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Properties of an aluminum based multilayered material versus package and processing performance

Eoin de Roiste

PROPERTIES OF AN ALUMINIUM BASED MULTILAYERED MATERIAL VERSUS PACKAGE AND PROCESSING PERFORMANCE.

By

Eoin de Róiste

A Thesis

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M. S. Degree Thesis

The M. S. Degree thesis of Eoin de Róiste
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for the thesis requirements for the
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August 4th, 1999

PROPERTIES OF
AN ALUMINIUM BASED MULTILAYERED MATERIAL
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DEDICATION

For Maria.

Thank you for your all your help and support plus your patience over the last two years since this course commenced.

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ABSTRACT

Due to a manufacturing issue within the company the lack of knowledge of a key material is highlighted. Research into the material is conducted under seal testing and mechanical properties. The material is multilayered, with aluminium foil as its core plus is top coated and has a sealant layer as its bottom coating.

Background literary review commences with a chronological assessment of packaging down through the ages and culminates with details on multilayered materials, concentrating on the type of material being researched.

A test plan details the research requirements. Seal testing consists of burst and peel testing of the material in its final package configuration and utilises two blister package sizes. A test on the variation of the actual burst tester itself is also undertaken.

The mechanical properties include basic measurement analysis, tensile testing and puncture resistance. These tests are conducted where appropriate on two versions of the multilayered material whose only difference is the thickness of the aluminium foil.

All results are analysed using a mixture of statistical as well as graphical techniques. Discussion on the results includes the statistics and leads to a number of conclusions on what effects seal strength, which blister produces the easier peel and where cost savings can be made with the current configuration. The conclusions also highlight further work that may lead to future cost savings by changing to different configurations.

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CHAPTER ONE: INTRODUCTION

The adages: "A little knowledge is a dangerous thing" and "Less is more," unfortunately when applied to the contemporary worlds of science and engineering it is the contrary that is true.

Against the backdrop of acronyms proclaiming the latest techniques that are sweeping the modern manufacturing processes lies the ancient hallmark of quality. Quality control is as old as industry itself. Integral to modern quality control is the art of problem solving.

What is problem solving and what makes for good problem solving? The forerunners in this field state that, "Any gap between the actual and the desired can be called a problem. Any effort to fill such a gap can be called problem solving. When people cannot find a way to solve a problem, it is usually because they lack sufficient understanding of the problem" (JUSE; Union of Japanese Scientists and Engineers, 1985)[1].

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[1] *TQC Solutions.*

It was in trying to fill such a gap that this author realised that a manufacturing/production process being in a stable state does not infer that there is a knowledge maintaining that status quo. It can mean that the overall knowledge of that process has not been examined or challenged, especially in the context of trying to resolve a problem hitherto unseen.

Within this context arose the thoughts for this thesis. There it was: a process that had been stable for years. Suddenly, as unknowns entered the equation there was unreliability. As the expert knowledge was examined and challenged it was found wanting in some quarters!

More knowledge and scientific data was needed to resolve the problem. As more data was obtained, either through research, analysis or through being provided by vendors, it became obvious how little was initially known. Thus it became necessary to do further research into a material which was so important to the company's core business. This key component, of multilayered structure, provides the basis for this research thesis.

The objective of this thesis is to perform three discrete sets of testing; seal testing, mechanical testing and barrier testing. Furthermore, to link the results of this research testing to actual requirements of the

material (and package) during it's manufacturing life cycle from packaging to distribution to shelf life.

A results review and discussion will assess the findings in order to determine whether current configurations and practices require change or what further work or study may be required, if any. These potential changes or further work are outside the scope of this thesis.

The initial problem that sparked this research was resolved using the techniques outlined in the book by JUSE referred to previously.

CHAPTER TWO: BACKGROUND

What is packaging? Not a question that can be easily answered in a few words. There are many descriptions and definitions that go along way towards answering the question. One thing is certain: if there is no product then there is no need for a pack.

Without packaging, the majority of products of any class or variety simply could not exist. Packaging plays a fundamental and critical role towards the health and welfare of people. It is an area that is irrefutably linked with the progression of civilisation. Given this link and given that civilisation is advancing so rapidly, nobody should be caught unawares at the rate at which packaging is changing. Yet few appreciate its importance.

While perhaps slightly idiomatic, Thomas Hine remarks in his foreword "Packages understand people much better than people understand packages. [Packaging's ability]… to bypass the intellect and induce a consuming forgetfulness is what makes it so effective. Although packaging pervades daily life and is found in every nook and cranny of the home and workplace, it flies beneath nearly everyone's analytical radar. It only comes to the fore when there's a problem. People think about packaging when they have

trouble getting it open, or when it's empty and it contributes to litter or overflowing landfills. But when packaging is working well, people rarely think about it apart from the product it contains". [2] This is a simple, modernistic yet incisive view of packaging but a view that can be related quite easily to the way packaging has been viewed down through the ages.

ANCIENT PACKAGING

Speculation abounds as to what the first package may have been. Answers more likely will come from anthropologists and archaeologists than from packaging engineers. Hardship was the watchword for the lifestyle of early man as he eked out his existence in nomadic fashion. Transportation and containment devices were the requirements of his time. Devices such as empty shells or animal skins and bladders, a wrap of leaves or hollow pieces of wood could have fitted into this category of the first package.

Evolvement from the wandering and predatory lifestyle brought about the development of communities and dwellings, around which animals were reared and plants grown, circa

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[2] Hine, Thomas, "*The Total Package: The Secret History and Hidden Meanings of Boxes, Bottles, Cans, and Other Persuasive Containers*"
Back Bay Books Little Brown & Company Limited © 1995

5000 B.C. Again it was transportation and containment devices that were required but these were more of the nature of fabricated sacks and bags for the likes of milk, honey and seed grain.

Pottery was in it's infancy, born by accident as someone trying to hasten the natural drying process of shaped river clay containers probably placed such a type of container or bowl into a fire. The result was a bowl that did not soften or revert back to its former state when filled with a liquid.

A well-known container from around 1800 B.C. was the Canaanite jar.[3] With a capacity of approximately 60 pints (30 litres) it had a rounded bottom and two small handles at the neck. When securely stoppered it could be piled on its side several layers deep. (As an aside, this container may also have been one of the first packages to have been a member of systematic and official recycling/reuse programmes. In some cases the neck was broken off and the empty jar used for the burial of babies. In other cases, the leaders of cities and villages in Egypt were responsible for collection of empty jars, refilling with water and placing them along the desert approaches to their country.)

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[3] Ibid.

By 1500 B.C. hollow glass objects had started to appear in Egypt and Iraq, then called Mesopotamia. These Egyptian glass objects were core formed (Figure 2.1). Hot glass was wrapped around a core. The cores were hand shaped from either clay or dung. Basic patterns like wavy lines or smooth areas could be introduced by working the soft hot glass. The core was removed from the container after the glass had cooled.

Metal can also be added to the above in order to complete the listing of packaging materials available many centuries ago.

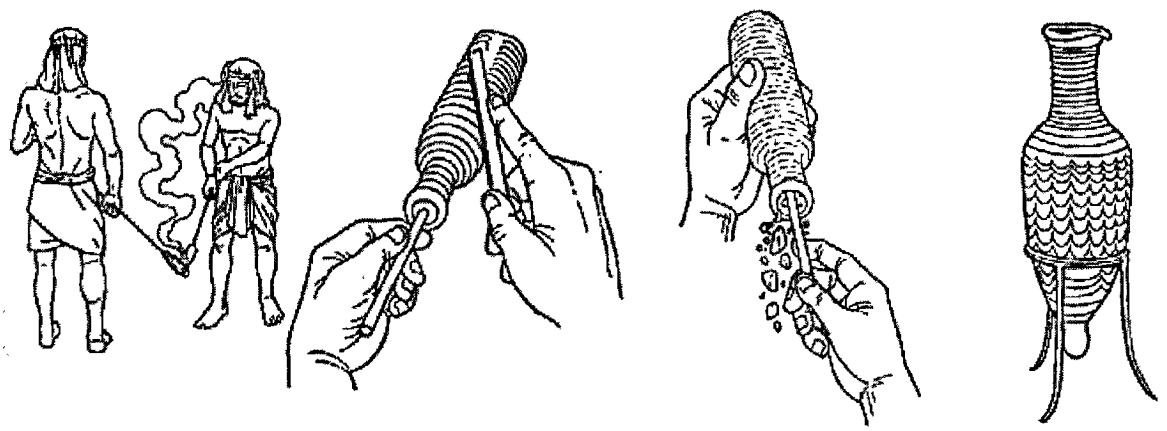

Figure 2.1. Forming a hollow glass container about a core.[4]

PACKAGING IN ROMAN TIMES

Glass, as mentioned above, is one of the oldest substances known to man. Arrowheads dating from the Bronze

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[4] Copeland, P. & Martin, H., *Story of Glass* Dover Publications, New York 1981.

Age were made from a glass-like material of volcanic basis.

Pliny, a Roman official, cavalry officer, advisor, and author of the 37 volume Natural History encyclopaedia, which was written to "set forth in detail all the contents of the entire world"[5] detailed sailors who discovered that blocks of salt from their cargo used to make fireplaces fused with the sand to form glass.

The discovery of the blowpipe in the first century B.C. occurred in Sidon, in Phoenicia (today's Lebanon). The blowpipe was a hollow rod used to inflate a gob of molten glass into a variety of hollow shapes and sizes. This invention brought glass out of homes of the nobility and by the third century of the Christian era articles of glass were in common use in most Roman households.

Barrels also originated at this time, more than two millennia ago. It is not clear who made the first barrel but it was preordained to command respect as one of the superior yet most common packaging forms for many centuries.

PACKAGING IN THE DARK AGES

The so called Dark Ages that succeeded the fall of the Roman Empire was not a great time for packaging and was

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[5] Internet www.cse.nd.edu/~theo/glossary/pliny.the.elder.html

devoid of any significant advances within Western Europe.

Any progress made in the intervening years was accredited to the civilisations of the Far East. The earliest records indicate that the first true paper was invented by T'sai Lun[6] at Lei-Lang, China in the year A.D. 105. The Muslims sacked Samarkand and captured a paper mill. The Muslims brought papermaking to Spain around 950.

The primary constituent of T'sai Lun's paper was the inner bark of mulberry trees. Egyptians had been making something similar by weaving together the split stalks of papyrus reeds, which eventually led to the name "paper" being used to describe the Chinese invention.

ADVENT OF PRE-PACKAGING AND BRANDING

In the 15th century the great age of exploration was heralded and from a packaging viewpoint it saw the commencement of book publishing. The first throw-away style packaging appeared around 1550, when German papermakers used inferior paper labels, printed with various designs to wrap their better paper products.

Goods were still being delivered in bulk and individual portions would be measured or weighed out by the shopkeeper. Medicines, cosmetics and teas were amongst the

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[6] Internet www.mediahistory.com/time

first products to be pre-packaged. By the early 1700's printed paper labels began to appear on glass phials for drugs and on wine bottles. Early medicinal containers were handblown glass bottles or phials that were wrapped in labels made from handmade paper, manufactured sheet by sheet and printed on a handpress. The designs were often no more than extensions of the manufacturers trade card and included such names as Singleton's eye ointment and Daffy's Elixir. It would be more than a century later before the printing technology had developed sufficiently to allow 'mass production' of such decorated labels and wrappings.

Antiquated as the above may seem, the labelling and marking of goods has a much longer history. Roman apothecaries were known to have dispensed drugs in small jars bearing the name of the drug and the seller. The aforementioned Canaanite jars were regularly stamped with inscriptions that detailed the date of the wine, the type of grape and where it was grown. Wines were sold in bottles with labels hanging loose from the neck, secured only by a fine chain.

As mentioned previously, goods were still being delivered in bulk and were generic in nature. This led to unscrupulous suppliers and shopkeepers to bulk out the products with other cheaper substances, or 'shortweigh'

quantities, in order to make larger profits. This practice was common, and when Horniman's began to sell teas in sealed fixed priced packets, grocers often refused to stock it and it had to be sold through chemist shops, who were more scrupulous. In the latter half of the 19th century, pre-packaging became more common, and was welcomed by shopkeepers as it made the manufacturer rather than the retailer responsible for the quality, quantity and hygiene of the product. It also reduced the time it took to serve customers weighing out and wrapping loose goods.[7]

To differentiate those generic and loose packed goods, some companies took to making identifying marks on their products. These marks were made with a blackening brush or with a hot branding iron. With time these marks became associated with certain goods and with high quality. With the progression in usage of individual packaging these companies then wanted their product to be identified with this high quality. The brand mark was derived from the bulk pack, e.g. a barrel, and copied and imitated onto unit packs or labels. This was an early form of product branding as well as the origin of the term "brand name".

The promotional value of a label or brand was not recognised until much later, therefore, early brand names

[7] Internet //www.londonfancybox.co.uk/education/d.history.html

were simply those of the maker; Yardley's (1770), Schweppes (1792), Perrier (1863) and Colgate (1873). Although about 1793 Guinness started using the Irish harp as a symbol on it's stout to help sales.

During the early 19th century the tin can was developed when the offer of a reward was made by Napoleon to anyone able to develop a method of preserving food. The money was claimed by a French chef. A year later the "tin canister" was invented by an Englishman. As people emigrated from England to America they brought the technology and beginnings of the American canning industry.

ARRIVAL OF THE CARTON

Louis Pasteur once said that, "In the fields of observation, chance favours only the prepared mind". Credit must be given to the Robert Gair Company of New York who were in such a prepared state back in the 1870's.

Up until then paper boxes had been handmade and required a great amount of labour for cutting and glueing. This precluded their use for anything other than luxury items. Inaccurate adjustment of a printing press, a mistake by the operator, caused the printing plate to cut though the paper instead of printing it. This led to the technique now used to cut and crease paperboard. Robert Gair's

contribution was combining both operations, previously done separately and by hand, onto the one machine.

The first major use of this new style box was to package biscuits just before the turn of the century. Within the next 25 years there were over 200 further manufacturers of folding cartons within America. It still remains one of the most popular types of rigid package.

PLASTIC PACKAGING

Who would think that the history of plastic packaging has its roots in the game of billiards, but that is exactly where the genesis of the material that changed the face of packaging began.

In the 1840's a white to brown latex sap similar to rubber was the material behind the formation of the Gutta Percha Company. The company used gutta percha for the manufacture of billiard balls.

Again, in the annals of history it is shown that the offer of a reward concentrates the mind enough to spring forth a technological advance. In 1863 a young printer from Starkey in the United States read a Phelan and Collander poster in Albany, New York, announcing a prize of $10,000 for anyone capable of producing a new material which could replace the ivory used for billiard balls, which was

becoming scarcer. John Wesley Hyatt committed himself to the search for "artificial ivory" or any new material that could meet the industrial demands. In 1869 he was successful with a compound that had a base of cellulose nitrate. Thus, celluloid was discovered. Its name coined by John's brother who worked with him on many experiments, and was patented on 12th July 1870.[8]

In the first decade of the 20th century Dr. Leo Hendrick Baekeland discovered phenol formaldehyde plastic, later known as Bakelite. This was the first synthetic resin. Bakelite's major packaging application was for closures.

For the packaging industry, the real breakthrough came with the invention of polyethylene. This took place in England just prior to the Second World War. An initial packaging use was as a wrapping. Early post-war uses saw the material being manufactured into squeezeable bottles. In the 1950's high density polyethylene was blow moulded into thin walled bottles. From these modest beginnings originated a proliferation of materials and manufacturing techniques and ultimately an endless array of shapes, forms and package types.

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[8] Internet //qlink.queensu.ca/~bsvs1/chem210/Page2.html & //npcm.plastics.com/hyatt.html

PRESENT TIMES

Nowadays packaging is seen to have no clear-cut boundaries. It has encroached into the realms of other subjects, sparking debates on its role in areas such as consumerism, marketing, advertising, environmental issues, health and safety.

It is not the intention of this author to delve into these topics. Nor should this list be taken as definitive or complete. Rather the intention is to highlight to the reader the extent to which "packaging pervades daily life and is found in every nook and cranny" and is now such an integral part of global issues and lifestyles.

The reiteration above of the quote by Thomas Hine brings us nicely back to the point that brought us on this chronological and retrospective journey. Yet, progress of society and packaging in a global context has been so rapid, and thus far so diverging, that extremes can be readily seen. At one extreme, it is easy to conceive that presently somewhere in the world humans are providing their own packaging or consuming on the spot just as they did in the initial insight on ancient packaging. At the other extreme, the Technical Association of the Pulp and Paper Industry (TAPPI) has just announced it will host "an interactive exhibit to showcase the paper industry's use of

science and technology at the 'Innoventions' attraction at Epcot® in The Walt Disney World® Resort."[9]

[9] Anonymous 05/1999 "Packaging World" web page. www.packworld.com/

CHAPTER THREE: MULTILAYERED STRUCTURES

BACKGROUND

In the previous chapter a general background was given, broadly detailing the history of general packaging. Any details on multilayered structures were deliberately omitted so that they could be included in their own right separately. This is done to emphasise the importance within the context of this thesis and not to emphasise or indicate any importance over any other type of packaging.

There is quite a listing of current manufacturing techniques for creating multilayered structures and a considerably longer listing of different structures made by these techniques. Broadly speaking the multilayered structures can be categorised into two:

1. When two or more discrete monofilms/layers are combined by means of heat and/or adhesive between the layers, then this structure is called a laminate.
2. When a multilayered material is manufactured by adhering two or more individual film layers within the body of an extrusion die, then this structure is called a coextrusion.

No single material can possess all of the desired properties or provide all of the desired requirements for

all products or applications. Hence the need to join together in order to combine the best of all properties. A multilayered structure can also be a combination of the above two general structures. As said, there is quite a listing of manufacturing techniques for creating multilayered structures and as is the case with the structures, manufacturing techniques can be given two general classifications, see Table 3.1.

Table 3.1.

COATING	**LAMINATING**
Roll coaters	Wet bonding
Knife, blade & bar coaters	Dry bonding
Slot orifice coaters	Hot melt bonding
Extrusion coaters	Extrusion / Coextrusion

For a more detailed explanation of any or each of the above techniques the reader should consult the general texts listed in the bibliography at the end of the Appendices.

For the purposes of illustration, a simple diagram showing the difference between extrusion coating and extrusion laminating is shown in Figure 3.1. As the name suggests a coating is the covering laid on a surface at one time. Laminating is the joining together of two or more

material layers. See point 1 above for the definition of a laminate structure.

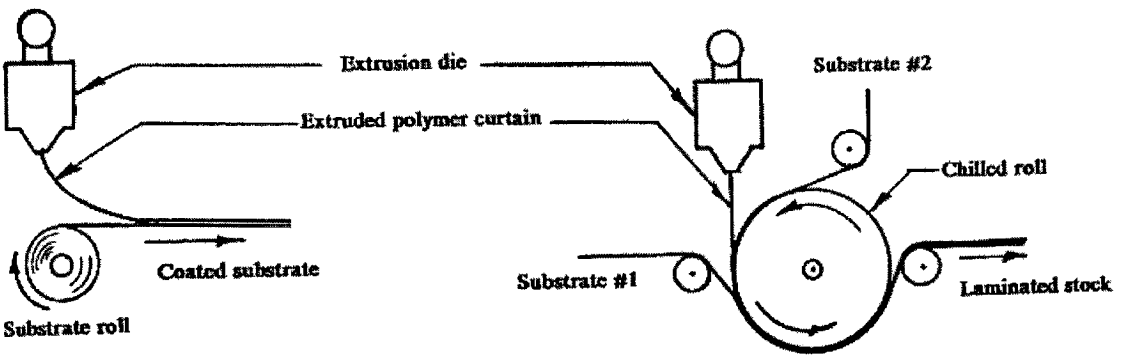

Figure 3.1. Extrusion coating (left) & laminating (right).[10]

The history of laminating and coatings is relatively short. Coatings were first on the scene when Kellogg's® Corn Flakes worked with wax as far back as 1912.

Extrusion coating, particularly with low-density polyethylene (LDPE), developed strongly in the immediate years succeeding the Second World War. Yet it was not until 1954 when LDPE was first applied to aluminium foil did the industry herald the arrival of flexible, high barrier packages.

Lamination has been in commercial use for more than four decades now, but it has only been in the last two that the vast assortment of combinations has been available.

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[10] Emblem, A. & Emblem, H., *Fundamentals of Packaging Technology* p.219
Revised UK edition © 1996 The Institute of Packaging

LIDDING

As mentioned, the list of multilayered structure formats is vast. This thesis is concerned only with those whose application is lidding and in particular the structure in use within our company today. Lidding is a very specialised aspect of packaging and, in general, lids are rarely composed of just one layer.

Most of the advances involving today's lidding were developed for the dairy industry. Initially aluminium foil lids were developed in the 1940's to replace waxed paper lids. For some fifty years foil lidding has brought added hygiene to milk packaging.

When cream and yoghurt were marketed, either in the early wax coated paper cup or in the later plastic cups, the initial lidding response was to imitate the milk bottle lids. However, as these cups were less rigid than the glass milk bottles this was not a dependable lidding solution. The introduction of the heat sealed foil lids, using heat seal lacquers as a coating, not only counteracted this problem but added extra rigidity to the container as well.

During the 1970's machine constructors and packaging producers developed the concept of form/fill/seal (FFS) machines for the dairy industry. With the advent of single serve, portion packaging and dispensing packages came the

requirement for a wider range of improved lidding materials.

Development was directed towards the heat seal lacquer formulations to give improved peelability combined with the security of a tight, reliable seal. In the 1980's coextrusions were being developed to progressively replace the lacquers, especially on the more aggressive products, e.g. juice and sauces. Not only did these improve the seal efficacy during pasteurisation and sterilisation but also brought an added dimension to convenience in the peeling operation.

Manufacturers and converters of these types of lidding materials did not solely concentrate on the sealing side. Development work carried out on the aluminium foil has steadily reduced thicknesses - presently metallised polymers are now being used as direct replacements to foil in some modern applications. Greater sophistication in alloy selection and conditioning has given better results in secondary activities such as printing, embossing and die cutting.

Advances are being continuously made on the printing and decorating side. High quality reproduction and multi-

colours are only two of the features that now come as standard within this industry.[11]

Sociological changes brought a new demand into alternatives. With microwaves more prevalent and single person households more widespread the call for packages that can be reheated for serving convenience brought forth development into non-foil options that achieved similar shelf life as those that contained aluminium.

All of these applications can be readily seen in most supermarkets and similar retail outlets. Heat seal lidding is now in widespread use in the packaging of chemicals, personal care products, food products, pharmaceutical and medical devices.

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[11] Aluminium Rolled Products Manufacturers Association
Design & Packaging Data Foil Files: No. 3, No. 10 & No. 12.

CHAPTER FOUR: THESIS MATERIAL STRUCTURE

MATERIAL AND PROCESSING OVERVIEW

There are a number of different lidding materials used currently within our company. Different vendors, situated in Europe and America, supply these materials. For clarification, the material is not one specific product that is supplied by various vendors as equivalent product. Rather, strictly speaking, the materials are different products that carry out the same function. For the purposes of this thesis the research and experiments have concentrated on just one specific lidding material.

At a macro level the company receives the material in roll form and processes this into two shapes or die outlines for its lidding. The die outlines form the same silhouette but differ in size, the larger being greater than 30% and 13% longer and wider respectively.

The materials themselves can be broadly broken down into four layers:

1. Top Coating
2. Aluminium Foil (Barrier Layer),
3. Tie Layer (Bonds layers on either side)
4. Sealant Layer.

This information is summarised in the following diagrams, see Figures 4.1. through 4.3.

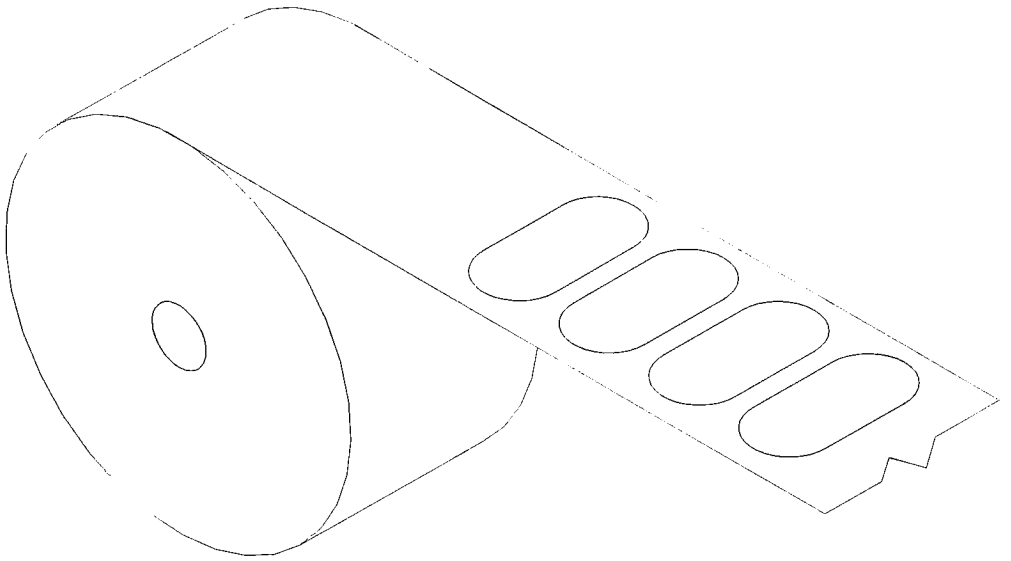
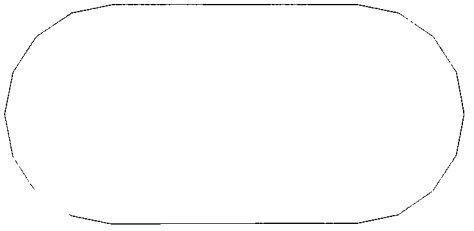


Figure 4.1. Material in roll form showing removed die cuts.

Die Outline - Top View



Small lid = 50mm * 29mm

Large lid = 67mm * 33mm

Figure 4.2. Die silhouette with approximate dimensions.

Material Cross-section
(Lines denote interfaces between layers)

Top Coating
Aluminium Foil Layer
Tie Layer
Sealant Layer

Figure 4.3. Block diagram of material layers (side profile)

The lids are subsequently sealed, to what are described internally as blister tubs. To state the obvious, the package contains the product at this point. Logically, there are two types of blister tubs to equate to the two different die outlines of lids, see Figures 4.4. and 4.5.

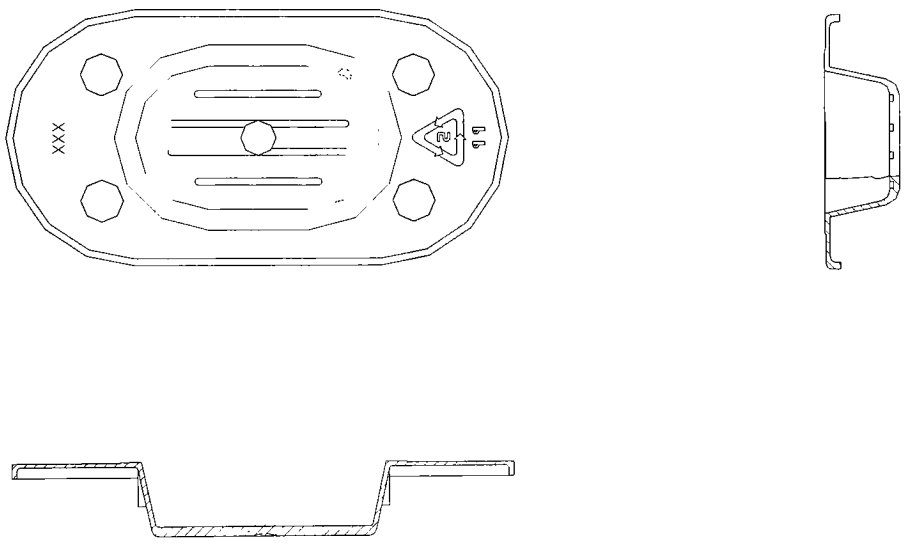


Figure 4.4. Large blister tub

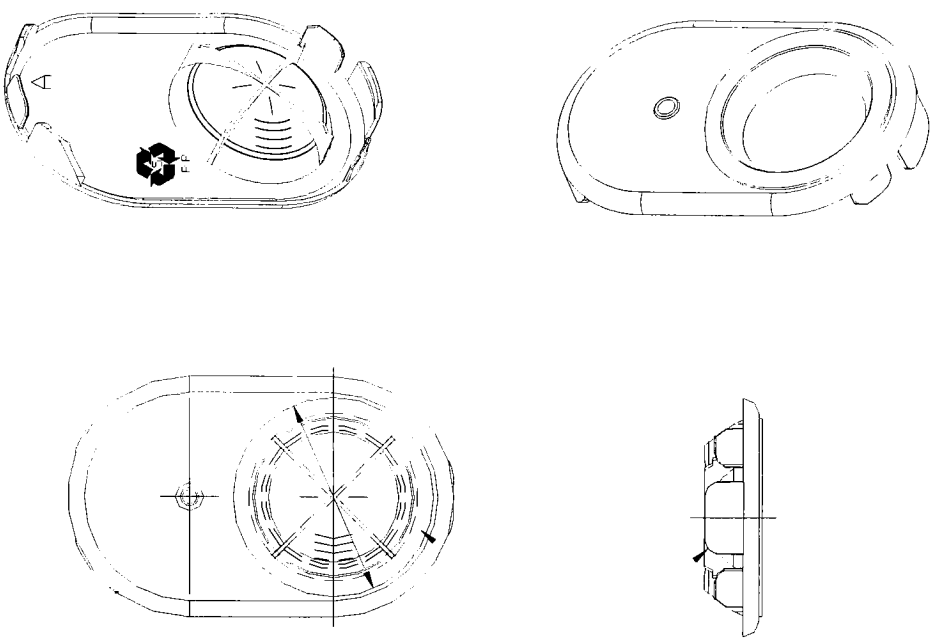


Figure 4.5. Smaller blister tub

The method of sealing the lidding material to the blister tubs is heat sealing utilising and controlling the three principle factors of temperature, time and pressure.

These three validated parameters are controlled within specific limits during production. Should the manufacturing process, either due to natural variation or known/unknown causes, deviate outside these limits then the process will automatically reject that package. Heat sealing parameters used in this thesis may be categorised using the following descriptions:

- HIGH when all three parameters are set to the upper allowable tolerance setting,
- NOMINAL (or NOM) when all three parameters are set to their nominal tolerance setting,
- LOW when all three are set to the lowest allowable setting and
- HIGH(+25%) or H(+25%) when all three have been set to a point that is 25% of the tolerance band, (i.e. {HIGH - LOW}*0.25), above the HIGH setting.

Due to the nature of our product, which is classified as a medical device, the product must arrive for use to the consumer in a sterile condition. Sterilisation as a manufacturing process step exerts forces, both internal and external, on the sealed package.

As package efficacy is paramount in the medical device industry there are inherent checks throughout the manufacturing process, one of which is a vacuum leak test. By its very nature this is a "stress" test on package seal quality and efficacy.

Both of these, sterilisation and vacuum leak testing, will be considered as experimental inputs within the context of testing during this thesis.

CHAPTER FIVE: PURPOSE, TESTING & SIGNIFICANT EFFECTS

PURPOSE

The reason behind this topic, as outlined in the introduction, was a desire to learn more about the material being used within our company. The purpose of the thesis itself is to link any new knowledge found to the material functioning in real-life situations such as package and processing performance.

A detailed test plan was drafted to cover the areas of package and processing performance, see Appendix A. Although only dealing with one material structure format, the testing does include comparisons between both of the die shapes mentioned previously. There are also comparisons between two samples of the same material that have, as their only difference, variation in the thickness of the aluminium foil.

BURST AND PEEL TESTING

The scope of the first battery of tests includes not only the short-term perspective of machine performance but also the potential for long term stability studies, including accelerated ageing and/or shelf life studies. A

review of burst and peel tests is given below. The longer duration studies are outside the scope of this thesis.

Burst and peel tests are carried out on packages that have been sealed at LOW, NOMINAL and HIGH plus HIGH(+25%). These tests are carried out after the packages have been removed from the manufacturing process at various distinct stages, e.g. before and after sterilisation. The test plan was designed to indicate any significant effects, if any, which the various stages of the manufacturing process might have on seal strength.

NOTE: Although covered in the test plan, two sterilisation cycles do not form part of the normal manufacturing process.

As a consequence of variations during the current manufacturing sealing process, packages may exhibit, on opening, adhesive or cohesive failure, see Figure 5.1.

Adhesive failure is where, on peeling, fracture occurs between the aluminium foil and the tie layer. Thus, tie layer and sealant layer remain adhered to the blister tub after opening. Cohesive failure is where, on peeling, fracture occurs between tie layer and sealant layer. Thus, the sealant layer remains adhered to the blister tub and the tie layer remains adhered to the foil layer. The material is designed to produce cohesive failure.

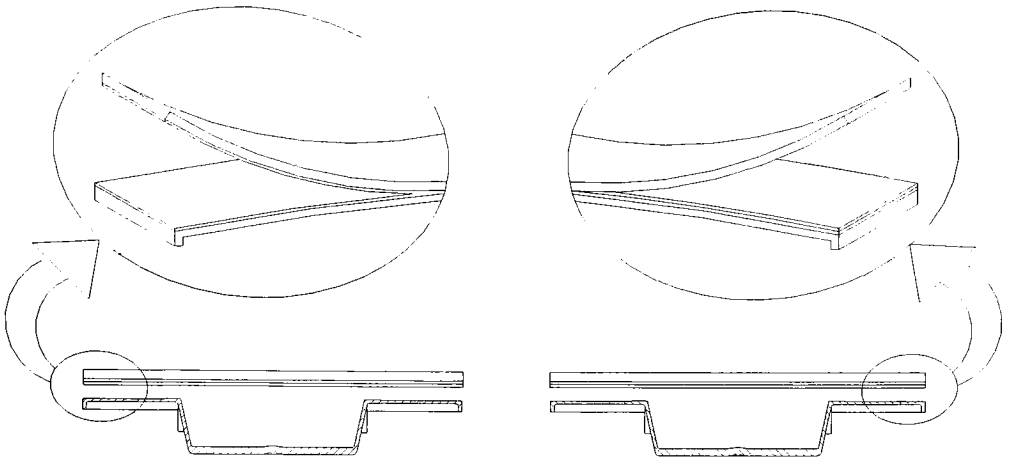


Figure 5.1. Cohesive failure (left inset) & adhesive failure (right inset).

The methodology for completing a burst test is to puncture a hole in the lidding and insert two needles into the package through which the pressurised air will flow and return for sensing. The package is held firm, the air is turned on and the pressure at which the seal bursts is noted. The tested package is removed and the procedure is repeated for the next one. A detailed description of the procedure is given in Appendix B.

The methodology for completing a peel test is summarised as follows. The multilayered material is clamped between two jaws while the base of the blister tub is secured separately. When the test rig is activated the jaws holding the material move at a constant rate in the vertical direction while the blister tub remains fixed in

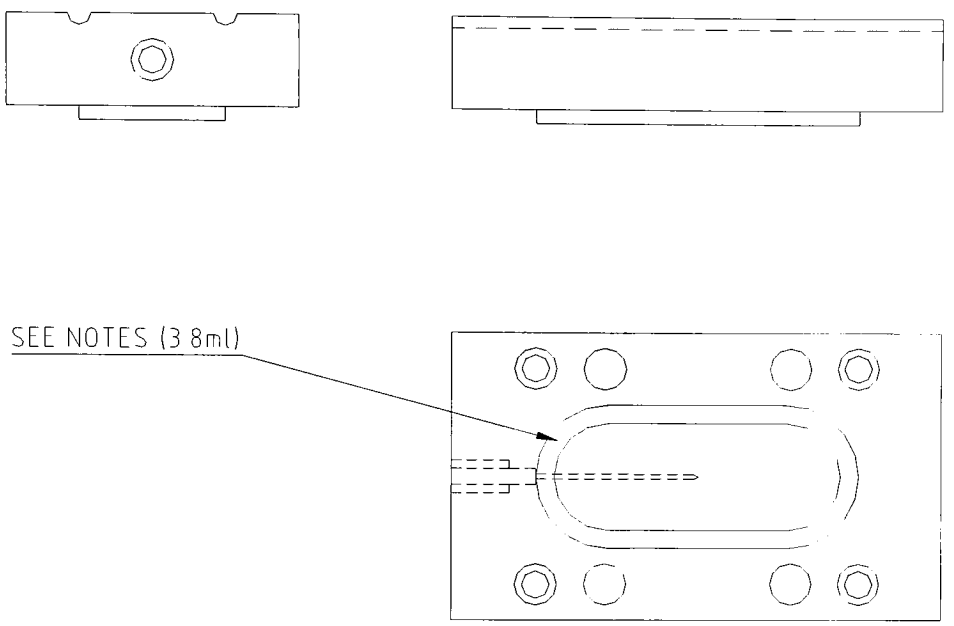
this direction but can move horizontally to maintain the peel angle. Thus, a peel is effected. The forces required to peel open the package are acquired automatically. This procedure is detailed in Appendix B.

MINIMUM SEAL WIDTH

Included in the "Peel and Burst" section of tests are attempts to reduce the seal width of the package to a minimum. There is a real significant potential for manufacturing depending on the outcome of this section. A positive outcome would lead to sample packages of a predetermined seal width being manufactured for stability study. See Figure 5.2 for diagrams of the tooling used to generate the different seal widths for both tub sizes. **NOTE**: Incomplete seal widths can occur in a sealing process due to a variety of reasons. Some examples of these are natural processing variation, poor quality components, dirt or debris on heat seal head and any loose, foreign matter getting trapped between the two surfaces being sealed.

Burst and peel tests on minimum seal width packages removed at the various stages of manufacturing are repeated for both blister sizes. This gives a direct opportunity to compare one versus the other and to see if one offers any

advantages over the other that may otherwise have gone unnoticed.



Notes:QTY 2 OFF. 1 @ 2mm SEAL WIDTH ; 1 @ 1mm SEAL WIDTH

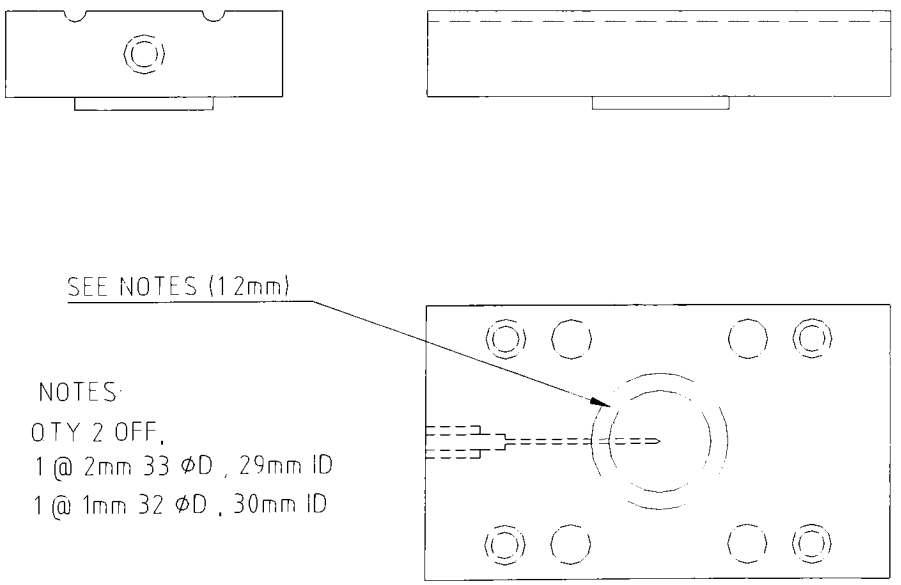


Figure 5.2. Diagrams of tooling that was used to generate the different seal widths for both blister sizes.

BARRIER TESTING

The thickness of aluminium foil used in the multilayered material under research gives complete barrier protection for oxygen and water. The barrier properties being investigated as part of this thesis are those of the blister tubs. The blister tubs have never been tested as a unique entity but rather as part of the overall package. The results of these tests for the thesis could have a potential impact on expiry dating.

The two tests being conducted are to calculate the oxygen and water vapour transmission rates (OTR & WVTR). Transmission rates for the above are independent of pressure, however, temperature and relative humidity can be important factors. The tests will be run at conditions close to actual shelf conditions.

Both transmission rates have, in the main, a direct relationship to thickness. The rates decline almost proportionately as thickness increases; i.e. twice the thickness gives about half the transmission rate. The following formula[12] can be used to get a close evaluation of either OTR or WVTR for multilayered materials for initial purposes if no testing equipment or service is available.

TRANSMISSION RATE =

$$\frac{1}{\frac{\text{Layer 1 (mils)}}{\text{Layer 1 TR}} + \frac{\text{Layer 2 (mils)}}{\text{Layer 2 TR}}}$$

where TR is the transmission rate value for a particular material at a thickness of 1 mil (.001 inches = 1 mil).

TENSILE AND PUNCTURE TESTING

The results obtained from this part of the research will have a bearing on the package throughout various stages of its lifecycle from packaging right through to distribution.

At the packaging machines tensile strength is an important characteristic as the lidding material is "pulled" through the die cutting process. Use of these results will allow manufacturing and process engineering to optimise, i.e. minimise, the amount of material remaining after die cutting. See Figure 5.3 for a diagrammatic explanation. Not only is this important from a cost perspective but it also has significance from an environmental standpoint; less waste material to deal with.

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[12] Hanlon.J., Kelsey.R., Forcinio.H., *HANDBOOK OF PACKAGE ENGINEERING* 3rd Edition p.106

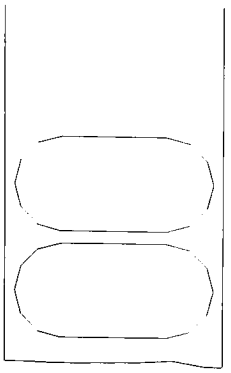

Figure 5.3. Material remaining after die cutting (skeleton)

The comparison between puncture testing of different gauge foil should give an indication as to how these lidding materials would actually perform at two separate stages of their life cycles. Initially, how they would process through the die cutting operation and latterly how either would stand up to the rigours of a distribution network.

The methodology for conducting these tests will be detailed in Appendix B. Standard tensile and puncture testing equipment was used - the lidding was cut to a particular size for the tensile test, whilst the puncture test was carried out on the material when it was still in its normal reel width format. For tensile testing the die cuts are held between a set of upper and lower jaws. The jaws then move in opposite directions at a fixed speed until the die cut is, quite literally, pulled apart. For

puncture testing the material is held between jaws and an arm descends in a pendulum like motion, piercing the sample. The resistance force offered by the material leads to the calculation of the puncture strength.

Finally, included under the heading of mechanical testing were some straightforward dimensional measurements of ten samples cut from the same die. This is significant in terms of machine variation and lid placement for the sealing operation. The dimensions were also used to calculate the cross sectional area for the different thickness of the two foils.

CHAPTER SIX: RESULTS

RESULTS SUMMARY

Once all of the tests were completed the raw data was formatted and documented. These are contained either in this chapter or in Appendices C, D and E. Results were obtained for all of the major headings in the test plan. Unfortunately, due to constraints that are outlined below, not all of the desired tests were completed. However, in one case a substitute test was executed and the results documented.

The raw data itself is not contained in this thesis as some test outputs were too bulky. For example, the quantity of peel graphs generated outnumbers the quantity of pages in this document itself. Instead the raw facts and figures were tabulated and documented.

Where possible statistical analysis was carried out on test results. Tests included Analysis of Variance (ANOVA), t-tests, correlation & Normal probability plots. For all analysis, a significance level of $\alpha = .05$ was used. Result analysis and discussion is contained in the next chapter. Summary tables are included in this chapter as well as complete tables where the actual number of results is small.

BURST AND PEEL TESTING

There was difficulty encountered in sourcing a burst tester in my country. Although carried out as a regular check some years ago within our company it was since superseded and the equipment disposed of. On contacting a number of pharmaceutical and medical device companies only one company was located with burst equipment. A testing and research facility outside the country was also located but as they had to be financially reimbursed for all tests, budgetary commitments could not cater for the quantity of burst tests required.

Concerning the actual testing itself, a "TEST-A-Pack" seal strength testing control system was used as detailed in Appendix B. The testing capabilities of this equipment reached a maximum at 52 PSI. A quantity of blister packages did not burst before reaching this maximum. This was normally consistent through out an experimental run, i.e. for the complete run of LOW sealing conditions blisters did not burst whether they had been subjected, or not, to vacuum leak or sterilisation. The same applied to the runs at NOMINAL, HIGH and HIGH(+25%).

Bursting only occurred for the runs of 1.00mm seal width and 2.00mm seal width. For the 2.00mm seal width run, two packages **d**id **n**ot **b**urst for the *Before Sterilisation*

condition. This is indicated in the results by the inclusion of **DNB** where the numbers should be. Appendix C contains all burst test results and analysis. An ANOVA comparing the results from the different processing conditions was conducted to see if these extra manufacturing and testing steps had any effect, Table 6.1. This was replicated for both the 1mm and 2mm seal width experimental runs.

Table 6.1. Burst test results summary

ANOVA of Burst Test Results	***Significant Difference***
1mm Seal Width - All Processing Conditions	NO
2mm Seal Width - All Processing Conditions	YES

The t-test was used to try and establish whether the different seal widths caused any significant effects on burst strength. The results are contained in Table 6.2 below.

Table 6.2. Burst test results, 1mm versus 2mm seal widths

t-test Comparison of 1mm versus 2mm Seal Width Results	***Significant Difference***
Before Sterilisation	YES
After Sterilisation	YES
After Vac. Leak & Sterilisation	YES
After 2 Sterilisations	YES

Further difficulties were encountered when attempting to burst test the smaller blister package. Due to the relative size of the blister well versus the needles, the test could not be accurately conducted. This is best illustrated in Figure 6.1. What occurred when attempting to conduct the test was that the area of lidding surrounding the hole produced by the needles enlarged under low pressure. This did not permit any further build up of pressure in the package, which may have forced seal failure as the air could easily escape through the holes.

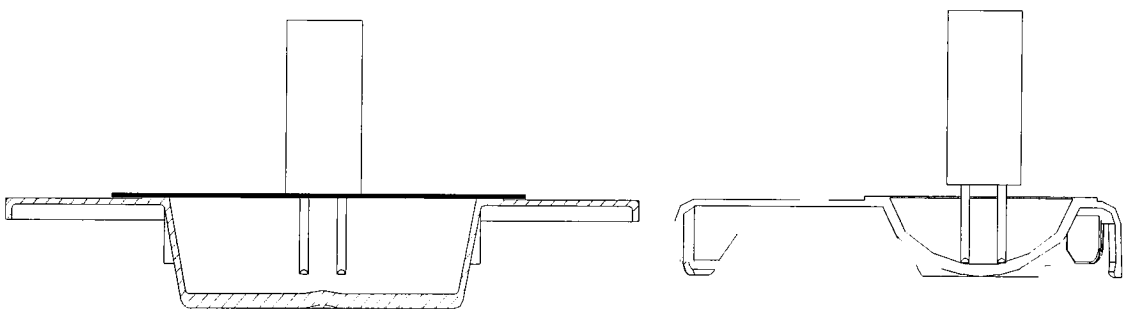

Figure 6.1. Illustration of burst testing both packages.

An extra test conducted using the burst tester, which was not originally planned, was to determine if the rate of fill of pressurised air made a difference to bursting pressures of the package. The results, by ANOVA, showed no statistical difference between four different fill rates. These results are contained in Appendix C also.

Peel testing provided the majority of the results and the opportunity for the greater amount of analysis. Tables below provide a summary. The complete results and analysis are contained in Appendix D. Table 6.3 indicates whether there was a statistical difference for either the large or small blister, for all sealing parameters and seal widths, covering the same processing condition.

Table 6.3. Peel test results summary

ANOVA of Peel Test Results	***Large Blister***	***Small Blister***
Before Sterilisation - All Sealing Parameters & Seal Widths	YES	YES
After Sterilisation - All Sealing Parameters & Seal Widths	YES	YES
After Vac. Leak & Sterilisation - All Sealing Parameters & Seal Widths	YES	YES
After 2 Sterilisations - All Sealing Parameters & Seal Widths	YES	YES

For Table 6.4 the ANOVA was conducted examining the results obtained within one set of sealing conditions but between the various processing conditions. The analysis was repeated for all of the sealing conditions and seal widths. As stated in the previous chapter, peel results would be used to make a comparison between both blister types. This comparison was made for each individual set of sealing parameters and included the results for that setting across the various processing conditions, see Table 6.5.

Table 6.4. Peel test results summary

ANOVA of Peel Test Results	***Large Blister***	***Small Blister***
LOW Sealing Parameters – All Processing Conditions	NO	YES
NOMINAL Sealing Parameters – All Processing Conditions	YES	YES
HIGH Sealing Parameters – All Processing Conditions	YES	YES
HIGH(+25%) Sealing Parameters – All Processing Conditions	NO	NO
1mm Seal Width – All Processing Conditions	YES	NO
2mm Seal Width – All Processing Conditions	YES	NO

Table 6.5. Peel test results, comparison between large and small blisters.

ANOVA of Peel Test Results	***Significant Difference***
LOW Sealing Parameters – All Processing Conditions	YES
NOMINAL Sealing Parameters – All Processing Conditions	YES
HIGH Sealing Parameters – All Processing Conditions	YES
HIGH(+25%) Sealing Parameters – All Processing Conditions	YES
1mm Seal Width – All Processing Conditions	NO
2mm Seal Width – All Processing Conditions	YES

BARRIER TESTING

This was a test recognised from the outset as needing very specialist equipment. This test is normally carried out on material sheets and such like. However, new ground

for the company was being covered by attempting to test both of the blister tubs in their injection moulded state.

The smaller blister caused difficulties and was beyond the scope and capabilities of the companies contacted. The tests were ultimately carried out at a testing facility outside the country on the larger blister only. All of the results are shown in Table 6.6 below.

Table 6.6. Barrier test results

	units	Sample 1	Sample 2	Sample 3
Oxygen Permeability	cc/pack.d	0.0943	0.0866	0.1053
Moisture Vapour Transmission Rate	g/pack.d	0.000139	0.000171	0.0000624

MECHANICAL TESTING

The tensile testing portion of this section proved to be straightforward. As regards the puncture resistance this proved to be a different matter. Even though this type of test is well recognised within the corrugate industry it is not readily used. The services of the external testing facility were required for this test.

Tensile testing was carried out in two directions on the die cuts: cross directional (side to side across the shortest distance) and longitudinal direction (from curve apex to curve apex across the longest distance). In the

overall context, i.e. looking at this from a roll viewpoint, cross directional of a die cut equates to the machine direction of rolls. The results, in order of descending tensile strength, are as follows:

1. 70μ foil cross directional
2. 60μ foil cross directional
3. 70μ foil longitudinal direction
4. 60μ foil longitudinal direction

The puncture results indicated, as expected, that 70μ foil had more resistance than 60μ foil.

Table 6.7. Puncture resistance summary results

Puncture Resistance Means	70μ = 50 N	60μ = 43 N

All of the results and analysis for mechanical testing are contained in Appendix E.

CHAPTER SEVEN: RESULTS ANALYSIS AND DISCUSSION

BURST AND PEEL TESTING

A number of statistical and graphical techniques were employed to analysis the results obtained from burst testing.

Using ANOVA the results indicated that the different processing conditions, e.g. sterilisation and vacuum leak testing, did not give a statistical significant difference at $\alpha = 0.05$ for 1mm seal width but gave a statistical significant difference at the same level for 2mm seal width.

The 1mm seal width product was visually inspected to see if there had been any weak points in the seal area but burst locations did vary. Therefore, it is reasonable to assume that the results represented the actual burst strength of the packages and that for this small sample processing conditions did not lessen the burst strength by a statistically significant amount.

The 2mm seal width results illustrated that the two processing conditions *sterilisation* and *vacuum leak testing & sterilisation* lowered the burst strength of the seal. Paradoxically, the results indicated that the processing condition of *2 sterilisations* gave a higher burst strength

over that for 1 cycle and that for vacuum leak testing. This increase did not reach the original level achieved for the results obtained before sterilisation. When checked, the six inter-relationships only showed up two that did NOT have a statistically significant difference. These were between:

1. *After Sterilisation* and *After Vac. Leak & Sterilisation*
2. *After Vac. Leak & Sterilisation* and *After 2 Sterilisations*.

The method used to discriminate amongst the six means was Fisher's least significant difference (LSD) procedure.

Summing up the results, it is felt that no conclusions can be drawn and further testing and analysis would be required to determine if the additional processing conditions cause any lessening of the package burst strength. Partly, some of this is inconclusive due to the large variance of the small sample sizes and any further work would need to use much larger sample sizes.

When comparing the 1mm versus the 2mm seal widths the results gave statistical significant differences for all processing conditions using the t-test. In all cases the results matched intuitive expectations with the 1mm seal width results being lower than their 2mm counterparts. It was noticed and is illustrated by the graphs in the results

section that there was a greater range in the results for the 1mm seal widths. This may be explained by the fact that greater difficulty was encountered when setting up the machine for this test. Alignment of the heat seal head was very complicated and time consuming. Also, with a 1mm seal width, any defects will have a greater percentage impact at this level. The greater range may be an indication that the mode of seal failure is alternating between cohesive and adhesive and is therefore not consistent within a tighter range.

To prove any of the above conclusively more tests would be required. The indications are, however, that 1mm seal widths would be too narrow for finished product specifications on the larger blister.

As stated previously an extra test was carried out at burst testing to see if fill rates have an impact on burst strength. They did not with the piece of equipment used. This is very useful information as the company intends to purchase a new burst tester and this test can be replicated as part of its validation procedure.

Peel test results provided the largest amount of data for analysis. ANOVA was conducted to see if there were any effects of processing conditions on results as well as to see if different seal parameters caused any effects.

The analysis indicated a lot of similarity in trends between the peel test and burst test. The results showed that different seal parameters gave rise to a statistical significant difference, for each of the four processing conditions, using ANOVA. This was the same whether it was the large or small blister. In addition, again as with the burst test results, the different processing conditions gave mixed results. Some showed a statistical significant difference while others did not. Once again it is inconclusive whether the processing conditions cause a lessening of the seal strength.

Theorising, one explanation could be that the controlled overpressure within the steam sterilisation chamber is, on account of the package's location, counteracting the internal pressures. These internal pressures are due to the high temperatures of sterilisation. Possibly, that is why the sterilisation cycle is not having an effect in some cases and is in others. This hypothesis would need to be verified and if proven then seal strength tests could be used as part of a validation for any sterilisation cycle profile changes.

Visually, from the peel graphs, there is a noticeable difference in the profiles for the two types of tubs. Examples of such profiles are contained in Appendix F. The

larger blister had the more expected theoretical profile - rising to an initial peak and staying at a plateau before dropping off at the end. The smaller blister produced a type of inverted parabolic curve.

When comparing the physical results themselves, i.e. large versus small blister peel results, for each set of seal parameters there was a statistically significant difference using the t-test for all of the data bar the 1mm seal width data. Given that, currently, both tubs have the same width of seal area this is certainly important information for any future blister design. Once package efficacy was proven by burst and peel tests and by stability over time then, from this work, a circular seal design would be preferential from an engineering standpoint as it gives an easier opening. A marketing viewpoint and customer feedback would need to be solicited before change.

No attempt was made to draw any correlation between the burst and peel test results obtained. Many more samples would need to have been tested in order to derive an empirical relationship. A simple correlation conversion, using easily measurable physical dimensions and basic mathematics is not possible.[13]

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[13] Wachala, Thomas P. *Correlating Tensile and Burst Tests in Pouches* Medical Device & Diagnostic Industry, February 1991.

BARRIER TESTING

Both moisture vapour transmission rate (MVTR) as well as oxygen permeability were measured for the large blister. When the actual MVTR result was analysed in conjunction with current fill volumes in manufacturing it would take in excess of 80 years at the test conditions for the blister to dry out. It is an understatement to say that this package, in terms of MVTR, could have its expiry dating reviewed. Product stability plus marketing and logistics will determine if changes can be accommodated. As the blister wall thickness is determined by hard tooling no action to reduce thickness will be taken on this result.

Regarding oxygen permeability, no data was available for to state how much oxygen over how much time is needed to effect product sterility in either the large or small blister package configuration. There is scope for further work under this topic!

MECHANICAL TESTING

Initially in this section some basic length and width measurements were taken from samples processed through the one die. This verified that the die cutting process followed a Normal Distribution. The data indicated a distribution spread for +/- 4 σ of +/- 0.1mm. This is more

than adequate for accurate placement onto the blister for sealing. Also, this data can be used to size future labels to create a more robust process.

Subsequent to this tensile testing was carried out on the material. As illustrated in the previous chapter, irregardless of material thickness (60μ foil and 70μ foil), the material demonstrated most strength in the cross direction of the die cut. This corresponds to actual machine direction. Given this information, there are two possible options to choose between in manufacturing:

1. Increase quantity of material on a roll then determine the minimum roll width (using tensile test results) required to pull material through die cutting. Then determine the minimum pitch of die cuts in order to prevent snapping of waste material or skeleton. Calculate the benefits.
2. Determine the minimum roll width, as above, for the current roll configuration. Then compute the minimum pitch and calculate the benefits.

While awaiting new tooling costs from the material vendor option two above, using the current roll width, has been implemented with cost savings to the company.

The results from puncture resistance testing for each material thickness closely approximated Normal

Distributions with a statistically significant difference of 7 N between their means. The thinner foil being almost 15% less resistant to puncture. Using formulae for shear[14] there will be a corresponding reduction in maximum punch load should the company change to 60μ foil.

When distributing individual products, i.e. those not contained in a secondary package, due diligence will also have to be paid to shock should the company change to 60μ foil. Should the need arise to make additions to the cushioning within the tertiary packaging to accommodate the 60μ foil then a cost benefit analysis should be carried out. Nothing stands out from the results to indicate that the down sizing of foil is not worth further consideration. If successful this would bring economic benefits as well as environmental benefits to the company.

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[14] Hannah, J. & Hillier, M.J. *Applied Mechanics* p.287

First metric edition published 1971 PITMAN EDUCATION LIMITED

CHAPTER EIGHT: CONCLUSIONS

The lidding material and its associated blisters were researched under the headings of:

1. Burst and peel testing
2. Barrier testing
3. Mechanical testing.

The conclusions drawn from the results presented were that:

a) Seal parameters, in the range tested, effect peel and burst strength. One proviso to that is the need to do further confirmatory work on burst testing as the test equipment only managed to cause failure on two sets of seal parameters and failed to burst any of the other packages.
b) It is inconclusive whether processing conditions effect seal strength to a statistical significant level. A repeat of the tests using larger samples is needed.
c) Rate of fill, for the type of burst tester used, does not impact burst results.
d) A circular seal configuration provides an easier peel.
e) The barrier properties of the current blister warrant revisiting expiry dating.

f) Dimensions due to die cutting, tensile strength and puncture resistance of this material all follow a Normal Distribution.

g) The amount of material remaining after die cutting (skeleton) can be further minimised to achieve cost savings. This has been implemented.

h) A full evaluation of a change to a thinner foil should be considered. This would lead to cost and environmental benefits.

i) Tests conducted as part of this thesis will be recommended to be included in future applicable validations.

Thesis Test Plan

Blister Type	PARAMETERS				FIRST					PEEL			
	Temperature	Time	Pressure	Target Seal Width	Total Qty.	Before Sterilisation	After Sterilisation	After Vac.Leak & Sterilisation	After 2 cycles	Before Sterilisation	After Sterilisation	After Vac.Leak & Sterilisation	After 2 cycles
3.8ml	LOW	LOW	LOW		40	5	5	5	5	5	5	5	5
	NOM	NOM	NOM		40	5	5	5	5	5	5	5	5
	HIGH	HIGH	HIGH		40	5	5	5	5	5	5	5	5
				1.0mm	40	5	5	5	5	5	5	5	5
				2.0mm	40	5	5	5	5	5	5	5	5
	H(+25%)	H(+25%)	H(+25%)		40	5	5	5	5	5	5	5	5
1.2ml	LOW	LOW	LOW		40	5	5	5	5	5	5	5	5
	NOM	NOM	NOM		40	5	5	5	5	5	5	5	5
	HIGH	HIGH	HIGH		40	5	5	5	5	5	5	5	5
				1.0mm	40	5	5	5	5	5	5	5	5
				2.0mm	40	5	5	5	5	5	5	5	5
	H(+25%)	H(+25%)	H(+25%)		40	5	5	5	5	5	5	5	5

Barrier Properties	MVTR	Oxygen
3.8ml Blister Type	5	5
1.2ml Blister Type	5	5

Mechanical Testing	70 micron Al. Foil	60 micron Al. foil	Dimensions
Tensile	10 * M/D 10 * C/D	10 * M/D 10 * C/D	10 * length 10 * width ave. csa 70μ
Puncture	10	10	ave. csa 60μ

PROCEDURE FOR THE PROCESSING OF BLISTER PACKAGES

1. Enter heat seal parameters to be used into the Programmable Logic Controller of the packaging machine. These are; *Seal Time, Temperature and Pressure.*

2. These parameters are defined as:
   Seal time is the total amount of time that the heat seal head is in contact with the material.
   Temperature is a measurement of the surface temperature of the heat seal head.
   Pressure is defined as the air pressure reading when the heat seal cylinder is extended and in the 'sealing position'.

3. Ensure the machine has stabilised at the new parameters before commencing test.

4. Check that there is no product or components in or around the packaging machine other than those to be tested.

5. Use the same mechanism and the same components to seal all packages within the test runs.

6. Enter the next set of parameters for the next test or change the heat seal head if testing for minimum seal width. Before changing a heat seal head always allow machine to cool down.

7. Sterilisation of the packages is achieved using a steam sterilisation cycle with a maximum temperature of 125°C for 30 minutes.

8. Drawing a vacuum of 25"HG with a hold time of 30 seconds constitutes vacuum leak testing of the packages. All packages are tested in an inverted position.

PROCEDURE FOR BURST TESTING

1. Equipment;

   TEST-A-PACK 2000 control console tester.

   ARO Sensing Probe. (Maximum PSI = 52)

2. Check that the display is reading zero.

3. Choose the rate of fill required for the test.

4. Centrally place blister package, right side up, on mounting plate under both needles of the sensing probe.

5. Lower probe and puncture through the lidding material.

6. Ensure that there is a flush fit between the lidding material and the flange of the sensing probe to ensure no air loss during testing. Double sided adhesive gaskets or tape is often used to achieve this.

7. Commence airflow until package fails, or not.

8. The tester will display the highest pressure sensed prior to burst. Record this value.

9. Remove tested package and repeat procedure.

PROCEDURE FOR PEEL TESTING

1. Equipment;

   Vinatoru peel tester

   Mecmesin force gauge

   Mecmesin dataplot software

2. Check that the force gauge is reading zero.

3. Place blister package in mounting plate and secure with attaching screw.

4. Lift peeled end of material and secure in clamp attached to force gauge.

5. Start the peeling operation. Speed setting should be approximately 7" per minute.

6. When peeling and data logging is complete print graph.

7. Remove peeled blister package and replace with new.

8. Repeat procedure.

PROCEDURE FOR BARRIER TESTING

1. Pre-conditioning: None

2. Conditioning: 23 ± 1°C, 50 ± 2%rh for a minimum of 24 hours.

3. Test Conditions: 23 ± 1°C, 50 ± 2%rh

4. Oxygen Permeability:

   Coulometric method using the Oxtran 2/20 apparatus with computer control. Each blister was adhered to a metal plate fitted with an inlet and outlet pipe for the carrier gas. Initially they were flushed with moist carrier gas after which the sensor was activated to detect the amount of oxygen that had permeated through the mounted samples.

   All measurements were made after a minimum 24 hour flushing period when the system was assumed to have reached equilibrium. Measurements were obtained to air and the results are quoted for 100% oxygen.

5. Moisture Vapour Transmission Rate

   ASTM F1249(1987) with modifications listed below.

Permatran W600 equipment. Each blister was adhered to a metal plate fitted with an inlet and outlet pipe for the carrier gas. The samples were then connected to the test equipment and were conditioned at the stated test conditions for a minimum 24 hour period prior to being tested. Any moisture picked up by the dry gas stream as each blister was being flushed was detected by an infra red sensor which produced a millivolt reading. This millivolt reading was then converted to the required test units. Each test consisted of a minimum four 2 hour test cycles.

6. Three replicate tests were performed for both of the above.

7. Individual results were quoted in all cases.

PROCEDURE FOR TENSILE TESTING

1. Equipment;

   Lloyd 3000S Universal test machine.

   500N load cell.

   Standard gauge length of 20mm or 45mm was employed as appropriate.

2. Samples were prepared by cutting parallel strips 10.0mm wide from the die cut blanks provided to within 0.1mm.

3. The samples were placed in soft jaws of the test machine.

4. Tests were carried out by remote control to a PC with manual zeroing of set-up position.

5. A 1.0 mm/min strain rate was applied to avoid rupturing the samples.

6. A digital log of the tests was kept as well as printed load versus cross-head position graphs. From these the UTS and yield stress were calculated. A uniform aluminium thickness was assumed.

7. The tests were replicated 10 times in both the cross direction and longitudinal direction.

8. The above procedure was repeated for both material thicknesses.

9. When conducting the statistical analysis for this test the following was carried out to eliminate any outliers that may have occurred due to damage in the test sample cutting operation.

   The maximum and minimum values recorded for each test were removed from that individual set of results.

   An average and standard deviation based on each of the new set of values were then calculated.

   If either the old maximum or minimum values exceeded this new average ± 3 standard deviations, then they were decreed to be outliers and removed from that set.

   If any were removed then a new average and standard deviation were calculated and used for the analysis.

   If none were removed then the average and standard deviation were calculated based on the old, complete set of test results.

PROCEDURE FOR PUNCTURE RESISTANCE TESTING

1. Equipment;

   Hounsfield tensile tester, 500N load cell and a test speed of 500mm/min.

   Two aluminium plates capable of being located to the underside of the cross-head platform of the tensile tester, with a 25.4mm diameter hole in the centre of the plates.

   Two sheets of carborundum paper with rough surfaces facing each other between the two plates.

2. Puncture Resistance to Def. Standard 81-75/1 Annex H.

3. The sheets are used to hold the test specimen.

4. A steel rod of diameter 12.7mm and 127mm long with one end tapered to 3.2mm radius is used as the probe. The length of the taper is 51.8mm.

5. A system is used which allows the wider end of the probe to be fixed rigidly to the compression load cell.

6. Five replicate tests per direction are completed.

7. Repeat procedure per material type.

SEAL TESTING

Large Blister Burst Test Results

Seal Width : 1.00mm	1	2	3	4	5	Range
Before Sterilisation	45.29	45.71	41.62	42.16	41.48	4.23
After Sterilisation	43.29	41.41	42.88	38.30	39.79	4.99
After Vac. Leak & Sterilisa	43.05	44.54	37.11	40.61	34.08	10.46
After 2 Sterilisation Cycle	45.86	43.66	36.31	35.09	38.69	10.77

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Before Sterilisation	5	216.26	43.25	4.29777
After Sterilisation	5	205.67	41.13	4.40273
After Vac. Leak & Sterilisa	5	199.39	39.88	18.40257
After 2 Sterilisation Cycle	5	199.61	39.92	21.78627

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	37.5089	3	12.5030	1.0230	0.4086	5.2922
Within Groups	195.5574	16	12.2223			
Total	233.0663	19				

Seal Width : 2.00mm	1	2	3	4	5	Range
Before Sterilisation	50.42	49.46	50.11	DNB	DNB	0.96
After Sterilisation	42.30	45.10	45.46	45.83	46.02	3.72
After Vac. Leak & Sterilisa	44.38	45.66	48.34	43.88	N/A	4.46
After 2 Sterilisation Cycle	47.03	49.02	45.55	47.19	46.40	3.47

Anova: Single Factor

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Before Sterilisation	3	149.99	50.00	0.24003
After Sterilisation	5	224.71	44.94	2.30602
After Vac. Leak & Sterilisa	4	182.26	45.57	3.98437
After 2 Sterilisation Cycle	5	235.19	47.04	1.64317

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	53.6008	3	17.8669	8.2278	0.0025	5.7394
Within Groups	28.2299	13	2.1715			
Total	81.8308	16				

Different Fill Rates	1	2	3	4	5	Range
@ Fill Rate = 9	49.05	46.27	43.33	44.37	37.67	11.38
@ Fill Rate = 7	42.13	47.46	47.91	42.66	43.57	5.78
@ Fill Rate = 5	42.09	44.25	43.75	42.43	41.68	2.57
@ Fill Rate = 3	42.52	43.07	39.35	41.03	43.44	4.09

Anova: Single Factor

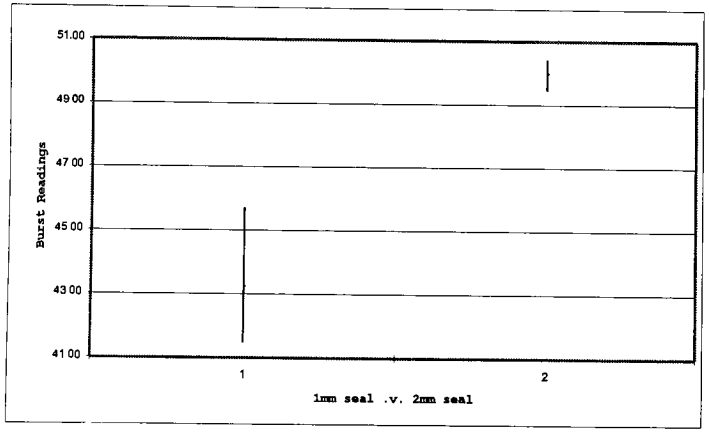
SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
@ Fill Rate = 9	5	220.69	44.14	17.80372
@ Fill Rate 7	5	223.73	44.75	7.48863
@ Fill Rate 5	5	214.2	42.84	1.2231
@ Fill Rate = 3	5	209.41	41.88	2.84567

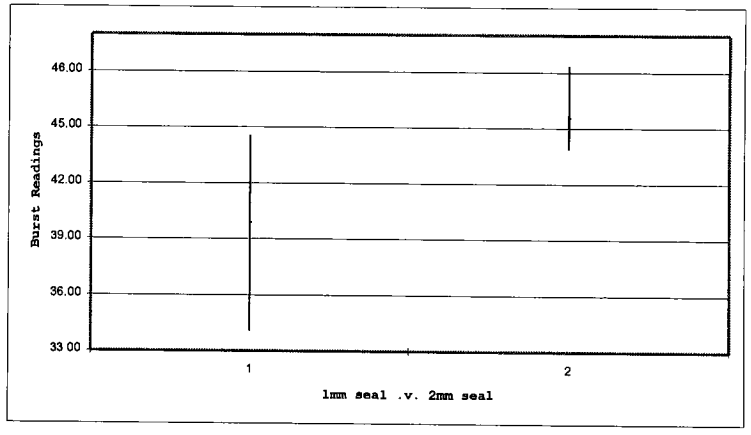
ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	24.8714	3	8.2905	1.1294	0.3668	3.2389
Within Groups	117.4445	16	7.3403			
Total	142.3159	19				

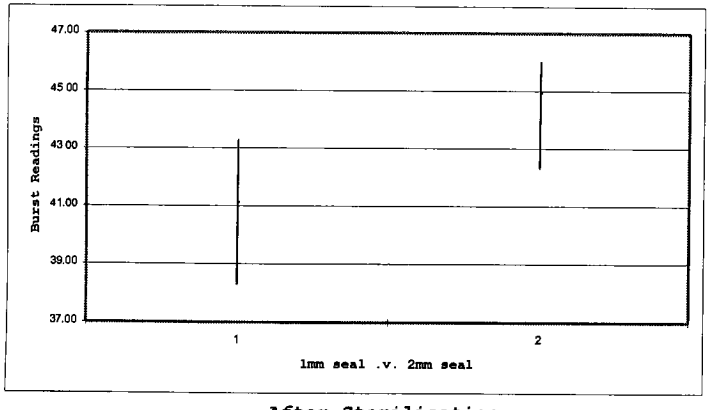
Comparison of Result Ranges
of
1mm Seal versus 2mm Seal



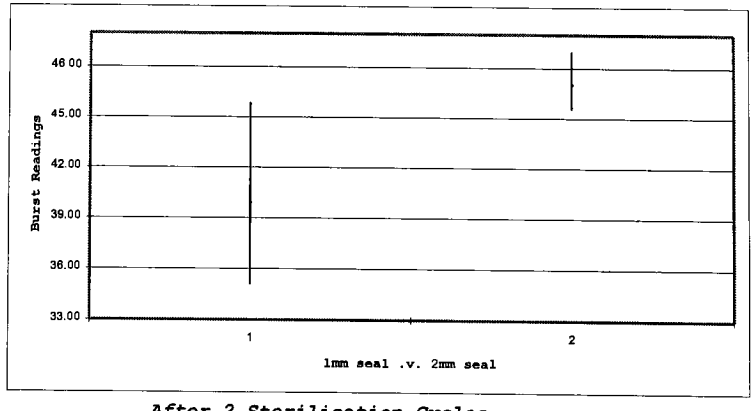
Before Sterilisation



After Vac. Leak & Sterilisation



After Sterilisation



After 2 Sterilisation Cycles

Two sample Student t-Tests
of
1mm seal results versus 2mm seal results

t-Test: Two-Sample Assuming Unequal Variances Before Sterilisation	*1mm seal*	*2mm seal*
Mean	43.25	50.00
Variance	4.2978	0.2400
Observations	5	3
Hypothesized Mean Difference	0	
df	5	
t Stat	-6.9582	
P(T<=t) one-tail	0.0005	
t Critical one-tail	2.0150	
P(T<=t) two-tail	0.0009	
t Critical two-tail	2.5706	

t-Test: Two-Sample Assuming Unequal Variances After Vac. Leak & Sterilisation	*1mm seal*	*2mm seal*
Mean	39.88	45.57
Variance	18.4026	3.9844
Observations	5	4
Hypothesized Mean Difference	0	
df	6	
t Stat	-2.6298	
P(T<=t) one-tail	0.0195	
t Critical one-tail	1.9432	
P(T<=t) two-tail	0.0391	
t Critical two-tail	2.4469	

t-Test: Two-Sample Assuming Unequal Variances After Sterilisation	*1mm seal*	*2mm seal*
Mean	41.13	44.94
Variance	4.4027	2.3060
Observations	5	5
Hypothesized Mean Difference	0	
df	7	
t Stat	-3.2875	
P(T<=t) one-tail	0.0067	
t Critical one-tail	1.8946	
P(T<=t) two-tail	0.0133	
t Critical two-tail	2.3646	

t-Test: Two-Sample Assuming Unequal Variances After 2 Sterilisations	*1mm seal*	*2mm seal*
Mean	39.92	47.04
Variance	21.7863	1.6432
Observations	5	5
Hypothesized Mean Difference	0	
df	5	
t Stat	-3.2873	
P(T<=t) one-tail	0.0109	
t Critical one-tail	2.0150	
P(T<=t) two-tail	0.0218	
t Critical two-tail	2.5706	

SEAL TESTING

Large Blister Peel Test Results

Blister Type	PARAMETERS Temperature	Time	Pressure	Target Seal Width	Total Qty.	PEEL Before Sterilisation	After Sterilisation	After Vac.Leak & Sterilisation	After 2 cycles
Large	LOW	LOW	LOW		20	5	5	5	5
	NOMINAL	NOMINAL	NOMINAL		19	5	5	4	5
	HIGH	HIGH	HIGH		20	5	5	5	5
				1.0mm	20	5	5	5	5
				2.0mm	20	5	5	5	5
	HIGH(+25%)	HIGH(+25%)	HIGH(+25%)		18	3	5	5	5

PARAMETER SETTINGS	LOW	NOMINAL	HIGH

Initial Peel Peak	Before Sterilisation	Before Sterilisation	Before Sterilisation
1	5.00	5.25	5.90
2	4.50	5.45	5.30
3	5.15	5.00	5.75
4	5.15	4.60	6.25
5	4.80	4.90	5.70
Sample mean	**4.92**	**5.04**	5.78
Sample std. dev.	0.28	0.33	0.34
Subsequent to Initial Peak	[redacted]	[redacted]	[redacted]
Upper peel	4.90	5.00	5.25
Range midpoint	4.05	4.13	4.45
Lower peel	3.20	3.25	3.65

Initial Peel Peak	After Sterilisation	After Sterilisation	After Sterilisation
1	4.55	4.90	5.20
2	4.80	4.75	5.10
3	4.45	4.65	5.35
4	4.20	4.65	5.25
5	4.50	4.55	4.70
Sample mean	4.50	4.70	**5.12**
Sample std. dev.	0.22	0.13	0.25
Subsequent to Initiel Peak	[redacted]	[redacted]	[redacted]
Upper peel	5.15	5.25	5.80
Range midpoint	3.90	3.73	4.30
Lower peel	2.65	2.20	2.80

Initial Peel Peak	After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation
1	4.45	4.85	5.25
2	4.40	4.70	5.20
3	3.90	3.80	5.25
4	3.65	3.20	5.25
5	5.15		5.25
Sample mean	**4.31**	4.14	**5.24**
Sample std. dev.	0.58	0.78	0.02
Subsequent to Initial Peak	[redacted]	[redacted]	[redacted]
Upper peel	5.00	5.55	6.00
Range midpoint	4.19	4.10	4.60
Lower peel	3.37	2.65	3.20

Initial Peel Peak	After 2 Sterilisations	After 2 Sterilisations	After 2 Sterilisations
1	4.75	4.20	4.90
2	4.35	4.70	4.30
3	4.65	4.15	4.70
4	3.20	4.30	4.60
5	4.70	3.95	4.50
Sample mean	**4.33**	**4.26**	4.60
Sample std. dev.	0.65	0.28	0.22
Subsequent to Initial Peak	[redacted]	[redacted]	[redacted]
Upper peel	5.60	5.85	5.55
Range midpoint	3.98	4.35	3.95
Lower peel	2.35	2.85	2.35

HIGH (+25%)	1.0mm	2.0mm

Before Sterilisation	Before Sterilisation	Before Sterilisation
6.30	3.15	4.37
6.00	3.15	4.20
5.10	2.70	3.80
	2.75	3.75
	2.85	4.20
5.80	**2.92**	**4.06**
0.62	0.22	0.27
[redacted]	[redacted]	[redacted]
6.05	3.00	4.20
4.10	3.35	3.38
2.15	1.70	2.55

After Sterilisation	After Sterilisation	After Sterilisation
5.20	3.02	3.80
5.50	2.20	3.75
5.50	2.00	3.65
5.65	3.30	3.25
5.30	2.50	3.50
5.43	**2.60**	**3.59**
0.18	0.55	0.22
[redacted]	[redacted]	[redacted]
6.15	4.37	4.75
4.68	3.04	3.35
3.20	1.70	1.95

After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation
5.35	2.12	3.60
4.35	2.32	3.70
4.50	1.61	3.75
5.75	1.27	3.65
5.00	1.91	3.85
4.99	**1.86**	**3.71**
0.58	0.41	0.10
[redacted]	[redacted]	[redacted]
6.30	4.13	4.25
4.82	2.77	3.18
3.33	1.40	2.10

After 2 Sterilisations	After 2 Sterilisations	After 2 Sterilisations
5.30	1.87	3.30
5.25	1.44	4.25
5.70	2.12	2.85
4.75	2.25	3.35
5.70	1.77	3.60
5.34	**1.90**	**3.47**
0.39	0.31	0.51
[redacted]	[redacted]	[redacted]
5.85	4.28	4.80
4.43	2.78	3.35
3.00	1.28	1.90

LARGE BLISTER

Anova: Single Factor LOW Sealing Parameters, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	24.6	4.92	0.07575
Column 2	5	22.5	4.5	0.04625
Column 3	5	21.55	4.31	0.33425
Column 4	5	21.65	4.33	0.42325

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	1.2025	3	0.400833	1.823005	0.183578	3.238867
Within Groups	3.518	16	0.219875			
Total	4.7205	19				

Anova: Single Factor NOMINAL Sealing Parameters, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	25.2	5.04	0.10675
Column 2	5	23.5	4.7	0.0175
Column 3	4	16.55	4.1375	0.605625
Column 4	5	21.3	4.26	0.07675

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	2.413599	3	0.804533	4.604567	0.0178	3.287383
Within Groups	2.620875	15	0.174725			
Total	5.034474	18				

Anova: Single Factor HIGH Sealing Parameters, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	28.9	5.78	0.11825
Column 2	5	25.6	5.12	0.06325
Column 3	5	26.2	5.24	0.0005
Column 4	5	23	4.6	0.05

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	3.5175	3	1.1725	20.21552	1.09E-05	3.238867
Within Groups	0.928	16	0.058			
Total	4.4455	19				

LARGE BLISTER

Anova: Single Factor HIGH (+25%) Sealing Parameters, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	3	17.4	5.8	0.39
Column 2	5	27.15	5.43	0.032
Column 3	5	24.95	4.99	0.33925
Column 4	5	26.7	5.34	0.15425

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	1.287444	3	0.429148	2.084689	0.148281	3.343885
Within Groups	2.882	14	0.205857			
Total	4.169444	17				

Anova: Single Factor 1mm Seal Width, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	14.6	2.92	0.047
Column 2	5	13.02	2.604	0.29908
Column 3	5	9.29	1.858	0.16647
Column 4	5	9.48	1.896	0.09338

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	4.169375	3	1.389792	9.174602	0.000913	3.238867
Within Groups	2.42372	16	0.151483			
Total	6.593095	19				

Anova: Single Factor 2mm Seal Width, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	20.32	4.064	0.07473
Column 2	5	17.95	3.59	0.04925
Column 3	5	18.55	3.71	0.00925
Column 4	5	17.35	3.47	0.26325

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.986535	3	0.328845	3.317645	0.046737	3.238867
Within Groups	1.58592	16	0.09912			
Total	2.572455	19				

LARGE BLISTER

Anova: Single Factor Before Sterilisation - All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	24.6	4.92	0.0757
Column 2	5	25.2	5.04	0.1068
Column 3	5	28.9	5.78	0.1183
Column 4	3	17.4	5.8	0.3900
Column 5	5	14.6	2.92	0.0470
Column 6	5	20.32	4.064	0.0747

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	28.134466	5	5.6269	50.1197	2.718E-11	2.6613
Within Groups	2.46992	22	0.1123			
Total	30.604386	27				

Anova: Single Factor After Sterilisation - All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	22.5	4.5	0.0463
Column 2	5	23.5	4.7	0.0175
Column 3	5	25.6	5.12	0.0633
Column 4	5	27.15	5.43	0.0320
Column 5	5	13.02	2.604	0.2991
Column 6	5	17.95	3.59	0.0492

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	27.6318	5	5.5264	65.3582	3.459E-13	2.6207
Within Groups	2.02932	24	0.0846			
Total	29.66112	29				

LARGE BLISTER

Anova: Single Factor After Vac. Leak & Sterilisation - All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	21.55	4.31	0.3342
Column 2	4	16.55	4.1375	0.6056
Column 3	5	26.2	5.24	0.0005
Column 4	5	24.95	4.99	0.3393
Column 5	5	9.29	1.858	0.1665
Column 6	5	18.55	3.71	0.0092

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	36.464976	5	7.2930	32.1600	1.188E-09	2.6400
Within Groups	5.215755	23	0.2268			
Total	41.680731	28				

Anova: Single Factor After 2 Sterilisations - All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	21.65	4.33	0.4233
Column 2	5	21.3	4.26	0.0768
Column 3	5	23	4.6	0.0500
Column 4	5	26.7	5.34	0.1543
Column 5	5	9.48	1.896	0.0934
Column 6	5	17.35	3.47	0.2632

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	35.190067	5	7.0380	39.8048	7.476E-11	2.6207
Within Groups	4.24352	24	0.1768			
Total	39.433587	29				

SEAL TESTING

Small Blister Peel Test Results

Blister Type	PARAMETERS Temperature	Time	Pressure	Target Seal Width	Total Qty.	PEEL Before Sterilisation	After Sterilisation	After Vac.Leak & Sterilisation	After 2 cycles
small	LOW	LOW	LOW		20	5	5	5	5
	NOMINAL	NOMINAL	NOMINAL		20	5	5	5	5
	HIGH	HIGH	HIGH		20	5	5	5	5
				1.0mm	20	5	5	5	5
				2.0mm	20	5	5	5	5
	HIGH(+25%)	HIGH(+25%)	HIGH(+25%)		20	5	5	5	5

PARAMETER SETTINGS	LOW	NOMINAL	HIGH
Initial Peel Peak	Before Sterilisation	Before Sterilisation	Before Sterilisation
1	3.25	3.75	3.50
2	3.60	3.35	3.83
3	3.65	3.40	3.45
4	3.95	3.20	3.40
5	3.38	3.25	3.55
Sample mean	**3.57**	**3.39**	**3.55**
Sample std. dev.	0.27	0.22	0.17
Subsequent to Initial Peak			
Upper peel	3.00	3.30	3.10
Range midpoint	2.40	2.53	2.49
Lower peel	1.80	1.75	1.87

HIGH (+25%)	1.0mm	2.0mm
Before Sterilisation	Before Sterilisation	Before Sterilisation
2.95	0.85	2.20
3.40	2.65	2.10
3.30	2.45	1.75
3.50	3.55	1.75
3.50	1.00	2.27
3.33	**2.10**	**2.01**
0.23	1.15	0.25
3.00	2.15	3.20
2.50	1.50	2.15
2.00	0.85	1.10

Initial Peel Peak	After Sterilisation	After Sterilisation	After Sterilisation
1	2.25	3.15	3.00
2	2.50	3.10	3.37
3	2.50	3.25	3.15
4	3.25	2.70	2.90
5	2.65	3.00	3.87
Sample mean	**2.63**	**3.04**	**3.26**
Sample std. dev.	0.38	0.21	0.39
Subsequent to Initial Peak			
Upper peel	2.84	2.75	2.90
Range midpoint	2.12	2.23	2.29
Lower peel	1.40	1.70	1.68

After Sterilisation	After Sterilisation	After Sterilisation
3.00	1.60	3.65
3.30	1.10	1.85
3.35	2.03	1.88
3.28	2.12	1.55
3.30	3.47	1.80
3.25	**2.06**	**2.15**
0.14	0.88	0.85
3.20	2.12	2.37
2.50	1.39	1.51
1.80	0.65	0.65

Initial Peel Peak	After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation
1	2.25	2.60	2.73
2	3.10	2.60	2.87
3	2.30	3.25	2.87
4	3.30	2.75	2.85
5	2.42	2.35	2.52
Sample mean	**2.67**	**2.71**	**2.77**
Sample std. dev.	0.49	0.33	0.15
Subsequent to Initial Peak			
Upper peel	2.35	2.27	2.73
Range midpoint	1.73	1.84	2.09
Lower peel	1.10	1.40	1.45

After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation	After Vac. Leak & Sterilisation
3.20	2.95	1.88
3.72	1.67	2.40
3.40	1.95	1.83
3.25	1.15	1.75
3.37	1.47	1.80
3.39	**1.76**	**1.93**
0.20	0.65	0.27
3.72	2.92	2.43
3.04	1.81	1.69
2.35	0.70	0.94

Initial Peel Peak	After 2 Sterilisations	After 2 Sterilisations	After 2 Sterilisations
1	2.65	3.05	3.05
2	3.00	3.25	3.30
3	2.70	2.75	3.10
4	2.80	2.85	3.35
5	2.72	3.20	3.60
Sample mean	**2.77**	**3.02**	**3.28**
Sample std. dev.	0.14	0.22	0.22
Subsequent to Initial Peak			
Upper peel	2.90	3.05	3.35
Range midpoint	2.29	2.40	2.45
Lower peel	1.68	1.75	1.55

After 2 Sterilisations	After 2 Sterilisations	After 2 Sterilisations
3.75	1.35	2.20
3.27	2.13	1.00
3.25	2.16	2.18
3.25	3.42	1.95
3.67	2.37	1.40
3.44	**2.29**	**1.95**
0.25	0.74	0.52
3.65	2.85	2.22
2.83	1.79	1.49
2.00	0.72	0.55

SMALL BLISTER

Anova: Single Factor LOW Sealing Parameters, All Processing Conditions

SUMMARY

Groups	Count	Sum	Average	Variance
Column 1	5	17.83	3.566	0.07253
Column 2	5	13.15	2.63	0.14075
Column 3	5	13.37	2.674	0.23938
Column 4	5	13.87	2.774	0.01888

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	2.91462	3	0.97154	8.241422	0.001528	3.238867
Within Groups	1.88616	16	0.117885			
Total	4.80078	19				

Anova: Single Factor NOMINAL Sealing Parameters, All Processing Conditions

SUMMARY

Groups	Count	Sum	Average	Variance
Column 1	5	16.95	3.39	0.04675
Column 2	5	15.2	3.04	0.04425
Column 3	5	13.55	2.71	0.11175
Column 4	5	15.1	3.02	0.047

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.159	3	0.386333	6.187521	0.005398	3.238867
Within Groups	0.999	16	0.062437			
Total	2.158	19				

Anova: Single Factor HIGH Sealing Parameters, All Processing Conditions

SUMMARY

Groups	Count	Sum	Average	Variance
Column 1	5	17.73	3.546	0.02833
Column 2	5	16.29	3.258	0.14837
Column 3	5	13.84	2.768	0.02262
Column 4	5	16.4	3.28	0.04825

ANOVA

Source of Variation	SS	df	MS	F	P-value	F crit
Between Groups	1.57714	3	0.525713	8.493975	0.001325	3.238867
Within Groups	0.99028	16	0.061893			
Total	2.56742	19				

SMALL BLISTER

Anova: Single Factor HIGH(+25%) Sealing Parameters, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	16.65	3.33	0.052
Column 2	5	16.23	3.246	0.01958
Column 3	5	16.94	3.388	0.04127
Column 4	5	17.19	3.438	0.06252

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.102015	3	0.034005	0.775617	0.524528	3.238867
Within Groups	0.70148	16	0.043843			
Total	0.803495	19				

Anova: Single Factor 1mm Seal Width, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	10.5	2.1	1.325
Column 2	5	10.32	2.064	0.78143
Column 3	5	8.79	1.758	0.41612
Column 4	5	11.43	2.286	0.55233

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.7182	3	0.2394	0.311427	0.816842	3.238867
Within Groups	12.29952	16	0.76872			
Total	13.01772	19				

Anova: Single Factor 2mm Seal Width, All Processing Conditions

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	10.07	2.014	0.06173
Column 2	5	10.73	2.146	0.72383
Column 3	5	9.66	1.932	0.07067
Column 4	5	9.73	1.946	0.10508

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	0.143455	3	0.047818	0.198972	0.895551	3.238867
Within Groups	3.84524	16	0.240328			
Total	3.988695	19				

SMALL BLISTER

Anova: Single Factor Before Sterilisation - All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	17.83	3.566	0.0725
Column 2	5	16.95	3.39	0.0468
Column 3	5	17.73	3.546	0.0283
Column 4	5	16.65	3.33	0.0520
Column 5	5	10.5	2.1	1.3250
Column 6	5	10.07	2.014	0.0617

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	13.30591	5	2.6612	10.0654	2.74098E-05	2.6207
Within Groups	6.34536	24	0.2644			
Total	19.65127	29				

Anova: Single Factor After Sterilisation - All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	13.15	2.63	0.1407
Column 2	5	15.2	3.04	0.0442
Column 3	5	16.29	3.258	0.1484
Column 4	5	16.23	3.246	0.0196
Column 5	5	10.32	2.064	0.7814
Column 6	5	10.73	2.146	0.7238

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	7.178746667	5	1.4357	4.6359	0.004213845	2.6207
Within Groups	7.43284	24	0.3097			
Total	14.61158667	29				

SMALL BLISTER

Anova: Single Factor After Vac. Leak & Sterilisation All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	13.37	2.674	0.2394
Column 2	5	13.55	2.71	0.1118
Column 3	5	13.84	2.768	0.0226
Column 4	5	16.94	3.388	0.0413
Column 5	5	8.79	1.758	0.4161
Column 6	5	9.66	1.932	0.0707

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	8.995576667	5	1.7991	11.9700	6.92214E-06	2.6207
Within Groups	3.60724	24	0.1503			
Total	12.60281667	29				

Anova: Single Factor After 2 Sterilisations - All sealing parameters

SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Column 1	5	13.87	2.774	0.0189
Column 2	5	15.1	3.02	0.0470
Column 3	5	16.4	3.28	0.0482
Column 4	5	17.19	3.438	0.0625
Column 5	5	11.43	2.286	0.5523
Column 6	5	9.73	1.946	0.1051

ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	8.397546667	5	1.6795	12.0819	6.41397E-06	2.6207
Within Groups	3.33624	24	0.1390			
Total	11.73378667	29				

PEEL RESULTS

t-test comparisons of large blister .v. small blister

t-Test: Two-Sample Assuming Unequal Variances

	LOW	*LOW*
Mean	4.515	2.911
Variance	0.24844737	0.252672632
Observations	20	20
Hypothesized Mean Difference	0	
df	38	
t Stat	10.1332438	
P(T<=t) one-tail	1.1801E-12	
t Critical one-tail	1.68595307	
P(T<=t) two-tail	2.3602E-12	
t Critical two-tail	2.02439423	

t-Test: Two-Sample Assuming Unequal Variances

	H(+25%)	*H(+25%)*
Mean	5.344	3.3505
Variance	0.2452614	0.042289211
Observations	18	20
Hypothesized Mean Difference	0	
df	22	
t Stat	15.893127	
P(T<=t) one-tail	7.628E-14	
t Cntical one-tail	1.7171442	
P(T<=t) two-tail	1.526E-13	
t Critical two-tail	2.0738753	

t-Test: Two-Sample Assuming Unequal Variances

	NOMINAL	*NOMINAL*
Mean	4.55526316	3.04
Variance	0.27969298	0.113578947
Observations	19	20
Hypothesized Mean Difference	0	
df	30	
t Stat	10.6090601	
P(T<=t) one-tail	5.6716E-12	
t Critical one-tail	1.69726036	
P(T<=t) two-tail	1.1343E-11	
t Critical two-tail	2.04227035	

t-Test: Two-Sample Assuming Unequal Variances

	1mm	*1mm*
Mean	2.3195	2.052
Variance	0.347005	0.685143158
Observations	20	20
Hypothesized Mean Difference	0	
df	34	
t Stat	1.1775186	
P(T<=t) one-tail	0.1235828	
t Criticel one-tail	1.6909235	
P(T<=t) two-tail	0.2471657	
t Critical two-tail	2.0322432	

t-Test: Two-Sample Assuming Unequal Variances

	HIGH	*HIGH*
Mean	5.185	3.213
Variance	0.23397368	0.135127368
Observations	20	20
Hypothesized Mean Difference	0	
df	35	
t Stat	14.5160771	
P(T<=t) one-tail	1.1112E-16	
t Critical one-tail	1.68957285	
P(T<=t) two-tail	2.2225E-16	
t Critical two-tail	2.03011041	

t-Test: Two-Sample Assuming Unequal Variances

	2mm	*2mm*
Mean	3.7085	2.0095
Variance	0.1353924	0.209931316
Observations	20	20
Hypothesized Mean Difference	0	
df	36	
t Stat	12.929901	
P(T<=t) one-tail	2.144E-15	
t Critical one-tail	1.6882973	
P(T<=t) two-tail	4.287E-15	
t Critical two-tail	2.0280913	

APPENDIX E

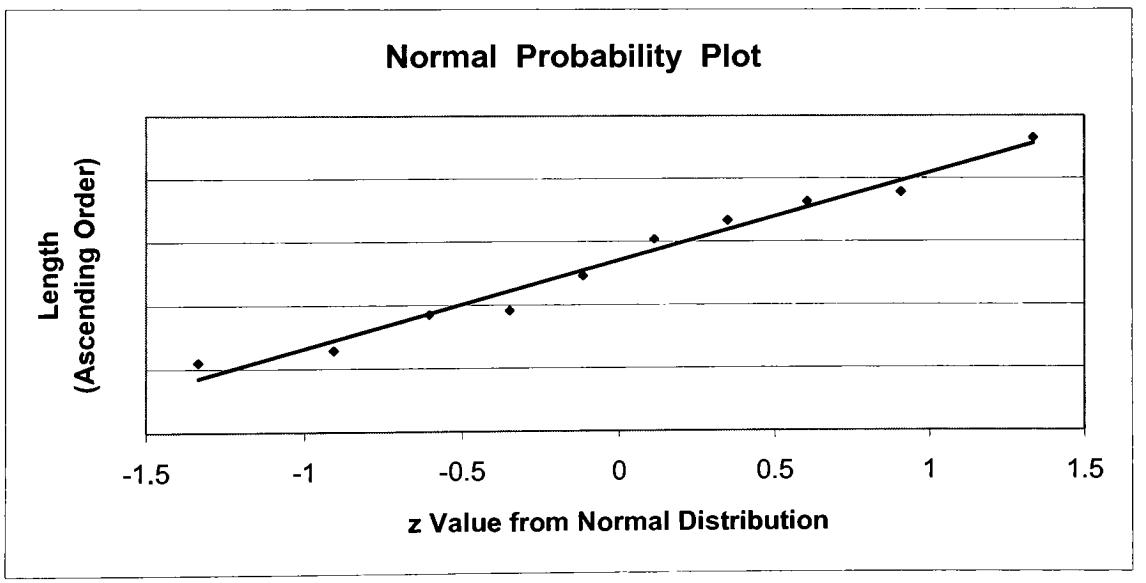
Mechanical Testing

Dimensional Results

Sample *i*	Length (mm)	Width (mm)	Length Sorted in Ascending Order	*i* / (n+1)	z value from Normal distribution
1	67.2383	33.2964	67.2221	0.0909	-1.3355
2	67.2221	33.3212	67.2259	0.1818	-0.9085
3	67.2371	33.3171	67.2371	0.2727	-0.6046
4	67.2757	33.3085	67.2383	0.3636	-0.3488
5	67.2259	33.2950	67.2493	0.4545	-0.1142
6	67.2493	33.3156	67.2608	0.5455	0.1142
7	67.2727	33.3191	67.2667	0.6364	0.3488
8	67.2667	33.2981	67.2727	0.7273	0.6046
9	67.2608	33.3031	67.2757	0.8182	0.9085
10	67.2929	33.3037	67.2929	0.9091	1.3355

Sample Mean	67.2542	33.3078
Sample Std. Deviation	0.0233	0.0099

Cross Sectional Area (mm^2) 70µ Foil	2.3315
Cross Sectional Area (mm^2) 60µ Foil	1.9985



For Normal Probability Plot : *Pearson* r = 0.9882

r^2 = 0.9765

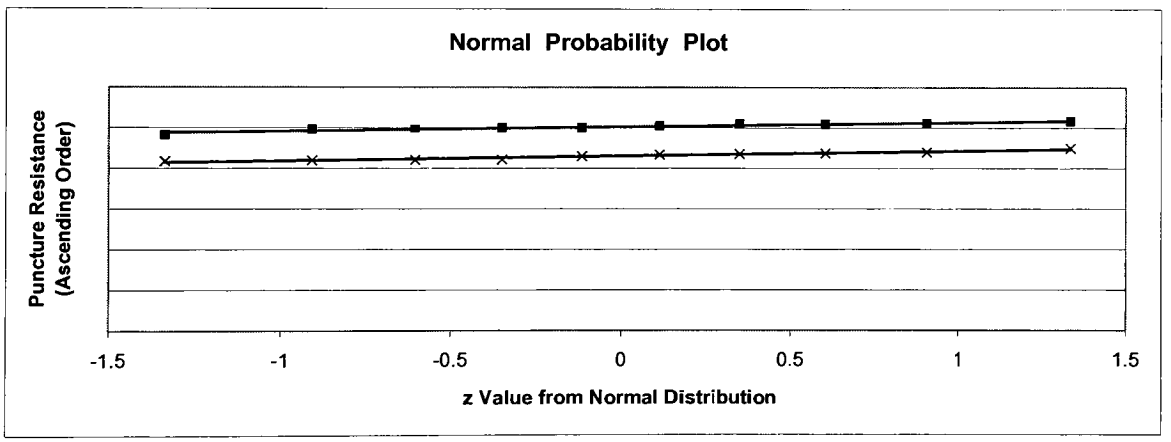
Mechanical Testing

Puncture Resistance Results

Sample *i*	60μ Foil (Newtons)	70μ Foil (Newtons)	60μ Foil Sorted in Ascending Order	70μ Foil Sorted in Ascending Order	*i* / (n+1)	z value from Normal distribution
1	43.7	50.9	41.8	48.3	0.0909	-1.3355
2	43.0	48.3	42.0	49.6	0.1818	-0.9085
3	45.0	51.1	42.1	49.9	0.2727	-0.6046
4	44.0	50.9	42.2	50.0	0.3636	-0.3488
5	43.4	49.9	43.0	50.0	0.4545	-0.1142
6	43.6	50.0	43.4	50.5	0.5455	0.1142
7	41.8	51.6	43.6	50.9	0.6364	0.3488
8	42.0	49.6	43.7	50.9	0.7273	0.6046
9	42.1	50.5	44.0	51.1	0.8182	0.9085
10	42.2	50.0	45.0	51.6	0.9091	1.3355

Sample Statistics		
Mean	43.08	50.28
Std. Deviation	1.0454	0.9378
Variance	1.0929	0.8796

Cross Sectional Area (mm^2) 70μ Foil	3.5196
Cross Sectional Area (mm^2) 60μ Foil	3.0168



Normal Probability Plot	*Pearson r*	*r 2*
Top Line on Plot = 70μ Foil	0.9707	0.9424
Bottom Line on Plot = 60μ Foil	0.9614	0.9244

t-Test: Two-Sample Assuming Unequal Variances

	60μ Foil	*70μ Foil*
Mean	43.08	50.28
Variance	1.0929	0.8796
Observations	10	10
Hypothesized Mean Difference		0
df	18	
t Stat	-16.2118	
P(T<=t) one-tail	1.74477E-12	
t Critical one-tail	2.5524	
P(T<=t) two-tail	3.48955E-12	
t Critical two-tail	2.8784	

Mechanical Testing

Tensile Test Results

70 µ foil (cross-sectional direction)				
Sample i	Load Break @ (Newtons)	UTS (Mpa)	i / (n+1)	z value from Normal distribution
1	38	54	0 0909	-1.3355
2	41	59	0.1818	-0.9085
3	45	64	0 2727	-0.6048
4	45	64	0.3636	-0.3498
5	46	66	0.4545	-0.1142
6	47	67	0.5455	0.1142
7	47	67	0.6364	0 3488
8	50	71	0.7273	0.6046
9	50	71	0.8182	0.9085
10	50	71	0.9091	1.3355
Sample Mean		65.57		
Sample Std Deviation		5 65		

70 µ foil (longitudinal direction)				
Sample i	Load Break @ (Newtons)	UTS (Mpa)	i / (n+1)	z value from Normal distribution
1	16	23	0.0909	-1.3355
2	23	33	0.1818	-0.9085
3	24	34	0.2727	-0.6048
4	24	34	0.3636	-0.3488
5	28	40	0.4545	-0.1142
6	32	46	0.5455	0 1142
7	32	46	0.6364	0.3488
8	33	47	0.7273	0.6048
9	35	50	0 8182	0.9085
10	42	80	0 9091	1.3355
Sample Maan		41.29		
Sample Sld. Devlation		10.59		

t-Test: Two-Sample Assuming Unequal Variances

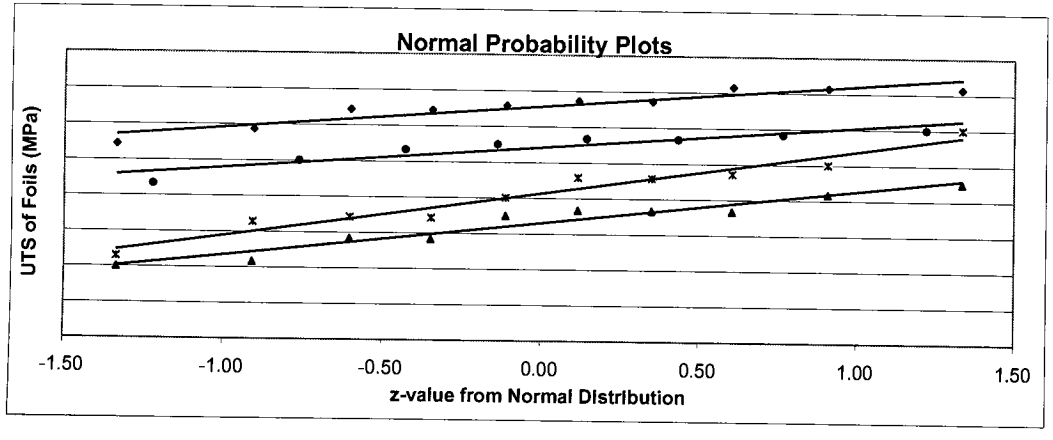
	70µ cross-sectional	70 µ longitudinal
Mean	65.57142857	41.28571429
Variance	31.9501	112.2222
Observations	10	10
Hypothesised Mean Diff	0	
df	14	
t Stat	6.3960	
P|T<=t| one-tail	8.32732E-06	
t Critical one-tail	2.6245	
P|T<=t| two-tail	1.66546E-05	
t Critical two-tail	2.9768	

60 µ foil (cross-sectional direction)					
Sample i	Load Break @ (Newtons)	UTS (Mpa)	UTS excl. outliers	i / (n+1)	z value from Normal distribution
1	35	58			
2	34	57	43	0.1111	-1.221
3	36	60	50	0 2222	-0.765
4	34	57	53	0.3333	-0 432
5	22	37	55	0 4444	-0.141
6	26	43	57	0 5556	0.141
7	30	50	57	0.6667	0.432
8	33	55	58	0.7778	0 765
9	32	53	60	0 8889	1.221
Sample Means		52.22	64.17		
Sample Std. Deviations		7.68	5.35		

60 µ foil (longitudinal direction)				
Sample i	Load Break @ (Newtons)	UTS (Mpa)	i / (n+1)	z value from Normal distribution
1	12	20	0.0909	-1 3355
2	13	22	0.1818	-0.9085
3	17	28	0.2727	-0.6048
4	17	28	0.3638	-0.3488
5	21	35	0.4545	-0.1142
6	22	37	0.6455	0.1142
7	22	37	0.6364	0.3488
8	22	37	0.7273	0.6046
9	25	42	0.8182	0.9085
10	27	45	0.9091	1.3355
Sample Maan		33 00		
Sample Std. Deviation		8 20		

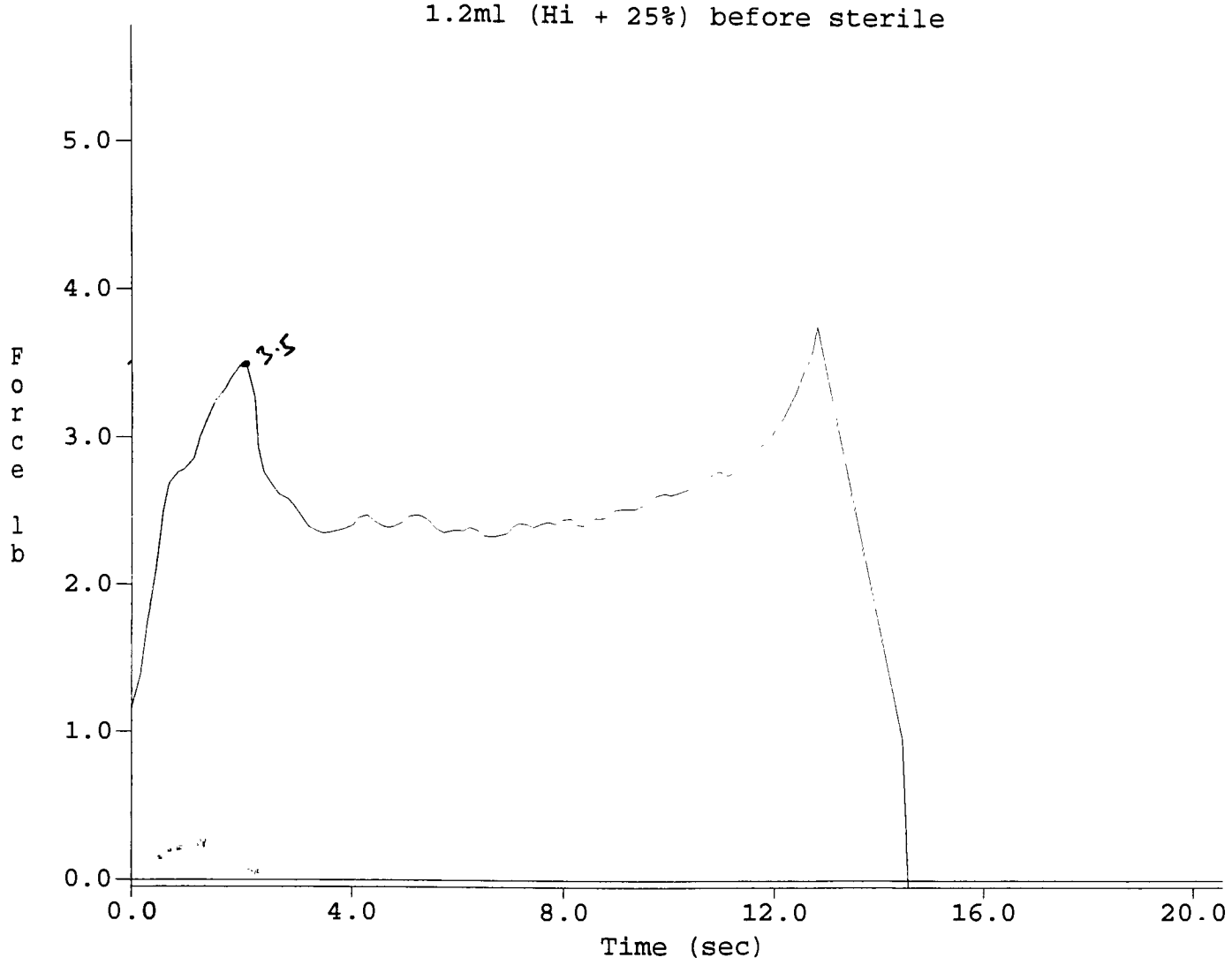
t-Test: Two-Sample Assuming Unequal Variances

	60 µ cross-sectional	60 µ longitudinal
Mean	54.16666667	33
Variance	28.67142857	67.16049383
Observations	8	10
Hypothesized Mean Difference	0	
df	15	
t Stat	6.599302131	
P(T<=t) one-tail	4 22329E-06	
t Critical one-tail	1.753051038	
P(T<=t) two-tail	8.44659E-06	
t Critical two-tail	2.131450856	

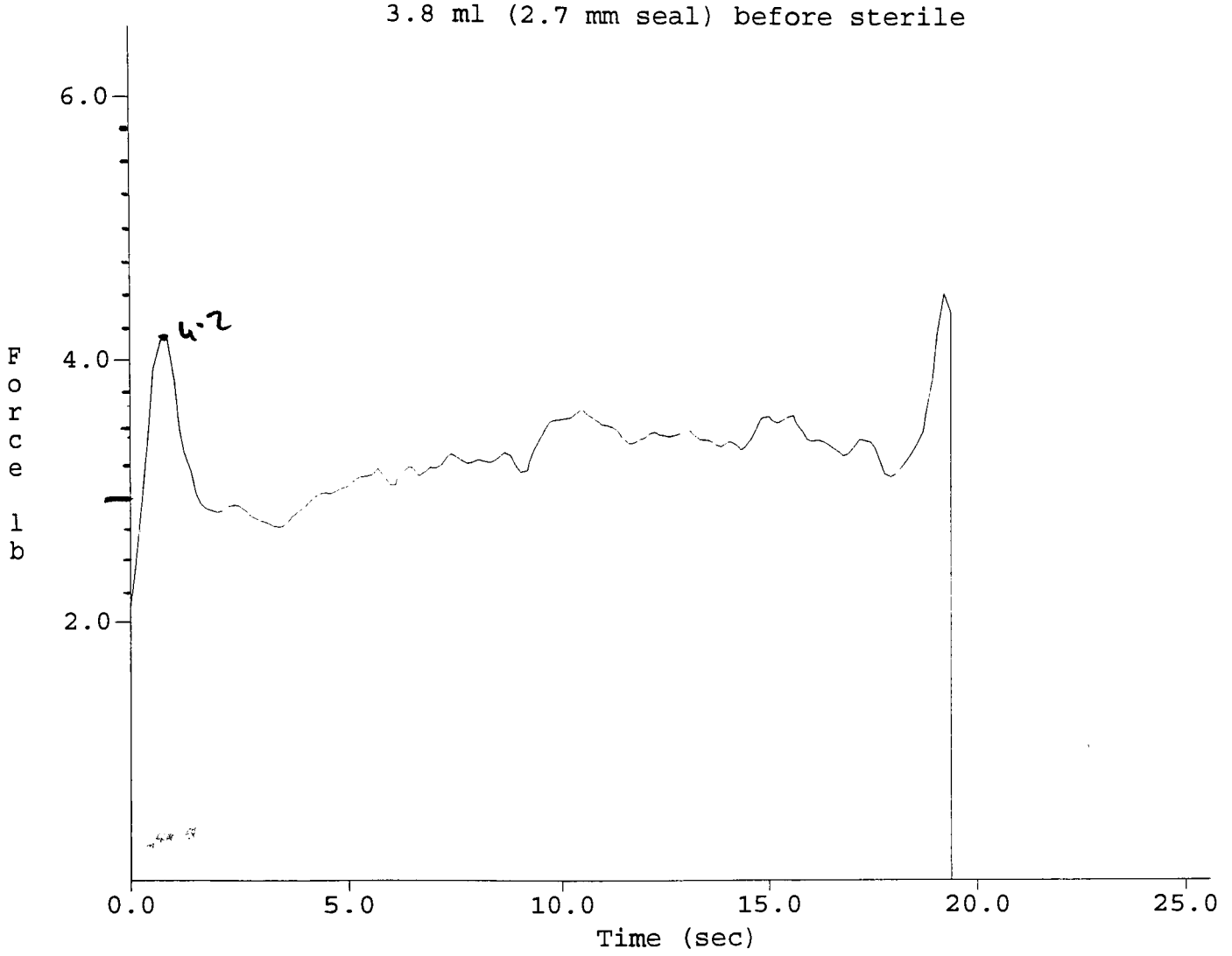


Normal Probability Plot			
Line Position	Description	*Pearson r*	r^2
Top	70 µ foil (cross-sectional direction)	0.9472	0.8972
2nd	60 µ foil (cross-sectional direction)	0.9399	0.8834
3rd	70 µ foil (longitudinal direction)	0.9791	0.9586
Bottom	80 µ foil (longitudinal direction)	0.9737	0.9480

1.2ml (Hi + 25%) before sterile



Maximum :	3.7890 lb
Average :	2.5837 lb
Minimum :	-0.0460 lb
Area :	37.5969 lb.sec
Top Marker Position (x) :	14.5600 sec
Top Marker Value (y) :	-0.0460 lb
Bottom Marker Position (x) :	0.0000 sec
Bottom Marker Value (y) :	1.1680 lb



Maximum :	4.5390 lb
Average :	3.2968 lb
Minimum :	2.1160 lb
Area :	64.0839 lb.sec
Top Marker Position (x) :	19.3900 sec
Top Marker Value (y) :	4.3980 lb
Bottom Marker Position (x) :	0.0000 sec
Bottom Marker Value (y) :	2.1160 lb

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