117].

Another strategy that has been employed to estimate the life span, is to look at the warranty period. The huge assumption that would have to be made, is that the warranty period would be a similar constant fraction of the life span for each sealing technology. However, the only warranty period found for the different sealing technologies (hot gas, dielectric, digital and ultrasonic sealing) is one year [117, 124-127]

Therefore, nothing useful can be stated about the difference in life span for the sealing technologies. This is the reason this interesting parameter is not included in the framework.

# 4.10.Safety ultrasonic sealing

Mechanical oscillations are created more than 20.000 times per second with ultrasonic sealing. The human ear can only hear noise below 20 kHz. So in theory, the ultrasonic sealing system would not be audible for the employees working with the packaging machine. To investigate whether this technology could be harmful to the operator of the machine, this section is written. When comparing ultrasonic waves to audible sound, it can be noted that ultrasonic waves don't travel as far as normal sound waves, since they are better absorbed by the air [128].

### Regulation

First, the present legislation will be investigated. If there is legislation present, this should be included in the consideration of a sealing technology. Although many machines generate noise within the ultrasonic frequency domain (often also unintentional) no clear legislation is present. There is some legislation present in Germany, France and Poland, but there is no general legislation from the European Union on ultrasonic noise. The Polish legislation is especially interesting to consider since this proposes a maximum admissible intensity is proposed. This is done based on the frequency of the appliance and an 8-hour working day. According to Polish legislation, a noise that has a frequency of 31,5 kHz can have a maximum sound pressure of 130 dB. It also states that 110 dB is the equivalent continuous sound pressure for and 8-hour working day. The regulations in general agree that the higher the frequency, the higher the allowed sound pressure [128, 129].

### Ultrasonic sealing system 35kHz

During the executed tests within this thesis, an ultrasonic sealing system of 35 kHz is used. This section investigates whether this application could be harmful to an operator of this machine. The sound pressures for ultrasonic welding for 31,5 kHz (which is the closest to 35 kHz), do not seem to be going above the allowed sound pressure levels stated in the Polish regulation, according to the results presented by Dudarewicz, et al. [129]. The same research shows that specifically for welding applications overexposure to ultrasonic noise has been measured. The result did not show a significant relationship between overexposure and hearing damage.

### Limitation

What makes this topic ambiguous, is that no evaluation criteria are available for ultrasonic noise. So determining if a certain ultrasonic application causes harm to human health in the form of hearing damage, is practically impossible. Next to this limitation, only hearing damage is measured to quantify the health risks of ultrasonic noise sources. It could however include other health risks since the ultrasonic noise can be absorbed by the human body and overexposure could cause damage to biological tissue [128].

### Conclusion

The health risks associated with ultrasonic noise are not well understood and the current application in an HFFS system does not seem to include health risks. This is based on the fact that regulations do not form a limitation, operators of this system already have to wear hearing protection and the noise is damped by the plastic films. Furthermore, no excessive sound pressures are expected. However, to draw clear conclusions on this topic, more (long-term) research is required.

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# 5. Testing

To better understand how ultrasonic sealing works and to be able to properly fill in the framework for selected types of sealing, some tests are required. First, an explanation of the testing methods will be offered. Afterwards, the tests will be described.

#### **Research process**

This research tries to get a more in-depth understanding of different kinds of sealing. Especially the comparison between ultrasonic and conduction sealing is of interest. Ultrasonic sealing is a relatively new technology, especially in combination with horizontal form fill and seal systems and mono-material applications. Therefore, probably not all research questions can be answered by looking into public literature. This means that research in the form of experimentation is probably necessary to answer these questions. These experiments need to be designed in a specific way to be useful. This chapter will describe what methods will be used to design the research that will be done.

Field, et al. [130] present a framework in their book that gives an overview of a scientific research process. This framework will be used as a basis in this thesis to answer research questions that require data collection. The different stages that are handled in the process are briefly described below.

An overview of the research process is illustrated in **Figure 20**. It starts with a question that needs to be answered. This research question may for example arise based on an observation that requires explanation. It is also possible that a gap in the literature is identified that needs clarification. Once the research question is formulated, one or multiple theories need to be created. The theories that are generated are usually based on literature. This will form a solid base to predict what the answer to the research question will be. This prediction is called the hypothesis. The hypothesis should be measurable, which also indicates that the research question should be formulated in such a way that the answer is measurable. In the hypothesis, a single or multiple (measurable) independent variables are identified that influence the (measurable) dependent variable. Once the hypothesis is created, data needs to be gathered to either confirm or deny the hypothesis. Data that needs to be gathered is based on the dependent and independent variables that are defined during the theory and hypothesis phase.

Once the data has been gathered, it needs to be analyzed and statistically proven whether the hypothesis can be confirmed or rejected. This is also the reason that the variables needed to be measurable. Statistics can only be used to state something quantitative about the data. If the hypothesis is rejected, the theory needs to be changed. Therefore, this data analyzing phase returns feedback to the theory generation phase [130].



Figure 20. Research process, based on Field, et al. [130]

The top	pics that will be discussed, are:	1.
		1.
1.	Design of Experiment	2.
2.	Testing methodology	
3.	Two-way ANOVA	3.
4.	Testing of the ANOVA assumptions	4.
5.	Test 1. Influence of temperature on seal strength	;····
6.	Test 2. Influence of pressure and amplitude on seal strength	5.
7.	Test 3. Mono-material sealing window CS	6.
8.	Test 4. Mono-material sealing window US	0.
9.	Test 5. Energy use US	7.
10.	. Test 6. Layer jumps	0
11.	. Test 7. Squeeze-out	8.
12.	. Test 8. Damage to the barrier layer	9.
13.	. Test 9. Easy peel US	10
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# 5.1.Design of Experiment

After the hypothesis is created, the Design of Experiment (DoE) might play a role. This can mainly be employed between the hypothesis and the data gathering phase, but it can also be useful during the data gathering and analysis phase. The DoE methodology offers a way to prepare the experiments [131]. More specifically, it is a tool to optimize variables for example to increase production efficiency [132]. By using DoE, statistics can be systematically applied to experiments [133].

In more detail, DoE tests what influence certain input variables (called 'factors') have on an independent variable (called 'responses). The factors can have different settings (called 'levels') and all different factor levels are specified in the DoE [131, 134]. The factor levels differ simultaneously and independently, which is why a predictive model can be created. This model is created by estimating the effect of the factors and the factor interactions on the response [26, 135]. There is a factor interaction if the effect of a factor on the response depends on another factor [134].

The estimations of the effects can be used to find optimum factor values that result in a maximum response value [134, 136]. DoE is an effective way to get a maximum amount of information with a minimum amount of data [134, 136]. In most applications, DoE is superior to for example the one-factor-at-a-time method [137]. That method works in a series of experiments designed to only test a single factor. So no factor interactions can be measured with this method [135].

### **Factorial design**

Often, Design of Experiment is used in combination with a factorial design. This means that factors are changed simultaneously [134, 136]. Two types of factorial design are full factorial and fractional factorial. A full factorial design tests every unique combination of factor levels that is possible. It is a systematic and efficient way to design the experiments [133]. That means for example that if a certain experiment has three factors with each two factor levels, 8 experiments need to be executed. The amount of experiments required can quickly grow to an amount that is too big with full factorial. In that case, some experiments need to be excluded [26]. This means that in that case only a fraction of the tests is selected, which is called fractional factorial design [131, 134]. This can decrease the amount of experiments at a low risk if the fraction is carefully selected [131, 137].

### 2k full factorial

2k full factorial design is one type of full factorial Design of Experiment (DoE). With this type of design, every factor has two levels [134]. One level is a 'high' setting and the other is a 'low' setting. These two settings should be far enough apart that a difference in response is expected, but not outside the likely operating range [134]. Often, this two-level variant of DoE is enough for an application like optimizing the process parameters [26].

This method has its limitations since only two factor levels are used in the analysis. So no trends (e.g. linear or exponential) can be observed between the factor and the response. Only whether the effect of the factor is positive or negative can be observed [26, 134]. Therefore, the relationship between the factor (or a factor interaction) and the response needs to be understood to find the optimum process output [26].

If the aforementioned relationship between factors and response is not known, this can be investigated by using a method called Response Surface Methodology (RSM).

### RSM

Response surface methodology (RSM) consists of a group of mathematical and statistical techniques [138, 139]. This methodology can be part of the DoE process. The RSM can be used to describe the relationship between several factors and a response variable. There are three types of RSM:

- 1. First order model
- 2. Second order model
- 3. Three-level fractional factorial

The first order is used to describe linear relationships. The second order is used to describe a combination of a linear and an exponential relationship and is usually employed when the first-order model shows a lack of fit. The output of the RSM is a surface that plots the response against two factors. This can be used to effectively find a maximum or minimal response and to understand the relations between the factors and the response[139].

If the nature of the relationship needs to be known, more than two factor levels are required. Threelevel factorial design could be used in this case. Each factor includes three levels with this type of design, a high, low and intermediate level. In case all possible combinations of factor levels are tested, this is called a complete or full factorial design. If this is not the case, it is called a fractional factorial design[139].

DoE is not only used to find the optimum factor values for a maximum response value but also for other applications like discovering interactions between factors, screening factors, quality control and designing robust products [134].

### **Types of DoE**

There are different types of DoEs, most used methods are: the Classical, Shainin and Taguchi method [26, 133, 137]. Tanco, et al. [137] evaluated the three methods based on a literature review. Their findings in combination with additional sources are discussed in Appendix 1. Types of DoE. The conclusion for this thesis is that the classic DoE seems to fit best. The Shainin method is too simplistic and not as statistically robust as the other methods. This seems only to be applicable for medium to high-volume processes that already have a high level of quality. Although the Taguchi method is more statistically valid and robust, success cannot be assured. This is due to the theoretical imperfections. In general, it is only advised to use the Taguchi method in case of tolerance analysis and analysis of the robustness of the noise factors [137].

### **DoE process**

To effectively go through a classical DoE process, different steps need to be taken. When doing research, DoE can mainly play a role between the hypothesis and the data collection phase (see **Figure 20**). One of the outputs of the hypothesis phase is a list of variables, so the factors and the response. This can be used as an input for the DoE. Generally speaking, the first step within the DoE process is to select the factor levels. Then, the experiments should be allocated in a random order [131, 134]. The experiments should be redone a few times with the same conditions. These experiments are called replications. This is necessary to decrease the possible noise that originates from uncontrollable variables.

In general, these two steps are the only two that happen between the hypothesis and the data collection. After the experiments have been designed, the experiments can be executed and the resulting data can be analyzed.

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#### In packaging

An example of how DoE can be useful in the field of packaging is described in the research from Hron and Macák [26]. They state that a 2k full factorial Design of Experiment can be useful to optimize the settings of the machine. The result indicates how much influence the factors (settings of the machine) have on the response (seal strength). With this information, the optimum factor values can be determined.

#### Conclusion

In conclusion, classical DoE in combination with the research process described by Field, et al. [130] seems to be an effective way to determine the factor values that result in a maximum response value. Other statistical methods are required to find the relation between the factors including their interaction and the response. Only when that relationship is known, the factors can be effectively optimized using classical DoE.
# 5.2. Testing methodology

There are different testing techniques available to evaluate the seal performance. The testing techniques that are encountered during the execution of the thesis will be briefly described. The first two (headspace and bubble test) will not be employed in this thesis, but are presented for completeness. The other methods (dye penetration, imaging and T-peel test) will be employed to answer research questions.

Next to an introduction of the testing techniques, an explanation of how these methods are employed during the tests will be given. This will describe the methods used for the tests.

### **Headspace analysis**

Another way to test the integrity of the seals of transparent materials is by analyzing the headspace of a modified atmosphere packaging (MAP). One tool to analyze the headspace gas composition is laserbased headspace analysis. This nondestructive measuring tool uses a technique based on a tunable diode laser absorption spectroscopy that indicates how much oxygen and carbon dioxide are present in the headspace [140]. This method is useful to monitor the change in headspace gases. In this case, this might be useful to determine how fast the modified atmosphere within the package changes from composition. This will give insights into the quality of the seals [141]. If for example there are some leakages in the packaging, the modified atmosphere will change quickly, much quicker than an integer package.

This method can inspect several hundreds of packages per minute on the production line [140].

### **Bubble test**

Another standard to determine if there are leaks in flexible packaging, is the so-called bubble test. This is the ASTM D3078 standard which is meant to find gross leaks. This standard applies to packages containing a headspace gas [142]. The test starts by placing a package in a water tank. A vacuum is created in the tank, which will cause the package to inflate a little. If bubbles can be observed coming out of the package, the package has a leakage. The test can detect gross leaks of more than 250µm wide [33]. The result of this test indicates whether there is a leak or not, so a qualitative result only [35].

There is a downside to this test because it is possible to detect bubbles while there are no leakages. This is possible if the gasses can travel through the packaging material. In the case of a thin film of porous material, this might be possible [143]. Therefore, it is necessary to have another test in place next to the bubble test. In this case, the dye penetration test is used. This combination of tests is also described by Guyer and Zednik [143]. They found that the packages that were falsely tested positive in the bubble test, were corrected by the dye penetration. They checked with other laboratory tests if there was a leak present in the packages. All packages that had a leak in them according to the bubble test and did not have a leak in them according to the dye penetration test, did not have a leak in them. Therefore it is expected that this combination of tests will give relevant insights into the quality of the packaging seals.

## Dye penetration

Dye penetration is an official standard to detect leaks in nonporous packaging. This is the standard ASTM F3039 which is a destructive and qualitative leak detection method. Its goal is to verify and locate leakages. In this test, a colorized liquid is injected into a transparent package and stays in contact with the seal for approximately 5 seconds [144]. This method can be used to detect channel leaks (a

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'tunnel') through the seal. The colorized liquid can run through these channels and can be visually observed [32]. Parameters that influence the outcomes of the dye penetration method are dye concentration, viscosity of the dye, temperature, pressure differential and surface tension of the dye [32].

The advantages of the dye penetration method through injection, are that it is widely used, it identifies the location of the leak, it is inexpensive, easy and it is widely accepted [32].

Disadvantages of this method are that it is not very sensitive (cannot detect channels smaller than 50  $\mu$ m), it is a destructive method and it is not in-line. Furthermore, it is messy, labor-intensive, relatively slow and it is not a quantitative method [32].

Moghimi and Park [32] described how they applied this method in their research. They injected the dye into the packaging and allowed the dye to stay in contact with each seal for 5 seconds.

#### Dye penetration application

Packages realized with different seal settings for conduction and ultrasonic sealing will be evaluated on their seal integrity. This will be done using the dye penetration test. If no leaks are observed, it can be concluded that no channels or leaks bigger than 50µm are present in the realized seals [32]. If a leak is however observed, the seal where the leakage appears cannot be seen as integer.

For some seals, it is already obvious that the seal is not integer. A gap can be observed within these seals. See for example **Figure 21**, it is obvious that this transverse seal (conduction sealing) is not closed. These seals will not be included in the operating window, while these seals will not be included in the dye penetration testing.



Figure 21. The side seal (conduction sealing) is open for a package (material 1 and 110°C)

Not all packages are subjected to the dye penetration test. To save time and resources an assumption is made. This assumption is that if a package does not show leakages, the packages with higher seal settings, higher seal strength and that do not show shrinkage or folds in the seal are expected to also be closed.

As mentioned in chapter 4.7 Squeeze-out, the higher the seal settings, the higher the squeeze-out. To close microleakages, squeeze-out is necessary [8, 10]. Combining both these principles would seem to indicate that the higher the seal settings, the better the microleakages are closed. This is only valid if no excessive squeeze-out is occurring [10]. No excessive squeeze-out has been observed for ultrasonic sealing (more than 15% thickness reduction at the seal). This is explained in chapter 5.11 Test 7. Squeeze-out. Therefore, the assumption seems plausible.

'Arrowpack push indicator dye test' packages are used to execute the dye penetration test. A test is placed in the created package. The blue dye fluid is pressed out and brought into contact with each seal for 5 seconds. See **Figure 22** for the use of an Arrowpack push indicator dye test.



Figure 22. Use of Arrowpack push indicator dye test [145]

# **T-peel test**

In ASTM standard F88 the T-peel test is described. This test systematically evaluates the seal strength. A sample is cut which includes a seal. This sample is placed into two 'peeling mouths'. These mouths will start to peel the seal. The seal forms a 'T' when it is placed in the oppositely placed peeling mouths. The maximum peeling force and the seal failure mode should be recorded [3]. The different seal failure modes can be found in **Figure 23**.



In this thesis, the tests will be executed using the KOPP Labormaster HCT 3000. This machine is shown in **Figure 24**. **Figure 25** shows how the T-peel tests are done by this machine. Stripes of 25mm will be cut from the sample packages containing a conduction or an ultrasonic seal. Each end of the sample is clamped between a cylindrical mouth. The bottom mouth will start to rotate (with a speed of 200mm/min) and will start to peel the seal.

After the bottom mouth has finished its round, the machine will show a graph with the time on the horizontal axis and the peeling force on the vertical one. The maximum peeling force is also given. The

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graph and the measurements can be saved. The peeling force must be at least 0,50N, otherwise the machine will not register any force.





Figure 24. KOPP Labormaster HCT 3000, the T-peel tester

#### Figure 25. Functioning of the T-peel tester (KOPP Labormaster HCT 3000)

A research that also performs T-peel tests uses 7 repetitions per treatment [146]. This will also be used as a minimum amount for this research. See the chapter Design of Experiment to see why repetitions are necessary and why not one test for each treatment is sufficient.

#### T-peel test failure modes

The failure mode that is usually observed during the testing, is the cohesive peel. This can be recognized by the material that is added to/ stripped of the seal area, which can be observed as a whitened seal area. If a weak seal is created, an adhesive peel might be observed (sometimes observed at conduction sealing). This seal failure mode is rarely observed during the T-peel tests. This makes sense since two of the same sealant layers are sealed together. This means that the seal interface is not a weak spot where the seal will probably fail.

Another failure mode that is (sometimes) observed during the T-peel tests, is delamination. In that case, a part of the film is detached (delaminated) from the rest of the film.

In **Figure 26** are some pictures of observed seals with different failure modes. The failure mode of each seal is recorded and can be found in 'Appendix 13. Testing result'.



Figure 26. Examples of different failure modes: cohesive (A and B), delamination (C) and adhesive peel (D)

## **Microscopic imaging**

Microscopic imaging can be used to magnify an object for visual observation. A digital microscope combines an optical microscope with digital multimedia and processing technology. The magnified representation of the object can be observed on a screen (e.g. computer monitor) [147].

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For this thesis, a digital microscope will be used to get an enlarged visual of the seal area. For the seal area to be properly investigated with the microscope, the samples need to be embedded in epoxy. The epoxy must be hardened for at least 24 hours. Then, the samples must be grinded and polished, to make sure that a clear picture will appear on the microscope. This is done with the Struers Tegramin-30 machine.

The last step that needs to be taken to create pictures using the digital microscope, is putting the sample on the microscope and adjusting the settings to get a clear image of the seal. This is done on the Keyence VHX 7000 digital microscope. The samples are cut parallel to the seal to get an image of the seal area. See **Figure 27** for the equipment used to create the microscopic images.



*Figure 27.* Equipment used for microscopic imaging Struers Tegramin-30 (left) [7] and Keyence VHX 7000 digital microscope (right) [12]

### **Test packages**

The packages that are created for the tests will be described in this section. The tests should answer the research questions concerning conduction and ultrasonic seals. The packages will be realized on a flowpack machine (an Omori bellpack machine) with an ultrasonic longitudinal seal and a conduction transverse seal.

#### Material

The materials used through the testing phase have the following specifications:

-	OPE / PE EVOH PE 25/50 (easy peel)	[Material 1]
-	OPE / PE EVOH PE 20/25 (easy peel)	[Material 2]
-	BOPP / PP 16/50 (easy peel)	[Material 3]
-	BOPP / PP EVOH PP 20/30 (lock seal)	[Material 4]

All materials are classified as mono-materials, either mono-polyethylene (PE) or mono-polypropylene (PP). The EVOH (ethylene vinyl alcohol) is in the films for its barrier properties. The easy peel sealant layers are created using controlled contamination with polybutylene (PB).

#### Conditioning

According to the ASTM F88 standard, the samples should be conditioned for at least 40h according to ASTM standard E171. That standard states that no meaningful physical changes in the packages are found when the packages are preserved in a laboratory or an office compared to conditioned surroundings [3, 148].

The packages created are shown in **Figure 28**. They are kept in the same box in a laboratory. It is assumed that the laboratory conditions don't noticeably affect the seal strength.

#### **Settings CS**

The temperature is varied for the conduction seals (pressure and time are constant). This is done with a temperature range that



Figure 28. The packages that are created are conditioned in a laboratory within a cardboard box

starts at 90°C and ends at 150°C or until the packages stick too much to the sealing jaws. This range is selected based on the test results from previous tests (see Appendix 12. Testing at Omori). A 10°C interval is selected based on the work from Bach, et al. [9], who also measured the relation between temperature and seal strength with the same interval. These samples are created to see what seal strengths can be achieved on the mono-materials using conduction sealing.

The maximum temperature at which seals are realized is 150°C. Above this temperature, the material sticks too much to the sealing jaws. Material 2 is tested for lower temperatures than the other materials. During testing, the film stuck too much to the sealing jaws to seal with higher temperatures than 110°C. At this temperature, shrinkage at the seal was visible. The seals that stuck to the sealing jaw sometimes ended up as packages that were all pasted to each other. To test the seal strength, the packages are detached from each other. Both the side that was connected to the other seals as well as the side that was not, were tested.

#### **Settings US**

The packages are created with variating temperature, amplitude and pressure. The rest of the variables influencing the seal strength are kept constant as well as possible. The settings used for ultrasonic seals are shown in **Table 1**. Some extra packages have been created with material 1. With these packages, the ultrasonic seal also has amplitudes lower than 70%. The reason for this is to check the relationship between amplitude and seal strength. To make that relationship obvious, the amplitude should be varied while keeping the pressure the same.

**Table 1.** The ultrasonic seal settings used to create the packages. Low settings are only tested for material 1 (mat. 1), the other settings are applied to all materials

		Amplitud	e (%)						
		30	40	50	60	70	80	90	100
Pressure	0,1					All			All
(bar)	0,3						All		
	0,5						All		
	0,7						All		
	0,9		Mat. 1	Mat. 1	Mat. 1		All		
	1,1					All			All
	1,3	Mat. 1	Mat. 1	Mat. 1	Mat. 1				All
	1,5								All
	1,7								All
	1,9								All
	2,1								All
	2,3								All

The reason for these specific values is based on a few elements. First, to determine the nature of the relationship between the factor and the seal strength, samples are realized to perform a 2k full factorial Design of Experiment. This is done with the factors pressure and amplitude. The factor levels for pressure are 0,1 bar and 1,1 bar and for the amplitude 70% and 100%. These values indicate a 'high' and a 'low' setting within the likely operating window. As mentioned before, the two levels should be far enough apart that a difference in response is expected, but not outside the likely operating range [134]. It is assumed that this is the case for the mentioned levels. This assumption is based on the data presented in Appendix 12. Testing at Omori.

As the 2k full factorial DoE cannot indicate the nature of the relationship between the factors and the response, more samples will be created. Since the pressure of the ultrasonic sealing system can be varied over a large range, the pressure will be varied for 80% and 100% amplitude. This will give insight into the nature of the influence the pressure has on the seal strength (e.g. linear or exponential).

#### Acceptable T-peel test ultrasonic seal

While performing the first T-peel tests on the ultrasonic seals, some extensive variation in maximum seal strength could be observed. In Appendix 3. Acceptable ultrasonic T-peel test samples it is explained that this huge variation is partly caused by the samples being put in the sealing mouths at an angle, increasing  $\theta$  (the angle that the seal differentiates from being exactly parallel to the peeling mouth as can be seen in **Figure 29**).

It is practically impossible to always place the sample perfectly parallel to the peeling mouths. The graph showing the peeling force against the time can indicate if the substrate was placed parallel enough or not. Therefore, three types of peels will be distinguished: parallel 1 (perfectly parallel), parallel 2 (almost parallel) and oblique. The graphs as well as the peeling type will be recorded for all tests (in addition to the maximum peeling force and the failure modes).



Figure 29. Explanation of the mouth-seal angle of the T-peel test

Parallel 1 and 2 will be included in the analysis, while tests classified as oblique will be excluded. This partly explains the variation in sample size for the ultrasonic seals.

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Note that it is recommended to read the appendix explaining what tests for ultrasonic sealing are acceptable before starting to read the tests (Appendix 3. Acceptable ultrasonic T-peel test samples).

# 5.3.Two-way ANOVA

To draw proper conclusions from the data, statistical methods should be used. These techniques will help to analyze the data retrieved from tests and say to what extent this data shows significant differences.

The statistical technique that is mostly employed in this thesis, is the analysis of variance (ANOVA). The ANOVA test shows how well a variable can predict the outcome of a dependent variable. This conclusion is represented in an F-ratio. This compares the amount of systematic variance in the data and the amount of unsystematic variance. The amount of systematic variance is explained by the independent variable and the unsystematic variance is the error [130].

The independent variable influences the dependent variable. The independent variable is called a factor in the ANOVA context. This factor has different so-called levels. These levels are the different options for the factor. For example, when there is a factor pressure and the influence of the pressure on the temperature will be measured at 1 bar, 2 bar and 3 bar, the factor pressure has three levels. The measurement at the treatment of 1 bar might be done a couple of times. The revision of the same treatment is called a replicate [149].

ANOVA is a special case of regression analysis and is usually used in the case of controlled experiments [130]. There are different kinds of ANOVA, for example, one-way ANOVA, which includes only one independent factor. When there are two or more factors and two or more conditions for these factors, a factorial ANOVA should be executed. This type of ANOVA can be used for empirical studies to investigate the differences between groups. The big difference with one-way ANOVA is that in factorial ANOVA, is that the groups are also tested for an interaction effect of multiple factors. The main effect is the effect of the factor on the dependent variable. The interaction effect is the interaction between the factors [149-151]. A factorial ANOVA in the case of two factors is called a two-way ANOVA. In this case, the two factors are expected to explain the variability within the dependent variables [150].

The hypotheses that are tested with a two-way ANOVA are [152]:

- There is no difference in the row's main effect
- There is no difference in the column's main effect
- There is no interaction

If these hypotheses can be rejected, that means that the two factors a have significant effect on the dependent variable and that both factors have an influence on each other.

Another interesting feature the ANOVA test can do is examine if there are trends between the factors and the independent variable, e.g. linear and quadratic trends [130].

#### Assumptions

Different assumptions are connected to the ANOVA test. These assumptions are [130, 149]:

- The variance of each factor should be similar, in other words, the variance should be homogeneous.
- The observations should be independent.
- The dependent variable should be measured on at least an interval scale.
- The distribution within groups should be normally distributed.

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# 5.4. Testing of the ANOVA assumptions

During the analysis part of this thesis, the results will be analyzed to draw conclusions from the gathered data. This is done using the programs Microsoft Excel and the statistical software SPSS. Different tests have been executed to answer different research questions. For all of them that are analyzed with the ANOVA test, the testing of the assumptions is done as described below. The assumptions are described in part 5.3 Two-way ANOVA. If the assumptions are not met, the results may not be valid [153].

#### Variance

Homogeneity, the variance within all groups must be equal. This is only required if the sample sizes are very unequal. If that is the case, the Levene's test can indicate whether this assumption is met [130, 154].

The Levene's test evaluates if the variation between certain groups differs. The test will be performed based on the mean. The null hypothesis of this test is that the variance between groups is homogeneous (variances are equal). So the outcome of the test should be insignificant (p>0.05) to not violate the assumption.

For this research, each treatment represents a group. The smallest group has 8 measurements and the biggest 14. Whether these sample sizes are very unequal is debatable. To be safe, the Levene's test will be performed and reported for the different ANOVAs that will be executed. If the result of this test is significant, the results might be invalid.

#### Measurement

There are two assumptions regarding the way of measuring. First, the observations should be independent. This is indeed the case. For each test a different plastic sample has been used, so there are no duplications. Secondly, the dependent variable (seal strength) should be measured on at least an interval scale. This assumption is also met since the seal strength is measured on a ratio scale, so the difference between measurements can be interpreted and a true zero value exists.

#### **Normal distribution**

The last assumption is that within each group, there should be a normal distribution [130]. This is only required in case of small sample sizes. The sample size per treatment is maximum of 14, which is seen as a small sample size [154].

So the normal distribution within the groups will be evaluated. This can be done by performing the Kolmogorov-Smirnov test or the Shapiro-Wilk test. Although both have low power for low sample sizes [155], the Shapiro-Wilk is a more appropriate method for small sample sizes [156]. Therefore this test will be performed. This test tests the null hypothesis that there is a normal distribution within a group. So if the results are significant, there is a high chance that there is no normal distribution within a group and the normal distribution assumption will thus be violated [130, 155]. The outcome of the Shapiro-Wilk test will be reported for each group included in the ANOVA. If the outcome is significant, the interpretation of the results for the specific group could be invalid.

# 5.5.Test 1. Influence of temperature on seal strength

## **Research question**

To find the optimal sealing process parameters, the seal performance will be evaluated. So both the seal strength and the seal integrity of different seals (US and CS) with variating parameters and materials will be investigated.

There are a lot of parameters that influence the seal strength, both for conduction and for ultrasonic sealing [9, 84]. To make a comparison between different materials it is however not workable to find the optimum for every parameter. Therefore, a limited amount of parameters are variated during the experiments, while the other parameters stay the same. This part of the research focuses on finding the influence some process parameters have on the resulting seal strength. The research question that will be answered through this research is presented below. The '[CS]' in front of the question indicates that the question is about conduction sealing.

[CS] What is the relationship between the seal strength and the temperature?

The answer to this question will show the relationship between some process parameters and the resulting seal strength. The insights gained from this will help to select the optimal process parameters for the conduction sealing process.

## Theory and hypothesis

A hypothesis is formulated for the research question based on public literature. The influence of temperature on seal strength in the conduction seal has been extensively investigated. The relationship between the seal strength and the temperature shows certain trends. These trends are already described in chapter 4.7 Squeeze-out. Based on that chapter a hypothesis is formed. It is expected that the seal strength increases with increasing temperature until a plateau is reached. If the temperature is further increased, it is expected that the seal strength will decrease again. Bamps, et al. [8] show this trend in their paper, this graph is also shown in **Figure 16**. This is only the case if the sealant layer is squeezed out of the sealing area and the next inner layer has less bonding capacity than the sealant layer.

This implies that it is also possible to see an increase in seal strength after the plateau if the bonding capacity of the next inner layer is bigger than that of the sealant layer. This is for example described by Nase, et al. [84] where an easy peel sealant layer (PP, LLDPE, LDPE & iPB-1) is squeezed out of the seal area. So it is expected that the relationship between the temperature and the seal strength will be similar to that visualized in **Figure 16** previously depicted.

### Method

The different methods used to answer the research question are extensively described in chapter 5.2 Testing methodology. In short, the temperature will be varied and the seal strength will be measured. To be specific, the methodology sections that apply to this test, are: 'T-peel test', 'Test packages' and 'Settings CS'.

## **Data collection**

The results of the conduction sealing seals are presented in Appendix 4.1. Test 1. Data analysis. As mentioned in the section 'Settings CS', material 2 is tested for different temperatures than the other materials.

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#### **Data analysis**

To answer the research question of what the relationship between the temperature and the seal strength is for conduction sealing, an analysis is required. This analysis is necessary to see if the material behaves as expected with heat sealing. If this is not the case, the measurements of the ultrasonic sealing part could be invalid due to a defect in the material used for the testing.

A boxplot that summarizes all the gathered data for conduction sealing is presented in **Figure 30**. What each material is can be found in the section 'Material'.

From this representation of the data can be observed that material 4 shows the highest seal strengths. The temperature in general seems to have a positive effect on the resulting seal strength. It can also be noted that the seal strength for material 2 is constantly 0N/25mm.



*Figure 30.* Boxplot of all gathered data for conduction sealing

To further investigate the influence of the temperature, a two-way ANOVA has been conducted with the factors temperature and material. The conclusion of this part is whether the temperature and the material have a significant effect on the seal strength.

A summary of the conclusions of the two-way ANOVA is presented in Table 2. One treatment represents one group, so each unique temperature and material combination forms a group (e.g. material 1 with 110°C is one group). The sample sizes are very similar (between n=7 and n=9), so a significant Levene's test is not problematic for the interpretations of the results. A normal distribution within the groups is however not the case.

The results of the analysis show that there seems to be a significant effect of all factors and their interaction on the remaining seal strength. Based on the F-test, the material seems to be the most dominant factor. See Appendix 4.1. Test 1. Data analysis for more elaborate results from the analysis (like the partial eta squared and the sum of squares).

Table 2. Results of the two-way ANOVA testing the influence of temperature and material on SS for CS

Material	Test	Significance Levene's*	Significance ANOVA**	F-test
ALL	Levene's test	<0,001		
	Influence of temperature		<0,001	36
	Influence of material		<0,001	116
	Influence of interaction		<0,001	37

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is  $\leq 0.05$ , the impact is significant.

A graphical representation of the results of the ANOVA is a graph that plots the temperature against the estimated marginal means for the seal strength for different materials (**Figure 31**). It seems that the temperature has a positive influence on the seal strength, although materials 2 and 3 don't seem to be increasing much.

Material 2 is an outlier compared to the other materials. This material got stuck to the sealing jaws too<br/>much. The film completely melted at temperatures above 110°C, which is why measurements could<br/>only be made for lower temperatures. At these low temperatures, the seal strength was (without<br/>exception) 0N/25mm. That is why later in this section the one-way ANOVA does not return useful<br/>results.10.12.



Figure 31. Temperature against estimated seal strength for different materials (error bars at 95% confidence)

To see what the exact influence of the temperature is, the materials will be separately evaluated. A summary of the gathered data per material can be found in **Figure 32**. What stands out is that material 2 exclusively returns a seal strength of 0N/25mm as mentioned before. Furthermore, it can be observed that materials 3 and 4 show a similar trend of the seal strength slightly going up and then down with increasing temperature.

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Figure 32. Boxplots of temperature against seal strength for CS for materials 1, 2, 3 and 4

A summary of the results of the one-way ANOVAs is given in **Table 3**. In this table can be observed that the temperature has a significant effect on all materials, except material 2.

Material	Test	Significance Levene's*	Significance ANOVA**	F-test
MATERIAL 1	Levene's test	<0,001		
	Influence of temperature		<0,001	84
MATERIAL 2	Levene's test	-		
	Influence of temperature		-	
MATERIAL 3	Levene's test	<0,001		
	Influence of temperature		0,019	3,4
MATERIAL 4	Levene's test	<0,001		
	Influence of temperature		<0,001	40

Table 3. Results of the one-way ANOVA testing the influence of temperature on SS for CS

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is ≤0.05, the impact is significant.

The results of the one-way ANOVAs are visualized in **Figure 33**. This shows similar results as already observed in the boxplots. Especially materials material 3 and material 4 show a clear graph of the changing seal strength. For material 1 the seal strength at 120°C and 130°C seems strange, since the temperature increases, but the seal strength decreases between these two temperatures. It is even the case that the seal strength at 130°C is 0N/25mm, which is also remarkable.

What is also remarkable is when looking at the Tukey post-hoc test, material 3 only shows a significant effect between 110°C and 130°C and between 110°C and 140°C. This is probably due to the high error margins that can be observed in the boxplot.



Figure 33. Temperature against the estimated seal strength for materials 1, 2, 3 and 4 with CS (error bars at 95% confidence)

When trying to describe the relationship between the temperature and the seal strengths, results from material 2 are discarded. For the other materials, the relation can be described as follows, with increasing temperature the seal strength increases linearly until slowly a local maximum is reached. After this maximum, the seal strength decreases again.

#### Conclusion

From the presented analysis can be concluded that the temperature has a significant effect on all materials except material 2. Therefore, there is no reason to mistrust the results of the ultrasonic tests with these materials.

The seals created with conduction sealing on material 2 were not closed. This was caused by the material sticking to the sealing jaw. The sticking has not been observed with ultrasonic sealing and the ultrasonic seals were closed. Therefore, it is assumed that also material 2 is suitable to perform tests on with the ultrasonic sealing system.

The nature of the relationship between the temperature and the seal strength is described as linearly increasing with a local maximum. This corresponds with the hypothesis that has been formulated.

It should be noted that some of these conclusions should be interpreted carefully since some Levene's tests and Shapiro-Wilk tests are significant.

# 5.6.Test 2. Influence of pressure and amplitude on seal strength

#### **Research question**

As mentioned before, the temperature cannot directly be set for ultrasonic sealing. This makes ultrasonic sealing a less intuitive technology to operate than conduction sealing. To still be able to find the optimal sealing process parameters, the seal performance will be evaluated. So both the seal strength and the seal integrity of different seals with variating parameters and materials will be investigated.

There are a lot of parameters that influence the seal strength, both for conduction and for ultrasonic sealing [9, 84]. To make a comparison between different materials it is however not workable to find the optimum for every parameter. Therefore, a limited amount of parameters are variated during the experiments, while the other parameters stay the same. This part of the research focuses on finding the influence some process parameters have on the resulting seal strength. The research questions that will be answered through this research are listed below. The '[US]' sign before the questions indicate that the questions regard ultrasonic sealing.

[US] What is the relationship between the seal strength and the amplitude?

[US] What is the relationship between the seal strength and the pressure?

The answers to these questions will show the relationship between some process parameters and the resulting seal strength. The insights gained from this will help to select the optimal process parameters for both the conduction and the ultrasonic sealing process.

#### Theory and hypothesis

In most studies that look at the seal strength of ultrasonic sealing, the pressure varies [9, 84]. However, one research has been found that also indicates the relationship between the amplitude and the seal strength. This is described in the research performed by D'huys, et al. [24]. The relationship that they found is visualized in **Figure 34** on the right. Based on this result, a hypothesis is created for the first question of Test 2. It is expected that the amplitude will have a positive effect on the seal strength. First, the effect is quite big, but this is slowly reaching a plateau.

It is good to first note that the pressure and the sealing force refer to the same parameter in the ultrasonic sealing context. In this thesis this parameter is referred to as 'pressure', but for example in **Figure 34** (copied from D'huys, et al. [24]), there is referred to the sealing force instead of the pressure on the horizontal axis.

The hypothesis for the second question, the relationship between pressure and seal strength of the ultrasonic sealing process, is also based on the research from D'huys, et al. [24]. It is expected that the sealing force influences the seal strength in a positive linear way. A representation of the expected relationship trend can be found in **Figure 34** on the left.



Figure 34. Influence of sealing force and amplitude on seal strength for ultrasonic sealing [24]

For easy peel films, the relationship between sealing force and seal strength seemed to be exponential and positive for the findings presented by Nase, et al. [84]. So it might also be possible that for easy peel films, the relation between sealing force and seal strength is exponential instead of linear.

#### Method

The different methods used to answer the research question are extensively described in chapter 5.2 Testing methodology. In short, the temperature will be varied and the seal strength will be measured. To be specific, the methodology sections that apply to this test, are: 'T-peel test', 'Test packages' and 'Settings US'.

#### **Data collection**

The descriptive statistics of the seals created with ultrasonic sealing are presented in Appendix 5. Test 2. Results. The realization of these seals went according to the test plan.

### Data analysis

The analysis of the data about ultrasonic sealing is done in the following section. This is done using ANOVA tests, descriptive statistics and boxplots. By first looking at the results, it stands out that the standard deviation is quite large. Especially if this is compared to conduction sealing. Bach, et al. [9] also observed this and think this might be caused by high sealing forces that are associated with uncontrollable melt flow or by the difference in crack and failure behavior.

#### Analysis of T-Peel test Results

To find the relationship between the pressure and the amplitude of the ultrasonic sealing system and the resulting seal strength, a DoE has been conducted (see chapter Design of Experiment for more information on this topic). As mentioned in the section 'Settings US' two factors (pressure and amplitude) with each having 2-factor levels (70%; 100%; 0,1 bar and 1,1 bar) are selected.

A summary of the analysis results of the Design of Experiment part can be found in **Table 4**. The Levene's test is significant for materials 2 and 4. The normality is evaluated per group by the Shapiro-Wilt test. The results of this can be found in Appendix 5.1. Test 2. Data analysis. Some groups show a significant result from this test. For these few groups, the results could be invalid. In this context, one group is a unique combination of pressure, amplitude and material.

What can be observed is that for these tests the amplitude seems to be the most dominant factor determining the seal strength. The effect of the amplitude is significant for all tests and the F-scores

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are higher than for the other factors. From this table, the pressure and the interaction effect don't seem to have a dominant effect. Sometimes the effect is significant and sometimes it is not.

Material	Test	Significance	Significance	F-test
		Levene's*	ANOVA**	
MATERIAL 1	Levene's test	0,463		
	Influence of pressure		0,003	10
	Influence of amplitude		<0,001	106
	Influence of interaction		<0,001	14
	effect			
MATERIAL 2	Levene's test	0,018		
	Influence of pressure		0,450	0,6
	Influence of amplitude		<0,001	180
	Influence of interaction		<0,001	3
	effect			
MATERIAL 3	Levene's test	0,705		
	Influence of pressure		0,050	4
	Influence of amplitude		<0,001	195
	Influence of interaction		0,269	1
	effect			
MATERIAL 4	Levene's test	0,026		
	Influence of pressure		0,134	2
	Influence of amplitude		<0,001	26
	Influence of interaction		0,699	0,2
	effect			

Table 4. Results of the 2k full factorial DoE analysis for materials 1, 2, 3 and 4 with US

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is ≤0.05, the impact is significant.

To visualize the analysis results of this section, some graphs are created. **Figure 35** shows the influence pressure has on the different amplitudes being represented by different lines. **Figure 36** includes graphs that have the amplitude on the horizontal axis and the different lines represent the different pressure levels.

A few observations can be made from these graphs. It has already been stated that the influence of pressure for materials 2 and 4 is not significant according to the Design of Experiment analysis. This can also be observed in **Figure 35**, where the lines in the graphs don't seem to have an angle. It is also interesting to see that for material 4 material, the pressure even seems to decrease the seal strength. Although the influence of pressure in that case is not significant, it is still remarkable.

As can be observed from **Figure 35**, from the measurements used, the pressure does not seem to have a significant effect on the resulting seal strength. This is different than **Figure 36**, where the lines seem to have a significant angle. This also confirms that the amplitude influences the seal strength positively.



Error bars: 95% CI MATERIAL 3 MATERIAL 4 Figure 36. Amplitude against estimated seal strength DoE for materials 1, 2, 3 and 4 with US (error bars at 95% confidence)

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Amplitude\_in\_percentage

Error bars: 95% CI

100

15,0

91

100

Amplitude\_in\_percentage

#### Trends

Now that it has been established that the amplitude influences the seal strength and that the pressure and the interaction effect slightly or don't influence the seal strength at 70 and 100% amplitude, the kind of relation that exists can be examined. This can for example show whether there is a linear or an exponential relationship between amplitude and seal strength.

#### Amplitude

The nature of the influence that the amplitude has on the seal strength will be investigated in this section. This is done for material 1 since this is the only material that has multiple measurements of the amplitude with a constant pressure. A one-way ANOVA is conducted with the factor amplitude and the response seal strength.

To see a general trend, a two-way ANOVA with the factors amplitude and pressure has been conducted with all the measurements from material 1. The results are summarized in **Table 5**. The Levene's test is significant, so drawing conclusions from the results should be done with care. As mentioned before, the normality is evaluated in Appendix 5.1. Test 2. Data analysis.

Table 5. Results of the two-w	ay ANOVA influence of pressure a	nd amplitude for material 1 and US
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Material	Test	Significance Levene's*	Significance ANOVA**	F-test
MATERIAL 1	Levene's test	<0,001		
	Influence of pressure		<0,001	16
	Influence of amplitude		<0,001	160
	Influence of interaction effect		<0,001	25

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is ≤0.05, the impact is significant.

The visual result of this analysis can be found in **Figure 37**. When looking at this figure, there does not seem to be a linear relationship between the amplitude and the seal strength, but rather exponential.



Figure 37. Amplitude against the estimated seal strength material 1 for US (error bars at 95% confidence)

## Influence of amplitude at restricted pressures (0,9 and 1,3bar)

To see what the exact relationship between the seal strength and the amplitude is, a one-way ANOVA will be conducted with a 0,9 bar and a 1.3 bar pressure. In **Table 6** a summary of the results can be found. The Levene's test is significant and the normal distribution within groups is evaluated in Appendix 5.1. Test 2. Data analysis The results of the analysis indicate that the amplitude is significant in both cases.

Table 6. Results of the one-way ANOVA influence of amplitude on SS for US

Material and treatment	Test	Significance Levene's*	Significance ANOVA**	F-test
MATERIAL 1	Levene's test	<0,001		
0,9 bar				
	Influence of amplitude		<0,001	216
MATERIAL 1	Levene's test	<0,001		
1,3 bar				
	Influence of amplitude		<0,001	120

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is ≤0.05, the impact is significant.

In **Figure 38** the results of the one-way ANOVA are presented. Note that the error bars are not included in the top graph. The graphs in the table that include error bars do not have a proportional horizontal axis. To visualize the trend, the graphs without error bars, but with a proportional horizontal axis are included. From these graphs can be observed that the amplitude seems to have an exponentially growing influence on the seal strength until the influence decreases again.



Figure 38. Result plots of the one-way ANOVA with varying amplitudes for material 1 with US (error bars at 95% confidence)

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#### Pressure

The influence of pressure on the seal strength is evaluated in the next part. The seal strengths of the four materials at 80% and 100% amplitude for different pressure settings are measured. For material 1, the seal strength is also evaluated at 40%, 50% and 60% amplitude. These measurements will help to understand the relationship that exists between the pressure and the seal strength. But first **Figure 39** will be analyzed (see **Table 5** for the data corresponding to this graph). In this figure, a graph can be observed that includes all measurements of material 1 plotting the pressure against the seal strength. What can be observed is that for higher seal strengths, the pressure does not seem to have much influence, but for lower seal strengths it seems to have an effect. To what extent this observation is correct will be evaluated in the following parts.



#### Estimated Marginal Means of SealStrength\_in\_Nper25mm

Figure 39. Pressure against the estimated seal strength material 1 for US (error bars at 95% confidence)

To evaluate whether this observation is correct, the gathered data will be analyzed. Especially the difference between the pressure effect for high and low amplitude values.

#### Influence of pressure on the seal strength at restricted amplitudes (40%, 50% and 60%)

From **Figure 39** can be observed that the pressure does seem to affect the seal strength lower in the graph, so at the lower estimated marginal means of the seal strength. The lower seal strengths are created with the lower amplitudes. Therefore, a one-way ANOVA will be executed that only includes measurements of material 1 with amplitudes of 40, 50 and 60%. The pressure levels are 0,9 and 1,3 bar. A summary of the results is presented in **Table 7**, which indicates that the influence of the amplitude is significant. Note that the Levene's test is also significant.

Table 7. Results of the one-way ANOVA influence of restricted amplitude levels (only 40, 50 and 60%) for material 1 with US

Material	Test	Significance Levene's*	Significance ANOVA**	F-test
MATERIAL	Levene's test	<0,001		
1				
	Influence of amplitude	<0,001		120

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is  $\leq 0.05$ , the impact is significant.

**Figure 40** illustrates the results of the two-way ANOVA, where it seems obvious that the higher the pressure, the higher the seal strength. When looking at this graph, it seems that the pressure gains influence (shows steeper lines) when the amplitude also increases. This conclusion can however not be drawn, since the seal strength of 50% and 40% at 0,9 bar are close to 0N/25mm. So it could be the case that the pressure has more influence on the seal strength at 40% than at 60% amplitude. This could for example be the case if the seal strength of 40% amplitude only starts to increase above 1,2 bar. To see at what amplitudes the pressure has the most influence, more measurements between 0,9 and 1,3 bar are necessary.





*Figure 40.* Pressure against estimated seal strength material 1 for 40, 50 and 60% amplitude for US (error bars at 95% confidence)

Influence of pressure on the seal strength at restricted amplitudes (80% and 100%)

Next to these measurements, it is necessary to look at the higher amplitudes to see if the pressure has a significant effect there. This is done for each material at 80% and 100% amplitude. A summary of the results of the one-way ANOVAs can be found in **Table 8**. From this table can be observed that in most cases, the pressure does not seem to have a significant effect on the seal strength.

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Material and	Test	Significance	Significance	F-test
treatment		Levene's*	ANOVA**	
MATERIAL 1 80%	Levene's test	0,967		
	Influence of pressure		0,777	0,4
MATERIAL 2 80%	Levene's test	0,405		
	Influence of pressure		<0,001	8,1
MATERIAL 3 80%	Levene's test	0,457		
	Influence of pressure		0,585	0,7
MATERIAL 4 80%	Levene's test	0,468		
	Influence of pressure		0,237	1,5
MATERIAL 1 100%	Levene's test	0,027		
	Influence of pressure		0,421	1,0
MATERIAL 2 100%	Levene's test	0,076		
	Influence of pressure		<0,001	17,4
MATERIAL 3 100%	Levene's test	0,096		
	Influence of pressure		0,311	1,2
MATERIAL 4 100%	Levene's test	0,011		
	Influence of pressure		<0,001	5,1

*Table 8.* Results of the one-way ANOVA influence of pressure for US (p<0,05)

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is  $\leq 0.05$ , the impact is significant.

The summary of the results only draws conclusions based on all measurements. It does not give insights into the difference between the treatments. To investigate whether there is a significant difference between each treatment group, a Tukey post-hoc test is performed. This test concludes that all treatments are significantly different if the effect of pressure is significant, except for material 2 at 80% amplitude. There is no significant difference between 0,3 and 0,9 bar and not between 0,5 and 0,7 bar for this group. This can also be observed when looking at the boxplots of the data, which is shown in **Figure 41**.



*Figure 41.* Boxplots of pressure against seal strength for 80% amplitude for materials 1, 2, 3 and 4 with US (error bars at 95% confidence)

To interpret the collected data and visualize it, boxplots have been created. This summarizes the data. What can be observed in the boxplots from the 80% amplitude treatments (**Figure 41**) is that there does not seem to be much difference in seal strength for different pressures.

Next to the boxplots, also a graphical representation of the results of the one-way ANOVA is given. The graphs that plot the pressure against the estimated seal strength can be seen in **Figure 42** for 80% amplitude treatments. From these tables the same conclusion can be drawn as from the boxplots, there does not appear to be a strong influence of pressure on the seal strength for 80% amplitude.



*Figure 42.* Pressure against the estimated marginal means of seal strength for 80% amplitude for materials 1, 2, 3 and 4 with US (error bars at 95% confidence)

The boxplots of the 100% amplitude treatments can be found in **Figure 43**. From these graphs can be observed that the error term is huge for some measurements (e.g. material 1; 100% and 2,3 bar). Furthermore, there does not appear to be a clear influence of the pressure on the remaining seal strength, except for material 2. At this material, especially with the higher pressures, the pressure seems to have an effect.

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*Figure 43.* Boxplots of pressure against seal strength for 100% amplitude for materials 1, 2, 3 and 4 with US (error bars at 95% confidence)

A graphical representation of the results of the one-way ANOVA with a 100% amplitude is presented in **Figure 44**. Only materials material 2 and material 4 have a significant influence on the seal strength (see **Table 8**). While taking a look at the results of the ANOVA, it is noticed that no clear trend can be observed. The seal strength response seems to be increasing for higher pressures, but this is not consequently observed.

For material 2 it indeed seems logical that the pressure has a significant effect, especially for higher pressures. Starting at 1,5 bar the seal strength seems to be increasing with increasing pressure.

The fact that material 4 at 100% amplitude shows a significant result is interesting since no clear relation can be observed. The seal strength first increases with increasing pressure and after 1,7 bar it decreases again.



*Figure 44.* Pressure against the estimated marginal means of the seal strength for 100% amplitude for materials 1, 2, 3 and 4 for US (error bars at 95% confidence)

#### Interaction effect amplitude and pressure

In previous tests, sometimes the significance of the interaction between factors has been reported. To evaluate the interaction effects (especially between the amplitude and the pressure) and to see if the material influences the seal strength, a three-way ANOVA of all measured data will be performed.

A summary of the results is presented in **Table 9**. While looking at this summary, it seems clear that the material indeed significantly influences the seal strength. More precisely, all factors and interactions seem to have a significant effect, except the interaction between all three factors. Based on the F-tests, it can be concluded that the amplitude has the most influence on the seal strength.

Whether the interaction effect between the pressure and the amplitude is significant will be evaluated now. This is indeed the case when looking at the results of the two-way ANOVA performed with material 1 with 40, 50 and 60% amplitude. In the two-way ANOVA performed on the Design of Experiment data, only two materials show a significant interaction effect between pressure and amplitude. The results of the three-way ANOVA performed on all measured data (**Table 9**) show that the third largest significant effect is the interaction effect between amplitude and pressure. This effect is even larger than that of the pressure (note that the Levene's test is significant). Concluded from this can be that the amplitude indeed influences the effect the pressure has on the seal strength. This conclusion has not been observed in public literature so forms a nice opportunity for future research (see chapter 9 Future research).

Table 9. Results of the three-way-ANOVA influence of pressure, amplitude and material on SS US

Test	Significance Levene's*	Significance ANOVA**	F-test
Levene's test	<0,001		
Influence of pressure		<0,001	7,7
Influence of amplitude		<0,001	94
Influence of material		<0,001	1689
Influence of interaction amplitude		0,002	5,2
and material			
Influence of interaction pressure		<0,001	4,9
and material			
Influence of interaction amplitude		<0,001	8,4
and pressure			
Influence of interaction amplitude,		0,188	1,6
pressure and material			

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is  $\leq 0.05$ , the impact is significant.

#### Conclusion

The conclusion of both the Design of Experiment and the part about trends will answer the following research questions: 'What is the relationship between the seal strength and the amplitude?' and 'What is the relationship between the seal strength and the pressure?'.

First the influence of the amplitude. The results of the analysis suggest that the influence of the amplitude on the seal strength is significant. The higher the amplitude, the higher the seal strength. The trend seems to follow the trend as already depicted in **Figure 38**. So the seal strength is first exponentially growing and then growing slower. This is in line with the hypothesis.

Secondly, the influence of the pressure on the seal strength has been evaluated. For high seal strengths and thus high amplitudes, the pressure does not seem to have a significant effect (for 80% and 100% amplitude). For lower seal strengths and thus lower amplitudes, the pressure does seem to have a significant effect. The nature of this relation could not be established, since only two points have been measured for lower amplitudes. What can be said, however, is that the nature of the relationship between pressure and seal strength for lower amplitudes is positive. This is in line with the hypothesis for non-easy peel films.

It should be noted that some of these conclusions should be interpreted carefully since some Levene's tests and Shapiro-Wilk tests are significant.

What is not included in the hypothesis is the dependence of the pressure on the amplitude. The nature of this relation seems to be the higher the amplitude, the lower the effect of the pressure. What the nature of this relation is, requires more research.

# 5.7.Test 3. Mono-material sealing window CS

#### **Research question**

To compare the mono-material sealing window of conduction and ultrasonic sealing, tests are required. This test will evaluate the sealing window of conduction sealing. The following research question will be answered:

[CS] What is the operating window for conduction sealing?

### Theory and hypothesis

To answer this research question, first, the meaning of the operating window needs to be established. This is defined as the temperature between the 'seal initiation temperature' and the maximum temperature that can be applied without causing damage to the film [23]. The 'seal initiation temperature' is the lowest temperature at which the films are sealed [40]. This window varies from a few degrees to 15°C or even more [28].

This window is dependent on the process and the material [28]. For example, the commonly used sealant layer PE has a broad operating window [37].

Another element that affects the processability of a material in HFFS systems and that is dependent on the sealing temperature, is sticking of the packaging to the sealing jaws. This failure is especially observed when the outer layer of a film is not heat resistant. This is for example the case with some mono-materials, see chapter 2.4 Mono-materials for an explanation about this. This failure causes the operating window to decrease for materials with an outer layer that is not very heat resistant. A possible way to overcome this problem is by using Teflon tape on the sealing jaws. This reduces the adhesion of a film to the sealing jaws [21, 52].

To get a rough feeling of what the operating window could be for conduction sealing, some operating windows mentioned in public literature are collected. A summary of this can be found in **Table 10**.

It is good to note that the operation window is also dependent on other parameters like dwell time and pressure. These parameters are not included in this table. The goal of this table is only to get a feeling for normal temperature operating windows for conduction sealing, so the other parameters are not required.

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Source	Seal initiation temperature (°C)	Maximum temperature (°C)	Operating window (°C)	Material	Notes
[8]	190	230	40	РНА	
[9]	110	160	50	BOPA/LLDPE and BOPA/mPE and BOPA/L(L)DPE	
[28]	100	130	30	LLDPE	Mono-layer, Teflon and BOPP on jaws
[44]	120	180	60	PET/AI/LDPE	
[44]	140	180	40	PET/AI/PET/AI/PET/ LDPE	
[98]	225	250	25	PET/AI/LLDPE and PS/EVOH/PS	Only upper jaw heated, silicone on lower jaw
[52]	110	180	70	BOPP/metBOPET/CP P	Teflon tape on jaws
[13]	140	170	30	OPE/water-based oxygen barrier/LDPE	Mono-material
[40]	120	150	30	OPP/CPP	Mono-material, Impulse heat sealing

Table 10. Selection of operating windows of conduction sealing reported in literature

This table shows that the operating window for conduction sealing differs substantially depending on the material and the application of tape on the sealing jaws. During the tests, no (Teflon)tape will be used on the jaws and mono-materials will be sealed. Therefore, the hypothesis is based on the operating window reported by Carullo, et al. [13]. In their research, a mono-material was sealed and it is not mentioned that tape was applied on the sealing jaws.

It is expected that the operating window for the conduction sealing of mono-materials is similar to the operating window reported by Carullo, et al. [13]. So an operating window of 30°C is expected. However, it is also expected that this heavily depends on the material. Especially the melting temperature of the sealant layer and the heat resistance of the outer layer.

#### Method

The different methods used to answer the research question are extensively described in chapter 5.2 Testing methodology. In short, the temperature will be varied and the seal strength will be measured. To be specific, the methodology sections that apply to this test, are: 'Dye penetration', 'Test packages' and 'Settings CS'.

### **Data collection**

Pictures of the samples that were tested with dye penetration can be found in Appendix 6. Results dye penetration test summary of the results of the dye penetration test for the conduction seal is presented in **Table 11**. In this table, the sealing window is presented for each material. When no dye was visible other than that within the package, the conclusion is draws that the seals were closed.

Note that material 2 has no sealing window since the conduction seals were all opened. This material stuck too much to the sealing jaws to seal at higher temperatures than 110°C as explained in 'Settings CS'.

The maximum sealing window that was possible to obtain was 40°C since the minimum sealing temperature was 110°C and the maximum was 150°C for materials 1, 3 and 4. Some seals stuck to the sealing jaw which caused the packages to paste together, see **Figure 45**. This negatively influenced the sealing window.



*Figure 45. Transverse conduction seals stuck to each other* 

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Table 11.	Results	of dye	penetration	test	CS
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Material	Lowest T (°C)	Highest T (°C)	Sealing window (°C)
MATERIAL 1	150	150	0
MATERIAL 2	-	-	-
MATERIAL 3	110	150	40
MATERIAL 4	120	150	30

#### **Data analysis**

While looking at the sealing window given it can be observed that the parameters for conduction and ultrasonic sealing are different. Therefore, these windows cannot directly be compared to each other.

What is possible to still make somewhat of a comparison is to look at the number of settings that are included within the sealing window. To accomplish this, the precision of the input values needs to be known. The conduction sealing machine used to realize the packages has an input preciseness for the temperature of one decimal (see **Figure 46**).

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	MENU	ADJUST	TEMP. SWI		RODUCT
ТЕМРЕ	RATURE				P300
ID SW	2		IAME	SET.	VAL.
1 00	CENTER	CENTER HEATER FRONT		0.0	0.0
2 00	CENTER	CENTER HEATER BACK		0.0	0.0
3	END HE	END HEATER UPPER		0.0	0.0
4 0	END HE	END HEATER LOWER		0.0	0.0
- 0				0.0	

**Figure 46.** Sealing interface conduction sealing machine. Temperature can be set with 1 decimal preciseness

That means that a sealing window of 40 degrees includes 400 setting options. A comparison and thus the actual analysis can be found in the next chapter (Test 4. Mono-material sealing window US).

#### Conclusion

The sealing window for mono-materials has been evaluated through the described dye penetration tests. The outcome of this is that for two tested materials, the sealing window is close to 0°C and that for two other materials the sealing window is 30°C and 40°C. Note that this is only based on the tightness of the seals and not the appearance or processability. So it is not taken into account if the seals stuck to the sealing jaw or showed traces of shrinking.

# 5.8.Test 4. Mono-material sealing window US

#### **Research question**

To compare the mono-material sealing window of conduction and ultrasonic sealing, tests are required. This test will evaluate the sealing window of ultrasonic sealing. The following research question will be answered:

[US] What is the operating window for ultrasonic sealing?

#### Theory and hypothesis

Just like conduction sealing, ultrasonic sealing also has certain parameters within which the created seals are integer. The range of the minimum and the maximum settings is defined as the operating window. This is influenced by all kinds of parameters, like pressure, amplitude, frequency and sealing time. The parameters that can easily be varied are the pressure and the amplitude. Therefore, these two parameters will be included to determine the operating window.

To get a feeling of what an operating window for ultrasonic sealing might look like, some operating windows mentioned in public literature are gathered and summarized in **Table 12**. It is good to note that not all parameters that influence the operating window are included in this table.

Source	Minimum pressure	Maximum pressure	Operating window	Minimum amplitude (μm)	Maximum amplitude (μm)	Operating window (μm)	Material
[9]	2,5N/mm	6N/mm	3,5N/mm	20	70	50	BOPA/LLDPE
[84]	2N/mm	6N/mm	4N/mm				LDPE & iPB- 1/ LLDPE/PP
[24]	2N/mm	6N/mm	4N/mm	20	35	15	PET/LLDPE- C4

Table 12. Selection of settings ultrasonic sealing from public literature

All materials stated in **Table 12** are not complete mono-materials. However, it is assumed that the data from this table can be used as a basis for the hypothesis. This is based on the knowledge that the ultrasonic sealing system heats the material from the inside, so other layers than the sealant layer have little effect on the operating window.

It is expected that the operating window of the ultrasonic sealing system for mono-material applications will be similar to that reported in **Table 12**.

#### **Data collection**

The results of the dye penetration tests for ultrasonic sealing can be found in **Table 13**. For the ultrasonic seals, two parameters are variated, unlike conduction sealing. The two parameters together form the sealing window. Pictures of the dye penetration test can be found in Appendix 6. Results dye penetration test.

Table 13. Results of dye penetration test US

Material	Lowest P (bar) and A (%)	Highest P (bar) and A (%)	Sealing window (bar) and (%)
MATERIAL	0,1 and 70	2,3 and 100	2,2 and 30
1			
MATERIAL	0,3 and 80 or 0,1 and 100	2,3 and 100	2,0 and 20 or 2,2 and 0
2			
MATERIAL	0,1 and 70	2,3 and 100	2,2 and 30
3			
MATERIAL	0,1 and 70	2,3 and 100	2,2 and 30
4			

#### **Data analysis**

The ultrasonic sealing machine used to realize the packages has an input preciseness for the amplitude of zero decimals and the sealing force of one decimal.

That means that a sealing window of 40 degrees includes 400 setting options. For the ultrasonic sealing window, the sealing force and the amplitude are included. 30% amplitude includes 30 settings and 2,2bar includes 22 settings. Together, it is possible to variate with 660 settings (30\*22). The sealing window in settings is given per material and for sealing technique in **Table 14**. The 400 settings option for material 2 with ultrasonic sealing is used. It is possible to realize closed seals with 400 different settings.

What can be observed is that the ultrasonic sealing technique can realize closed seals for a broader range of settings than conduction sealing techniques.

Material	Sealing window CS (settings)	Sealing window US (settings)
MATERIAL 1	0	660
MATERIAL 2	0	400 (or 22)
MATERIAL 3	300	660
MATERIAL 4	400	660

Table 14. Sealing windows given in the number of settings for CS and US

### Conclusion

The sealing window of ultrasonic sealing is measured for four materials in this analysis. The sealing window of conduction and ultrasonic sealing are compared and it can be concluded that (based on the amount of settings) ultrasonic sealing has a wider sealing window than conduction sealing.

Note that this comparison based on the amount of settings is not ideal. This method is selected since it is a method that can be determined. This does not mean that this method is flawless since nothing is stated about the accuracy of the sealing technology machines. There might for example be fluctuations of 5°C for the conduction sealing machine, while the temperature can be set with one decimal preciseness.

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# 5.9.Test 5. Energy use US

#### **Research question**

To be able to compare the consumed energy of ultrasonic sealing to other technologies, the average consumed power during the sealing should be estimated. To get more insight into what exactly influences the used energy, the following research questions need to be answered:

[US] What is the relation between pressure and consumed energy?

[US] What is the relation between amplitude and consumed energy?

#### Theory and hypothesis

The hypothesis of the relationship between energy, amplitude and sealing force (pressure) is based on the research performed by D'huys, et al. [24]. The relationships that they found regarding the energy are shown in **Figure 47**. The same trends are expected to be observed in the experiments that will be done. That means that a positive linear trend is expected between the pressure and the energy. A positive exponential relationship is expected between the amplitude and the energy.



Figure 47. Relationship between sealing force and seal energy (left) and amplitude and seal energy (right) [24]

#### Method

For different ultrasonic sealing settings, the energy will be gathered. This will be done by reading it from the ultrasonic generator interface while the HFFS system is running. The amplitude is variated between 30 and 100% with steps of 10% (except 90%) and the pressure between 0,1 and 2,3 bar with steps of 0,1 bar.

#### **Data collection**

The used energy differed during the process. The average value was taken, but this can be plus or minus 2W depending on the time in the process. It is assumed that the material does not influence the consumed energy. The results can be observed in **Table 15**.

Pressure (bar)	Amplitude (%)	Energy (W)
0,1	60	80
0,1	70	85
0,1	100	90
0,3	80	100
0,5	60	95
0,5	80	100
0,7	80	100
0,9	40	100
0,9	50	105
0,9	60	105
0,9	80	105
1,1	100	110
1,1	70	110
1,3	30	115
1,3	40	115
1,3	50	115
1,3	60	115
1,3	100	115
1,5	100	125
1,7	100	128
1,9	100	130
2,1	100	135
2,3	100	140

 Table 15. Consumed energy for different ultrasonic sealing settings

#### **Data analysis**

As can be seen from the results, the consumed energy is not the same for each setting. Whether this is caused by the changing pressure or amplitude and what parameter mostly influences the energy will be investigated.

First, it will be proven that the seal strength is influenced by the changing energy. By looking at the results and observing a certain trend, the ideal sealing settings can be selected. This is the highest seal strength for the lowest energy, so the highest energy efficiency.

A 2-way-ANOVA is conducted to see what the influence of the material, and energy is on the seal strength. The graph that visually represents the results can be seen in **Figure 48**. The Levene's and the Shapiro-Wilt test were significant for this ANOVA. Therefore, no finite conclusions can be drawn from the results. When carefully looking at the results, it can be observed that the energy shows a significant effect on the seal strength. When looking at the Tukey post-hoc test, there is no significant difference between all energy levels. This can also be observed in the graph, where some seal strengths are similar for different energy inputs.

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Error bars: 95% Cl

*Figure 48.* Energy against the estimated seal strength for different temperatures. NOTE: the steps are not equal on the horizontal axis (error bars at 95% confidence)

What can be observed is that the lowest energy (85W) shows a peek at the start. So with a low energy, high seal strengths are realized showing the highest energy efficiency. The settings used for this are 0,1 bar pressure and 70% amplitude.

All the materials will be looked at separately to see if there are big differences among them. The boxplots function as a graphical representation of all gathered data. They are presented in **Figure 49**. They show similar trends.



Figure 49. Boxplots of energy against seal strength for materials 1, 2, 3 and 4 with US (error bars at 95% confidence)
A 1-way-ANOVA per material is executed to see if the energy has a significant influence on the seal strength for all of them. The results are summarized in **Table 16**. The normality among groups is evaluated through the Shapiro-Wilk test. One group in this case is one value of energy with a specific material (e.g. material 1 with an energy of 80W is one group). A few treatments return a significant Shapiro-Wilk test, as can be seen in Appendix 8. Results dye penetration test layer jump.

The Levene's test for each material is significant, while the sample size differs quite a lot with a minimum of n=7 and a maximum of n=61. Therefore, interpreting the results of this analysis should be done carefully. The equal variance assumption for the ANOVA test has been violated. The complete graphs that were obtained from the analysis can be found in Appendix 7.Test 5. Results energy analysis.

Material Test		Significance Levene's*	Significance ANOVA**	F-test
MATERIAL 1	Levene's test	<0,001		
	Influence of energy		<0,001	15
MATERIAL 2	Levene's test	<0,001		
	Influence of energy		<0,001	40
MATERIAL 3	Levene's test	<0,001		
	Influence of energy		<0,001	17
MATERIAL 4	Levene's test	0,011		
	Influence of energy		<0,001	10

Table 16. Results of the one-way-ANOVA influence of energy on SS US for materials 1, 2, 3 and 4

\*If the significance of Levene's test is  $\leq 0.05$ , the variance between groups is not homogeneous, which means that drawing conclusions from the ANOVA should be done carefully.

\*\*If the p-value of the ANOVA is ≤0.05, the impact is significant.

If we carefully take a look at the results, it can be observed that for all materials the influence of the energy on the seal strength is significant.

To graphically see what the expected trends are based on the ANOVA test, the graphs in **Figure 50** are presented. They show similar trends as the boxplots. It can be concluded that the energy positively influences the seal strength, with some exceptions. Especially the high seal strength at low energy (85%) is noteworthy.

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Figure 50. Energy against the estimated seal strength for materials 1, 2, 3 and 4 with US (error bars at 95% confidence)

To see what parameter (amplitude or pressure) has the most effect, what kind of relation there is between the parameter and the energy and to confirm that the parameters influence the energy, the following analysis is performed.

Since only one measurement of energy is done per setting, comparing group means is impossible. So no ANOVA, but when looking at the boxplots of pressure and amplitude against the energy (see **Figure 51** and **Figure 52**), it can be observed that the relationship could be linear. Especially the influence of pressure on the energy.

Therefore, a multiple linear regression analysis will be performed to see what parameter mostly influences the energy consumption.

The results of this analysis are that the pressure and the amplitude have a significant influence on the energy. The model suggests the following formula:

#### Energy = 83 + (22,9 \*pressure) + (0,04\*amplitude)

The  $R^2$  and adjusted  $R^2$  are both 0,97. It can be concluded that the pressure influences the energy most, way more than the amplitude.



Figure 51. Boxplot of energy against pressure



Figure 52. Boxplot of energy against amplitude

# Conclusion

From this part can be concluded that the energy statistically influences the seal strength. The pressure mostly influences the seal strength, while the amplitude slightly affects the consumed energy. The nature of the influence the pressure and amplitude have on the energy seems to be positive and linear.

This might also explain the peak in seal strength at 85W. The pressure there is low, while the amplitude is high. It has already been established that the amplitude significantly influences the seal strength, while it has minimal influence on the energy use.

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# 5.10.Test 6. Layer jumps

As already stated in chapter 4.6 Layer jump, a transition in thickness can form a struggle during sealing. As well as conduction sealing as for ultrasonic sealing this can be difficult. Layer jumps in combination with conduction sealing are sufficiently described in the literature, so performing tests on this is not deemed valuable. However, layer jumps in combination with ultrasonic sealing are not elaborately described in the public literature and thus some tests are valuable to fill in the framework for the parameter 'layer jumps'.

#### **Research question**

To properly say something about ultrasonic sealing with a layer jump in the seal area, a research question needs to be formulated and answered. The answer will be based on scientific literature and the analysis of tests. The research question is formulated as follows:

[US] Is a layer jump in the seal area problematic for ultrasonic sealing technology?

First, a hypothesis will be formulated based on theory. Later, a test will be performed to confirm or deny the hypothesis.

#### Theory and hypothesis

The theory on ultrasonic sealing of layer jumps is already described in chapter 4.6 Layer jump. Based on that literature a hypothesis is formulated. The article of Dun, et al. [99] states that layer jumps can be problematic for ultrasonic sealing. Only if the material reaches high seal strengths at low input energy it is possible to create closed seals. This statement forms the hypothesis, although no quantification of the requirement is offered. This makes rejection and acceptance of the hypothesis challenging.

#### Method

To answer the research question, packages with a layer jump need to be realized and tested. This will be done using dye penetration and microscopic imaging testing. For an elaborate explanation of both methods, see 'Dye penetration' and 'Microscopic imaging'. For the microscopic imaging, the samples are not cut perpendicularly to the seal (like described in the testing method), but parallel to the seal. Otherwise, the layer jump would not be visible.

Layer jumps will be created on the same HFFS system and with the same samples as described in 'Test packages'. One difference is that the sample is created until 1,1 bar. Another difference is that for this test, an extra film is inserted into the already existing film to create a layer jump. Another folded film is inserted into the other folded film. At a certain point, the ultrasonic sealing system will have to seal four instead of two layers. The film is inserted with the sealant layer to the outside. A cross-section of the four-layered film is presented in **Figure 53**. The material used is material 4 (BOPP PP EVOH PP 20/30 without easy peel layer). The material is not made to also seal the outside layers to each other.



Figure 53. Simplified representation of the placement of the films with layer jump. Orange represents the sealant layer

# **Data collection**

The results of the dye penetration tests can be found in Appendix 8. Results dye penetration test layer jump.

When looking at the cross-section of an ultrasonic seal (see **Figure 54**) it can be observed that the seal is not closed throughout the entire cross-section. The ultrasonic seal is realized by locally sealing at four points. Three samples were placed in one cup of epoxy resin. An effort was made to place the samples with layer jump at the same height to realize (after polishing) a visible sealed part for each sample. However, not all pictures are taken exactly at the place where the seal is closed, since this was probably impossible.



Figure 54. Microscopic image of ultrasonic seal cross-section (sample 7)

# Data analysis

The results of the dye penetration test show that the packages are not properly sealed at the fourlayer fraction. This is in line with the expectation since the material was not expected to seal the heatresistant outer layer to another outer layer.

Before taking a look at the microscopic images, it is good to first see what one layer of material looks like under the microscope. In **Figure 55** can be observed that unsealed material consists of 3 light-colored layers with two thin dark-colored layers in between. When sealed together, the two light-colored layers that face each other seem to merge. So in theory, the amount of dark-colored layers between light-colored layers divided by two gives the total amount of layers.

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Figure 55. The layers of material 4 with conduction sealing (sample 29)

Keeping this in mind, it is surprising to find that these thin dark-colored layers cannot be observed so clearly for 3 and 4-layered seals.

For the 3-layered seal, it looks like the following layers can be observed from left to right: one layer without clear dark layers, a layer with clear dark layers, a gap and another layer with clear dark lines. For the most left material layer, the material structure seems to be damaged during the sealing. This could be caused by too high pressure.

The four-layered picture shows the following layers from left to right: two layers with only 3 dark layers, one layer with clear dark layers, a gap and another layer with clear dark layers. What can also be observed is that the light-colored layer connecting the second and third film layers are relatively thick.



Figure 56. Material 4 with 3 layers (left) and 4 layers (right) (sample 109)

So what can be observed in both cases, is that on the left of the seal, the layers are fused very well, maybe even too well and the layers on the right are not sealed together. This suggests that the energy exerted by the ultrasonic sealing system is absorbed by the first (few) sealing interface(s), but does not properly get through the last one.

The material seems to be damaged at one part of the seal and opened at the other end. This is observed for sample 109, which has been created with relatively low settings (0,1 bar and 70%). The question now is whether this is also observed for higher pressures. Therefore, sample 114 will also be investigated in more detail because of its high settings (1,1 bar and 100%). In **Figure 57** a picture made from sample 114 can be observed. This is a picture that shows four layers. The layers that can be

observed from left to right are one layer with clear dark lines, a gap, another layer with clear dark lines, two layers without clear dark lines. This is very similar to the four-layered version of sample 109. Even the fact that the layer connecting layers two and three is relatively thick can be observed. This is probably caused by the fact that this is where the two heat-resistant outer layers face each other.





Figure 58. Clear example of a layer jump with a gap (sample 109)

When looking at the microscopic pictures, another observation can be made. The layer jumps themselves are very obvious and in all cases include a visible gap (filled with fluid from the polishing process). An example of this is given in Figure 58. Another example is given in Figure 59, which shows layer jumps at two different places (from 2 to 3 and from 3 to 4 film layers). The sealant layers of the two remaining layers should fill the gap. This is however difficult since very local pressure is required and the sealant material cannot be connected to for example Figure 59. Layer the exposed EVOH layer from the middle film that is facing the gap.



jumps at two different places (sample 112)

## Conclusion

Ultrasonic sealing generates heat at the seal interface. When multiple seal interfaces are present, this technology seems to reach its limitations based on the data analyzed. No clear differences are observed between different settings of ultrasonic sealing. This is in line with the hypothesis that the material did not show strong seal strengths at low seal settings. To find an answer to the research question it needs to be known whether layer jumps are problematic for ultrasonic sealing. The answer to this question is that it can indeed be problematic based on the analyzed data.

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# 5.11.Test 7. Squeeze-out

# **Research question**

It is interesting to look at the nature of the influence pressure and amplitude have on squeeze-out since it can help to explain how the seals are performing with different (especially higher) sealing settings. This might also be used to decrease the risk of squeeze-out for ultrasonic sealing.

No public literature has been found that exactly describes the relationship between the amplitude, pressure and squeeze-out. What is described, however, is that the amplitude and the pressure positively influence the amount of squeeze-out, as is described in chapter 4.7 Squeeze-out.

The research question that needs to be answered is:

- [US] What is the relationship between pressure and squeeze-out?
- [US] What is the relationship between amplitude and squeeze-out?

The investigation of seals created with conduction sealing will be limited. The combination of conduction sealing and squeeze-out is already extensively described in literature and the relationship between the sealing parameters and the squeeze-out is already known. So, conduction sealing will only be investigated to check the normal behavior of the material.

## Theory and hypothesis

In chapter 4.7 Squeeze-out, literature on squeeze-out is reviewed. Based on this chapter, hypotheses are formed for the research questions.

From the literature, it can be concluded that for higher pressures higher squeeze-out rates are expected [9, 84]. The exact relationship between the pressure and the squeeze-out is not described for US. This forms the hypothesis for the first research question of test 7.

For the second research question, the same conclusions can be drawn for the relationship between the amplitude and the squeeze-out. The higher this amplitude, the higher the expected squeeze-out rates [9]. Also for this parameter, the exact relationship with squeeze-out is not known.

## Method

To answer the research questions, multiple samples will be investigated under a light microscope. See the section 'Microscopic imaging', 'Test packages', 'Settings CS' and 'Settings US' for the method used. Not all created packages are included in the analysis. The samples used in this test are presented in **Table 17**. It can be seen that especially the ultrasonic seals are investigated. Conduction sealing has been more extensively researched in combination with squeeze-out than ultrasonic sealing.

Code	Material	Type of sealing	Amplitude (%)	Pressure (bar)	Temperature (°C)
U1	MATERIAL 1	Ultrasonic	70	0,1	
U6	MATERIAL 1	Ultrasonic	100	1,1	
U7	MATERIAL 1	Ultrasonic	100	0,1	
U8	MATERIAL 1	Ultrasonic	70	1,1	
U206	MATERIAL 1	Ultrasonic	100	2,3	
C25	MATERIAL 4	Conduction			110
C29	MATERIAL 4	Conduction			150

Table 17. Samples tested for squeeze-out

The samples created with conduction sealing are created to verify that the method used to process the ultrasonic seals shows reliable results. What the squeeze-out should do in combination with conduction sealing is known. If this is not confirmed by these tests, drawing conclusions for seals realized with ultrasonic sealing, might not be valid.

#### Quantification of squeeze-out

A challenging part of the tests regarding squeeze-out is the quantification of the squeeze-out. Morris and Scherer [10] describe a method in their paper that does this for conduction sealing. The area of squeeze-out visible at the seal cross-section is taken as a measure. In **Figure 60** the squeeze-out from a microscopic picture and a simplification of this that are used in their research, can be seen. The simplified version is used to calculate the squeeze-out with the following formula:

$$\% Squeezeout = \frac{bc}{2h_0w} * 100\%$$

Where b is the highest height of the squeeze-out measured from the seal interface, c is the distance between the end of the squeeze-out and where the seal has a constant thickness, h0 is the initial half separation distance between the plates, and w is half of the width of the plates.



*Figure 60.* Observed squeeze-out at the seal interface (left) and simplified representation of squeeze-out (right) [10]

It is good to note that this method has a limitation. It can only estimate the squeeze-out when the squeeze-out is visible in the picture. This became more difficult as the temperature increased since the clear trapezoidal squeeze-out form disappeared then. So, before this method is selected, it should first be checked whether the squeeze-out is in a trapezoid-like form visible in the pictures and can be quantified in a similar way as Morris and Scherer [10] showed.

If this is not possible, Aghkand, et al. [157] describe another way to determine whether there is squeeze-out. They state that if the thickness of the unsealed layers close to the seal area combined, is around the same as the thickness of the sealed area, no significant squeeze-out occurs. This method will be employed if the squeeze-out does not show a trapezoid-like shape.

# **Data collection**

The results are presented in Appendix 10. Squeeze-out test pictures. These include pictures of every seal as a whole and separate, enlarged pictures of the squeeze-out area.

Sample U1 could unfortunately only be observed clearly from the left side of the seal. The right part did not become visible. Therefore, only the squeeze-out of one side can be determined for this sample. Sample U8 also does not show clear pictures.

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# **Data analysis**

Looking at the microscopic pictures, a few things can be observed. The different material layers can be observed. The seals are closed locally for the seals created with ultrasonic sealing. This means that per seal there are four little local seals created with an unsealed area in between. This opens the possibility that squeeze-out can occur at all four local sealing sites.

The seals created with conduction sealing did not show a significant decrease in thickness at the seal area. Therefore it is concluded that no significant squeeze-out is present at those seals. This is in line with the expectations since no extensive heat or pressure was used to realize these seals, so no squeeze-out was expected. Therefore, there is no reason to deem the results from the ultrasonic sealing tests in this chapter as invalid.

In **Figure 62** the most left local sealing site is presented. First, it can be observed that at the bottom of the created seal, an indent is visible. This shows that the thickness of the seal is lower than the combination of the two separate layers. This



**Figure 61.** Microscopic image of a seal created by conduction sealing showing no significant squeeze-out (sample 25 CS)

suggests that squeeze-out has occurred. Furthermore, when following the different material layers of the bottom layer, it can be observed that one layer seems to be disrupted at the seal area and pushed to the left. This also suggests a squeeze-out flow (especially to the left). Lastly, a little clomp of material can be observed on the left. This seems to be squeezed-out material. To conclude, all these points indicate a presence of squeeze-out, but no trapezoid-like shape can be distinguished.



#### Figure 62. Visible squeeze-out (sample 6 US)

The indent visible in **Figure 62** can be observed four times in the entire seal in **Figure 63**. Figure 64 shows that indeed squeeze-out can occur at one of the two inner local sealing sites. So there are four local places where squeeze-out can occur. Maybe it seems logical that squeeze-out for the outer sealing sites would mainly be to the outside, but Figure 64 shows that this does not have to be the case.



Figure 63. Cross section of an ultrasonic seal with four indents (sample 7 US)



*Figure 64.* Squeeze-out visible at the inside of the seal, at one of the two inner sealing sites (left) and towards the other local sealing sites (right) (sample 7 US)

Looking at the clomp of squeeze-out in **Figure 62**, it is clear that this does not have the trapezoid-like form used by Morris and Scherer [10] to quantify the squeeze-out. It is difficult to differentiate where the separation between squeeze-out material and non-squeeze-out material is. Therefore, the determination of the exact squeeze-out area would not be trustworthy.

What can be done, is apply the method described by Aghkand, et al. [157] which states that the squeeze-out can be quantified by comparing the thickness of the unsealed layers close to the seal area to the thickness of the sealed area. If this only deviates a little, no significant squeeze-out occurs. What a small deviation is, is not further quantified by them.

The results of the analysis comparable to what Aghkand, et al. [157] performed, are presented in **Table 18**. This is done for each ultrasonic seal, so four local seals per sample. The coding of the table is illustrated by the first example. U6.1 is the first seal from the left from the ultrasonic seal from seal 6. U6.2 is the second seal from the left from the same treatment. What stands out is that sample U206.1 has a negative difference. This is caused by the air gap that is visible at this local seal (this seal site is not properly closed). U8.3 is also not sealed, and the films were too far apart to even bother measuring.

It is expected that the seals realized with conduction sealing do not show significant squeeze-out. So the threshold when squeeze-out is happening should be above 4%. At sample U7.3 squeeze-out of the EVOH layer seems to be occurring, so squeeze-out should be significant. This causes the threshold

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value to be 5%. If the difference is below this value, no significant squeeze-out is occurring, if the difference is above this value, a significant squeeze-out is expected.

Sample	Film 1 (µm)	Film 2 (µm)	Film 1+2 (μm)	Measured thickness (μm)	Thickness reduction
U6.1	72	81	153	140	9%
U6.2	71	85	156	138	11%
U6.3	69	77	146	140	4%
U6.4	68	83	151	136	10%
U7.1	77	73	150	138	8%
U7.2	81	73	154	142	7%
U7.3	81	73	154	146	5%
U7.4	81	73	154	138	10%
U8.1	77	85	162	146	10%
U8.2	77	81	158	146	7%
U8.3					
U8.4	77	81	158	146	7%
U206.1	77	69	146	154	-5%
U206.2	77	71	148	138	6%
U206.3	77	71	148	135	9%
U206.4	77	71	148	131	12%
C25	50	50	100	96	4%
C29	52	52	104	104	0%

Table 18. Squeeze-out analysis results

A two-way-ANOVA has been executed to see if there is a significant effect from the pressure and the amplitude on the percentage of difference in thickness. For this test, all measurements from **Table 18** have been included, except the measurements that were not sealed, so samples U8.3 and U206.1. If the effect of pressure and amplitude on the percentage of difference would have been significant, the influence of the factors on the squeeze-out could have been proven together with which parameter mostly influenced the squeeze-out could have been determined. However, the influence of pressure and amplitude is not significant.

## Conclusion

Squeeze-out did not seem to occur at conduction sealing. This is in line with the hypothesis, so there is no reason to mistrust the results of the analysis for ultrasonic sealing.

Based on the executed tests and their analysis in combination with the literature, it can be concluded that squeeze-out seems to be occurring at ultrasonic sealing. This part is in line with the hypothesis. It can occur at all local sealing sites. To determine if pressure and amplitude influence the amount of squeeze-out, more research is necessary. To determine this, the squeeze-out should be determined and a wider variation of sealing settings should be used. For now, it is too preliminary to draw conclusions.

# 5.12.Test 8. Damage to the barrier layer

Multilayer mono-material films can be sealed to create a flexible packaging system. Since most layers in the film have the same basic material, the melting temperatures will be similar. The temperature difference will at least not be as far apart as some multi-material films show. This can cause sealing issues. An example of a problem that might occur is the damage to the barrier layer.

As the permeation of gases and vapors is one of the key parameters that determine the shelf life of a product and thus the performance of a packaging, the destruction of a barrier layer might be highly undesired [13]. So it is worth investigating whether and when the barrier layers are damaged during the sealing. This will be done for conduction sealing as well as for ultrasonic sealing. The focus of these tests will be on the ultrasonic sealing performance since less is known in the academic world about this sealing principle.

# **Research question**

The research questions that need to be answered to find out if the barrier-layer destruction can cause problems for ultrasonic and conduction sealing processes are:

[US] Can the EVOH layer be damaged during ultrasonic sealing?

[CS] Can the EVOH layer be damaged during conduction sealing?

Based on public literature and testing, these questions will be answered. The barrier layer EVOH is chosen, since this is one of the most applied barrier layers in plastic food packaging [158]. EVOH is also included in the films that are already investigated, so this choice is also based on practical availability.

# Theory and hypothesis

Carullo, et al. [13] show that it is not inherent to a mono-material to have a barrier layer damaged after sealing. They show a mono-material with excellent moisture and oxygen barrier properties. So the EVOH layer might stay intact during sealing.

Another research shows that EVOH is suitable for high-pressure applications regarding food processing [158]. This same research indicates that thermal sterilization is not suitable for products with a package containing EVOH. The crystallinity of the EVOH is disrupted during this treatment. The sterilization was performed at 120°C for 20 minutes. It is mentioned that not the high temperature, but the pressurized water was probably responsible for the higher oxygen permeability after sterilization. The water would have gone through the outer layer and reached the EVOH layer, thus compromising its high barrier properties (for more information, see the chapter EVOH). This seems to suggest that high pressure without water will not damage the EVOH layer.

No clear research has been found in public literature that addresses the possibility of this specific problem. In practice, however, this has been found. In Appendix 11. Destruction of EVOH layer some pictures can be found that illustrate a damaged EVOH layer due to too high sealing settings of the ultrasonic sealing process. It is confirmed in an interview with an ultrasonic seal expert that layers of a mono-material might merge if the pressure of the ultrasonic sealer is too high [117]. This might cause damage to the EVOH layer.

The hypothesis is formulated based on the fact that EVOH is so widely used in sealing applications. It is expected that no EVOH damage will be present for the conduction seals. It is expected that some EVOH damage will be visible for the ultrasonic seals, based on the information gathered in practice (see the pictures in Appendix 12. Testing at Omori).

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## Method

To check if the seal area still has the original layers present in the film, testing is necessary. This will be done using the digital microscope on the same sample used in a previous test (see chapter 5.11 Test 7. Squeeze-out). For more information about the microscopic imaging method, see the section 'Microscopic imaging'.

## **Data collection**

Pictures of the results can be found in Appendix 10. Squeeze-out test pictures. Pictures of sample 1 with ultrasonic sealing are not clear. No conclusion can be drawn from this picture. Sample U8 is not very clear, because of the visible liquid drops. The quality is high enough to draw quantitative conclusions about the EVOH destruction.

# Data analysis

The results can now be analyzed to answer the research questions. Conduction seals will be analyzed first, followed by the seals created with ultrasonic sealing.



*Figure 65.* No barrier destruction visible for conduction sealing realized at 150°C (sample C29)

*Figure 66.* Disruption of the EVOH layer at one of the inner sealing sites (sample U6)

In Figure 65 a picture of a seal realized with conduction sealing at the highest seal settings can be found. This picture clearly shows the different layers and no disruptions or inconsistencies can be found within these layers. The same goes for the other conduction seal samples. Based on this, it is concluded that no destruction of the EVOH layer happens for conduction sealing.

The most interesting results for different ultrasonic sealing settings are summarized in Figure 67. This shows that for material 1, all samples seem to have a non-continuous EVOH layer. The lines that can be observed near the seal interface are somewhat lighter than the surrounding material, such as the EVOH layer. In the table, only disruptions are shown at the border of the seal but are also visible at the second and third (inner) sealing sites (e.g. see Figure 66).



*Figure 67.* Microscopic images of material 1 ultrasonic sealing with different settings to see the destruction of the EVOH layer for samples 6, 7, 8 and 206

# Conclusion

It is concluded that no destruction of the EVOH layer happens for conduction sealing at the tested material. This is in line with the expectations. It should however be noted that this can only be concluded for normal sealing pressure and 150°C. It cannot be concluded whether EVOH destruction will cause a problem at even higher pressures and higher temperatures. So there is no reason to mistrust the results of ultrasonic sealing with this material and squeeze-out.

With normal sealing settings on material 1, ultrasonic sealing resulted in a disrupted EVOH layer. This is in line with the hypothesis. What the effect of the disruption of the EVOH layer is, is not known. It might influence the barrier properties of the film. To draw these kinds of conclusions, more research is necessary.

# 5.13.Test 9. Easy peel US

Easy peel in combination with ultrasonic sealing will be described in this section. First, easy peel realized with controlled contamination will be described. After that, creating an easy peel through ultrasonic sealing, without the need for special material will be described. Note that this chapter thus has a different setup than the other testing chapters.

# **Controlled contamination easy peel**

Nase, et al. [84] described as firsts the combination of easy peel films (controlled contamination) and ultrasonic sealing in public literature. They showed that with heat sealing a plateau is reached when looking at the maximum peeling force with different temperatures. This is compared to the maximum peeling force that ultrasonic sealing shows for different sealing forces.

The maximum peeling force decreases both for ultrasonic sealing and conduction sealing in an exponential way when the *percentage* of contamination in the sealant layer is increased. The *thickness* of the sealant layer influences the peeling force for conduction sealing positively. For ultrasonic sealing, however, the sealant layer thickness seems to have no significant effect on the maximum peeling force [84].

For the ultrasonic sealing, no significant effect could be measured due to the high standard deviation. This is caused by the inhomogeneous presence of the contaminants in the sealant layer. This is also caused by the fact that the ultrasonic sealing process is a dynamic process that decreases reproducibility according to Nase, et al. [84].

Nase, et al. [84] conclude by proposing that ultrasonic sealing might not be the adequate sealing mechanism to seal easy peel seals with controlled contamination. Their argument for this statement is that contaminants are pushed out of the seal area if the sealing force is too high.

# Ultrasonic sealing creating an easy peel seal

During the testing performed at the company Omori Europe, a surprising result was that it was possible to realize an easy peel seal with ultrasonic sealing on a material that was not made for easy peeling (see chapter 2.8 Easy peel for more information about easy peel). This is confirmed by an ultrasonic sealing technology supplier [117]. This section will test whether this is indeed possible and will speculate on the cause of this.

As mentioned before, an easy peel seal is a seal that can easily be opened without the use of any tools. An easy peel seal is classified as 6 to 10N/15mm by Sängerlaub, et al. [29].

Material 4 which has already been used for the testing of the influence of the pressure and amplitude on the seal strength for ultrasonic sealing, is the only material that does not include an easy peel mechanism through controlled contamination. The results of the tests are presented in the section Test 2. Influence of pressure and amplitude on seal strength. These T-peel tests returned the seal strength in the unit N/25mm. That means that an easy peel in this case is one between 10 and 17N.

In Figure 68 a scatter plot from all the measurements on material 4 is presented. In this figure, a horizontal line can be seen at 17N/25mm. All measurements below the line are considered to be easy to peel. From this figure can be concluded that it indeed is possible to create an easy peel film, especially for lower pressures and amplitudes. However, most measurements are not considered easy to peel.



Figure 68. Scatter plot of all measurements material 4 with a line at 17N/25mm

From the chapter Test 4. Mono-material sealing window US can be concluded that all ultrasonic seals from material 4 were properly closed according to dye penetration tests.

It is expected that at even lower sealing settings, more measurements will be considered easy peel seals. To be certain and to properly prove that ultrasonic sealing can create easy peel seals, more research is required.

# Speculating

As already mentioned, it is expected that it is possible to realize easy peel seals with the ultrasonic sealing technology on materials that don't include a controlled contamination easy peel mechanism. It is expected that this is possible due to the heat mechanism.

Ultrasonic sealing technology heats the materials from the inside. In Figure 69 a cross-section of a seal can be observed. This seal is realized with an anvil containing four lines. It is visible in the figure that there is no continuous seal, but rather four different local sealing sites. This is also observed during the testing and can be seen in the graphs from the T-peel tests (e.g. see Appendix 3. Acceptable ultrasonic T-peel test samples).



*Figure 69.* Cross section of an ultrasonic seal with four indents (sample 7 US)

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It is expected that the seal is so easy to peel because there is only a thin sealing site that needs to be peeled. It might very well be the case that the seal is considered to be easy to peel, because four very thin seals are peeled, instead of one wide one.

Another possibility that the peeling of this seal is considered to be easy is that delamination occurs at the four sealing sites. This way, the delamination must be initiated with some force, but the rest of the seal will peel easily if the adherence between the different layers is low.

However, to draw a conclusion on whether and why an easy peel seal can be realized with ultrasonic sealing on materials that do not include an easy peel mechanism, more research is required.

# 6. Framework

In this chapter, the framework will be presented. Before this can happen, other types of sealing must be introduced. These are the types of heat sealing other than conduction and ultrasonic sealing that will be introduced. This chapter will contain the following sections:

- 1. Selecting other types of heat sealing
- 2. Introduction of other heat sealing mechanisms
- 3. Framework

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# 6.1.Selecting other types of heat sealing

Next to the conventionally used conduction and ultrasonic sealing technologies, other technologies will be included in the framework. This will create the opportunity to create a complete overview of the possible choices. The framework user can then make a proper choice of sealing technology.

# Selecting sealing technologies for the framework

To see what heat-sealing technologies should be included in the framework, a well-funded selection phase needs to take place. To achieve this, we will look at public literature describing heat-sealing technologies. These technologies will be presented together with the amount of time they are mentioned. This way, a valid selection of sealing technologies can be made.

#### Method

The method that determines what papers to include for the heat-sealing selection process, is based on the paper by Wee and Banister [159]. They explain how to select papers for a literature review. They strongly recommend being explicit about the methodology used and how the papers have been selected that are included. The paper suggests that if all literature concerning the topic is reviewed, it should be clear what database has been used in combination with what search terms and selection criteria. This methodology will be used to determine what papers to include in the heat sealing technologies selection.

The database that is used is Google Scholar. The search terms are carefully considered since these highly influence the papers that are found in the database. To make a selection from the gathered papers, the papers should include heat sealing technologies used in the packaging industry. Therefore the term that is used for sealing is formulated as:

"heat seal\*" AND "packaging" AND "sealing technolog\*" OR "sealing technique\*" OR "type\* of sealing" OR "sealing method\*" AND intitle:Seal OR intitle:Sealing

The first term includes an asterisk to include heat seal, heat seals and heat sealing terms. Synonyms for sealing technologies have been selected based on how the section describing the sealing technologies is titled in different papers [8, 9, 21, 24, 31, 39-41, 97, 160]. The asterisk after technolog is to include the terms technology as well as technologies. The other asterisks are there to also include the plural.

In total Google Scholar returned 49 hits to the search term. Citations were not included in the search and only English articles were selected. Not all sources were useful, therefore a few selection criteria have been formulated to filter out the usable sources. These criteria are:

- 1. The paper should be publicly available or available to the University of Twente
- 2. The article showed up more than once
- 3. Include at least 3 heat sealing technologies

The first criteria concerning accessibility is there for practical reasons. The second criteria filters out the double articles that Google Scholar sometimes returns. The last criteria is there to only include the articles that mention more than two sealing technologies. Some articles compare a 'special' kind of sealing technology to conventional heat sealing. These articles are not included, since these articles do not include a list of heat-sealing technologies, while this is what we are looking for. The articles that are excluded from the analysis are shown in **Table 19**.

#	Source	Criteria 1	Criteria 2	Criteria 3	2
1	[105]			X	2.
2	[161]			X	3.
3	[162]		X		
4	[163]			X	
5	[164]			X	5.
6	[165]			X	
7	[166]			Х	6.
8	[167]			Х	7.
9	[168]			Х	
10	[169]			X	8.
11	[170]	X			9.
12	[171]			X	
13	[172]			X	10.
14	[173]			X	
15	[44]			X	11.
16	[174]			X	12.
17	[175]		X		
18	[176]			X	
19	[30]			X	
20	[177]			Х	
21	[178]			X	
22	[179]			X	
23	[180]			X	
24	[181]			X	
25	[182]			X	
26	[105]		X		
27	[183]	X			
28	[184]	X			

Table 19. The articles that do not meet the criteria and are excluded

The rest of the articles are included in the analysis. The result of the analysis can be found in **Table 20** where the articles are presented together with the heat-sealing technologies that are mentioned in them.

#	Source	CS	US	HGS	IMS	HWS	INFS	INDS	DS	PS	LS	RS	MS
1	[23]	Х	Х	Х		Х		Х		Х			
2	[39]	Х	Х		Х	Х		Х	Х				
3	[185]	Х	Х		Х			Х					
4	[8]	Х	Х	Х	Х		Х	Х	Х				
5	[160]	Х	Х	Х	Х	Х		Х	Х				
6	[2]	Х	Х	Х	Х	Х		Х	Х				
7	[186]	Х	Х	Х	Х								
8	[71]	Х	Х					Х					
9	[74]	Х	Х								Х		
10	[187]	Х	Х	Х	Х								
11	[188]	Х	Х			Х		Х	Х		Х		
12	[189]	Х	Х	Х	Х	Х		Х	Х			Х	Х
13	[24]	Х	Х	Х	Х	Х	Х	Х	Х				
14	[41]	Х	Х	Х	Х								
15	[40]	Х	Х	Х	Х								
16	[190]	Х	Х	Х									
17	[191]	Х	Х						Х				
18	[192]	Х	Х	Х									
19	[175]	Х	Х	Х	Х	Х		Х	Х	Х		Х	Х
20	[193]	Х	Х						Х		Х		
21	[194]	Х	Х	Х	Х	Х		Х	Х	Х		Х	Х

**Table 20.** The articles that meet the criteria and are included, together with what types of sealing they mention. Types of sealing included are: Conduction sealing (CS), Ultrasonic sealing (US), Hot gas sealing (HGS), Impulse sealing (IMS), Hot wire sealing

The amount of times the sealing technologies are mentioned in the articles is summarized in **Table 21**. From this table can be concluded that the most mentioned types of heat sealing are conduction and ultrasonic sealing. This is also where this thesis focuses on. Four sealing techniques are rarely mentioned: magnetic, laser, radiant and infrared sealing. These will therefore not be included in the framework. Hot wire sealing is mentioned more often but is also not included in the framework. The other types will be included.

Table 21. Sealing technologies with the amount of time they are mentioned in the 21 selected articles

Sealing technology	Amount of references in literature
Conduction sealing	21
Ultrasonic sealing	21
Hot gas sealing	14
Impulse sealing	13
Induction sealing	13
Dielectric sealing	11
Hot wire sealing	9
Magnetic sealing	3
Laser sealing	3
Radiant sealing	3
Infrared sealing	2

Mostly, the articles found use the same terminology to refer to a certain kind of sealing. However, some articles used a different terminology than those presented in the tables. So to be explicit hot bar sealing has been included in the conduction sealing, hot air sealing in the hot has sealing and electric field loss in the dielectric sealing category.

# Limitation

Some limitations are associated with the selection of heat-sealing techniques. For this selection, a small literature review has been executed. The techniques that were mentioned most in heat sealing technology lists in literature were selected. To find the publications that included lists of heat sealing techniques, a search term together with a database have been selected. A different search term could have resulted in different findings. Also, only one database has been used to find the publications (Google Scholar). This database includes a high number of publications, but not all. Including more databases in the search could have resulted in more publications and thus different results. So although it is expected that the conclusions from the selection method are valid, there are some limitations.

A type of sealing that is not mentioned in the public literature sources stated in the table, is digital sealing. This is a new type of sealing, which might have great applications to seal mono-materials. This sealing technology is quite new but might be interesting to include for future applications. Therefore, this sealing technology will be included in the framework.

The framework will be filled in as completely as possible, but less extensive as conduction and ultrasonic sealing, since these two types are the focus of this thesis. The information will be based on public literature and information offered by industry.

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# 6.2.Introduction of other heat sealing mechanisms

Heat sealing is the most common way to seal in flexible packaging [105], but other non-heat sealing methods exist such as cold, adhesive, and solvent sealing. [189]. Advantages of heat sealing compared to the other kinds include for example the fast cycle times, no adhesives required, the possibility to create peelable seals and the fact that it is the most cost-effective sealing method. There are also some disadvantages associated with this sealing method, one of these disadvantages is for example the possibility of heat transfer to the product [106].

The selected heat sealing technologies other than conduction and ultrasonic sealing will be introduced in this part. This will be followed by an explanation of how well they score compared to the other technologies for each parameter that is included in the framework. For certain sealing technologies, nothing useful can be said about a parameter. It will be indicated if this is the case.

To successfully fill in the framework, a comparison between the different types of sealing is necessary. This is difficult since this is dependent on a lot of unknown and hard-to-estimate variables. However, often a comparison to conventional conduction sealing is possible.

The parameter squeeze-out is not incorporated for these heat-sealing technologies, since determining this would require new research. It is a good possible future research opportunity.

# Hot gas sealing

The hot gas sealing technology exerts heat to the inside and pressure to the outside of two films to fuse them. The heat is introduced to the sealant layer by heated gas (up to 500°C). After the sealant layers are heated, pressure is exerted on the films to seal them. This is done by two cooled rolls [2, 21, 31, 160]. A simplified visualization of the sealing process can be found in Figure 70. This process is suitable for sealing thermoplastic materials continuously and without making heated contact [195]. It is also possible to exert the gas to the outside of the film [196].

The type of gas used can differ depending on the application. It can be nitrogen, carbon dioxide or normal air [31]. If the hot gas is air, the sealing technology is called hot air sealing and for this technology, maximum sealing temperatures of 350°C are mentioned by the industry [196].



Figure 70. Hot gas sealing principle

Hot gas sealing is used when conduction sealing is not suitable. Possible scenarios where conduction sealing is not suitable are when the sealing jaws have the risk of destroying a package or when a packaging material does not conduct heat well (e.g. due to high thickness). In these cases, hot gas sealing can be employed since it can apply the heat directly to the seal interface. The big advantage of hot gas sealing is that no contact is required. Examples of when this sealing technology is usually applied are thick plastic coated paper and cardboard [21, 31, 160], but also for example mono-material films and woven bags [124, 195].

## Hot gas sealing: mono-material sealing

The temperatures associated with hot gas sealing are significantly higher than normal conduction sealing. According to one source from industry, sealing mono-materials is not suitable with hot gas sealing because of this reason [124]. Other sources claim that hot gas sealing (hot air sealing to be

more specific) is mainly applied to mono-materials because this technique doesn't require physical contact with the heated elements. Typical applications that are mentioned by industry are mono PE and mono PP films [196, 197].

In conclusion, it is possible to seal mono-materials with this sealing technology and it might provide some advantages once the desired sealing temperatures are achieved.

## Hot gas sealing: energy consumption

Hot gas sealing requires (way) higher temperatures than conduction sealing (ranging from 200°C to 300°C) [196, 198] and it has to conduct the heat through the air. Air does not conduct heat well [197]. Because of these reasons, the energy efficiency of hot gas sealing is poor. At least worse than conduction sealing.

#### Hot gas sealing: start cost

Machinery options start around \$15,000 (≈€14.000) for tube sealing with hot air [199]. It is confirmed by a supplier that the price of hot gas sealing equipment is higher than conduction sealing equipment [197].

#### Hot gas sealing: production speed

Maximum production speeds reported by hot gas sealing technology suppliers are around 50m/min [124, 200]. So 50 meters of film can be sealed within a minute.

#### Hot gas sealing: maintenance time

Less maintenance is required for hot gas sealing compared to conduction sealing since there is no physical contact between the heated elements and the film while sealing. This reduces wear, tear, cleaning and thus the need for maintenance [196, 197].

#### Hot gas sealing: layer jump

No successful application has been reported of hot gas sealing in combination with a layer jump. This technology is only incorporated in continuous sealing operations, where layer jumps don't occur [124, 196, 197]

#### Hot gas sealing: contamination

Hot gas sealing can handle some contamination. Especially the dry powders, since the hot gas blows away the contamination from the sealing area [124].

#### Hot gas sealing: limitations

A limitation associated with hot gas sealing technology mentioned by the industry is that some safety issues arise when it is applied to films that include paper. The air applied is hotter than that of conduction sealing jaws, which brings along the risk of burning the paper [196].

Another limitation of this technology is that the hot gas might oxidize the seal interface of the films. This might compromise the seal performance of the sealant layer. Another limitation is that this system is less efficient than conduction sealing since there is no direct contact [31].

#### Hot gas sealing: opportunities

Hot gas sealing can successfully seal materials that are too thick to properly seal with conduction sealing. Also, films that don't conduct heat well can be sealed with this technology.

#### Hot gas sealing: continuous or intermitted

Hot gas sealing is only applicable for continuous sealing [124, 196, 197].

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#### **Impulse sealing**

Impulse sealing is similar to conventional conduction sealing. The main difference between both technologies is that impulse sealing does not constantly heat the sealing jaws like conduction sealing does [39].

Impulse sealing technology works as follows. First, two films are placed in between the sealing jaws. Secondly, the sealing jaws are heated by a short and strong electric current. This heats the sealing jaws and lets the sealant layer melt. While the jaws are heated, pressure is being exerted on the films. Thirdly, the pressure is still exerted on the films while the seal area cools down. Finally, the sealed films are released [21, 31, 39, 106, 160]. A simplification of the steps included in the sealing process is visualized in Figure 71.



Figure 71. Simplification of sealing steps impulse sealing

On at least one of the seal jaws is a thin nichrome (a combination of nickel and chromium) resistance wire that generates heat when an electrical current is sent through. This wire is wrapped with PTFE (Teflon) tape to prevent it from sticking to the films or the sealing jaws. This tape also helps to adjust the heat flow. The width of the heat-resistant wire/strip determines the width of the seal [21, 39, 106, 160, 201].

Just like conduction sealing, the main parameters that determine the seal strength are sealing time, pressure and temperature. Usually, the current is controlled instead of the temperature [21, 31, 202].

Applications of this technology that are reported are the sealing of mono-layer films and thin laminated films to create pouches and sachets [160, 203].

#### Impulse sealing: mono-material sealing

Impulse sealing can seal mono-layer films and thus mono-material films [203]. One supplier even claims that impulse sealing can better seal mono-materials than conduction sealing since it includes a pressurized cooling time. This will help to reduce the sticking and shrinking associated with sealing mono-materials. This controlled cooling is even mentioned as one of the main advantages of impulse sealing [201].

#### Impulse sealing: energy consumption

In general, impulse sealing is regarded as more energy efficient than conduction sealing. Impulse sealing also requires a heat-up time, but a significantly shorter one [2]. One source even mentions heat-up times below one second [201]. Both technologies electrically heat the sealing jaws, it is therefore deemed valid to assume that the energy used during the actual sealing is similar. So overall, impulse sealing is seen as more energy efficient than conduction sealing. This is confirmed by

VALDAMARK [204]. Energy consumed during the sealing operation itself is compared in the framework, so it is concluded that impulse sealing consumes a similar amount of energy as conduction sealing.

One source reports an example of a sealing operation where 450W is consumed during sealing [205]. Other sources indicate maximum powers of 650W and 750W [206, 207]

#### Impulse sealing: start cost

One independent source claims that impulse sealing technology is associated with low implementation costs [160]. One source estimates the cost of one seal unit (only functioning sealing jaws) to be €3500 [201]. So it is expected that it has similar investment costs as conduction sealing since this technology is also associated with low implementation costs.

#### Impulse sealing: production speed

The production speeds that impulse sealing can reach are differently reported in practice. Two sources claim to be able to reach 120 cycles per minute [201, 205]. Another source indicates that it can process 12 meters of material per minute [206]. The 12 meters per minute can be compared to other types of sealing, so this value will be used in the framework.

#### Impulse sealing: maintenance time

Higher maintenance costs are associated with impulse sealing. This is mainly caused by the heating element burning out [31]. Although an impulse sealing system supplier claims that one of the advantages of impulse sealing is that the sealing jaws are easy to replace, it agrees that the technology often requires operation attention [204].

An advantage associated with impulse sealing compared to conduction sealing regarding maintenance, is the fast cooling times due to the thin heat sources [2]. This means that maintenance can happen quickly after sealing [201].

To conclude, the maintenance time and costs are expected to be higher for impulse sealing than conduction sealing, mainly based on the article from Hendrickson [31].

#### Impulse sealing: layer jump

Impulse sealing is expected to perform similarly to conduction sealing when looking at sealing layer jumps. It is possible to add a silicone part on top of the sealing jaws to close the gap that exists at layer jumps [201]. One supplier even mentions that this is one of the main advantages of using impulse sealing [201].

#### Impulse sealing: contamination

Impulse sealing can handle a small amount of contamination. An independent source even mentions that impulse sealing performs better than conduction sealing in combination with contamination in the seal area [21]. Impulse sealing is not able to expulse contamination away from the seal area, but it can encapsulate the contamination within the seal [201].

#### Impulse sealing: limitations

There are some limitations to the impulse sealing technology. For example, there is no control system. That is why excessively high temperatures can be reached. This brings along the risk of burning through the material and creep of the heat-resistant wire [31, 106].

The realized seals of impulse sealing are relatively narrow. This is an advantage as well as a possible seal integrity risk and thus a limitation [21, 31, 160].

This technology is especially suitable for small badges and low machine speeds [202, 204], although another supplier claims that it is possible to implement impulse sealing at high machine speeds [201].

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#### Impulse sealing: opportunities

With controlled impulse sealing (this incorporates a control in the heating element), more precise temperature control (especially after sealing) is possible [31]. This type of sealing is usually applied to packages with a narrow seal area [31, 39].

#### Impulse sealing: continuous or intermitted

Impulse sealing is generally an intermitted process since it requires pressurized cooling time. So it generally works in cycles. It is however possible to make this process continuous if the seal bars run along with the film once the seal bars are closed [201].

#### **Induction sealing**

Induction sealing heats the films entirely differently than conduction sealing. For induction sealing to work, an electricity-conducting layer is required that heats up when exposed to a rapidly changing magnetic field. The induction sealing system includes an induction coil that is supplied with AC power. This creates a rapidly changing magnetic field. The heat is generated within the conductive layer caused by Eddy currents. This heat is conducted to the sealant layer, which causes the films to seal. This also means that this technology does not require contact between the sealing equipment and the films [8, 21, 31, 39, 71, 208]. A simplified representation of the induction sealing process can be found in Figure 72.



Figure 72. Induction sealing of a film on a bottle. Note that the magnetic field changes direction rapidly (adopted from [2])

Parameters that control the sealing for induction sealing are the length of the magnetic field, the production speed (both determining the sealing time) and the strength of the magnetic field. Usually, the power of the electric field generator is varied to find the optimum seal settings [31].

A typical application is films with an aluminum layer included. This is often applied on the closure of the lid on sauce bottles. Other common applications include the inner seals of bottles/jars for tamper evidence and laminated paperboard cartons [31, 39, 160, 209].

#### Induction sealing: mono-material sealing

Induction sealing technology can seal mono-materials as long as an aluminum layer is included [209]. This technology does not require a large difference in melting point between the outer and inner layer of the film, so there is no reason this technology shouldn't be able to effectively seal mono-materials.

#### Induction sealing: energy consumption

Two sources are found that compare the energy consumption of induction sealing to conduction sealing. One source states that conduction sealing machines are more energy efficient [210], while another source states that induction sealing only uses a fraction of the energy used by conduction sealing [211]. Both compare the sealing technologies for sealing films on a bottle.

In general, an advantage of using induction sealing is reduced energy consumption [212]. All in all, this seems conflicting information, but it is expected that during the sealing operation (so without warm-up times) both technologies consume a similar amount of energy.

#### Induction sealing: start cost

As already mentioned, the price is dependent on many factors. Induction sealing technology is seen as economical [209]. However, conduction sealing equipment is more affordable [210]. Good to note is that induction sealing can be used for different-sized bottles and caps, while conduction sealing requires different equipment for different dimensions [213].

#### Induction sealing: production speed

Induction sealing technology can seal for example a film on a bottle without stopping the bottle. Conduction sealing technology cannot achieve this in the same way. Either the bottles must stop for the sealing time or the sealing jaw must run along with the bottle during the sealing time [211, 213]. So for similar applications, the production speeds that can be achieved with conduction sealing should be higher than with conduction sealing.

To compare both technologies the maximum reported packaging speeds will be used. For induction sealing 80 packs per minute have been reported [209].

#### Induction sealing: maintenance time

The maintenance time and costs associated with induction sealing are low. The induction technology requires no physical contact, which reduces the required maintenance related to for example wear, tear and cleaning. The maintenance is even described as 'easier' than conduction sealing [209]. Another supplier of induction sealing technology even goes further and states that induction sealing almost requires no maintenance at all [211].

Furthermore, the equipment does not heat up while operational, so maintenance can happen straight away if it is required.

#### Induction sealing: layer jump

No implementation of layer jumps has been reported.

#### Induction sealing: contamination

No information has been found on how well induction sealing can seal through contamination, but what is known, is that there is less risk of contamination compared to conduction sealing [211]. Induction sealing requires no physical contact. This means for sealing a film on a bottle, that the cap can already be on the bottle before the sealing happens.

If contamination occurs, it is expected that it performs worse than other sealing technologies. This is based on the hypothesis that less squeeze-out will occur at induction sealing. No pressure is exerted

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on the film while it is sealed. This technology is therefore not able to squeeze-out minor contaminants. This is a hypothesis and needs to be checked to draw finite conclusions from this.

#### Induction sealing: limitations

The main limitation associated with induction sealing technology is that it requires a conductive layer in the film. Usually, this is an aluminum layer, which highly influences the recyclability of the film. If an aluminum layer of more than 1  $\mu$ m is included in the film, the film is seen as not suitable for recycling [214].

The quickly changing magnetic field heats metals close to it. This also forms a limitation for the packaging machine materials close to the induction coil. Therefore the material close to the induction coil must be non-conductive [31].

The last limitation worth mentioning is that only a circular edge can be sealed on. This is due to the eddy current that only transmits current at the circumference of a circular metallic layer. This allows heat sealing the film to the rounded edge of a rigid plastic [2, 160]

#### Induction sealing: opportunities

Some opportunities are associated with induction sealing, especially since it does not require contact between the sealing equipment and the film [31, 39]. So when for example sealing a film on a bottle, the cap does not have to be separated from the bottle [209]. An inherent advantage of non-contact sealing is that the risk of damaging the container due to excessive pressure is taken away. Another advantage that comes with the non-contact operation is that it is a more hygienic operation that does not require daily cleaning of the sealing equipment as conduction sealing does [213].

One supplier mentions that the films required for induction sealing can be thinner than conduction sealing [211].

#### Induction sealing: continuous or intermitted

The induction sealing technology is mainly used in continuous operations. In theory, it would also be possible to realize the seals with an intermitted operation since the technology only seals when it is powered.

## **Dielectric sealing**

The sealing principle of dielectric sealing (also called electric field loss sealing and radio frequency sealing) is similar to that of induction sealing. The big advantage of dielectric sealing is that it doesn't require a conductive layer in the film. Dielectric sealing systems can heat polar polymers by introducing them to a quickly changing electrical field. The frequency of change can range from 50 to 80MHz according to theory, but a typical frequency of 27,12MHz is reported by industry [215]. This changing electrical field is often combined with some pressure to create a seal. The electric field is realized with two electrodes and the pressure by the dies. These electrodes are coated with an insulator. The changing electric field causes excitation of the polar molecules which causes them to heat [8, 31, 39, 160]. A supplier of dielectric sealing technology mentions that the material heats most at the interface of the two films [215]. A simplified representation of the process can be found in Figure 73. Once the materials are sealed, the power will be turned off to allow the seal to cool down under pressure [160, 215]. Both the seal time and the cool time determine the seal performance [216].



Figure 73. Simplified representation of the dielectric sealing technology (adopted from [1, 2])

The sealant layer must be polar for this sealing technology to be effective [21, 31]. The dielectric loss is correlated with the polarization of the material [2]. Polar bonds are bonds between atoms that have an electronegativity difference of more than 0,4. A polar bond includes a small charge that is associated with the molecules [217]. PE and PP are examples of polymers that are not polar and thus are not suitable for this application. Examples of polar materials that are thus suitable for dielectric sealing include EVA, nylon and PVC [31].

Dielectric sealing is usually applied in a context where conduction sealing is not possible because the material is susceptible to deformation under high temperatures. Examples of materials that are dielectrically sealed are paper, multilayer sheets, PVC, PET and cellulose [2, 31, 126, 160, 216]. It is possible to include a metal layer in the film, as long as it is embedded in polar material [126]. A typical application is medical bags [215].

#### Dielectric sealing: mono-material sealing

Dielectric sealing can seal mono-materials, as long as the sealant layer is polar. Therefore, it does not seem suitable for mono PE and PP applications. However, a mono PET application is already reported to be successful [125].

#### Dielectric sealing: energy consumption

In general, dielectric sealing consumes a little more energy than conduction sealing [125] Another source states that it uses less energy than conduction sealing, if both are implemented on intermitted sealing. Dielectric sealing does not use power when not sealing. According to this source, this makes dielectric sealing overall more energy efficient [126]. The sealing process requires around 250W to 300W [216]. The power consumed during sealing is compared in the framework, therefore it is concluded that dielectric sealing requires more energy than conduction sealing.

#### Dielectric sealing: start cost

For dielectric sealing, RF generators, are seen as expensive [97].

#### Dielectric sealing: production speed

Dielectric sealing works in cycles. In each cycle, a film is sealed, although more than one film may be sealed during one cycle [215]. Production speeds of 10 cycles per minute [215, 216] up to 20 cycles per minute [125] are reported. This is with the note that one cycle includes press time, sealing time and cooling time. The sealing time itself can be as short as 2 seconds [126]. This works in cycles and a comparable production speed is thus hard to indicate. Another source has been found that can produce 60 packs per minute [218]. This value will be used in the framework to compare the sealing technology to other sealing technologies.

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#### Dielectric sealing: maintenance time

There is a broad scala of maintenance operations that can happen on the dielectric sealing system. It is expected that it requires less maintenance than a conduction sealing system. This is based on the manual from a dielectric sealing system describing the maintenance. The only maintenance operation that needs to happen daily is draining the trap on the air regulator. Other operations like cleaning are not necessary for the cleaning equipment [215].

#### Dielectric sealing: layer jump

No information is found on this topic.

#### Dielectric sealing: contamination

Dielectric sealing can seal through contamination in the seal area, except if the contamination is metal. It cannot expulse contamination from the seal area, but it can encapsulate it. Compared to other sealing technologies, like contamination, dielectric sealing performs quite well [125, 126].

#### Dielectric sealing: limitations

An inherent limitation of the dielectric sealing technology is that it requires a polar sealant layer to be able to seal.

Furthermore, a limitation is that the equipment requires long warm-up times if the dielectric sealing equipment is heated [215].

#### Dielectric sealing: opportunities

There are some great possibilities associated with dielectric sealing. For example, it is possible to realize tear seals with this technology. Next to this, the seals have a great appearance [215]. Another supplier states that the machines are very reliable and exemplifies this with a machine that has been running daily for 40 years already [125].

#### Dielectric sealing: continuous or intermitted

This system requires some pressurized cooling time, so it is an intermitted sealing technique [160, 215]. If the sealing jaws with electrodes run along with the film, it could be implemented in a continuous system in theory.

## **Digital sealing**

Conduction sealing experiences some trouble sealing mono-materials and sealing layer jumps. Digital sealing can be the ideal type of sealing when trying to seal mono-materials or layer jumps. First, in this part, an explanation of why these mechanisms can cause trouble for conduction sealing will follow. Afterward, digital sealing itself will be explained together with the framework parameters.

Mono-materials are difficult to seal for this sealing technology, since conduction sealing jaws have large temperature fluctuations, sometimes even more than 10°C [219]. Mono-materials only have a small seal window, so cannot consistently be sealed without compromising the production speed with these huge temperature fluctuations [219, 220]. This is confirmed by my observations during testing, see Appendix 12. Testing at Omori.

Sealing layer jumps can also create difficulties for conduction sealing. This is caused by the difference in width of the film and thus pressure exerted on one part of the film compared to the other side. A possible solution for this is introducing sealing bars that can be adjusted to have different temperatures in different regions [52].

This is exactly what digital sealing offers, it consists of multiple heating elements of around 5mm by 5mm (co-called heat pixels) that can be independently controlled (see Figure 74). Each heat pixel also includes a temperature sensor which is located close to the heating surface. This allows this sealing technology to control the temperature more precisely than for example conduction sealing. It also allows differentiation of the heat within the sealing jaw. The precise



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*Figure 74.* Digital sealing visualization of the heat pixels (source: [5])

temperature control allows this technology to effectively seal mono-materials, while the differentiation in heat allows this technology to effectively seal layer jumps [219, 220]. A simplified representation of this technology in combination with a layer jump can be found in **Figure 75**. The higher temperature at the four layers of film assists the sealing in this area [221].



Figure 75. Simplified representation of digital sealing of a layer jump

Digital sealing was invented by the company Watttron [222] and this invention earned them the 2024 Food Tec award [220]. Digital sealing is not mentioned in public literature. When searching for "Digital seal\*" AND "packaging" in the database Google Scholar, from the 21 hits no relevant articles show up. Only articles regarding the digital seal of a document and digital seal testers are shown as results. It can be concluded that this is a new type of sealing that has not received (m)any attention from a scientific perspective. So all information presented in this part is based on interviews, emails and websites from Watttron or sources referring to Watttron. It should be noted that this is a limitation from this part since no independent source is referred to.

This technology can be applied in the transverse and the longitudinal seal to for example VFFS and HFFS systems. It can also be implemented in already existing packaging machines. Products that are realized, include for example cups, trays, pillow bags and pouches [5, 127, 219, 221].

#### Digital sealing: mono-material sealing

As already mentioned, digital sealing is suitable for sealing mono-materials. The sealing window is the same as conduction sealing in terms of temperature, but it does not require a sealing window as large as conduction sealing [221]. This technology only has temperature fluctuations of 1°C compared to 10°C for conduction sealing [5, 127]. So it can be concluded that digital sealing is suitable for mono-material applications.

#### Digital sealing: energy consumption

In general, digital sealing consumes less energy than conduction sealing. It requires lower warm-up times (due to lightweight heat pixels) and digital sealing can precisely turn on the heat where necessary and turn it off where it isn't. This prevents energy spillage that cannot be achieved by conduction sealing [127, 221, 223]. An estimation of the reduction in energy consumption is 30 to 50% compared to conduction sealing in continuous operations [5, 127, 219, 221]. It is therefore concluded that digital sealing is more energy efficient than conduction sealing.

#### Digital sealing: start cost

Reading through all the advantages of this technology might make one wonder why this technology is not more often employed. This might have to do with the investment costs. Depending on the application, a digital sealing system is 2 to 10 times more expensive than conduction sealing [127]. In general, it is more affordable than ultrasonic sealing technology, according to Bach [127]. She spends her PhD on investigating the differences between conduction and ultrasonic sealing [9]. This increases her reliability, although she now works for Watttron.

#### Digital sealing: production speed

The production speed for the brought sealing window material is expected to be similar to conduction sealing since both technologies work similarly. It might be the case that the production speed must be lowered to seal mono-materials with conduction sealing. In that case, digital sealing might reach higher production speeds (10 to 20%) [127].

The time it takes to warm up the system is lower for digital than for conduction sealing [5]. For the framework only the production speeds during sealing are used, so the warm-up time is neglected. It is expected that digital sealing reaches similar production speeds as conduction sealing.

#### Digital sealing: maintenance time

Digital sealing scores slightly higher on maintenance time compared to conduction sealing, since the system can cool down quicker [221]. This way, maintenance can happen quickly. Furthermore, the changeover times are lower [223]. However, both technologies need to be cleaned regularly. Digital sealing is generally more complicated to repair than conduction sealing technology. The sealing jaws can be sent to the supplier to be repaired, so it is recommended to have a spare sealing jaw to minimize downtime [127]. It is concluded that both technologies perform similarly regarding maintenance.

#### Digital sealing: layer jump

Digital sealing is suitable for closing layer jumps, as mentioned before. It can adjust the temperature locally. This helps to successfully close the gap that usually arises at the layer jump [127]. So, digital sealing performs better than conduction sealing regarding layer jumps.

#### Digital sealing: contamination

Digital and conduction sealing are expected to perform similarly when contamination is introduced in the seal area. This is expected since both technologies work similarly with two heated sealing bars introducing heat into the seal area while also exerting pressure. One source mentions that digital sealing can handle liquid contamination slightly better than conduction sealing [127].

If contamination is present in the seal area, the temperature sensors report a deviation from the target value. These deviations can be detected and these packages can be taken out. So digital sealing allows for contamination registration [127, 219].

All in all, it is concluded that digital sealing performs similarly to conduction sealing in case of contamination in the seal area, although digital sealing might perform slightly better in case of liquid contamination.

#### Digital sealing: limitations

A limitation of digital sealing technology is that the maximum temperature that can be reached is 250°C [219]. More limitations are not found, this might be explained by the fact that only the suppliers of this technology are the only source used and no independent sources.

#### Digital sealing: opportunities

There are some opportunities associated with digital sealing. It can for example be implemented in an existing VFFS or HFFS system to seal mono-materials or layer jumps.

Another advantage of this technology is that it has a built-in quality control system in the form of temperature sensors that compare the measured temperature with the target value.

#### Digital sealing: continuous or intermitted

Digital sealing can be applied on both continuous and intermitted sealing [127].

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# 6.3.Framework

	Seal mechanism	Mono-material sealing	Energy consumption	Start costs	Production speed	<b>Maintenance</b> time	Layer jump	Squeeze-out	Contamination	Intermitted (I) / continuous (C)	Limitations	Opportunities
Explained in chapter:		5.7 and 5.8	4.2	4.3	4.4	4.5	4.6	4.7	4.8			
Conduction sealing	Heated jaw + pressure	-	+/-	++	+/-	-	+/-	+	-	I&C	High temperature fluctuations Mono-materials	Low cost Intuitive
Ultrasonic sealing	Vibrating horn on anvil + pressure	++	++	-	+	+		+/-	++	I&C	Layer jumps EVOH damage	Heat sensitive products Implement on existing machine
Hot gas sealing	Hot gas + pressure from rolls	+	-	+/-	++	+	/		+	С	Safety risk with paper applications, oxidation of seal risk	Thick films and films that don't conduct heat well
Impulse sealing	Heat (resistance wire) + pressure (also while cooling)	+/-	+/-	++	-		+/-		+/-	I (C)	No control system	More precise temperature control Narrow seal Small badges/ low speeds
Induction sealing	Quickly changing magnetic field	+	+/-	+	+/-	++	/			I&C	Requires conductive layer No metal close Circular edge required	Non-contact Thinner films can be sealed
Dielectric sealing	Changing electrical field (+ pressure)	+	-		-	+	/		-	I (C)	Only polar materials (no PE and PP)	Realize tear seals with great appearance
Digital sealing	Heated jaw + pressure	+	+	+/-	+/-	-	+		-	I&C	Only up to 250°C	Implement in existing machine Quality control

++ Very positive
+ Positive
+/- Neutral
- Negative
-- Very negative
/ No implementation reported
#### Explanation conduction and ultrasonic sealing

An explanation of why the framework for conduction and ultrasonic sealing looks like this will be given in the next part. This can be considered a partial conclusion of the thesis. Most parameters are evaluated based on the nature of the impact the sealing technology has on the parameter. This nature can be (very) positive, (very) negative, neutral or no implementation has been reported. As an example, ultrasonic sealing scores 'negative' for the parameter cost, since the cost of implementation for the ultrasonic sealing system is relatively high. This part will explain why the comparison in the framework between conduction and ultrasonic sealing looks like it does.

#### Seal mechanism

Conduction sealing can fuse two films of material by applying heat and pressure to the outside of the film. The heat has to conduct through the film layers to heat the sealant layer. The pressure is required to bring both films into intimate contact. Parameters that mainly influence the seal strength are pressure, temperature and sealing time.

Ultrasonic sealing uses a mechanically vibrating horn to heat the two films from the inside. The horn vibrates on the anvil with the film in between. Heat is generated in between the films by intermolecular and interfacial friction. Parameters that mainly influence the seal strength are the sealing time, amplitude and frequency of the horn and the sealing force (pressure).

#### Mono-material sealing

The sealing window in which mono-materials realized with conduction can be sealed, is limited. This is caused by the fact that conduction sealing heats the material from the outside, thereby requiring a high difference in melting temperature between the outer and inner layers of the film. This is hard to realize with mono-materials. This is also confirmed by the dye penetration tests performed on four different materials. All materials showed a lower sealing window for conduction sealing compared to ultrasonic sealing.

The ultrasonic sealing technology does not require a high difference in melting point between the sealant and outer layer. This makes this technology very suitable for mono-material applications, for which the sealing window is large. Especially compared to conduction sealing.

#### **Energy consumption**

Conduction sealing heats the material from the outside using heated sealing jaws. These jaws are continuously heated and therefore emit a lot of energy to the environment. This makes the conduction sealing process energy efficiency poor. Only keeping the sealing jaws at a constant temperature is estimated to consume 240W. Note that the framework thus only compares the power used during the actual sealing and neglects the energy consumed to heat the sealing jaws.

The energy efficiency of the ultrasonic sealing system is described in the literature as being very high. Ultrasonic sealing especially uses less energy compared to conduction sealing with low machine speeds, thick materials and materials with an aluminum layer included. Sealing mono-materials with an ultrasonic longitudinal seal consumes around 110W. The seal setting pressure mostly influences the energy consumption, but the amplitude also influences it. So ultrasonic sealing only uses around (110/240=) 46% of the power that conduction sealing uses.

#### Start cost

It can be concluded that the difference in investment costs is huge between ultrasonic and conduction sealing. An extra investment of around €53000 is required to employ an ultrasonic sealing system

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instead of a conduction sealing system. Note that this only counts for the longitudinal seal. Conduction sealing is considered to be an economical sealing option.

#### **Production speed**

Higher production speeds can be achieved with ultrasonic sealing compared to conduction sealing. Comparing the typical production speeds of 80 (for conduction sealing) and 110 (for ultrasonic sealing) packs per minute indicates the order of magnitude of their difference. However, both conduction and ultrasonic sealing technologies are considered to be suitable for high-speed applications, as indicated by industry.

#### Maintenance time

Regarding maintenance, ultrasonic sealing is preferred over conduction sealing. Mainly due to the sealing jaws being hot during sealing and taking a long time to cool down. Also, since there is no heat exerted on the material from the outside by the ultrasonic sealing system, less cleaning is required compared to conduction sealing.

#### Layer jump

For conduction sealing, layer jumps can be sealed with sufficient pressure. The sealing jaw profile can be altered and a rubber membrane can be added to make the gap that exists at a layer jump smaller as illustrated by Hauptmann, et al. [17].

Ultrasonic sealing generates heat at the seal interface. When more than one seal interface is present, this technology reaches its limitations. This does not seem to be solvable by optimizing the seal settings. So ultrasonic sealing does not seem suitable for sealing layer jumps.

#### Squeeze-out

Squeeze-out is usually not problematic and often desired up to some degree within the conduction sealing context, as long as the seal settings are not extremely high.

Ultrasonic sealing generates heat at the seal interface, thereby enlarging the risk of squeeze-out. Squeeze-out is observed at ultrasonic sealing with normal settings. Squeeze-out is possible at all local sealing sites. The amplitude and the pressure positively influence the squeeze-out. For the tested settings (that range from low to very high), no squeeze-out problems could be observed.

#### Contamination

Conduction sealing can be used in combination with contamination. Only a slight decrease in seal strength is observed. Solid particles, like powders, are not suitable for conduction sealing.

Ultrasonic sealing is known for its ability to seal through some contaminants. It especially performs well in combination with powder contamination. An indirect advantage of ultrasonic sealing is that it does not melt the contaminants. The differences between conduction and ultrasonic sealing are quite small, except in the case of loose materials (like powders).

#### Limitations

#### Ultrasonic sealing

The disruption of the EVOH layer for mono-material applications is worth mentioning since it might affect the suitability of a sealing technology for mono-materials. No conclusions have been drawn on this topic yet, but for ultrasonic sealing, EVOH disruption has been observed. Whether this happens with other materials as well and whether this is problematic for the barrier properties is still unknown.

#### Conduction sealing

Conduction sealing is not ideal for sealing mono-materials, because of the high-temperature fluctuations within the sealing jaw. Mono-materials can only be sealed within a small temperature window. The seal jaws must keep the temperature within this window.

#### **Opportunities**

#### Ultrasonic sealing

As already mentioned, an indirect advantage of ultrasonic sealing is that it does not melt the contaminants.

A combination of ultrasonic sealing technology with other sealing technologies is possible. An example is demonstrated by a company that has a machine in its portfolio that employs an ultrasonic sealing system at the longitudinal seal and a conduction sealing system at the transverse seal. They claim to be able to realize production speeds of up to 170 packs per minute [113].

#### Conduction sealing

Conduction sealing is intuitive to operate [105].

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#### **Explanation other types of sealing**

This part will further explain why the framework looks like it does for the parameters maintenance time, start cost and production speed. Why the different types of sealing relate to each other like they are presented in the framework should be clear after reading this section. How the sealing technologies are presented in the framework for the other parameters should be clear from the information presented per sealing technology in the chapter Introduction of other heat sealing mechanisms.

#### Comparison: Maintenance time

Ultrasonic sealing only requires around 5% of the maintenance costs compared to conduction sealing. It is expected that this fraction will be the same for the maintenance time. The maintenance operation that significantly adds to the time for conduction sealing maintenance, is the cleaning. If this is not required anymore (e.g. because a sealing technology does not require heated physical contact), the maintenance time should drop a lot. Hot gas, dielectric, ultrasonic and induction sealing don't have the same contact between a heated element and the film. It is expected that this will take away the need for frequent cleaning and the maintenance time for these sealing technologies are thus lower. Induction sealing doesn't make contact at all with the film. That is why it is expected that this technology requires the least maintenance time

#### Comparison: Start costs

To fill in the framework, it is necessary to make a comparison of the costs between the different sealing technologies. This is difficult to quantify since it might indicate whether a sealing technology is less affordable than conduction sealing, but it is hard to exactly know how it compares to other sealing technologies. Standard prices of sealing technology equipment are not found. Therefore, Alibaba has been used to compare the technologies. Semi-automatic machines have been selected for each sealing technology together with the price that is presented. It is expected that the ratios between the prices are similar for mass production implementations. The findings are presented in **Table 22** and are used as a basis for filling in the framework for the starting costs.

Sealing technology	Price (\$)	Source
Conduction sealing	140	https://www.alibaba.com/product-detail/YOUNGSUN-Automatic-Horizontal-Continuous-Solid- Ink_1600829058840.html?spm=a2700.galleryofferlist.topad_classic.i6.756c1913f3gEzP
Ultrasonic sealing	1050	https://www.alibaba.com/product-detail/15khz-2600w-Digital-Welding-Ultrasonic- Welder_1600230547705.html?spm=a2700.galleryofferlist.normal_offer.d_title.54a331ads4o5Nx
Hot gas sealing	700	https://www.alibaba.com/product-detail/QS-1000-protective-cloth-hot- sealing_1600453454974.html?spm=a2700.galleryofferlist.normal_offer.d_price.423169b2ByCn1 d
Impulse sealing	185	https://www.alibaba.com/product-detail/KSA-450-Table-Style-Semi- Automatic_1600268512483.html?spm=a2700.galleryofferlist.normal_offer.d_price.48a2f3efPYX yF8
Induction sealing	300	https://www.alibaba.com/product-detail/New-fully-automatic-electromagnetic-induction- aluminum_1600957839230.html?spm=a2700.galleryofferlist.p_offer.d_price.1fe05de6P6xsQq&s =p
Dielectric sealing	2499	https://www.alibaba.com/product-detail/Double-Heads-Radio-Frequency-Welder- For_1600847238893.html?spm=a2700.galleryofferlist.normal_offer.d_price.518185bdqCBYjz
Digital sealing	280- 1400	2 to 10 times more expensive than conduction sealing [127]

Table 22. Comparison of different semi-automatic sealing technologies' prices

#### Comparison: Production speed

To reliably compare the different types of sealing on production speed, it is necessary to quantify the production speed in the amount of meters of material per minute. The long seal dimension of a typical packaging (e.g. sliced cheese) has been estimated at 0,2m per package to go from packs per minute to meter of material per minute. It is therefore expected that every package can reach the same production speed as a package from this dimension. The production speeds used to fill in the table are listed in Table 23.

Table 23. Production s		6.		
Sealing technology	Typical production speed (pack/min)	Typical production speed (m/min)	Source	<b>.</b>
Conduction sealing	80	16	[110]	8.
Ultrasonic sealing	110	22	[88]	9.
Hot gas sealing		50	[124, 200]	10.
Impulse sealing		12	[206]	11.
Induction sealing	80	16	[209]	12.
Dielectric sealing	60	12	[218]	
Digital sealing		16		

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# 7. Example case

In this chapter, an example of how to use the framework will be given. This will be done by presenting an imaginary case and explaining how the framework can be employed to facilitate the heat-sealing technology decision-making process.

## 7.1.Scenario

The imaginary case used for explaining the framework use is presented here, but it is based on a website text [4].

A company that specializes in packaging around sliced cheese has lowered the environmental impact of the packaging as a high priority. They already changed their packaging from a rigid tray with a flexible lid to an envelope packaging (see **Figure 76** for an example of an envelope packaging). This transition in the packaging concept already reduced the weight of the package.



Figure 76. Example of an envelope package [4]

The current package's general material composition is

OPET-PE-EVOH-PE. The EVOH layer is there to function as an oxygen barrier. OPET has a relatively high melting temperature compared to PE, which allows for efficient conduction sealing. One drawback of this packaging solution is that it is not suitable for efficient recycling as it ends up in a mixed stream (in the Netherlands) [214].

To lower the impact of the packaging, the company wants to change to a mono-material. One supplier offers a mono-material film with enough barriers to protect the cheese. In combination with Modified Atmosphere Packaging (MAP), a long enough shelf life can be guaranteed [224]. The general material composition is BOPP-PP-EVOH-PP, with the EVOH layer being less than 5% of the weight of the package. This qualifies as a mono-material and is better suitable for recycling than the previous material [15]. The company wants to keep the geometry of the packaging system as it is (envelope form).

The question from the company is what type of heat sealing technology they could best choose to realize the envelope packages with the mono-material. That is where the framework can help.

## 7.2.Framework use

To answer this question, important parameters from the framework should be selected first. The parameters are listed below together with an explanation of why the parameters are included or not.

Include:

- Mono-material sealing window, for this application the sealing of the mono-material is of high importance since a mono-material will be used.
- Energy consumption, the company is trying to minimize the impact their packaging solution has on the environment. This is also influenced by energy consumption (so this should be minimized).
- Start costs, the investment should also be financially interesting for the company.
- Production speed, the sliced cheese should be packed fast, at least 60 packs/minute (estimated to be 12m/min of packaging material with each package being 20cm long)
- Maintenance time, the maintenance time should be as short as possible.

- Layer jump, the envelope geometry includes a fold which creates a fold at the transverse seal (if sealed after folding, which is done at e.g. Omori flow wrappers). Note that this is only required for the transverse seal, the longitudinal seal doesn't require the ability to seal monomaterials.
- Contamination, it is not expected that contamination will enter the seal area often, but it should be included in the overview to get a complete picture and comparison.

Excluded:

- Squeeze-out, it is not expected that squeeze-out will be problematic, as no excessive seal settings will be used.
- Intermitted/continuous, it does not matter if the process is intermitted or not, as long as the set production speeds can be reached.

The framework with all included parameters is presented in **Table 24** with a color addition to make it more comprehensible at first look.

	Mono- material sealing	Energy consumption	Start costs	Production speed	Maintenance time	Layer jump	Contamination
Conduction sealing	-	+/-	++	+/-	-	+/-	-
Ultrasonic sealing	++	++	-	+	+		++
Hot gas sealing	+	-	+/-	++	+	/	+
Impulse sealing	+/-	+/-	++	-		+/-	+/-
Induction sealing	+	+/-	+	+/-	++	/	
Dielectric sealing	+	-		-	+	/	-
Digital sealing	+	+	+/-	+/-	-	+	-

Table 24. Example use of the framework

The next step that needs to be taken is the determination of what parameters are more important than others. This could be done by for example adding a weighting factor to the parameters and assigning a number to each score (e.g. a 1 for '-' and a 2 for '+/-'). This method however has its drawbacks, since the sealing technologies are compared to each other on a non-linear scale and not on the same scale for each sealing technology. This is exemplified by the parameter start cost. The start cost is for (\$140) conduction sealing (++), for (\$300) induction sealing (+) and for (\$700) hot gas sealing (+/-), which does not show a linear trend (as is shown in Figure 77).

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**Figure 77.** Graph plotting the score against the price (\$) for start cost of conduction (++), induction (+) and hot gas sealing (+/-)

Therefore no weighing factors will be used. There will be looked at what parameters have the most influence and what sealing technologies thus are least suitable. First, induction sealing and dielectric sealing are excluded from consideration, since no conductive or polar layer is included in the film.

The most important parameter in this example case is the mono-material sealing window. If this window is too small, the material cannot be closed properly thereby possibly compromising the shelf life. *Conduction sealing* can also be excluded based on this requirement.

For the transverse seal, a layer jump must be sealed. This also excludes ultrasonic sealing for this application. Hot gas sealing has no implementation known for the transverse seal and it is questionable whether this is even possible, so this technology can also be excluded from the transverse seal. Only impulse and digital sealing are left for the transverse seal. Digital sealing will be chosen as the preferred option, mainly because it outperforms impulse sealing when it comes to mono-materials.

For the longitudinal seal, no layer jumps are required. Therefore, ultrasonic sealing will be chosen as a sealing technology. It outperforms the other types on the parameters 'mono-material sealing window' and 'energy consumption', which are important parameters for this company case.

So the advice for the company, based on the framework, will be to use digital sealing on the transverse seal and ultrasonic sealing on the longitudinal seal.

# 8. Limitations

This thesis comes with a few limitations. These limitations will be described below. The limitations might arise from for example the testing techniques or from interpretations of conclusions.

## 8.1.Framework

The main limitations of this thesis are about the framework itself since that is the main outcome of the thesis. The limitations inherent to the framework will be discussed in this section.

First of all, not all available (heat) sealing technologies are included. The goal of the framework is to adequately assist in the choice of sealing technology. It might be the case that one of the sealing technologies that is not included in the framework (e.g. hot wire or cold sealing) is the optimal option for a certain application. If that is the case, using the framework will give the wrong conclusion. Most conventionally used heat sealing technologies are however included in the framework, so it is expected that the chance of ending up with a wrong conclusion is small, but should still be considered as a limitation.

Secondly, a limitation of the framework is not only that not all sealing technologies are included, but also that not all parameters are included. Parameters affecting the sealing technology decision-making might be left out of the framework. This might lead to invalid conclusions when using the framework. An illustrative example is the life span of a certain sealing technology. This parameter is not included in the framework (see chapter 4.9 Life span for the reason), while this is a very interesting parameter to consider when choosing a type of sealing.

Thirdly, the framework is not fully validated. This means that it is not yet confirmed that this framework can indeed successfully be employed as a tool to choose a heat-sealing technology. The data gathered to fill in the framework comes from literature sources, testing and sources from industry. Therefore, it is expected that this framework is valid since it includes insights from different angles. The framework also seems to work based on the example in chapter 7 Example . So it is expected that the framework is validation through case studies should be noted. A validation plan is already presented in chapter 9.1 Future research on the framework.

Lastly, to help fill in the framework, sealing technology suppliers have been contacted. These suppliers have the motivation to promote their sealing technology and let it appear better than they are. Although it is tried to minimize the impact that this can have by comparing different answers to each other and by looking at independent sources, this might have added some bias to the results presented in the framework. Especially for the sealing technologies other than conduction and ultrasonic sealing. For these two sealing technologies, sufficient independent sources have been found and tests have been performed. This limitation therefore should be considered when interpreting the framework for other types of heat sealing than conduction and ultrasonic sealing.

Especially for digital sealing the aforementioned limitation should be considered. This heat-sealing technology has not been mentioned in public literature. The only source for filling in the framework for this type of sealing is the company Watttron, which is the supplier and inventor of digital sealing.

## 8.2. Testing limitations

Those were the limitations that count for the framework. More specific limitations about the testing that should be noted when interpreting that part of the thesis will be mentioned here.

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#### Dye penetration test

The dye penetration test has been used to check the seal integrity of different packages. This method is dependent on the observations of the operator within a short period (5s per seal). Not all leakages might be detected, due to human error. This might have implications for the results.

Furthermore, an assumption has been made about the seal integrity of packages realized with higher seal settings than packages that were closed. It is assumed that packages realized with higher seal settings and also show a higher seal strength than a closed package, are also closed. In this case, a closed package is one that did not show leakages at the dye penetration test. Why this assumption is deemed valid, is explained in the section 'Dye penetration application'. If it turns out this assumption is not valid, this would have implications for the results about the seal integrity.

#### **Samples**

The samples are not conditioned, as proposed by ASTM standards F88 and E171. These standards suggest conditioning the to-be-peeled samples for at least 40 hours. The samples used for this thesis are stored in a cardboard box in a laboratory environment for at least 40 hours. However, standard E171 implies that no meaningful physical changes in the packages are found when preserved in a laboratory environment [3, 148]. Therefore, it is expected that this will have a neglectable effect on the outcome of the tests, but should still be mentioned as a limitation.

#### Type of peel classification US

Each T-peel test for a seal created with ultrasonic sealing is classified. It can be classified as 'parallel 1' (4 steep peaks), 'parallel 2' (4 clear peaks), and 'oblique' (4 peaks cannot be distinguished). This classification brings along an additional source of bias. When the four peaks are for example considered steep and when not, are subjected to human perception. It is tried to minimize the bias by comparing the graphs of different types of peels with each other, to keep the classification consistent. However, it should be noted as a limitation that there is a degree of bias caused by this. This might have implications for the interpretations of the results from the T-peel tests from ultrasonic sealing.

The degree of bias is also decreased by adding both parallel 1 and parallel 2 tests into the analysis. So the distinction between these two classifications has no effect. The differentiation between tests considered as parallel oblique does have an influence.

#### **Material 2 conduction sealing**

The seals created with conduction sealing on material 2 were not closed. This is caused by the material sticking to the sealing jaw during the sealing operations. The sticking has not been observed with ultrasonic sealing and the ultrasonic seals were closed. Therefore it is assumed that material 2 is suitable to perform tests on with the ultrasonic sealing system. This assumption should be noted as a possible limitation.

#### Layer jump, squeeze-out and barrier layer destruction

For testing regarding the layer jump, squeeze-out and the barrier layer destruction for conduction and ultrasonic sealing, only one type of material has been used. If more materials would have been used, the conclusions of the tests would be more reliable. This is a limitation for the interpretation of the conclusion for these tests.

#### **ANOVA** test

During the analysis, the Shapiro-Wilk and Levene's tests were performed for all ANOVAs. Sometimes, these tests showed significant results. If the Shapiro-Wilk test showed a significant result for a certain group, there was no normal distribution within the group. If Levene's test was significant, there was no homogeneous variance among the different groups. Both tests affect the interpretation of the results. They might cause the outcome of the ANOVA to be invalid in case one of them (or both) is significant.

## 8.3.Testing results

There are some limitations associated with the results of the tests and the interpretation of them. These limitations will be presented here and only influence the interpretation of the results for conduction and ultrasonic sealing.

#### Mono-material sealing window test

The sealing windows conduction and ultrasonic sealing are compared based on the amount of settings that they can be set on. This comparison is not ideal. However, this method is selected since this is the most practical comparison. It is difficult to compare conduction and ultrasonic sealing operating windows since they both operate with completely different sealing parameters. This method is however flawed since the accuracy of the sealing technology machines is not considered. There might for example be fluctuations of 5°C for the conduction sealing machine, while the temperature can be set with one decimal preciseness.

Therefore, when filling in the framework, there is also looked at how the sealing technologies work. For example, ultrasonic sealing heats the material at the seal interface, which makes it ideal for sealing mono-materials. This already takes away the consequences of this limitation, but it should still be noted.

#### **T-peel tests**

Theoretically, the pressure and the amplitude should fully explain the variation in seal strength for ultrasonic sealing. The same counts for the temperature for conduction sealing. These parameters are the only factors that are varied, the rest of the context is kept constant as much as possible. This is however not completely true, since there is a deviation visible when looking at the T-peel results. This should be noted as a limitation. Bach, et al. [9] also observed a high standard deviation for ultrasonic sealing and think this might be caused by high sealing forces that are associated with uncontrollable melt flow or by the difference in crack and failure behavior.

The variation in seal strength for the same treatment might have different causes. It might for example be caused by variations in (layer) thicknesses within the same film. Another option is the placement of the samples in the peeling mouths. The seal should be completely parallel to the peeling mouths, as well as the upper as lower mouth, as is shown in Appendix 3. Acceptable ultrasonic T-peel test samples. This is practically impossible and is therefore a limitation of the research. It is proven that the angle between the seal and the peeling mouths influences the measured maximum seal strength and the peeling behavior for ultrasonic sealing. This might also be the case for seals created with conduction sealing. Also, environmental elements like temperature and humidity as well during the realization of the packages and the testing might affect the maximum seal strength result. Lastly, the accuracy of the sealing technology equipment for the realization and measuring of the seals affects the deviation in maximum seal strength within one treatment.

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#### Squeeze-out

While determining the amount of squeeze-out for the ultrasonic seals, microscopic images of the seals have been observed. The squeeze-out area could not be quantified since no trapezoid-like squeeze-out shape could be distinguished. This creates the limitation that no trends can be observed between for example the amplitude and the amount of squeeze-out.

The squeeze-out has only been determined qualitatively (yes or no). This is based on a thickness reduction of more than 5% at the seal area. The 5% value is substantiated but is not based on prior research. It could be the case that this threshold value is higher or lower, which might influence the conclusion about squeeze-out.

#### Layer jumps

The microscopic pictures made to evaluate the layer jumps might not all be taken in a properly sealed area. When looking at the cross-section of an ultrasonic seal, it can be observed that the seal is not closed throughout the entire cross-section. The ultrasonic seal is realized by locally sealing at four points. The pictures used for the analysis of the layer jumps are made parallel to the seal. Effort was made to take a picture of the closed part of the seals. This was checked by looking at the two-layered section of the picture. However, it might be possible that the visible part of the seal was at the edge of the closed seal, thereby giving the wrong impression about the seal (integrity). This is a limitation of the analysis of layer jumps.

### 8.4.Other limitations

There are other limitations than the ones already mentioned. More specific limitations not concerning testing and the framework that should be noted when interpreting the other parts of the thesis will be mentioned here.

#### Easy peel

One of the requirements for a seal to be considered 'easy peel', one should be able to peel the seal without the use of any tools. This requirement is quite ambiguous since strength differs per person. This brings some bias into the quantification of when a seal can be considered to be easy to peel. This should be considered as a limitation, especially when looking at the chapter describing the possibility of creating an easy peel seal with ultrasonic sealing without controlled contamination in the material (see chapter 5.13 Test 9. Easy peel US).

#### Flexible mono-material recycling

The focus of this thesis is on flexible plastics. It should be noted that most recycling processes focus on rigid plastics. This sometimes means that the flexible plastics are neglected and do not end up in a recycling stream [14]. This limitation should be considered for flexible mono-materials. This might mean that switching from a multi-material to a mono-material does not decrease the environmental impact of a flexible plastic packaging system. It is supported by governments to switch to mono-materials, also for flexible plastic packaging systems, so it is expected that this limitation does not count for Europe.

#### Contamination

Chapter 4.8 Contamination is mainly based on a paper from Bach, et al. [9]. This paper also knows some limitations that should be considered when interpreting the results of that chapter. First, the seal settings are not optimized for contamination. The sealing technologies could therefore show

higher performance in combination with contamination in the seal area than suggested by their paper. There is one more limitation, the seal integrity is not evaluated for the seals, only the seal strengths. The seal performance consists of the seal integrity and the seal strength, so their research only shows a part of the seal performance in case of contamination. These limitations also apply to the contamination of conduction and ultrasonic sealing in the framework. Although there, the work of Bach, et al. [9] is combined with other (independent) sources. So the impact of this limitation will be lower there.

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## 9. Future research

This thesis presents a comprehensive framework that compares different types of heat-sealing technologies. This is based on scientific literature, sealing technology suppliers and testing. While gathering all the information, some directions for future research arose. In this chapter, the different directions will be described.

## 9.1. Future research on the framework

The framework is already quite complete and seems usable for a proper heat-sealing technology selection. However, the framework has not been validated. It is recommended to validate the framework and adapt it accordingly.

It is recommended to ask experts for feedback on the framework. This might add some nuance to the framework and validate it. The most important validation should be done through testing and case studies. This will confirm whether the framework is also valid in practice or if there are some changes required. It might for example be the case that a limitation is not added, but which can cause problems in practice. The framework is based on information from literature, tests and practice and requires case studies to be validated.

To increase the usability of the framework, the scores (e.g. '+' or '-') should be changed to numbers on a linear scale. Then, the sealing technologies can be compared. This also opens up the opportunity to calculate a score per sealing technology based on a weighing factor for the parameters. This is also illustrated by Colvin [97], where the sealing technology with the highest score is the optimal choice.

## 9.2.Squeeze-out

Nase, et al. [84] and Bach, et al. [9] both mostly refer to the sealing force(s) as being the driving parameter that influences the squeeze-out. During testing, however, the amplitude also seemed to influence the squeeze-out for the ultrasonic sealing process. To fully grasp the (nature of the) influence that different parameters have on the amount of squeeze-out for ultrasonic sealing, more research is required.

It is expected that parameters that influence the temperature at the seal interface, also influence the amount of squeeze-out. Also, other parameters might influence the squeeze-out, like the anvil design, because the seal width also influences the squeeze-out for conduction sealing [10]. So a hypothesis for future research might look like this: it is expected that the frequency, amplitude, sealing force, sealing time and anvil geometry influence the squeeze-out occurring at ultrasonic sealing.

## 9.3.Squeeze-out for other sealing technologies

The squeeze-out of other sealing technologies has not been evaluated. Squeeze-out however has some effect on how well for example contamination in the seal area can be handled by a sealing technology. Therefore, it might be interesting to dive deeper into the combination of squeeze-out and other heat sealing technologies than conduction and ultrasonic sealing.

It is expected that the squeeze-out is dependent on the presence of pressure and the heat mechanism. The more pressure is exerted, the higher the squeeze-out will probably be. That for example also means that for induction sealing, limited to no squeeze-out is expected. How hot the seal interface gets (heat mechanism) is also expected to influence the squeeze-out. The hotter the interface, the easier the material will flow out of the seal area.

With further research on this topic, it should be determined when squeeze-out can form problems in combination with ultrasonic sealing. It could for example be the case that with extensively high seal settings, squeeze-out can become problematic (just like with conduction sealing).

# 9.4.Effect of amplitude on the influence of pressure on the seal strength

Another interesting future research direction is related to the interaction effect between amplitude and pressure. During the testing of the effect that the amplitude and the pressure have on the seal strength, the interaction effect between these two factors turned out to be significant.

It seemed to be the case that for lower amplitudes, the pressure has a significant effect on the seal strength, but for higher amplitudes not. To know what the (nature of the) influence of the amplitude on the influence that the pressure has on the seal strength is, more research is required.

This could be evaluated by testing the seal strength of different ultrasonic seal samples realized at a wider variety of amplitudes and pressures. The differences between the influence of pressure at high amplitudes can be compared with the influence of pressure at low pressures.

It is expected that for higher amplitudes the pressure indeed has a neglectable effect since a plausible hypothesis can be formulated to explain this phenomenon. The hypothesis is that for low amplitudes, the pressure is indeed necessary to bring the films into intimate contact. The horn only vibrates a little bit at lower amplitudes. It seems plausible that pressure is required to bring these small oscillations into enough contact with the film to properly seal. It makes sense that in that case, the more pressure, the closer the horn is pressed in the film, the stronger the seal.

For higher amplitudes, the horn will have larger oscillations. The pressure is in this case not required to bring the horn into enough intimate contact with the films. The amplitude alone already realizes that. Therefore, the pressure has a neglectable effect at these higher amplitudes. Note that this is all speculative and requires more research to draw conclusions about the validity of the hypothesis.

## 9.5.T-peel test seal angle

It is proven that the angle between the seal and the peeling mouths influences the measured maximum seal strength and the peeling behavior for ultrasonic sealing, as is shown in in Appendix 3. Acceptable ultrasonic T-peel test samples. What the exact relation between the angle and the measured maximum seal strength exactly is, is interesting to determine. This way, if the angle between the seal and the peeling mouths is recorded for each T-peel test, the measured maximum seal strength can be adjusted accordingly. This way, less deviation within the measured seal strength is expected.

This could be evaluated by measuring the maximum seal strength of ultrasonic seals and capturing the angle between the peeling mouths and the seal. This could be done for seals created at different settings and with different materials to increase reliability. The result could indicate a certain trend between the angle and the seal strength, thereby indicating their relationship.

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## 9.6.Damage to the barrier layer

While looking at the microscopic images that have been created, it became apparent that the EVOH layer might have been damaged during the ultrasonic sealing process. In **Figure 78** one of these pictures can be seen. It can be observed that at the indent, the EVOH layer is not continuous. More research is required to see what the effect of these interruptions might be.

This could be done by performing oxygen permeability tests with the seal area included in the sample. This could be done for different ultrasonic seal settings and this way, the effect of the seal settings (amplitude and pressure) on the EVOH layer damage effect can be explained.



*Figure 78.* Damage to the barrier layer visible at sample 7 (0,1bar; 70%; material 1)

If the EVOH layer is indeed damaged, this might affect the oxygen permeability, since EVOH acts as the main protector against oxygen within the film of material 1. If the damage has any effect, this would become visible at an oxygen permeability test. It is not expected that the damage to the EVOH layer has a significant effect on the oxygen permeability since successful shelf life tests have been reported with oxygen-sensitive products packed in a mono-material with an EVOH layer.

## 9.7. Easy peel and ultrasonic sealing

It has been suggested in this thesis that ultrasonic sealing can create easy peel seals without the need for controlled contamination in the film. However, to draw conclusions on whether this is indeed consistently possible and how this exactly works requires more research.

In this research, it should also be established whether the T-peel test is a proper method to determine how easy a seal is to peel. It might for example be the case that an ultrasonic seal and conduction seal that show the same maximum seal strength, show different peel experiences.

# 10. Conclusion

In this thesis, a framework has been developed that can be used as a tool to choose heat-sealing technology. This has been done by looking at scientific literature, by performing tests and by consulting sealing technology suppliers. All information combined forms the framework that compares a selected amount of heat sealing technologies (conduction, ultrasonic, hot gas, impulse, induction, dielectric and digital sealing) for a selected amount of parameters (heat mechanism, mono-material sealing, energy consumption, start costs, production speed, maintenance time, layer jump, squeeze-out, contamination, intermitted or continuous, limitations and opportunities). The framework is the main conclusion of this thesis and can be found in chapter 6.3 Framework.

The focus of this thesis was on the sealing technologies conduction and ultrasonic sealing. These are investigated more than the other technologies. The framework could not fully be filled in for ultrasonic sealing without testing. The results of these tests give more insight into the principles at work at ultrasonic sealing. Ultrasonic sealing can seal mono-materials that can incorporate easy peel (without controlled contamination material) as long as no layer jump is present in the seal. Ultrasonic sealing might cause damage to the barrier layer of a material.

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