CLOSURE INTEGRITY TESTING OF HEAT SEALED ASEPTIC PACKAGING USING SCANNING ACOUSTIC MICROSCOPY

by

Bruno P. Jarrosson

Thesis submitted to the Faculty of the
Virginia Polytechnic Institute and State University
in partial fulfillment of the requirement for the degree of

MASTER OF SCIENCE
in
Food Science & Technology

APPROVED:

Joseph E. Marcy

Dr. Joseph E. Marcy, Chair

Kenneth C. Diehl Jr — John C. Duke

Dr. Kenneth C. Diehl — Dr. John C. Duke

February 11, 1992
Blacksburg, Virginia

CLOSURE INTEGRITY TESTING OF HEAT SEALED ASEPTIC PACKAGING USING SCANNING ACOUSTIC MICROSCOPY

by

Bruno P. Jarrosson

Joseph E. Marcy, Chair

Food Science & Technology

(ABSTRACT)

The objective of this study was to determine the possible application of ultrasonic inspection for non-destructive, on-line evaluation of the integrity of heat sealed, flexible package structure commonly used in packaging of aseptic and shelf-stable food products.

A scanning acoustic microscope (SAM), Olympus UH-3, and image analysis system were used to establish the operational parameters to ultrasonically inspect the heat seal closure of various flexible packages. The frequency range, attenuation, and focal length (Z-value) were determined respectively for paper laminate containers, plastic and plastic/aluminum pouches and plastic trays with plastic or plastic/aluminum lidding materials.

The SAM images of channel leakers, blisters and wrinkles were sufficiently characteristic to allow their identification. The same should be possible in an on-line, ultrasonic testing device through proper design of the

transducers and scanning mechanism of the inspection system and by monitoring of the ultrasonic signal. Channel leakers of 20 μm diameter were successfully detected in all package structures with the exception of the paper laminate which scattered the ultrasonic waves. The frequency used for inspection ranged from 30 to 100 MHz and best results were obtained when focussing at the seal bottom surface. As a general rule, lower frequencies were used for inspection of relatively thick seals or laminates containing an aluminum layer. Geometry, thickness, surface characteristics, and laminate composition of the seal to be inspected were found to affect SAM's performance. The SAM was able to detect defects as small as 20 μm when working in the pulse mode, using focussed transducers of frequency ranging between 30 and 100 MHz. However, because smaller defects could not technically be manufactured at the seal interface, this value is not definite and it is believed that smaller defects could be successfully detected, especially in the higher frequency range. For optimum results, seals to be inspected should be free of embossment, flat, and should remain parallel to the surface of the transducer during inspection. Finally, bio-tests showed that a 20 μm channel leaker in a seal of 5 mm width was of sufficient size to cause post-process contamination in Meal Ready to Eat (MRE, plastics/Al structure) pouches.

ACKNOWLEDGEMENTS

I wish to express my sincere gratitude to my research advisor, Dr. Joseph E. Marcy, for his suggestions, guidance and assistance throughout this study and the preparation of this thesis.

I would like to extend further appreciation to my committee members, Dr. Kenneth C. Diehl and John C. Duke, for their interest, advises, and inputs throughout my research work.

Very special thanks to Marshal H. McCord for his priceless advises and explanations on the basics of scanning acoustic microscopy, to John Chandler and Harriet Williams for their constant support and patience. I also would like to acknowledge CAPPS members who provided me with the packaging materials necessary for my research.

Finally, I want to address my deepest appreciation to my parents, Mr. and Mrs. Jarrosson, for their constant support throughout my studies in the U.S., and to my fiance, Eva Moral, for her encouragement, support, and patience.

Table of Contents

Title..i
Abstract...ii
Acknowledgements...iv
Table of Contents...v
Lists of Figures..xii
List of Tables..xiv

I. INTRODUCTION...**1**

II. LITERATURE REVIEW...................................**5**

- 1. Convenience Foods......................................5
- 2. Aseptic..6
  - 2.1. Historical Perspective.............................6
  - 2.2. Aseptic Packaging..................................7
    - 2.2.1. Requirements...................................8
    - 2.2.2. Flexible Packaging............................10
  - 2.3. Market place and Future of Aseptic................11
- 3. Heat Sealing..13
  - 3.1. Basic Principles..................................13
  - 3.2. Heat Sealing Systems..............................14
    - 3.2.1. Hot Jaws Sealing..............................14
    - 3.2.2. Impulse Sealing...............................15

3.2.3. Induction Sealing....................15
3.2.4. Dielectric Sealing...................17
3.2.5. Ultrasonic Sealing...................17
3.3. Heat Sealant Polymers..........................18
3.3.1. Films and Coatings...................19
3.3.2. Heat Sealant Properties..............20
3.3.2.1. Sealing Properties.........21
3.3.2.2. Strength Properties........21
3.3.2.3. Product Holding Properties.................22
3.4. Flexible Packages Defects....................23
3.4.1. Seal Delamination....................23
3.4.2. Seal Contamination...................24
3.4.3. Seal Deformation.....................25
4. Package Integrity....................................26
4.1. Historical Perspective.........................26
4.2. Purposes of Package Integrity Testing........27
4.3. Requirements to Ensure Flexible Package Integrity..............................29
4.3.1. Critical Points of Package Integrity Testing....................29
4.3.2. Equipment Design and Process Parameters............................30
4.3.3. Quality Control......................30
4.4. Destructive Testing............................31

4.4.1. FDA/BAM Tests for Retortable Pouch Integrity........................31
    4.4.1.1. Visual Examination.........32
    4.4.1.2. Internal Pressure Tests....33
    4.4.1.3. Seal Strength Test.........34
    4.4.1.4. Bond Strength Test.........35
    4.4.1.5. ASTM Physical Abuse Test...35
4.4.2. FDA/BAM Tests for Aseptic Semi-Rigid Package Integrity.........36
    4.4.2.1. Visual Examination.........36
    4.4.2.2. Electrolytic Testing.......37
    4.4.2.3. Ink Penetration Test.......38
4.4.3. Miscellaneous Tests..................38
    4.4.3.1. Helium Leak Test...........39
    4.4.3.2. Bio-Test...................40
4.5. Non-Destructive Testing.......................40
4.5.1. Purposes of Non-Destructive Testing in Food Packaging............41
4.5.2. Non-Destructive Testing Methods Currently Available..................42
    4.5.2.1. Pressure Differential Tests.......................43
    4.5.2.2. Package deformation Tests.......................44
    4.5.2.3. Acoustic Tests.............45

4.5.2.4. Voltage Differential Tests........................45

4.2.4.5. Ultrasonic Tests...........46

5. Scanning Acoustic Microscopy..........................46

6. Ultrasonic Testing of Materials.......................47

6.1. Basic Principles................................47

6.2. Advantages and Disadvantages of Ultrasonic Inspection.........................48

6.3. General Characteristics of Ultrasonic Waves..49

6.3.1. Frequency, Wavelength, Velocity and Amplitude.........................49

6.3.2. Wave Propagation.....................50

6.4. Major Variables in Ultrasonic Inspection.....52

6.4.1. Frequency.............................52

6.4.2. Acoustic Impedance...................53

6.4.3. Refraction at Interface and Mode Conversion...........................56

6.5. Attenuation of Ultrasonic Waves..............57

6.6. Pulse-Echo Inspection Method.................59

6.6.1. System Components for Pulse-Echo Ultrasonic Inspection................59

6.6.2. Presentation of Pulse-Echo Data......61

6.6.2.1. A-Scan.....................62

6.6.2.2. B-Scan.....................62

6.6.2.3. C-Scan....................63

III. OBJECTIVES OF THE STUDY....................................64

IV. MATERIALS AND METHODS....................................65

1. Materials....................................65
    1.1. Bricks....................................65
    1.2. Pouches....................................65
    1.3. Trays....................................66
2. Heat Sealing....................................66
3. Defects Manufacture....................................67
4. Scanning Acoustic Microscopy....................................70
5. Bio-Test....................................71
6. Defect Detection and sizing....................................73
    6.1. Optical Microscopy....................................73
    6.2. Electrolytic Testing....................................73
    6.3. Ink Penetration Test....................................74

V. RESULTS AND DISCUSSION....................................76

1. Scanning Acoustic Microscopy....................................76
    1.1. Defect Detection Results....................................76
    1.2. Operational Acoustic Parameters....................................78
        1.2.1. Frequency Range....................................78

1.2.2. Attenuation............................80
1.2.3. Z-Value and Focal Length.............81
1.3. Acoustic Evaluation of Packaging Structures....................................82
1.3.1. Bricks..................................82
1.3.2. Extrusion Laminated Pouches..........83
1.3.3. Plastic/Aluminum Laminated Pouches..............................84
1.3.4. Coextruded Plastic Lidding...........87
1.3.5. Plastic/Aluminum Lidding.............92
1.4. Criteria for Choosing Ultrasonic Inspection as a Non-Destructive Methods for Heat-Seal Integrity Testing.............................92
1.4.1. Attribute Properties.................95
1.4.2. Assessment Procedure Requirements.........................95
1.4.3. Application Constraints..............97
1.4.3.1. Time of Inspection.........97
1.4.3.2. Seal Geometry and Structure..................97
1.4.3.3. Topography................100
1.5. Machine Specifications for an On-Line Testing Device................................101
2. Bio-Testing.....................................101
3. Channel Leaker Size Determination..................104

3.1. Optical Microscopy....................104

3.2. Electrolytic Testing....................105

VI. SUMMARY AND CONCLUSIONS....................109

VII. SUGGESTIONS FOR FURTHER RESEARCH....................111

VIII. REFERENCES....................112

VITA....................122

LIST OF FIGURES

Figure 1. Curved-Bar Sealer Allowing Sealing Through Contaminated Seals................................16

Figure 2. Wavelength, Compression, Rarefaction and Amplitude of a Typical Sinusoidal Wave...........51

Figure 3. Essential Components of a Basic Pulse-Echo, Ultrasonic, Inspection System....................60

Figure 4. Typical A-Scan of a Package Seal.................77

Figure 5: SAM Image at 100 MHz of a 20 μm Channel Leaker Located at the Seal Interface of an Extrusion Laminated Plastic Pouch................85

Figure 6: SAM Image at 50 MHz of a 20 μm Channel Leaker Located at the Seal Interface of a Plastic/Aluminum Laminate Pouch..................86

Figure 7: SAM Image at 100 MHz of a 20 μm Channel Leaker Located at the Seal Interface of a Tray Sealed with Coextruded Plastic Lidding..................88

Figure 8: SAM Image at 50 MHz of a 20 μm Channel Leaker Located at the Seal Interface of a Tray Sealed with Coextruded Plastic Lidding..................90

Figure 9: SAM Image at 30 MHz of a 20 μm Channel Leaker Located at the Seal Interface of a Tray Sealed with Coextruded Plastic Lidding..................91

Figure 10: SAM Image at 50 MHz of a 20 μm Channel Leaker Located at the Seal Interface of a Tray Sealed with Plastic/Aluminum Lidding....................93

Figure 11: SAM Image at 30 MHz of a 20 μm Channel Leaker Located at the Seal Interface of a Tray Sealed with Plastic/Aluminum Lidding....................94

Figure 12: Electrolytic Testing Current Intensity (mA) Over Channel Leaker Diameter (mm)..............106

LIST OF TABLES

Table 1: Operating Frequencies of the Scanning Acoustic Microscope and their Respective Wavelength in Water..54

Table 2. Sealing Temperature, Pressure, and Dwell Time for Different Packaging Structures................68

Table 3: Frequency Range, Z-Value, and Attenuation Settings Used for the Detection of a 20 μm Channel Leaker Located at the Seal Interface of Five Types of Package............................79

Table 4: Acoustic Impedances of Various Materials Used in Semi-Rigid Food Packaging.................99

Table 5: Percentage of Pouches Showing Positive Swelling, Turbidity and Presence of CO_2 After the Bio-Test...............................103

Table 6: Regression Data and Equation Calculated from Data Measured at Various Voltages and Salt Concentrations..................................107

I. INTRODUCTION

Packaging has always played a leading role in shaping the food industry and today is no exception. Convenience in food packaging was first introduced with the canning process developed by Nicolas Appert in 1795. For the first time food was preserved by the exclusion of harmful microorganisms, making the package not only a convenience, but an integral part of the food preservation process. Changes brought by today's trends in consumer behavior, advances in food processing, and new packaging technologies are sparking a second revolution in convenience foods. Shelf-stable, microwaveable entrees and chilled, modified atmosphere packaged (MAP) entrees lead a long list of convenience foods being made possible by new packaging technology (Morris, 1989).

The method most commonly used to close these new plastic containers is to heat seal a barrier lidding structure to the bowl or tray using conductive or inductive heating equipment. Methods to non-destructively test seal integrity of rigid container such as metal cans or glass jars has lead to confidence by both processors and consumers in these packaging forms. However, there does not exist today a way to evaluate semi-rigid and flexible containers in a similar manner. These convenient easy-to-open containers are of increasing demand in the U.S. and worldwide. Approximately 35 billion food containers are used every year in the U.S. of which approximately 12 billion are semi-rigid and flexible containers with heat seal closures. Foods most often packaged in this type of container include creamers, jams, margarine, juices, pudding, applesauce, yogurt, sauces and salad dressing. This group of foods does not pose potential public health problems because they are high-acid foods or are refrigerated during storage and distribution. However, use of semi-rigid and flexible packaging for low-acid foods (pH 4.6 or higher) has brought the issue of heat seal integrity into question by both food processors and regulatory agencies. Shelf-stable, low-acid foods packed in multilayer plastic containers depend upon the hermetic seal in providing a commercially sterile product to the consumer (Lampi, et al., 1976).

According to a 1989 Schotland research study (Morris, 1989), the microwaveable, shelf-stable segment of the U.S. food market accounted for $250 million in sales in 1988 and will reach $696 million by 1996. Morris (1989) predicts sales in this category will not be limited by consumer purchases through the end of this century, but rather by lack of packaging equipment. Although the future of this new generation of convenience foods might appear to be bright, it will be essential for the food processors to implement on-line seal inspection to assure the same high degree of safety as found in traditional rigid containers (Morris, 1989).

Development of the retortable pouch, which first prompted the investigation of heat seals, can be used as a historical perspective to the problem and importance of heat seal inspection for low-acid, shelf-stable foods. Retortable pouches were developed during the 1960s as a heat sealable replacement for the metal can. They were potentially less expensive, lighter in weight, easier to dispose, more convenient to use than metal cans, and produced a superior product in many cases. However, the regulatory requirement for 100% visual inspection, both before and after thermal processing, was one major impediment to the pouch's success. Visual inspection was deemed necessary because of general lack of confidence in the integrity of heat seals and because an objective non-destructive method to assess seal quality had not been developed. Fortunately, heat sealed semi-rigid packages have not been subjected to the same requirement, but there still is not a system for continuous, non-destructive evaluation as is available for rigid containers (Downes, 1989).

Consequently, a better understanding of heat seal closures and development of an on-line, non-destructive inspection system is needed for the advancement of semi-rigid, heat sealed containers. A scientific examination of heat seals should lead to an improved control of heat seals and better container integrity. As more shelf-stable foods are packaged in heat sealable containers, a reliable method to non-destructively test these containers will be needed by both food processors and regulatory agencies.

II. LITERATURE REVIEW

1. Convenience Foods

Increasing demands for convenience and new technologies such as modified atmosphere packaging, aseptic processing, and microwave heating make it a challenging world for packaging materials. Today, packages should not only contain and protect, but also be easy to use, light-weight, shatter resistant, microwaveable, and recyclable. Because of these new demands on packaging, the opportunities for plastics in food and beverage packaging are numerous. By the year 2000, plastic's share of the packaging market will increase to 30% versus 17% in 1989. Plastics are expected to replace a substantial amount of glass containers and metal cans. This substitution is already apparent on supermarket shelves and was made possible through complex multilayered packaging structures. In order for plastic packaging to keep growing as fast as it has, three critical hurdles will have to be overcome by the packaging industry. Material costs will have to be reduced and recyclability taken into account by developing more effective resins and structure. Finally, and not the least, plastic containers will have to prove themselves as reliable and efficient in protecting and maintaining the quality and integrity of the foods they contain, as metal cans and glass containers have proven to be (Fox, 1989).

2. Aseptic

The aseptic process consists of the independent sterilization of a food product and its container, followed by filling and hermetic sealing in a sterile environment (ASTM, 1989).

2.1. Historical Perspective

The concept of aseptic packaging of foods is not new. The first aseptic process to be patented was developed in Denmark in 1921 by J. Nielsen, and the first food to be successfully aseptically processed was cream packed in a glass container by the Avoset Foods Corporation in the early 1940s. The first commercialized aseptic filling plant to open in the United States was built by James Dole Inc. in 1950 (Reuter, 1989). While the aseptic process developed rapidly in Europe in the 1970s with the introduction of Tetrapak Inc.'s paperboard/foil brick, the later did not appear in the United States until the FDA approved hydrogen peroxide for sterilization of coated paper board containers in 1981 (Reuter, 1989).

Because the materials used in aseptic packaging are not subjected to high temperature and pressure, a variety of light-weight, flexible packaging materials such as plastics and paper became available for aseptic packaging (Hammond and Potts, 1989). However, aseptic packaging being a new and more complex technology than conventional packaging methods, it is more vulnerable and more susceptible to potential errors (Reuter, 1989).

2.2. Aseptic Packaging

An hermetically sealed aseptic container is a package maintaining the commercial sterility of its content by preventing the entry of microorganisms which is necessary for the preservation of aseptically package foods (ASTM, 1989).

In order for a food to remain commercially sterile, the package in which it is contained must prevent post-process contamination until opened. The canning process has proven itself reliable and established that the number of food poisoning outbreaks associated with post-process contamination of canned food is extremely small compared to the total canned food production. Aseptic packaging, being a relatively new process, does not have the benefit of the safety record of canning or of the technologies which have been developed to assure container integrity. Nevertheless, aseptic packaging presents a major advantage in that the process does not require cooling in the container, which is a major source of post-process contamination in canning due to pressure differential between the container and its environment. Sealing of containers used in aseptic is mostly done by heat sealing in which two compatible polymeric material surfaces are fused together. While numerous methods are available for defect detection in empty containers, the inspection of filled and sealed containers is most often performed by visual inspection and sampling. Because empty containers can be adequately inspected, often on-line, the seal, formed after filling, is the critical point of inspection in a aseptic container (Downes et al., 1985).

2.2.1. Requirements

Requirements for packaging materials used in aseptic are identical to those for other types of food packaging (Maros, 1985). Prior to 1958, the Food and Drug Administration (FDA) had to prove that substances present in foods were not rendering the food injurious to health. In 1958, with the Food Additives Amendment, it became the responsibility of food processors to prove the safety of all food additive components including any materials used in packaging. Consequently, unless generally regarded as safe (GRAS) or approved by the FDA prior 1958 (prior sanction), all materials used for aseptic packaging must fulfill a numbers of requirements before being approved by the FDA or the food processor. They must be unaffected by the sterilization system, provide sufficient barrier to moisture and gas transfer, prevent transfer of any tainted or off-flavors, or contain toxic or harmful substances, and finally be hermetically sealable and stable during the product shelf-life (Dunkelberger, 1985).

Beside fulfilling the food processors and regulatory agencies requirements, the container and packaging materials must also satisfy the consumers' demands. In a world where changing lifestyles and new technologies sparked a second convenience food revolution, the consumers expect packaging, not only to protect and contain, but also to be microwaveable, light-weight, shatter resistant, squeezeable and easy to open (Drimmer, 1985). The most important property of food packaging should not be overlooked, for as Dunkelberger (1985) said: "no package or packaging materials will achieved commercial success if it does not adequately meet its primary obligation-to protect its intended contents".

2.2.2. Flexible Packaging

Flexible containers used in aseptic packaging can be grouped into three basic categories based on the Flexible Package Integrity Committee of the National Food Processor Association (NFPA): paperboard packages, flexible pouch packages, and plastic packages with heat sealed lids. A fourth category, plastic cans with double seamed metal ends, was defined but will not be discussed since it does not apply to aseptic packaging (NFPA, 1989).

The first flexible packaging structure to be used in aseptic was a paperboard package consisting of a polyethylene (PE)/paperboard/PE/foil/adhesive/PE laminate. It is currently used in capacity ranging from 200 to 1894.4 ml. The packaging materials are sterilized either as rolled stock or after forming using a combination of hydrogen peroxide and heat as sterilants (Hammond and Potts, 1989). Plastic containers used in aseptic packaging can be either thermoformed or injection molded. A wide variety of thermoplastic materials can be used in aseptic packaging as long as they have sufficient thermal stability and are cleared for food contact by the FDA. Because in thermoform-fill-seal process, the packaging materials are sterilized as a rolled stock before being formed, filled and sealed under aseptic conditions, this process is favored in aseptic packaging as opposed to depositfill-seal process where prefabricated containers are sterilized before being filled and sealed under aseptic conditions (Hahn, 1989).

2.3. Market Place and Future of Aseptic

Ever since the FDA approval of hydrogen peroxide for sterilization of flexible packaging in 1981, aseptic packaging of foods and beverages has rapidly expanded (Milliner, 1985). With only 10 years on the U.S. market and 3 billions packages in 1989, aseptic packaging was the first packaging technology to take over such a large volume of the market in such a short time (Brody, 1989). This success of aseptic packaging on the American market has been mainly in the area of high-acid products where post-process contamination of the product by pathogenic bacteria represents a low risk due to the low pH of the product (Downes, 1989).

While in its infancy, aseptic packaging of foods and beverages was mainly limited to fruit juices in boxes and single serving applesauce. Food processors are now trying to expand the current aseptic market to a new range of products and consumers. Both the adult beverage market and low-acid food products are regarded as "untapped, fertile territory" (Trager, 1991). If aseptic packaging of low-acid food products has yet to be fulfilled, as Swientek (1991) pointed out "extensive and collective research efforts plus new technology point to a bright future". Low-acid food products, some of which containing small particulate, have been successfully processed since 1988. A mathematical model for predicting heat transfer into particulate developed by the NFPA allowed the design of acceptable methodologies for validating thermal processes for low-acid foods containing particulate (Swientek, 1991). While the opportunity for growth of low-acid aseptic packaging might appear very good from a technological point of view, as Milliner (1985) mentioned, "aseptic low-acid foods will have to out-perform the other options available to the consumer" in order to be successful. Nevertheless, in a survey on food packaging done by Food Processing magazine in 1989, 23.4% of 958 companies indicated their intention to launch aseptic products in the 90s. Both high and low-acid foods, ranging from juices to entrees and side dishes, were specified as targets (Rice, 1990).

3. Heat Sealing

3.1. Basic Principles

Heat sealing is the primary method of closing non-rigid packages and can be defined as a process in which two compatible polymers are fusion welded by providing heat at their interface, and maintaining them in close contact by applying pressure for a given period of time (Downes et al., 1985; Young, 1986). A fusion weld is defined as a bond obtained by combining two materials through melting or other means so that the sealing interface becomes indistinguishable (Lampi et al., 1976; Downes et al., 1985).

Flexible packaging materials for heat sealing can be categorized as unsupported films consisting of one or more thermoplastic materials, and coextruded or laminated materials consisting of non-thermoplastic materials with a thermoplastic inner ply for sealing purpose (Downes et al., 1985; Young, 1986).

The quality of a seal is controlled through the sealing temperature, pressure and dwell time. In order to obtained a good seal, it is crucial that both surfaces to be sealed be exactly parallel and equal in length, and that the sealing temperature be within range of the melting temperature of the sealant (Downes et al., 1985).

3.2. Heat Sealing Systems

Heat sealing can be achieved by a number of techniques mainly varying in the way the sealing interface is heated. These techniques include hot jaws, impulse, induction, dielectric and ultrasonic sealing (Downes et al., 1985).

3.2.1. Hot Jaws Sealing

Hot jaws or bar sealing consists of two sealing jaws, one or both being heated by a resistance. It is commonly used in the food industry for sealing flexible pouches or lidding materials to plastic cups and trays. The obtention of an hermetic seal being desired in most food packaging applications, the use of a sealer with a flat-faced heater bar opposed to a bar covered with silicone rubber is most common. The flexibility of silicone rubber is desired to compensate for variations in the flange thickness of the container as well as sealer bars that are not perfectly parallel, allowing for uniform pressure during sealing. The edges of the heater bar are often gently rounded to prevent puncturing the packaging materials. In order to seal despite grease or water contamination, either the silicone rubber bar or heater bar can be curved allowing the pressure to expand from a line to a band (Young, 1986).

Figure 1 illustrates the ability of such a sealing bar to seal through grease and moisture contamination, whereas a flat bar sealer fails to do so (Lampi et al., 1976).

3.2.2. Impulse Sealing

In impulse sealing, heat is applied to the seal interface by an electric current passing through a nichrome ribbon laid over one or both sealing bars. A flexible surface such as silicone rubber is usually present over the surface of the sealing bar and the nichrome ribbon(s) covered with an electrically insulating, high temperature, release film such as teflon coated fiberglass. Although impulse sealing cannot operate at high speeds compared to hot jaws sealing, it presents the advantage of allowing the seal to cool down while remaining under pressure. This characteristic is especially useful when sealing materials with poor hot tack property. The high temperature release film presents another advantage in that it prevents unsupported thermoplastic films to stick to the heated bar and fall apart when the bars are opened (Downes et al., 1985; Young, 1986).

3.2.3. Induction Sealing

Induction sealing can only be used when the lidstock

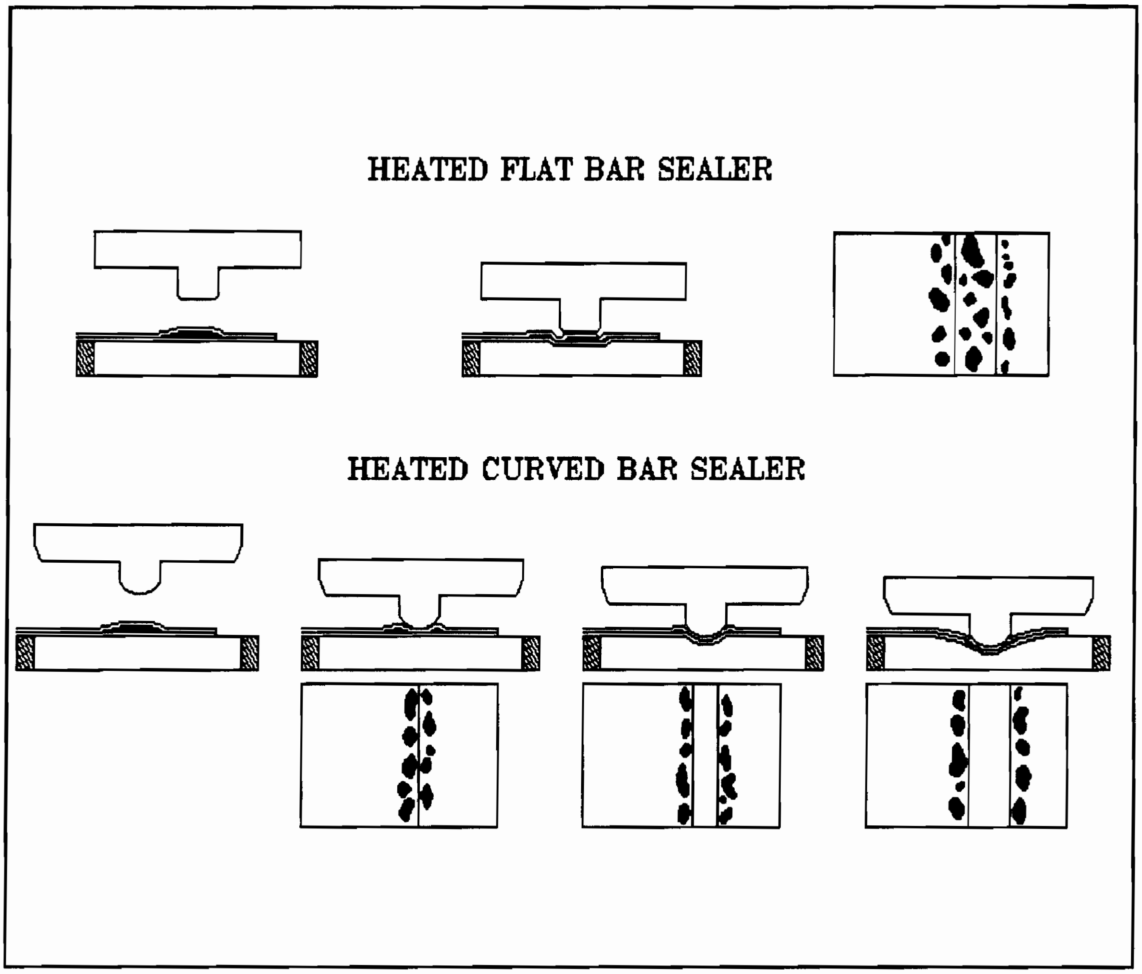

FIGURE 1: CURVED-BAR SEALER ALLOWING SEALING THROUGH CONTAMINATED SEALS (Lampi et al., 1976).

contains an electrically conductive material such as aluminum. The heat energy is directly applied to the seal interface by submitting the seal to an alternating electrical field which generates an inductive current in the conductive layer of the lidstock and heats the seal interface (Downes et al., 1985; Young, 1986; Toner, 1987). Induction sealing's most common application is the production of tamper-evident seal over the top of bottles (Young, 1986).

3.2.4. Dielectric Sealing

Dielectric sealing application in food packaging is limited to materials containing dipoles and is mainly used with polyvinyl chloride (PVC) and PVC coated papers. Heating of the seal interface is achieved by friction when submitting the material to a high frequency electric field while held under pressure, causing the dipoles of the sealing material to align themselves with the electromagnetic field. Dielectric sealing cannot be used for polyolefins such as PE or polypropylene (PP) (Downes et al., 1985; Young, 1986).

3.2.5. Ultrasonic Sealing

Ultrasonic transducers have been used successfully to seal thermoplastic packaging materials. The ultrasonic waves sent through the plastic materials cause them to vibrate, generating enough heat at the seal interface to fuse the plastic materials together (Bongo, 1978; Downes et al., 1985; Young, 1986). Ultrasonic sealers consist of a piezoelectric transducer generating mechanical waves/ultrasound of 20 KHz frequency which are transmitted to the plastic materials via an ultrasonic horn. As in conventional heat sealing, the heat provided to obtained the desired seal is controlled by the pressure and length of time (dwell time) applied to the plastic materials as well as the ultrasonic horn amplitude. The main inconvenience of such a sealing method lies in the power required to generate enough heat to achieve sealing. Since ultrasonic sealing is not very energy efficient, it is used only where other sealing methods cannot be applied (Bongo, 1978), such as sealing biaxially oriented films, aluminum foil or materials that are too thick to permit heat transfer (Young, 1986).

3.3. Heat Sealant Polymers

Heat sealants used in packaging are thermoplastic polymers that repeatedly soften when heated and harden when cooled. Thermoplastic polymers most commonly used in heat sealing of aseptic food packaging are PE (n = CH2=CH2) and PP (n = CH2=CH-CH3). Where low sealing temperatures are required low density PE (LDPE) and PE-ethylene vinyl acetate (EVA) copolymers are used while, when high temperature performance is required, high density PE (HDPE), PP, and ionomers are used (Downes et al., 1985). These thermoplastics polymers can be applied as coatings or films and classified as copolymers such as EVA, ethyl methyl acrylate (EMA) or Surlyn (ionomer of PE), and as homopolymers such as PE and PP. This last category represents 99% of all heat sealants used in the food industry (Martin, 1986).

3.3.1. Films and Coatings

Heat seal coating polymers for films and paper include vinyl acetate-vinyl chloride copolymers, nitrocellulose, acrylics, or poly-vinyldiene chloride (PVDC) and are applied to the substrate as solvent solutions or water emulsions. Vinyl acetate-vinyl chloride copolymers and emulsion coatings of low molecular weight EVA are frequently used on films and foils lid stock for rigid and semi-rigid (cups) containers (Martin, 1986).

Heat sealants films are incorporated in the package structure through lamination, extrusion coating or as components of coextrusions. They are mostly polyolefins and more precisely either PE or PP. PE can be found in a variety of density, melt index, and molecular weight distribution. The melting point of polymers is greatly affected by their degree of crystallinity. For this reason, HDPE and medium density PE (MDPE) which have a high degree of crystallinity have a sharp melting point and therefore are difficult to use as heat sealants. On the contrary, LDPE having a low degree of crystallinity, is ideal for heat sealing because it melts over a wide temperature range, thus slight variations in sealing bar temperature do not affect the performance of a seal (Giacin et al., 1984). Increasing density results in increased sealing temperature, heat resistance, strength and stiffness, and decreased sealing range, clarity, and barrier properties. Decreasing melt index has the same effects with the exception that barrier properties are not affected and chemical resistance is increased. Increasing the molecular weight distribution results in an increase of the sealing range (Martin, 1986).

Oriented polypropylene (OPP) is widely used as a heat sealant film in multilayer flexible packaging because it has good hot tack and seal strength properties as well as seal range (Martin, 1986).

3.3.2. Heat Sealant Properties

When choosing a heat sealant for a given application, its sealing properties, strength properties and finally product holding properties should be considered.

3.3.2.1. Sealing Properties

Sealing properties include seal initiation temperature, seal range, and hot tack. Seal initiation temperature refers to the minimum temperature at which fusion takes place. In the case of multilayer containers, the seal initiation temperature is affected by the conductivity of the other components of the containers, which more often than not are poor thermal conductors. Consequently, heat seal initiation temperature are determined experimentally at a standard pressure and dwell time which refers to the time in seconds during which the seal is maintained under pressure. The seal range refers to the effective range of temperatures at which fusion is obtained under a set dwell time and pressure. Finally the hot tack refers to the forces holding the polymer(s) together at sealing temperature immediately after removing the pressure during sealing (Martin, 1986).

3.3.2.2. Strength Properties

Strength properties of heat sealants include impact, tensile, tear and seal strength. Impact strength of a film refers to its ability to withstand shock loading. Tensile strength is a measurement of the force required to stretch a material at a constant rate to the breaking point and it is reported in pounds per square inch of cross section; in other words, it refers to the maximum stress that a material can sustain. Tear strength is a measurement of the energy absorbed by a test specimen in propagating a tear which is initiated by cutting a small nick in the sample (Briston, 1986). Finally, seal strength is the force necessary to separate a seal and it is reported in pound-force per inch width. It is often incorrectly used by food processors as a measurement of container integrity, and since it is possible to have high seal strength even if a container is leaking, seal strength cannot be taken for a measure of seal continuity (Martin, 1986).

3.3.2.3. Product Holding Properties

Product holding properties of heat-sealants refers to chemical resistance and barrier properties. Both these properties are important factors when assessing the suitability of a sealant for packaging a particular product (Martin, 1986).

3.4. Flexible Packages Defects

In 1984 the NFPA created the Flexible Package Integrity Committee to assess post-process microbial contamination caused by leakage. Flexible containers are categorized as paperboard packages, flexible pouches, plastic packages with heat sealed lid, and plastic packages with double-seamed metal ends. Defects, good manufacturing practices (GMPs) and testing methods are defined for each package category and the defects are classified as either "critical" when loss of integrity is apparent, "major" when loss of integrity is suspected, or "minor" when not having any adverse effect on integrity (Kopetz, 1987; NFPA, 1989; Denny, 1989).

When considering heat seal closure, the defects defined for each package category, excluding plastic packages with double-seamed metal ends, can be classified as being caused by delamination, contamination, or deformation of the seal.

3.4.1. Seal Delamination

Defects caused by seal delamination include channel leakers and incomplete seals. Channel leakers are defined by the NFPA as "a patch of non-bonding across the width of the seal creating a leak" while incomplete seals are defined as "a portion of the seal that has a lack of adhesion between lid and body". Seal delamination occurs in a blister, but in this case is usually brought about by seal contamination (NFPA, 1989).

Seal delamination can be avoided by carefully monitoring operational heat seal parameters. Insufficient temperature, dwell time, and/or pressure will results in non-fusion of the seal or delamination. Such a problem is frequently encountered when operating the sealing equipment at too a high speed, which does not leave enough time for the sealing bars to regain the required sealing temperature (Downes et al., 1985). Other factors resulting in seal delamination are uneven temperature and pressure distribution across the sealing area. Such problems can be avoided by using sealing bars designed to provide an even pressure distribution around the seal, and made of a material as conductive as possible to prevent uneven temperature distribution (Downes et al., 1985).

3.4.2. Seal Contamination

Defects caused by seal contamination include contaminated seals and blisters which are respectively defined by the NFPA as "foreign matter in the seal area such as, but not limited to, water, grease, or food" and as "a void within the bonded seal" (NFPA, 1989).

Seal contamination can be minimized by increasing seal width, carefully monitoring and designing filling operation, vacuumization, if any, and package handling prior sealing (Lampi et al., 1976). If seal contamination remains a problem despite proper monitoring of these operations, sealing bars should be designed to seal through gross grease or water contamination, and a defect detection system should be used whenever available. (Lampi et al., 1976; Downes et al., 1985).

Effects of seal contamination on retortable pouch seal quality has been studied by the Natick Development Center by enclosing pieces of rubber (1.6 x 1.6 x 0.8 mm) into seals of 32 and 64 mm width. The study showed that contamination had no apparent effect on burst strength, but that 11% of the 32 mm width seals failed during retorting (Lampi et al., 1976).

3.4.3. Seal Deformation

Defects caused by seal deformation are classified as wrinkles or convolutions by the NFPA, and are respectively defined as "a fold of material in the seal area" and "a slight visual impression in the seal indented on one side and raised on the other side" (NFPA, 1989).

The most reliable way to avoid seal deformations such as wrinkles during sealing is to hold the area of the container to be sealed under tension to assure that the opposing surfaces to be sealed are flat and parallel (Downes et al., 1985; Young, 1986). However, this is not always possible, in which case alternatives are, either to increase the thickness of the sealant, or to use PE/EVA copolymer or ionomers as sealant. These changes allow for better tolerance of small wrinkles by improving flow properties during sealing (Young, 1986).

4. Package Integrity

4.1. Historical Perspective

The issue of container integrity has been recognized as critical ever since the canning process was developed; Nicolas Appert himself mentioned the problem of leaker spoilage of canned foods (Put et al., 1980). Although no recorded date can be found in literature to determined when containers were first inspected for integrity, it is believed that such inspection started in 1940, with the first reported case of botulism outbreak. Furthermore, Kopetz (1987) reported C. botulinum type E as the only toxin type implicated in an outbreak involving container leakage, and this was not recognized as an etiological agent until 1936.

In 1964, in order to answer a question asked by the FDA commissioner, regarding the safety of canned food containers, the NFPA declared the cans used at the present time as being as safe as the cans used in the past. Most of the regulations on the acceptance quality levels for canned food container defects were first developed by the United State Department of Agriculture (USDA) and canning industries in a cooperative effort. These regulations were used later on to develop the guidelines for examination of container integrity published in the Bacteriological Analytical Manual (Kopetz, 1987). Finally, a metal can integrity task force was formed in 1982 by the NFPA and Can Manufacturers Institute (CMI) to establish "Good Manufacturing Guidelines for Quality Assurance of Metal Food Cans". "Hold for Investigation" (HFI) guidelines for low acid foods were developed and work was done to improve leak detection in filled containers. In 1984, after recognizing the increasing importance of flexible containers, the NFPA subsequently created the Flexible Package Integrity Committee (Kopetz, 1987; NFPA, 1989; Denny, 1989).

4.2. Purposes of Package Integrity Testing

When considering the issue of package integrity in the food industry everyone agrees that proper package testing procedures should be developed to assure the hermeticity of the package. An hermetically sealed container is defined under the 21 Code of Federal Regulations (CFR) 113.3 as "a container that is designed and intended to be secure against the entry of microorganisms and thereby to maintain the commercial sterility of its contents after processing" (Wurts, 1985). From the consumer's point of view, package integrity and package quality are often perceived as synonymous, consequently, there is no faster way to unsell a consumer than with a leaky package (Kopetz, 1987). The bottom line when considering package integrity is to "keep the bugs out" (Wurts, 1985), meaning that the diameter of a microhole in the walls or seal of a package should not exceed 0.5 μm (Wurts, 1985; Axelson et al., 1990).

Consequently, any testing methods should, in theory, have such sensitivity. However, due to other factors such as hole length and surface properties, growth possibility in the vicinity of the hole, and bacteria concentration, this tolerance limit probably lies higher than 0.5 μm (Axelson et al., 1990). Gilchrist et al. (1985) reported that cans with direct hole diameter greater than 5 μm became contaminated when cooled in water containing 10^6 CFU/ml of E. coli LT 13. Similar work on retortable flexible pouches having a thickness of 127 μm showed that a direct hole of at least 20 μm was necessary for post process contamination to occur (Gilchrist et al., 1989).

4.3. Requirements to Ensure Flexible Package Integrity

4.3.1. Critical Points of Package Integrity Testing

When considering the flexible containers used in aseptic packaging which consist of a laminate of two or more films, the probability of leakage through discontinuities is almost insignificant since it is very unlikely to have discontinuities located on top of one another. For this reason, the major cause of leakage in flexibles is through defective seals (Amini and Morrow, 1979). Hermetic sealing being crucial to maintain the sterility of an aseptic container, therefore, it is important that the sealing surface form a good fusion weld. When such a weld is obtained, the sealing interface should not be visible, and package failure upon application of tensile forces should not occur at the seal interface but between plies of the packaging material (Downes et al., 1985). Because ease of opening is also critical to the consumer, optimum sealing conditions cannot always be used (Amini and Morrow, 1979). This challenging problem has been partially solved by several seals, one of which is the EPOCH seal where the heat seal and opening mechanisms are separated (Hatano and Taira, 1991).

4.3.2. Equipment Design and Process Parameters

In order to ensure container integrity on a production line, the packaging materials need to be carefully inspected for defects and care should be taken in handling empty containers. The filling machine should be designed to avoid possible spillage on the seal area and heat seal parameters set to obtain fusion of the seal. The sealing machine should be designed to ensure that sealing bars and container holder are perfectly parallel. The sealing bar should be designed to allow even temperature and pressure distribution around the seal and to minimize contamination of the seal area (Downes et al., 1985).

4.3.3. Quality Control

The design of an adequate quality control (QC) program for inspection of packaging after filling is necessary since acceptable defect levels are unattainable by close monitoring of production equipments and parameters alone. Because non-destructive testing methods for 100% on-line inspection of filled, heat sealed containers are not yet available, seal integrity has to be evaluated using standard testing methods such as seal strength, pressure testing, bubble test, gas detection, electrolytic testing, ink penetration, and bio-testing (Downes et al., 1985). It is of crucial importance that all testing methods used in a QC program be carefully standardized because only effective in-plant test methods and a QC program can maintain seal integrity performance in flexible package at a level matching that of metal cans. Standard procedures for most methods used in leak detection are published in the ASTM standards, Section 15, Volume 9 under the title *General Products, Chemical Specialties, and End Use Products*, as well as in Chapter 24 of the *Bacteriological Analytical Manual* (BAM) published by the Association of Official Analytical Chemists (AOAC).

4.4. Destructive Testing

4.4.1. FDA/BAM Tests for Retortable Pouch Integrity

The importance of closure performance and integrity of containers used for packaging of thermally processed foods has long been recognized. Whether a closure consists of a tight mechanical clinch such as for cans, or a fusion weld as for most flexible and semi-rigid packages, characteristics of a reliable closure and parameters which affect them need to be well defined to keep the defect rate as low as possible. However, this is not sufficient to ensure package integrity, and consequently testing procedures need to be developed to assess seal quality. The common can with a 0.01% failure rate is often used as a performance standard for other food containers. This was the case with the retortable pouch.

Burke and Schulz (Lampi et al., 1976) compared the metal can and retortable pouch by submitting them to a series of abuses simulating handling by a military distribution system. Results showed both cans and pouches resisted mishandling equally with even slightly better results for the pouch. Lampi et al. (1976) reported until on-line, non-destructive leak/seal inspection systems are available, quality assurance inspection plans need to be established and good seals must be defined based on fusion, burst and tensile testing and visual examination.

4.4.1.1. Visual Examination

Although visual examination might appear subjective and prone to human errors, when applied with proper standards and adequate monitoring of well designed equipment , it has proven to lower defect level to acceptable failure rates (Downes et al., 1985). Visual examination allows for the detection of obvious defects such as misaligned seals, burnt and contaminated seals, wrinkles, delaminations, and leakers (AOAC, 1984; Downes et al., 1985; Wurts, 1985).

4.4.1.2. Internal Pressure Tests

An internal pressure tests is used as a measure of a package's ability to withstand manufacture, distribution and storage. This test is based on the fact that when adequate sealing is obtained, the sealant is allowed to flow from the center of the seal to its edges creating a bead of material. The sealant, if evenly distributed around the seal, increases the container's resistance to internal pressure (Downes et al., 1985). Two types of tests are used to determine the ability of a pouch to withstand internal pressure. In the first case, the pouch is placed under a specified pressure for a predetermined length of time by introducing water or air at a controlled rate. The pressure is then released and the pouch inspected for failure. In the second case, the pouch is placed under increasing pressure by introducing air or water at a controlled rate until failure occurs, at which point the time and pressure are recorded as the results of the test. The three main testing devices used for internal pressure testing are the pouch air burst tester, the Reynolds Metals Flex-Can Hydro Pressure Tester, and the FMC Burst Tester for pouches and semi-rigid trays (AOAC, 1984). The internal pressure test can also be modified for leak detection by pressurizing the pouch with air and carrying the test under water (Wurts, 1985).

4.4.1.3. Seal Strength Test

Seal strength or tensile test is used as a measurement of forces required to separate a seal and is reported in newton per centimeter width. Such a test is important when a package is designed to open easily. It is frequently used to measure the sealing quality of a container, and to determine the optimal sealing parameters and sealing equipment performance. Although, this test is of obvious relevance to the integrity of a package, it cannot be used for defect detection since the stress created by most leakers, blisters, wrinkles or contamination is masked by the strength of the adjacent areas (Lampi et al., 1976; Downes et al., 1985). In other words, seal strength measurements are useful to assess seal quality, but are not sufficient to ensure seal integrity since seal strength is not a measure of seal continuity (Martin, 1986).

The effects of storage time and temperature on retortable pouches has been studied by the Natick Development Center. Pouches consisting of Polyester/Aluminum Foil as outer and middle plies and HDPE or PP as inner ply were stored over a period of 27 months at 22 and 38°C, and their respective seal and bond (between inner and middle ply) strength measured every three months. The results of the study showed that the retort process decreased seal strength and bond strength, but that storage and storage temperature had little or no effect

(Lampi et al., 1976).

4.4.1.4. Bond Strength Test

The performance of retortable pouch laminate being highly dependent on its ability to function as a single unit, it is necessary to measure the forces holding the laminate's plies together. Bond strength is measured by first delaminating the plies using an adequate solvent, and placing the free ends of the sample in the grips of a tensile testing machine, such as an Instron (Canton, MA) (AOAC, 1984). As mentioned above, bond strength was left unaffected by storage time and temperature, while a slight decrease in the bond strength was noted after retorting (Lampi et al., 1976).

4.4.1.5. ASTM Physical Abuse Test

ASTM physical abuse tests are used to simulate expected distribution conditions such as handling and shipping. After performing these tests, seal and bond strength testing as well as burst testing are usually repeated (AOAC, 1984).

4.4.2. FDA/BAM Tests for Aseptic Semi-Rigid Package Integrity

Aseptic meaning free of all microorganisms capable of growth and multiplication under normal storage and distribution conditions, a container should prevent any kind of bacterial penetration in order to remain aseptic. Because the average size of a bacteria is 0.5 µm, the walls and seals of an aseptic container should not have any discontinuity with a diameter exceeding this size. Contamination of a container will also be affected by a number of factors such as surface properties and length of the discontinuity. Consequently, the smallest defect of significance to allow post process contamination of an aseptic container is probably considerably higher (Axelson et al., 1990).

4.4.2.1. Visual Examination

Visual examination of aseptic semi-rigid package for integrity has the same purpose as for retortable pouches. It has proven to be effective in lowering failure rate to acceptable levels when coupled with appropriate equipment design and control (Downes et al., 1985).

4.4.2.2. Electrolytic Testing

Electrolytic testing is used as an indicative method for leak detection based on the fact that any real leak in a container made of insulating materials will allow current flow provided that the leak is filled with a conducting liquid (AOAC, 1984; Wurts, 1985; Axelson et al., 1990). Axelson et al. (1990) reported that the intensity of the current passing through a given leak could be correlated to its size, if the hole length and surface properties as well as voltage, salt concentration, and type of electrodes used during testing remained constant. Results showed that electrolytic testing has the ability to detect seal or body leaks as small as 0.8 μm in diameter, as long as their length is no longer than 1 mm and direct current is used for the measurement. For this same reason and because it can be performed relatively quickly electrolytic testing has been reported as an effective on-line testing method (Wurts, 1985). An inconvenience of this test method lies in the fact that when testing packaging laminates containing an aluminum layer, a defect exposing that aluminum ply will give a reading even if a true leak is not present (Axelson et al., 1990).

4.4.2.3. Ink Penetration Test

In a dye test or ink penetration test, leaks are detected using a dye solution of low surface tension to facilitate penetration of capillary leaks. Dye solutions commonly used for this test are fluorescein dye solution (Gilchrist et al., 1989) and rodhamine B or safranin O powder dissolved in isopropanol (AOAC, 1984). The ink penetration test is normally used for packages that gave a positive results when submitted to the electrolytic test. Containers to be inspected are emptied, cleaned and dried, and a few drop of dye solution are applied to all critical points (AOAC, 1984). In some cases the containers are sealed and pressure applied manually to facilitate ink penetration. The dye test has been reported by Gilchrist, et al. (1989) to detect leaks down to 20 μm in diameter.

4.4.3. Miscellaneous Tests

Two other tests are commonly used in the food industry besides those recommended by the FDA. These test are the helium leak test and the bio-test.

4.4.3.1. Helium Leak Test

Helium leak testing was first developed for cans based on an helium leak test widely used by the American Society for Metals. After being compared with conventional leak tests (vacuum and fluorescein dye tests), the helium leak test was found to be more sensitive for detecting leakage through the double seam. Furthermore, when testing can with leakers of known diameters ranging from 1 to 20 μm, a linear relationship was established between the percentage of helium present in the head space of the can and the hole diameter ($r=0.81$) (Gilchrist et al., 1985). This same method was later modified for inspection of flexible containers, and reported to be sensitive enough to detect leaks as small as 3 μm in diameter (Gilchrist et al., 1989). Because exchange between food enclosed in a flexible package and the outside environment can occur through leakage and permeation (Amini and Morrow, 1979), helium leak test and other similar gas detection control systems are all based on the assumption that gas permeation through the material is negligible. Consequently, serious errors from gas permeation can be expected if the package being tested has poor gas barrier properties (Axelson and Calvin, 1991).

4.4.3.2. Bio-Test

Bio-testing is based on the principle that a leaky container will allow bacteria present in the vicinity of the leak to contaminate its content. This leak testing method was first designed for metal containers, but was modified for integrity testing of retortable pouches by Maunder et al. (1968). Bio-testing has been used as a modification of the electrolytic test (Downes et al., 1985) and reported to have a sensitivity similar to that of the dye test but a better precision (Gilchrist et al., 1989). Even if bio tests are the most stringent testing methods, package integrity should not be assessed only based on their results (Garret, 1985).

4.5. Non-Destructive Testing

Outside the packaging industry, non-destructive evaluation of plastic and metals parts has long been necessary to decrease production cost by reducing the number of defective products. Leak testing can be achieved by detecting and pinpointing leaks or by measuring leakage rate from a system.

Leaks can be classified as real leaks referring to a through wall discontinuity in the container, or as a distributed leak referring to permeation. Methods for leak detection in a pressurized or vacuumed container include direct sensing methods such as acoustics, bubble testing and flow detection, and gas detection using infrared gas analyzer, mass spectrometer or gas chromatograph. In all these methods, detection relies on pressure difference between the container and the surrounding environment to cause a fluid to flow in or out of the container being inspected. Therefore, these methods truly detect leakage and not a leak. Consequently, the sensitivity of such methods depends on the existing conditions of pressure, temperature and fluid flow (Anderson, 1989).

4.5.1. Purposes of Non-Destructive Testing in Food Packaging

Before the development of non-destructive testing methods, the only way for a food packager to ensure package integrity was to rely on properly designed statistical control in which a lot of containers were accepted or rejected based on random sampling. Evaluation of such samples was done using destructive testing, resulting in their loss whether or not they were found defective. On a long term basis, such testing procedures cost food companies a significant amount of money due to loss of time, products, and packaging materials (Rice, 1988). This resulted in a rapid development of non-destructive testing methods for the inspection of metal cans

(Kelsey, 1990b).

With the increasing popularity of convenience foods, the demand for flexible packaging has skyrocketed (Stauffer, 1990). Although one might easily agree that the heat sealed closure of flexible container is very probably more reliable than the mechanical closure of the metal can, one must consider what was once said by Wurts (1985): "double seaming technology is to heat sealing technology as the old pro is to the rookie". Consequently, before flexible containers and their closures are recognized as safe, they must prove themselves as being at least as reliable as the metal cans (Wurts, 1985). This resulted in an increasing demand for reliable, 100%, on-line, non-destructive testing methods (Stauffer, 1990).

4.5.2. Non-Destructive Testing Methods Currently Available

On-line, non-destructive tests currently available can be classified based on their operating principles: pressure differential, package deformation, acoustic and voltage differential tests (Kelsey, 1990b).

4.5.2.1. Pressure Differential Tests

Pressure differential or pressure decay testers detect leakers in rigid and flexible containers based on their inability to remain at constant pressure when placed in a vacuumed or pressurized chamber when a leak is present. Pressure differential testers rely on very sophisticated and sensitive pressure transducers to detect slight decreases or increases in pressure in empty or filled containers.

Inspection of empty containers is carried out in three steps process, the empty containers being first pressurized to a preset level. In the second step, the container and pressurized air are allowed to stabilized before the actual leak detection is accomplished. This last step is carried out by measuring any change in pressure in the container. Time required for inspection of empty containers is mainly controlled by the container size and the materials it is made of (Stauffer, 1988; Kelsey, 1990a,b). Stauffer (1988) reported that test time for small containers seldom exceed 2.6 seconds, with 1.5 seconds as the normal, with up to 18 seconds being required for larger containers and drums, allowing for line speed of 700 containers per minute using rotary inspection equipment (Stauffer, 1988).

Inspection of filled containers range from 1 to 3 seconds per container depending on container size, shape and characteristics (Stauffer, 1988). Testing is carried out by placing the filled container in an air tight chamber and creating a pressure differential between the outside and the inside of the container. Both container and pressure are allowed to stabilize for a short period of time, after which potential leaks are detected by monitoring any pressure decay in the chamber (Kelsey, 1990a,b; Stauffer, 1990). This methods has been reported to have sensitivity similar to that of destructive tests such as bubble or ink penetration test, and under ideal conditions to be able to detect leakers of less than 25 μm in size (Stauffer, 1990).

4.5.2.2. Package Deformation Tests

Leak detection by measurement of package deformation has its main applications with containers having internal vacuum, that is mainly metal and glass containers. Presence of a leak is detected by proximity sensors which determine the presence of internal vacuum by measuring the concavity of the metal lids. Such machines can operate at speeds up to 1,200 containers per minutes (Kelsey, 1990b).

Cups and trays with flexible lids are heated gently in order to cause swelling due to expansion of the head space gas, while vacuum is naturally created in brick-packs during folding of the container to produce the brick. The lid or wall profile are then measured using an electromagnetic pulse to excite vibrations in the aluminum foil layer that must be present in the lidding or brick materials, with leaks detected based on the inability of the defective container to maintain lid profile (Andres, 1982; Rice, 1989; Kelsey, 1990a). Such a method is only effective when leakers are big enough to allow sufficient loss of vacuum or pressure at the time of inspection (Andres, 1982; Stauffer, 1988).

4.5.2.3. Acoustic Tests

The first acoustic test was developed to inspect metal cans, and consisted of mechanically tapping the top of a can and monitoring the vibration tones to measure the degree of internal vacuum (Andres, 1982; Kelsey, 1990b).

4.5.2.4. Voltage Differential Tests

Voltage differential testers were first developed to inspect ampules at speeds of up to 300 per minute by submitting the surface of these container to a high-voltage electrical current. Any leak present in these ampules or insufficient glass wall thickness creates a spark between the high moisture content of the ampule and the electrode used for inspection resulting in the rejection of the container

(Kelsey, 1990b). A similar system for integrity testing of packaged foods containing at least 50% moisture was developed and inspects up to 60 packages per minute. It can accept most types of packaging material, from 30 μm (top web) to 1,000 μm (bottom tray) as long as they do not contain any conductive materials (Anonymous, 1991).

4.5.2.5. Ultrasonic Tests

An unusual leak detection method used in fields other than packaging but that might have some potential applications in packaging is the use of ultrasonic sound as a leak detector. Such method is based on the fact that air or gases upon escaping a container placed under vacuum or pressure will create a turbulent ultrasonic sound which can be detected (Kelsey, 1990b).

5. Scanning Acoustic Microscopy

Acoustic microscopy, according to L. W. Kessler (1989), can be defined as "high resolution, high frequency ultrasonic inspection techniques that produce images of features beneath the surface of a sample". As in standard ultrasonic inspection, the ultrasound waves sent into the sample require continuity of the material to propagate. The main difference between both methods lies in the frequency range at which they operate, as well as the use of a sapphire lens in scanning acoustic microscopy to focus the ultrasound waves (Kessler, 1989).

Consequently, flaws detection in scanning acoustic microscopy is done based on the same principle found in standard ultrasonic inspection. For this reason ultrasonic testing will be discussed in order to explain scanning acoustic microscopy.

6. Ultrasonics Testing of Materials

6.1. Basic Principles

Ultrasonic inspection is one of the most widely used method of non-destructive inspection, and is based on the production of high frequency sound waves into the material being inspected to detect surface and subsurface flaws. Because ultrasonic waves are mechanical waves propagating through materials by oscillations or vibrations of their atomic or molecular particles, any flaw or discontinuity in the molecular structure of a given material will cause the wave to be reflected (Halmshaw, 1987; Bar-Cohen et al., 1989).

6.2. Advantages and Disadvantages of Ultrasonic Inspection

Ultrasonic inspection presents many advantages for it has a penetrating power which allows the detection of flaws located as far as 6 meters deep in a material. Its ability to detect flaws as small as 1 μm makes it highly sensitive when compared to other non-destructive methods. It is also accurate in determining flaw location, size, shape and nature. Another advantage of importance is the electronic operation of ultrasonic inspection system. This allow for quasi instantaneous results and interpretation making this technique ideal for automation, rapid scanning and in-line production monitoring. Finally, the ultrasonic inspection is made even more appealing when considering portability and safety of this technique as well as the access required for inspection (Bar-Cohen et al., 1989).

The disadvantages of this technique mainly lies in its sensitivity to material properties which makes it difficult to inspect materials that are not homogeneous, irregular in shape, very small or thin, or have a rough surface. Furthermore, a coupling fluid is needed during inspection to ensure an effective propagation of the ultrasonic waves energy between the transducer and material being inspected (Bar-Cohen et al., 1989).

6.3. General Characteristics of Ultrasonic Waves

6.3.1. Frequency, Wavelength, Velocity and Amplitude

As mentioned earlier, ultrasound are mechanical waves propagating through a material by oscillation of its particles. If the mechanical movements of the particles have a regular motion (sinusoidal), a wave can be assigned a frequency, wavelength, velocity and amplitude (Bar-Cohen et al., 1989).

The frequency, f, of a wave is measured in hertz (Hz) and it refers to the number of oscillations of a given particle per second. Within the range of human hearing sound waves have frequencies between 10 and 20,000 hertz. Above this frequency, they are referred to as ultrasound or ultrasonics (Halmshaw, 1987; Bar-Cohen et al., 1989; Krautkrämer and Krautkrämer, 1990).

The wavelength of a wave, λ, is the distance between two successive points having an identical state of motion, such as, for example, two successive compressions or depressions. The wavelength, amplitude and compression and depression of a typical sinusoidal wave are illustrated in figure 2.

Wavelength and frequency are inversely proportional, and their product gives the velocity, c, at which the wave propagates in a material. The velocity of a wave is characteristic for a given material and remains constant for any frequency and any wavelength (Bar-Cohen et al., 1989; Krautkrämer and Krautkrämer, 1990).

The relation between frequency, wavelength and velocity of a sound wave travelling in a given material is given by the following equation (Halmshaw, 1987; Bar-Cohen et al., 1989; Krautkrämer and Krautkrämer, 1990):

$$c = f\lambda.$$

The amplitude of a sound wave is defined as the height of a crest or the depth of a rarefaction in relation to the surface at equilibrium. The amplitude of a sound wave decreases as it propagates through a material. Figure 2 illustrates the wavelength, compression, rarefaction and amplitude of an ultrasonic wave (Bar-Cohen et al., 1989).

6.3.2. Wave Propagation

Ultrasonic waves propagating in a material can be classified based on the mode of particle motion in relation to their direction of propagation. Longitudinal waves are the most common and important waves used in ultrasonic inspection, where both particle motion and direction of propagation occur along the same axis. These waves propagate in solids, liquids

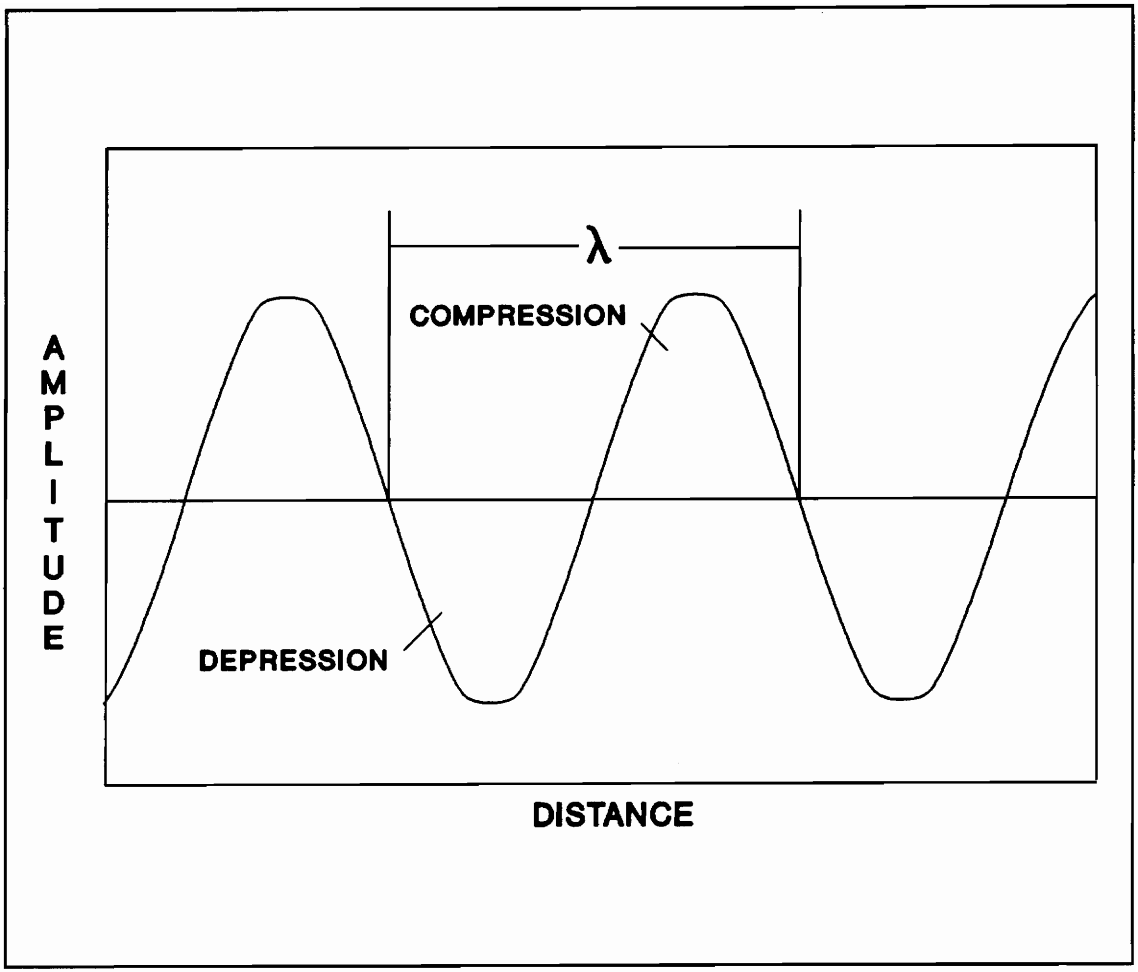

FIGURE 2: WAVELENGTH, COMPRESSION, RAREFACTION AND AMPLITUDE OF A TYPICAL SINUSOIDAL WAVE (Bar-Cohen et al., 1989).

and gases and are also referred to as compression waves. Other waves commonly used in ultrasonic inspection and propagating in solid materials are the transverse or shear waves. The particle motion for such waves occurs along an axis perpendicular to the direction of propagation. Transverse waves do not propagate in liquids or gases and have a velocity of approximately half of that of longitudinal waves. Finally, in Rayleigh or surface waves the particle motion is elliptical and it occurs only in the surface layer of large solids (Halmshaw, 1987; Bar-Cohen et al., 1989;Krautkrämer and Krautkrämer, 1990).

6.4. Major Variables in Ultrasonic Inspection

When considering ultrasonic inspection for any given application, a number of variables must be considered in order to obtain optimum results. These variables include both the properties of the material(s) being inspected as well as the characteristics of the ultrasonic wave used for inspection (Bar-Cohen et al., 1989).

6.4.1. Frequency

The choice of a particular frequency for an ultrasonic inspection application can have both adverse and favorable effects on its performance. As a general rule, the use of high frequencies improves the system's ability to detect and distinguish between small defects, and therefore increases both its sensitivity and resolution. In order to obtain the best chances of detection and/or best acoustic image in SAM, the wavelength in water of the frequency of inspection should be of the order or smaller than the defect to be detected. The wavelength in water for the operational frequencies of the SAM are listed in Table 1.

The major drawback when using high frequencies is a reduction of the depth of penetration, because attenuation of the ultrasonic wave is much stronger at high frequencies. Consequently, a compromise between the depth of penetration and the resolution and sensitivity needed must often be made when choosing a frequency for a given application (Bar-Cohen et al., 1989).

6.4.2. Acoustic Impedance

The acoustic impedance, Z, of a given material is the product of its density, ρ, given in grams per cubic centimeter, and the longitudinal wave velocity, V_l, given in centimeters per second (Bar-Cohen et al., 1989). Therefore:

$$Z = \rho V_l$$

TABLE 1: SAM OPERATING FREQUENCIES AND THEIR RESPECTIVE WAVELENGTH IN WATER.

FREQUENCY (MHz)	WAVELENGTH IN WATER (μm)
30	49.43
50	29.66
100	14.83
200	7.42
400	3.70
600	2.47
800	1.85
1000	1.48

When considering a sound wave propagating from one material to another, the percentage of energy reflected from their interface is a function of the ratio of their respective acoustic impedances and angle of incidence. In the case of an ultrasonic wave incident on a perpendicular, flat, smooth boundary between two materials of acoustic impedances, Z_1 and Z_2, the percentage of energy reflected from their interface can be calculated as follow (Halmshaw, 1987; Bar-Cohen et al., 1989):

$$E_R = \left(\frac{Z_1 - Z_2}{Z_1 + Z_2}\right)^2 x\ 100$$

In this case, where all the energy is either reflected or transmitted, the percentage of energy transmitted can be calculated as follow (Halmshaw, 1987; Bar-Cohen et al., 1989):

$$E_T = 100 - E_R = \frac{4\ Z_1\ Z_2}{(Z_1 + Z_2)^2}\ x\ 100$$

6.4.3. Refraction at Interface and Mode Conversion

In ultrasonic inspection, the transmission and reflection of an ultrasonic wave at an interface is greatly affected by its angle of incidence. At normal incidence, the transmission and reflection of ultrasonic waves occur without any change in beam direction. However, at any other angle of incidence, both wave refraction and mode conversion are observed. The phenomenon of wave refraction refers to a change in direction of the transmitted wave propagation, while mode conversion refers to a change in the nature of the wave motion. The angle of refraction of a transmitted wave can be calculated according to following equation, also known as Snell's law (Halmshaw, 1987; Bar-Cohen et al., 1989; Krautkrämer and Krautkrämer, 1990):

$$\frac{\sin\alpha}{\sin\beta} = \frac{c_1}{c_2}$$

Where:-α is the angle of incidence,

-β is the angle of refraction,

-c_1 is the velocity of the incident wave,

-c_2 is the velocity of the refracted wave.

6.5. Attenuation of Ultrasonic Waves

The intensity of an ultrasonic wave propagating through a material is reduced by various mechanisms including acoustic impedance effects, absorption, scattering and diffraction at interface.

The acoustic impedance effects, as explained in section 6.4.2., occurs when an ultrasonic wave propagating from one material to another is partially reflected due to the acoustic impedance mismatch. As the difference in acoustic impedance between two materials increases, the energy reflected from their interface increases, and therefore the intensity of the ultrasonic wave transmitted into the second material is decreased (Bar-Cohen et al., 1989).

The absorption mechanism depends on material properties such as grain size and grain orientation, and on the frequency used for inspection. Attenuation of the intensity of an ultrasonic wave occurring through absorption is the result of the conversion of mechanical energy into heat. In most cases, absorption losses increases with frequency, but at a rate much slower than scattering. Absorption effects can be reduced both by increasing the intensity of the ultrasonic wave sent into the material and by increasing amplification of the detected signal (Halmshaw, 1987; Bar-Cohen et al., 1989).

Scattering of ultrasonic waves is directly related to the homogeneity and grain size of the material being inspected. Scattering can be considered negligible when the grain size is less than one hundredth of the wavelength. Above this size inclusions such as grains or pores tend to partially deflect the ultrasonic wave beam resulting in attenuation of its intensity. In addition to attenuation, scattering produces small echoes with different times of flight resulting in the production of background noise or "grass". Therefore, unlike absorption, scattering effect cannot be reduced by increasing the ultrasonic wave intensity or amplification, since the grass increases simultaneously. The only way to reduce scattering is to reduce the frequency of inspection which results in an increase in wavelength but also decreases sensitivity and resolution (Bar-Cohen et al., 1989; Krautkrämer and Krautkrämer, 1990).

Diffraction phenomena occurs when an ultrasound wave propagating in an homogeneous medium passes the edge of a reflecting surface causing the wave front to bend. Bending of the wave front, in case of a reflector of the order of one wavelength, produces an interference pattern immediately behind the reflector. Diffraction of ultrasonic waves should be considered when developing inspection procedures for a given application (Bar-Cohen et al., 1989).

6.6. Pulse-Echo Inspection Method

The methods used in ultrasonic inspection include the frequency-modulation method, transmission method, and pulse-echo method. This last method gives the most information and it is the most widely used in non-destructive evaluation of material. The pulse-echo method is used both for detection and location of flaws and for thickness measurements (Halmshaw, 1987; Bar-Cohen et al., 1989).

6.6.1. System Components for Pulse-Echo Ultrasonic Inspection

The essential components of a basic system for pulse-echo inspection are illustrated in Figure 3.

In a typical pulse-echo, ultrasonic inspection system a pulser (1) consisting of an electronic signal generator is used to generate the burst of alternating voltage of about 500 to a 1000 volts necessary to stimulate a transducer (2). This transducer consists of a piezoelectric element, which, when submitted to an electric charge, mechanically deforms thereby creating a sound wave. Vice versa, it has the ability to develop an electrical charge when submitted to a pressure change, and consequently can be used both as a transmitter and a receiver. A couplant (3) is used to promote ultrasonic transmission between the transducer and the surface of the

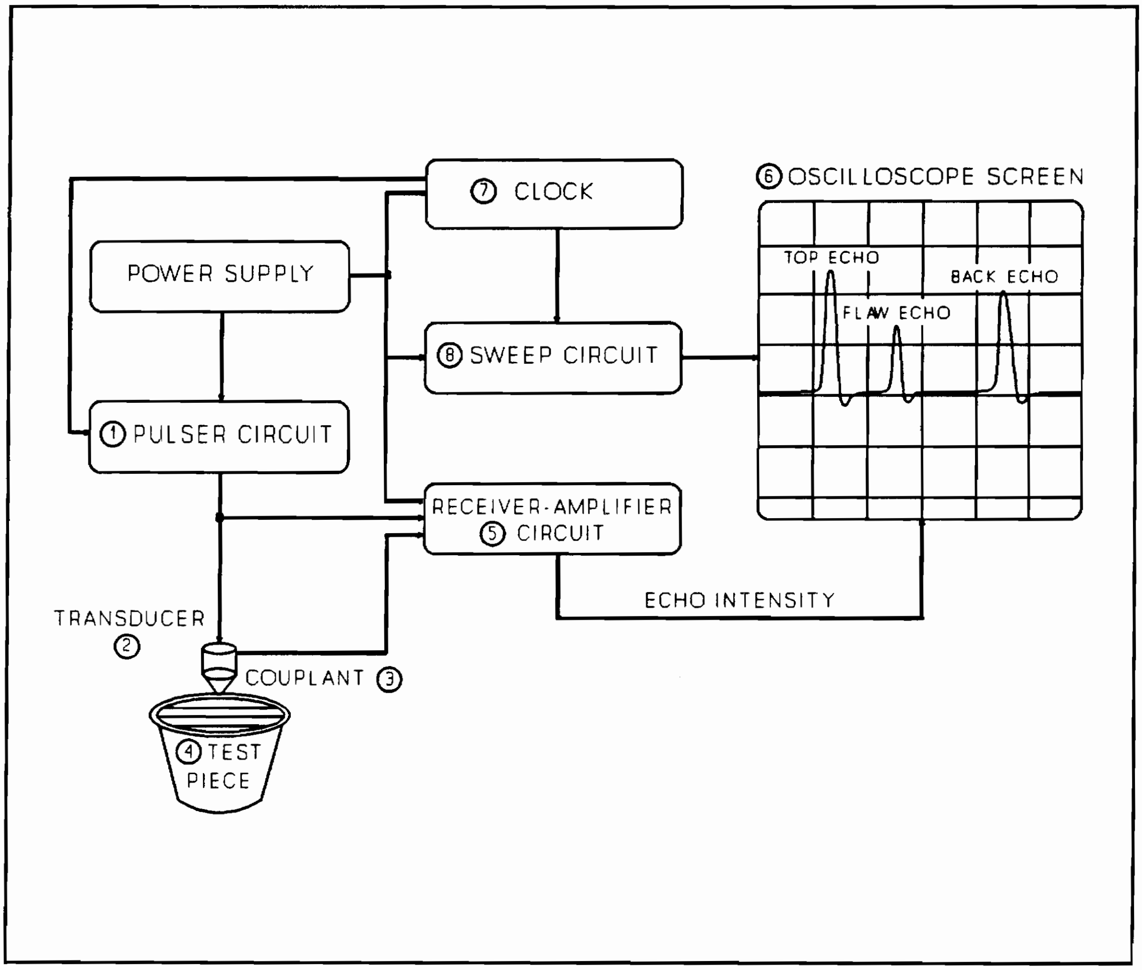

FIGURE 3: ESSENTIAL COMPONENTS OF A BASIC PULSE-ECHO, ULTRASONIC, INSPECTION SYSTEM (Bar-Cohen et al., 1989)

material being inspected (4). Ideally the acoustic impedance of a couplant should match that of the material as closely as possible to minimize energy losses through reflection.

After being propagated through the material, the energy reflected at interfaces within the material is collected by a receiver, which turns the acoustic signal into an electric output. As mentioned above, a single transducer is often usedas transmitter and receiver in pulse-echo method. The electric output is then amplified by an electronic device (5), which is also used to demodulate or modify the signal when necessary. Finally, the electric output is displayed on an indicating device such as an oscilloscope (6), and recorded on a computer printout or thermal paper. An electronic clock (7) is used to synchronize the entire system and to control the various components (Halmshaw, 1987; Bar-Cohen et al., 1989).

6.6.2. Presentation of Pulse-Echo Data

As mentioned above, the output obtained from a pulse-echo inspection is usually displayed on an oscilloscope. Based on whether the data is collected on a single point, line or total surface of the sample, the displays are respectively referred to as A, B, or C-scan.

6.6.2.1. A-Scan

The A-scan data is collected over a single point on the surface of the material being inspected and basically consists of a plot of the signal amplitude versus time. Therefore, an A-scan gives a quantitative display of signal amplitudes and time-of-flight data, allowing defect detection and sizing, as well as location when knowing the longitudinal wave velocity of the material being inspected (Bar-Cohen et al., 1989).

6.6.2.2. B-Scan

On a B-scan, data is displayed as a plot of time-of-flight versus distance representing the position of the transducer along a line on the surface of the material being inspected in relation to its original position. Although the signal amplitude is not measured directly as in A-scan, it is often indicated by the relative brightness of the echo on the oscilloscope screen. Consequently, a B-scan can be used to determine the size, location and, to a certain extend, shape of a defect (Bar-Cohen et al., 1989).

6.6.2.3. C-Scan

The C-scan is collected over an area of the material surface and provides a semi-quantitative or quantitative display of signal amplitudes. Consequently, a C-scan can be used to map out both the location and size of a defect within a plan view of the material being inspected. C-scan displays vary; some produce a shaded-line scan in which echo intensity is recorded as a variation in line shading on a gray scale, while others indicate flaws by an absence of shading so that each flaw shows up as a blank space on the display. Flaw depth normally is not recorded on a C-scan, although it can be measured semi-quantitatively, in such cases the flaw depth is displayed with a color scale (Bar-Cohen et al., 1989).

III. OBJECTIVES OF THE STUDY

The goal of this study was to determine the ability of ultrasonic inspection techniques to be used for on-line, non-destructive evaluation of the integrity of heat sealed, flexible package structure commonly used in packaging of aseptic and shelf-stable food products.

The specific objectives of this study were:

1. Establish operational parameters for ultrasonic inspection of heat seal closure of various flexible packages using acoustic microscopy.

2. Establish parameters to ultrasonically identify various seal defects.

3. Establish seal characteristics affecting ultrasonic inspection.

4. Establish limits of detection of ultrasonic inspection using scanning acoustic microscopy.

5. Develop machine criteria for a non-destructive, on-line, ultrasonic inspection device.

IV. MATERIALS AND METHODS

1. Materials

The packaging materials used throughout this research consisted of five structures commonly encountered in flexible packaging of aseptic and shelf-stable products. These fives structures are used for bricks, pouches and trays.

1.1. Bricks

The bricks packaging material was provided by International Paper Inc. (Raleigh, NC). The laminate consisted of, from the inside to the outside, PE, paper, PE, aluminum foil and PE.

1.2. Pouches

The structure of the pouch materials consisted of extrusion laminated plastics and plastic/aluminum laminate. The plastic pouches were provided by Cryovac Inc. (W.R. Grace & Co., Cryovac Division, Duncan, SC) and consisted of linear LDPE (LLDPE)/Nylon laminate of 0.0026" thickness (Cryovac, P-640-2.6 mil LLDPE).

The plastic/aluminum laminate pouches were meal ready to eat (MRE) retortable pouches (LC Flex 70460, 8 oz., 4¾" x 8⅛") and were provided by the Jefferson Smurfit Corporation (Schaumburg, IL). The laminate consisted of 48 ga. PE terephthalate (PET)/adhesive/12.7 μm foil/adhesive/76.2 μm PP.

1.3. Trays

The plastic trays were provided by Rampart Packaging Inc. (Williamsburg, VA), and consisted of coextruded PP/regrind/adhesive/EVOH/adhesive/regrind/PP of 1.32 mm thickness. The lids consisted either of plastic/aluminum or coextruded plastic laminates. The former was provided by American National Can Company (Barrington, IL) and consisted of PET/PVDC/PP (M-6262), while the later was provided by Alcoa Technical Center (Pittsburgh, PA) and consisted of PET/foil/polyolefin (DF-31964).

2. Heat Sealing

The different packaging materials used in throughout this research were heat sealed using a hot jaws sealer (Packaging Aids Corporation, San Rafael, CA), PAC Barrier Heat Sealer, Model Series PB-H, modified in order to obtain seals of the desired quality and width. The original sealing jaw was replaced by a custom designed aluminum bar of 5 mm width. Aluminum and teflon bars were cut so as to provide materials to cover the bottom jaw in addition to the original rubber bar. The sealing temperature, pressure and dwell time, as well as the material covering the bottom jaw of the sealer, used for each of the five packaging structures are recorded in Table 1.

3. Defects Manufacture

Channel leakers of 20 μm diameter were manufactured at the heat sealed interface of the five type of packaging materials. This was done by placing a 20 μm polytetrafluoroethylene (PTFE) fiber (DuPont, Wilmington, DE) at the seal interface prior to sealing. The teflon fiber was maintained straight and perpendicular to the seal by taping its ends inside and outside the pouch or tray. After sealing and cooling, the teflon fiber was removed by pulling on either end of the fiber, and the seal area to be inspected cut out of the pouch or tray.

Channel leakers of 100, 130 and 210 μm diameters were manufactured in a similar way at the interface of MRE pouches using platinum wires (Fisher Scientific, Pittsburgh, PA). This was done by placing a platinum wire of desired diameter at the seal interface prior sealing. The platinum wire was

TABLE 2. SEALING TEMPERATURE, PRESSURE, AND DWELL TIME FOR DIFFERENT PACKAGING STRUCTURES.

PACKAGE TYPE	STRUCTURES	TEMPERATURE	DWELL TIME	PRESSURE
BRICKS[a]	PAPER/Al/ PLASTICS	177°C	4.5 sec.	552 kPa
POUCHES[b]	PLASTICS	149°C	4.5 sec.	552 kPa
	PLASTICS/Al	177°C	3.5 sec.	552 kPa
TRAYS[c]	PLASTICS LIDDING	177°C	3.0 sec.	552 kPa
	PLASTICS/Al LIDDING	177°C	2.5 sec.	552 kPa

Bottom Jaw: [a] Teflon in all cases.
[b] Teflon for leaker above 20 μm, rubber for 20 μm leaker.
[c] Aluminum in all cases.

previously coated with a high temperature, releasing agent, polytetrafluoroethylene (PTFE), Dry Film Vydax Mold Release (McMaster, New Brunswick, NJ), in order to facilitate removal, and the bottom jaw of the sealer covered with a teflon bar to avoid delamination around the leaker. After sealing and adequate cooling the platinum wire was removed by pulling on its end.

Blisters and wrinkles could not be manufactured and reproduced in a consistent manner. Consequently, the blisters and wrinkles inspected in this study were those obtained randomly during sealing of the various materials.

Carbon fibers of 8 μm diameter were embedded at the heat sealed interface of the five type of packaging materials. This was done to simulate seal contamination and to determine the ability of the SAM to detect defects as small as 8 μm in diameter.

The size of the carbon and teflon fibers were confirmed by measuring their diameter using an optical microscope, Olympus System Microscope, Model BH-2 (Opelco, Washington, D.C.), equipped with an internal grid (Olympus Eyepiece, 10x/18L with Internal Grid), while the platinum wire diameters were checked by measuring their respective diameter with a micrometer, Mitutoyo, series 193 (McMaster, New Brunswick, NJ).

4. Scanning Acoustic Microscopy

Acoustic operational parameters for detection and identification of heat seal defects were determined for each of the five packaging structures studied in this research using a scanning acoustic microscope (SAM), Olympus UH3, and image analysis system to detect a 20 μm channel leaker located at their interface.

The size of the smallest defect/leak necessary to allow bacteria to enter a container being unknown, the smallest reproducible channel leaker manufactured was used as a reference to determine the acoustic operational parameters to inspect the five packaging structures. These included the frequency of inspection, attenuation setting, and Z-value which represents the distance at which the transducer is lowered from its point of origin to obtain the desired acoustic image. The point of origin is the position of the transducer when this one is focussed on the surface of the seal to be inspected.

Each sample was inspected immersed in water at room temperature (22-23°C), and kept as flat and horizontal as possible during inspection. In the case of the pouches, the seal sections containing the defects were glued with cyanoacrylate glue on glass slides used in optical microscopy (Fisher Scientific, Pittsburgh, PA). The optimum operational parameters to ensure defect detection and identification were determined for each type of package structure by changing the frequency, attenuation and focal point settings until the best possible image resolution was obtained. Other types of defects such as wrinkles, blisters or contaminated seals were inspected, whenever possible, to determine the possibility to identify defects based on their acoustic signature.

The seal characteristics affecting ultrasonic inspection were determined by inspecting embossed seals and by comparing the results obtained for each type of packaging materials.

5. Bio-Test

Five sets of retortable pouches were manufactured for bio-testing: one hundred and fifty MRE pouches containing 20 μm channel leakers, in addition to three sets of 50 MRE pouches respectively containing channel leakers of 100, 130 and 210 μm. Each pouch was numbered and the diameter and position of the channel leaker indicated. A fifth set of 50 retortable pouches containing no leaks were manufactured as controls and were processed with the other four sets. All pouches were double sealed to ensure their integrity, filled with 80 ml of Tryptic Soy Broth (Fisher Scientific, Pittsburgh, PA) and sealed, using the hot jaws sealer with the bottom jaw covered by the rubber bar, leaving as little headspace as possible. All pouches were commercially sterilized for 25 minutes at 121°C in a vertical still retort (Model RDTI-3, Dixie Canner Equipment Co., Athens, GA) with 159±7 kPA (23±1 p.s.i.g.) head pressure during heating.

Gas producing E. coli 9637 were grown overnight at 37°C (98.6°F) in 114 liters (30 gal) of Tryptic Soy Broth, reaching a concentration of 4 x 10^8 E. coli/ml. The channel leaker in each pouch was exposed by cutting along the edge of the second seal. Care was taken not to cut along the edge of the first seal since the pressure applied during cutting was found sufficient to close the 20 μm channel leaker. Before being placed in the inoculated Tryptic Soy Broth, the flanges of each MRE pouch were wiped with the E. coli broth using a sterile cotton swab to ensure adequate wetting of the seal. After being allowed to stand in the broth for 2 hours with agitation every 15 minutes, the pouches were rinsed with tap water and incubated at 37°C for 24-48 hours. A pouch was recorded positive for the bio-test if it was (1) swollen, and (2) the broth was turbid. To further confirm the results of the bio-test, the gas headspace of 20% of the MRE pouches of each set were analyzed with a gas partitioner to confirm the presence of carbon dioxide and hydrogen. Finally all pouches were submitted to an ink penetration test to confirm that, in case of a positive bio-test, the only leaker present in the pouches were those manufactured.

6. Defect Detection and Sizing

In order to confirm the results of the bio-test, several methods were used to determine the presence of the channel leakers and their size.

6.1. Optical microscopy

The diameter and continuity of the 20 μm channel leaker was confirmed by delaminating 20% of the pouches used in the bio-test and measuring their diameter under an optical microscope, Olympus System Microscope, Model BH-2, equipped with the same internal grid used to measure the teflon fiber diameter.

6.2. Electrolytic Testing

Electrolytic testing as described in the *Bacteriological Analytical Manual* (AOAC, 1989) was used to confirm the presence of the channel leakers after their manufacture in the Cryovac and MRE pouches. An attempt was made to correlate the diameter of a leaker to the intensity of the current flow by plotting a standard curve for channel leakers of known diameter. Six sets of ten retortable pouches (Cryovac) were sealed at 149°C for 4.5 seconds at 552 kPa pressure using a 5 millimeters aluminum hot bar. The bottom jaw was covered with teflon to obtain a smooth seal. Channel leakers of 20, 100, 130, 210 and 260 μm were respectively manufactured during sealing in the six set of pouches. The pouches were then inspected using the electrolytic test, as mentioned in the B.A.M. (Section 24.52, AOAC, 1984), using a 1% salt solution and 9 volts alternating current. Electrolytic testing was repeated using 18 volts alternating current and 2% salt solution. The same procedures was repeated using MRE pouches.

6.3. Ink Penetration Test

The ink penetration test was used to ensure that a positive bio-test was not resulting from any other leaks than that manufactured during sealing, and that, in case of a negative bio-test, the manufactured leak was not clogged.

The ink penetration test used in this study was a modification of the AOAC ink test described in the *Bacteriological Analytical Manual* (AOAC, 1984). The dye solution used in the ink penetration test was prepared by dissolving 2.5 g of Safranin O (Sigma Chemical Company, St Louis, MO) in to one liter of isopropanol. The dye solution was allowed to stand overnight and was filtered using a glass funnel and medium filter paper. The MRE pouches used for the bio-test were emptied, washed and dried, and a few drops of the dye solution were applied to the critical point. The pouches were allowed to stand for 15 minutes and their seals inspected visually for ink penetration.

V. RESULTS AND DISCUSSION

1. Scanning Acoustic Microscopy

1.1. Defect Detection Results

As in standard ultrasonic inspection, defects located within a seal are detected based on their capacity to interfere with the transmission of an acoustic wave sent throughout the thickness of the seal. Because of the difference in acoustic impedance between plastics and air, any voids such as leakers and blisters interfere with the transmission of the acoustic wave, showing as an echo on an A-scan display. Consequently, defect detection can be achieved, in theory, by monitoring or gating such an echo on an A-Scan display. However, as illustrated in Figure 4, it is often difficult to differentiate between the surface echo (1) of the seal and the defect echo (2) since the seals commonly encountered in food packaging are relatively thin. Because any defects located within a seal affect the bottom reflection of the seal by reducing its intensity, and this one being constant within noise level for a given seal, the easiest and most reliable way to detect any defect is to monitor any changes in the intensity of the acoustic reflection from the bottom of the seal, or of the acoustic reflection of any

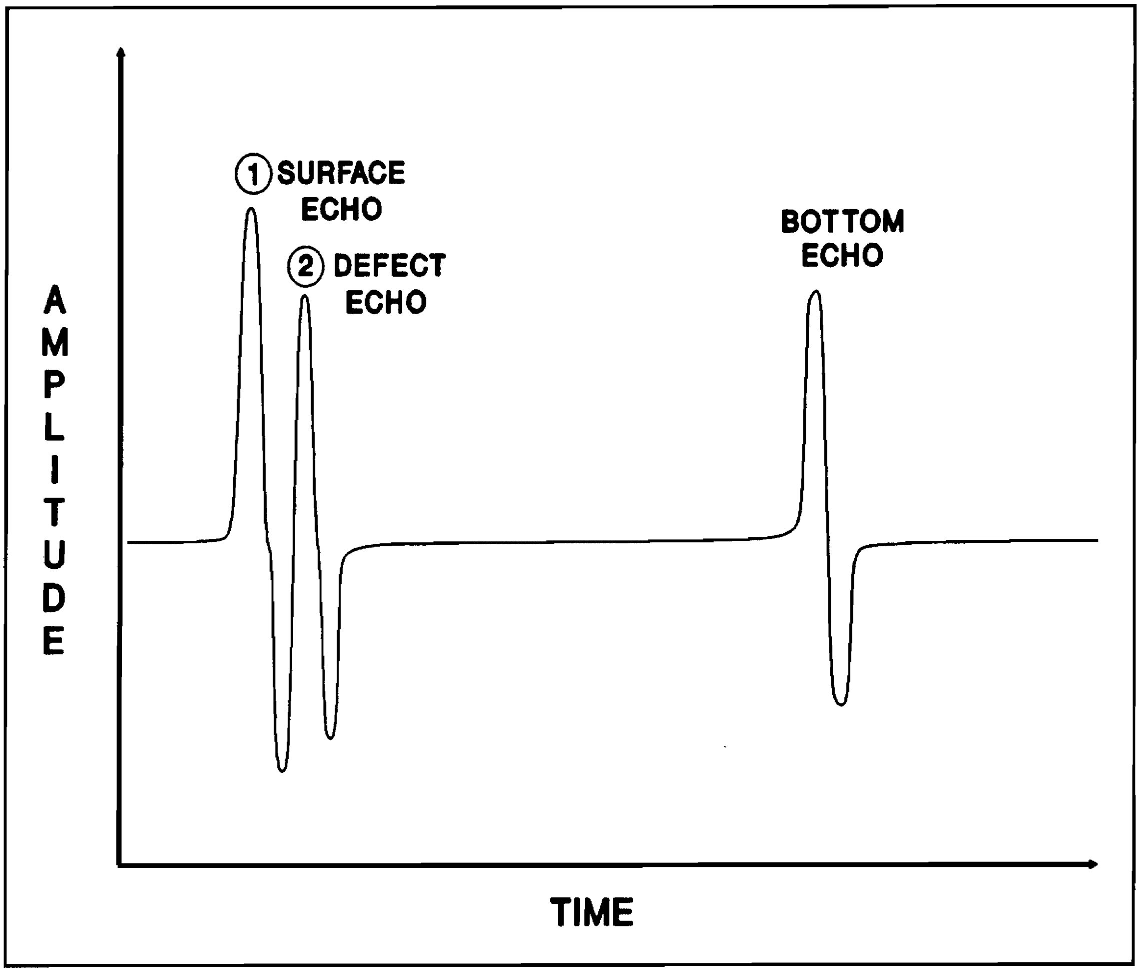

FIGURE 4: TYPICAL A-SCAN OF A PACKAGE SEAL.

interface located below the seal interface.

1.2. Operational Acoustic Parameters

The frequency range, attenuation and Z-value (transducer position) used to detect a 20 μm channel leaker located at the seal interface of the five packaging structures are recorded in Table 2.

1.2.1. Frequency Range

The frequency used for the detection of a defect located at the interface of a seal is affected by three parameters: the level of sensitivity desired or the size of the smallest defect of interest, the thickness of the seal to be inspected, and the composition of the seal structure.

The defect used as a reference for this research being a channel leaker of 20 μm diameter, the first frequency to be investigated was 100 MHz, since its wavelength in water was of 14.83 μm.

However, frequencies having a wavelength in water greater than the defect to be detected can be used successfully if the defect is still of sufficient size to affect the ultrasound to allow its detection. Consequently, the limits of detection at a given frequency is not limited by the size of one wavelength

TABLE 3: FREQUENCY RANGE, Z-VALUE, AND ATTENUATION SETTINGS USED FOR THE DETECTION OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF FIVE TYPES OF PACKAGE.

PACKAGE	STRUCTURES	FREQUENCY	Z-VALUE	ATTENUATION
BRICKS	PAPER/Al/ PLASTICS	UNABLE TO DETECT A 20 μm LEAKER AT THE SAM OPERATIONAL FREQUENCIES		
POUCHES	PLASTICS	100 MHz	-200 μm to -550 μm	3 dB
	PLASTICS/Al	50 MHz	-200 μm	18 dB
TRAYS	PLASTICS LIDDING	50 MHz	-1.30 mm to -1.50 mm	0 dB
	PLASTICS/Al LIDDING	30 MHz	-1.30 mm to -1.90 mm	0 dB

in water but rather by the refraction, diffraction and scattering of the acoustic signal by the defect. Others limiting factors at a given frequency are the seal structure and thickness; these will be discussed later along with other package properties affecting ultrasonic wave propagation.

1.2.2. Attenuation

The attenuation settings for the scanning acoustic microscope were determined by the intensity of the signal returning to the transducer. When this one was too high, it was necessary to attenuate the signal to prevent saturation of the amplifier. The attenuation settings are a characteristic of both the electronics used to amplify and modify the signal output and of the seal being inspected. Consequently, the settings recorded for the different packages cannot be taken as definite values. As a general rule it can be said that higher attenuation settings are necessary with thin homogeneous materials or as the frequency is decreased. When considering the pouches, it can be noticed that the plastic/Al structure has an attenuation setting of 18 dB, thus even though this one contains an aluminum layer, when the attenuation level for the plastics structure is of only 3 dB. This difference between attenuation levels can be explained by the difference in the frequency of inspection, respectively 50

MHz for the plastics/Al structure and 100 MHz for the plastic structure.

1.2.3. Z-Value/Transducer Position

The focussed transducers used for inspection all have a well defined focal length in water. When working with the SAM, the transducer is first focussed on top of the surface of the seal and the Z-value set to zero. The Z-value represents the distance at which the transducer is moved from its point of origin to obtain the desired acoustic image. Because of the refraction phenomena occurring when the acoustic signal is transmitted in the seal, the Z-value is not a true measurement of the position of the focal point within the seal.

Because any defects are detected based on their ability to affect the bottom reflection of the seal, it was important to obtain a bottom reflection of sufficient amplitude to allow its detection. The obtention of a clear bottom echo is affected by the packaging materials as well as the thickness of the seal. At a given frequency the intensity of the acoustic signal reaching the bottom of a seal can be optimized by focusing the transducer at the bottom surface of the seal. When considering the Z-values for the four structures successfully inspected in this research, it is clear that, in all cases, the transducer was focussed close to the bottom of the seal being inspected since the Z-values were greater than the thickness of the seals.

However, for imaging purpose the best resolution is achieved by focusing the transducer on the defect of interest, usually located at the seal interface. The purpose of the on-line inspection being to detect defects and not to image them, the transducer would be preferably focused at the bottom of the seal on an on-line testing device.

1.3. Acoustic Evaluation of Packaging Structures

The acoustic images obtained during the acoustic evaluation of the five different types of packaging structures were generated from echo-amplitude data collected over an X,Y scanned field of view, and having similar time of flight as long as the gate length is kept reasonably small.

1.3.1. Bricks

The brick packaging material provided by International Paper Inc. consisted of a PE/paper/PE/aluminum/PE laminate and had a seal of the order of 700 μm thickness. The acoustic wave required to detect the 20 μm channel leaker needed to travel through the seal and back to the transducer with sufficient intensity to be differentiated from the background noise. Detection of a 20 μm channel leaker located at the seal interface was unsuccessful due to the low intensity of the acoustic signal returning to the transducer. Attenuation of the acoustic signal was explained both by scattering caused by entrapped air in the paper layer and by the aluminum layer which caused most of the energy of the acoustic signal to be reflected, leaving it with too little energy to reach the bottom of the seal. Furthermore, the energy transmitted encountered the same phenomena when travelling back toward the transducer.

Better results might have been obtained by using a through-transmission technique with a transmitter and receiver on opposite sides of the seal. The SAM used was based on the pulse-echo technique where a single transducer serves as transmitter and receiver. Therefore, it was not possible to determine whether or not the through-transmission technique would allow the detection of a 20 μm channel leaker.

1.3.2. Extrusion Laminated Pouches

A 20 μm channel leaker was successfully detected in the extrusion laminated plastic pouches at a frequency of 100 MHz. Figure 5 shows the SAM image of the seal and defect as well as a B-scan collected along a line perpendicular to the channel leaker. Even though the working frequency was of 100 MHz, an attenuation setting of 3 dB was necessary due to the homogeneity of the packaging structure which consisted only of materials having similar acoustic impedances (between 2 and 3 x 10^5 $g \cdot cm^{-2} \cdot s^{-1}$) and allowed for good transmission of the acoustic signal. Furthermore, the seal thickness was of the order of 160 μm which was not sufficient to cause a significant attenuation of the acoustic signal. It can be noted that the Z-value was -280 μm which placed the focal point below the bottom of the seal if refraction of the acoustic signal is not taken into consideration. The 20 μm channel leaker appears clearly on the B-scan as it causes a strong reduction in the intensity of the bottom echo of the seal.

1.3.3. Plastic/Aluminum Laminated Pouches

The plastic/aluminum laminated MRE pouch seals had a thickness of the order of 230 μm. Detection of a 20 μm channel leaker was successful at a frequency of 50 MHz. Figure 6 shows the SAM image of the seal and defect as well as a B-scan collected along a line perpendicular to the direction of the channel leaker. The Z-value of -200 μm showed that the focal point was not located at the bottom of the seal. An attenuation setting of 18 dB was necessary to prevent the saturation of the amplifier. The plastic/aluminum laminate

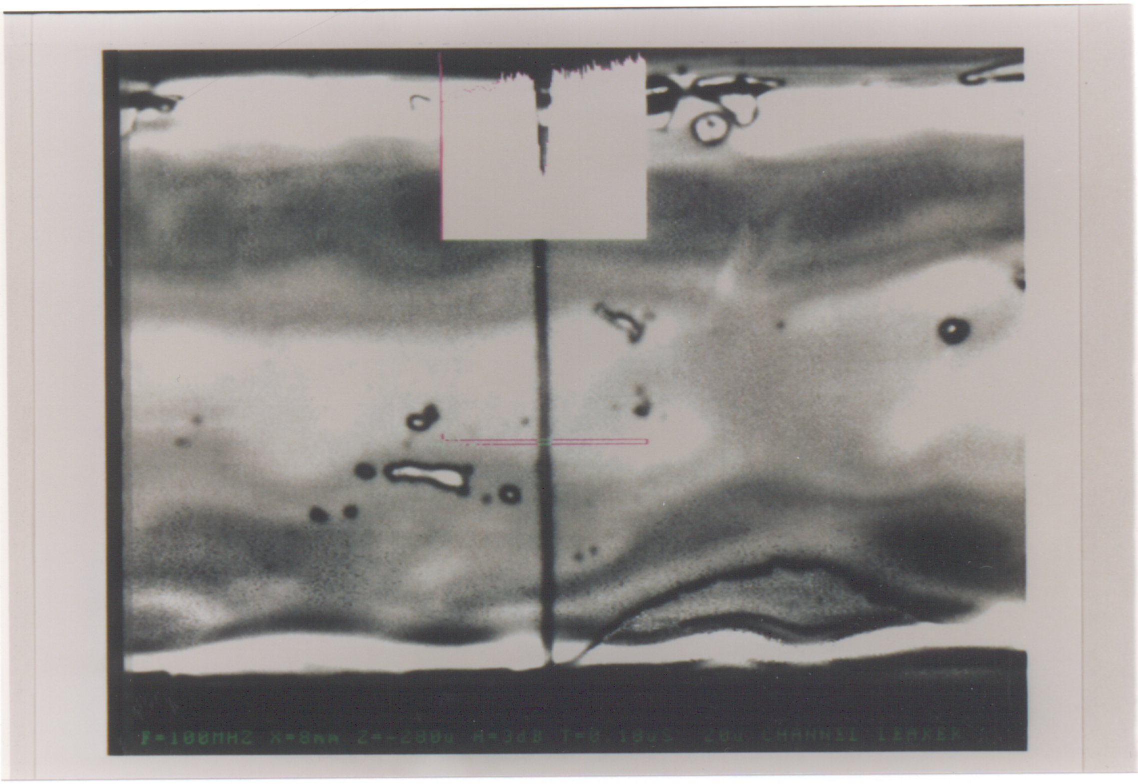

FIGURE 5: SAM IMAGE AT 100 MHz OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF AN EXTRUSION LAMINATED PLASTIC POUCH.

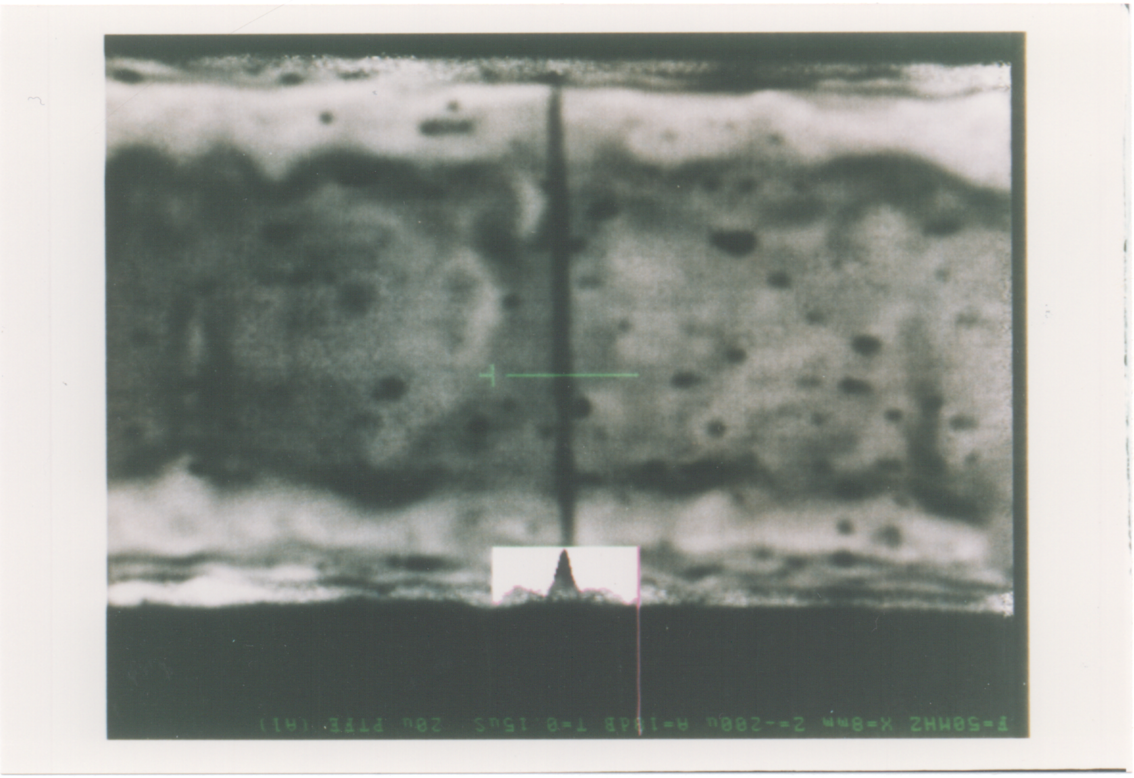

FIGURE 6: SAM IMAGE AT 50 MHz OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF A PLASTIC/ALUMINUM LAMINATE POUCH.

presented relatively non-homogeneous acoustic properties since the plastics have an acoustic impedance ranging between 2 and 3 x 10^5 $g \cdot cm^{-2} \cdot s^{-1}$, while aluminum acoustic impedance is 17 x 10^5 $g \cdot cm^{-2} \cdot s^{-1}$. Such a difference in acoustic impedance between plastic and aluminum layers caused a strong reflection of the acoustic signal when reaching the plastic-aluminum interface. Therefore, it was possible that the echo gated for the obtention of the SAM image might have been an echo obtained from the first or second plastic aluminum interface. This would explain both the Z-value and the high attenuation setting. Finally, after being observed with an optical microscope, the black dots appearing in several places on the SAM image were identified as being part of the green pigmentation used to color the pouches. This pigmentation non-homogeneity should be avoided since the black dots observed on the SAM image caused a signal reduction of the same order than the channel leaker and consequently would be able to mask defects located at the seal interface.

1.3.4. Coextruded Plastic Lidding

Trays sealed with the coextruded plastic lidding had seals of the order of 1.45 mm in thickness. Detection of a 20 μm channel leaker was unsuccessful at a frequency of 100 MHz. When looking at Figure 7 which shows the SAM image obtained at

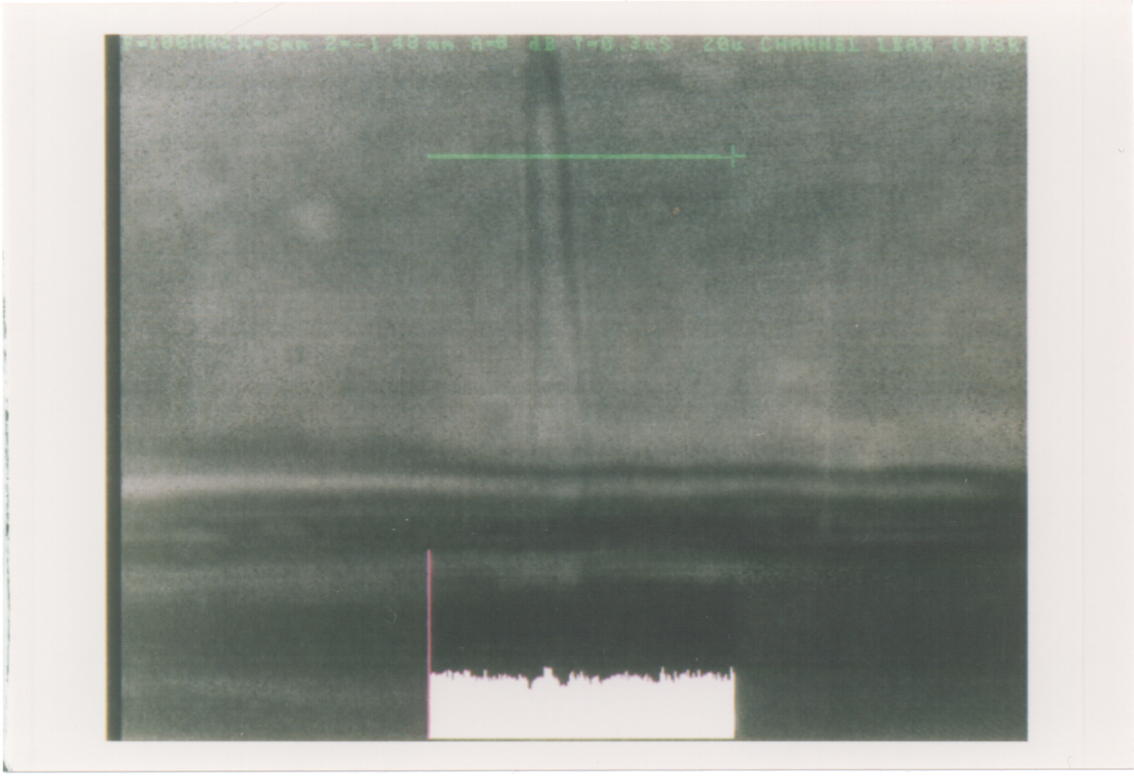

FIGURE 7: SAM IMAGE AT 100 MHz OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF A TRAY SEALED WITH COEXTRUDED PLASTIC LIDDING.

that frequency, the 20 μm channel leaker starts showing because its interfere with the transmission of the acoustic signal. However, interferences effects are not strong enough to be detected on the B-scan since they are of the same intensity as the background noise. Figure 8 shows the SAM image of the same seal inspected this time at 50 MHz frequency. The 20 μm channel leaker appeared clearly both on the SAM image and the B-scan. The acoustic signal returning to the transducer did not need to be attenuated because the seal thickness did not allow for a strong bottom reflection. The intensity of the bottom reflection of the seal might have been increased by lowering the focal point to the bottom of the sample, which was not done since the Z-value of -1.31 mm was smaller than the thickness of the seal. This Z-value setting was chosen to improve the resolution of the acoustic image. Finally, Figure 9 shows the SAM image of that same seal obtained at a 30 MHz frequency. The 20 μm channel leaker appears larger both on the SAM image and B-scan because the intensity of the bottom reflection was increased by working at a lower frequency. For that same reason the attenuation setting needed to be increased to 18 decibels. The 20 μm channel leaker appears bigger than its actual size on both Figures 8 and 9 due to the refraction and diffraction of the acoustic signal and because the focal point of the transducer was not located at the seal interface but somewhere below it.

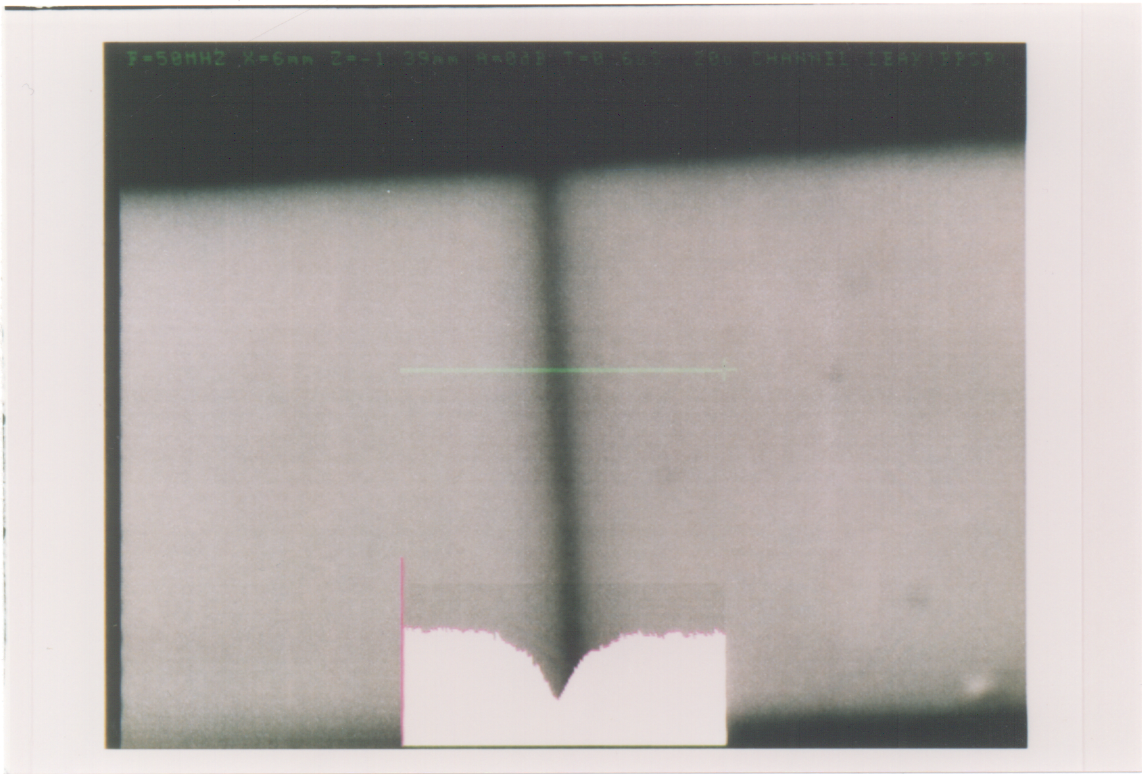

FIGURE 8: SAM IMAGE AT 50 MHz OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF A TRAY SEALED WITH COEXTRUDED PLASTIC LIDDING.

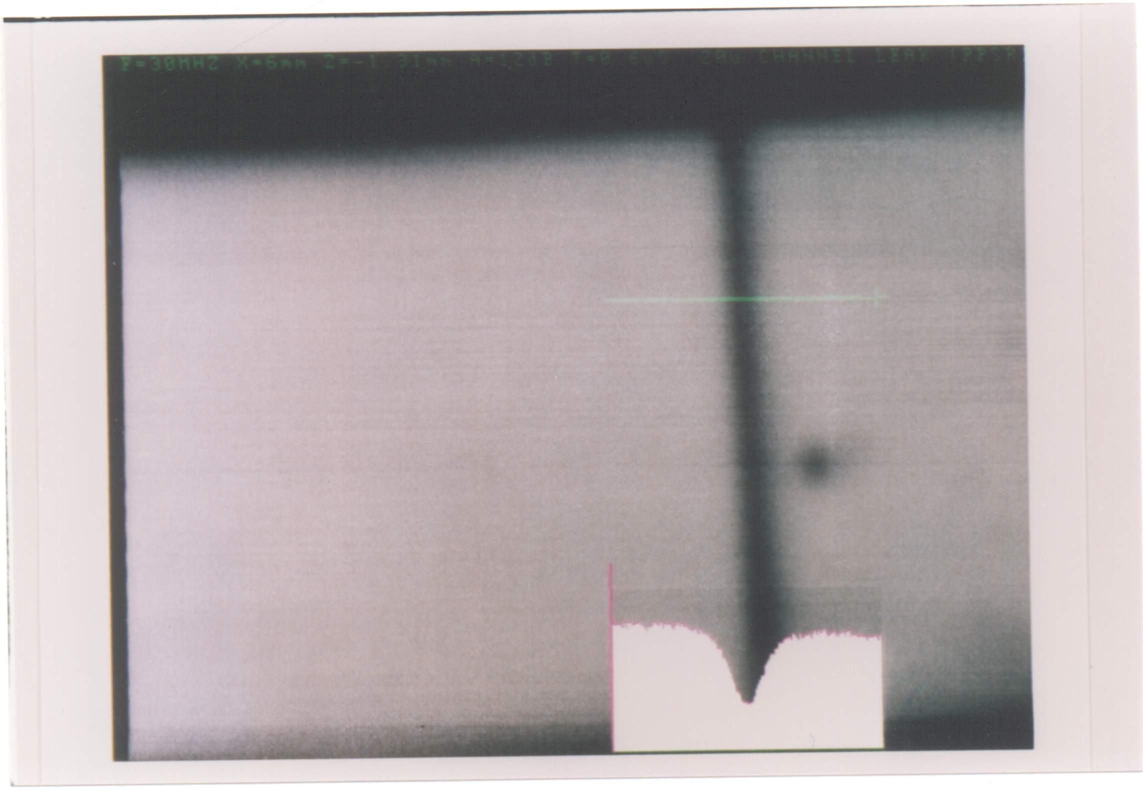

FIGURE 9: SAM IMAGE AT 30 MHz OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF A TRAY SEALED WITH COEXTRUDED PLASTIC LIDDING.

1.3.5. Plastic/Aluminum Lidding

Trays sealed with the plastic/aluminum lidding had seals of the order of 1.35 mm thickness. The first frequency at which a 20 μm channel leaker was successfully detected was at 50 MHz. At this frequency the 20 μm channel leaker was visible both on the SAM image and B-scan as shown in Figure 10. However, the intensity of the bottom reflection at that frequency was low as a result of the attenuation of the acoustic signal by the materials, thus, even though the position of the focal point during inspection was as close as possible to the bottom of the seal (Z-value = -1.60 mm) to maximize the bottom echo intensity. The frequency of inspection was lowered to 30 MHz and the 20 μm channel leaker detected more easily since, as shown in Figure 11, the contrast between the 20 μm channel leaker and the rest of the seal was increased, both on the SAM image and B-scan.

1.4. Criteria for Choosing Ultrasonic Inspection as a Non-Destructive Methods for Heat Seal Integrity Testing

When considering ultrasonic inspection for a non-destructive methods to detect defects such as channels leakers, blisters or wrinkles, a number of criteria were taken under consideration. These can be classified as attribute

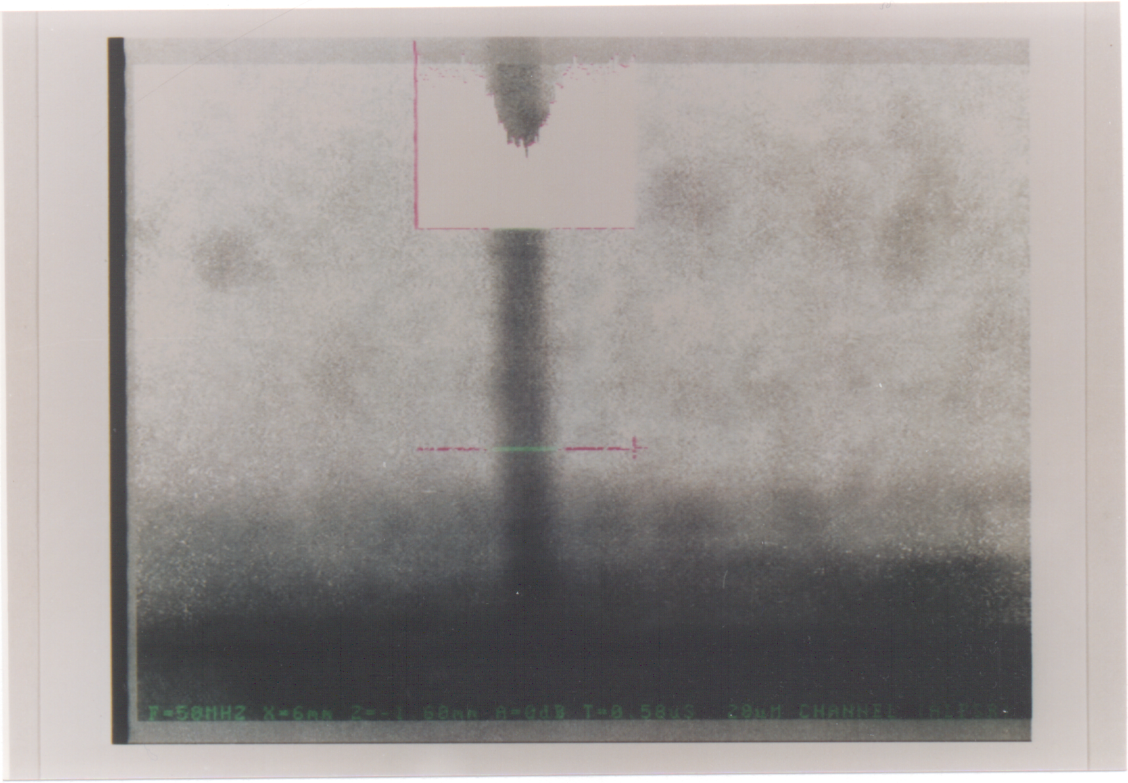

FIGURE 10: SAM IMAGE OF 50 MHz OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF A TRAY SEALED WITH PLASTIC/ALUMINUM LIDDING.

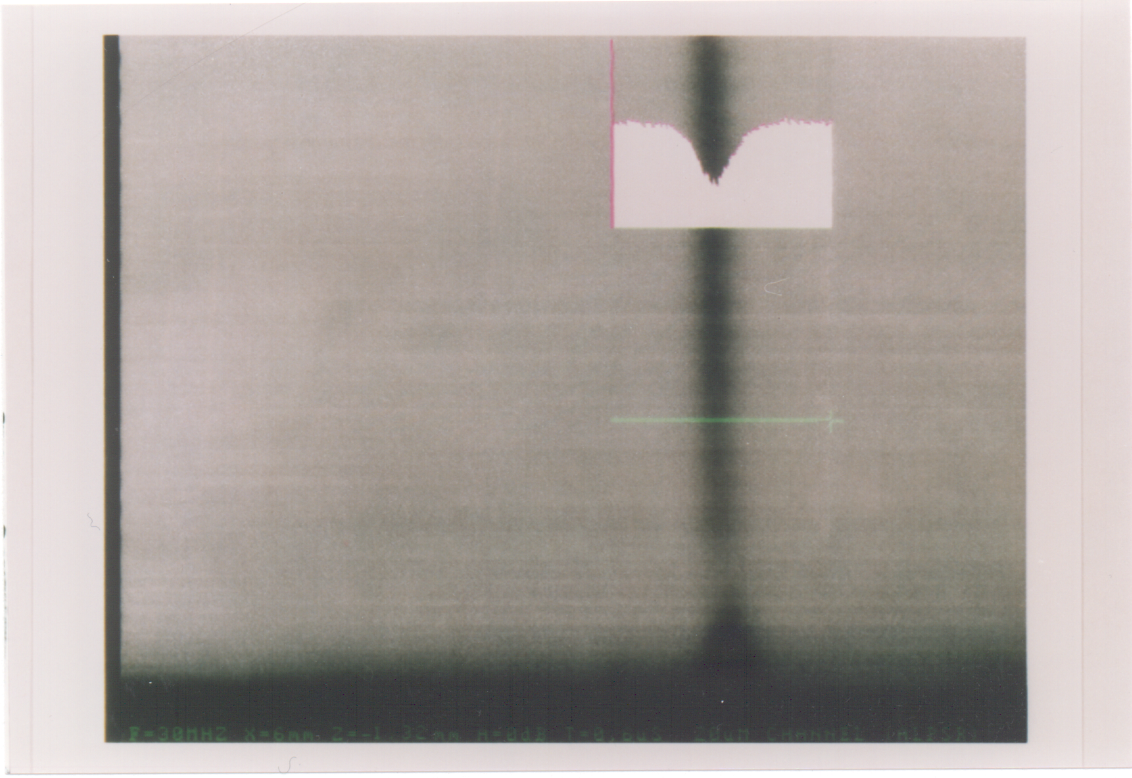

FIGURE 11: SAM IMAGE AT 30 MHz OF A 20 μm CHANNEL LEAKER LOCATED AT THE SEAL INTERFACE OF A TRAY SEALED WITH PLASTIC/ALUMINUM LIDDING.

properties, procedure requirements, and application constraints.

1.4.1. Attribute Properties

Attribute properties refers to the physical properties of the package and its contents which may be exploited as means of detecting lack of seal integrity. A good example of a non-destructive testing technique where attributes properties of the package and its content were combined to allow defect detection is the voltage differential tester. For such methods to be applicable the container cannot contain any conductive materials and its content must have at least 50% moisture. Ultrasonic inspection as explained earlier detects defects located at the seal interface of a container based on their capacity to interfere with the transmission of an acoustic wave sent throughout the thickness of the seal. For this reason ultrasonic inspection appears to be a method of interest since it will work regardless of the package content.

1.4.2. Assessment Procedure Requirements

Procedure requirements refers to the size, shape, location and orientation of the defect to be detected. In case of heat seal closure, these requirements are: the ability to detect any defect that allow post-process contamination, as well as those defects which might cause heat seal to fail during transportation and distribution. These defects have been identified as seal delamination such as channel leakers or blisters, seal contamination, and seal deformation such as wrinkles. Because such defects occur during sealing, they are located at the seal interface and oriented in a plane parallel to the surface of the seal.

Although it is not known at the present time whether or not ultrasonic inspection is able to detect seal contamination, the results of this research showed that defects such as channel leakers, blister and wrinkles can be successfully detected using scanning acoustic microscopy. The differentiation between defects that affect the integrity of a package and those that do not is possible by scanning the width of a seal using several transducers. By analyzing the data points collected by these transducers, it should be possible to distinguish between true leakers and blisters. Wrinkles resulting in a change in the topography of the surface of the seal could be differentiated with channel leakers by gating both the top and bottom reflection of the seal. Wrinkles cause a shortening of the travel time of the top echo while channel leakers located at the seal interface do not cause such a change.

1.4.3. Application Constraints

Applications constraints are those characteristics susceptible to change from one application to another. They refers to requirements such as the geometry and topography of the seal to be inspected, its structure and time available for inspection.

1.4.3.1. Time of Inspection

The time available for inspection might become a limiting factor on very high speed processing lines but, in the opinion of the author should present an improvement compared to the speed of inspection of non-destructive methods such as pressure differential. Ultrasonic wave propagation being done at the speed of sound, the speed of inspection for a given package will be limited mainly by the scanning mechanism, data interpretation and physical dimension of the seal to be inspected.

1.4.3.2. Seal Geometry and Structure

The geometry of the seal should only be a limiting factor for ultrasonic inspection when considering its thickness. As the thickness of a seal increases it is necessary to decrease the working frequency to compensate for the attenuation of the acoustic signal in the material(s). It is known at this time that a 20 μm channel leaker can be successfully detected in most seal materials with the exception of paper laminate. However, since the wavelength decreases as the frequency increases, small defects are easier to detect at high frequency, and consequently, seal thickness might become a limiting factor if a defect smaller than 20 μm is to be detected.

The structure of the seal is also determinant as an application constraint. Ideally, a seal should be as homogeneous as possible, meaning that it should be made of materials having similar acoustic impedances to facilitate ultrasonic inspection. Defects are easier to detect when working with laminated or coextruded plastics material because most plastics used in the food packaging industry, as shown in Table 4, have acoustic impedances ranging between 2 and 3 x $10^5 g \cdot cm^{-2} \cdot s^{-1}$. These acoustic impedances are also ideal since water which is used as a coupling fluid, has an impedance of 1.48 $10^5 g \cdot cm^{-2} \cdot s^{-1}$, and therefore little energy is reflected at the water/plastic interface.

If the seal structure contains an aluminum layer, it can represent either an asset or an inconvenience for inspection depending on its position in the seal. If the aluminum layer is placed between the surface to be inspected and the seal

TABLE 4: ACOUSTIC IMPEDANCES OF VARIOUS MATERIALS USED IN SEMI-RIGID FOOD PACKAGING.

MATERIAL	LONGITUDINAL WAVE VELOCITY ($10^5 cm\cdot s^{-1}$)	ACOUSTIC IMPEDANCE ($10^5 gm\cdot cm^{-2}\cdot s^{-1}$)
ALUMINUM	6.37	17.00
NYLON 6,6	2.68	2.90
POLYESTER	1.84	1.92
POLYETHYLENE	2.67	2.94
POLYPROPYLENE	2.65	2.42
POLYSTYRENE	2.40	2.52
WATER	1.48	1.48

interface, its high acoustic impedance compared to plastics will cause a strong reflection of the acoustic signal due to the impedance mismatch. Therefore it will be necessary to work at a lower frequency. On the contrary, if the aluminum layer is place below the seal interface, it will provide a good background reflection to detect flaws located at the seal interface.

If the seal structure contains a paper layer, it was found impossible to detect small defects located at the seal interface due to scattering of the acoustic signal by air entrapped in the paper. Detection was limited by the intensity of the echo reflected off the defect surface in relation to the attenuation of the material. Embossments on the surface of a lid can have a similar effect and are discussed with the topography of the seal.

1.4.3.3. Topography

In order for the defect detection to be successful, it is important to avoid any features on the surface of a seal that might cause scattering of the acoustic signal or prevent detection. Embossment on the surface of a seal or any surface roughness will cause the acoustic signal to be scattered, especially if the embossment pattern is of the same order or greater than the working frequency wavelength. Ideally, a seal should be perfectly flat and should be kept in the same plane as the transducer operating surface. Consequently, ideally the container should be registered with the transducer surface within one wavelength of the frequency of inspection to prevent any variation in the time of flight of the top and bottom echo of the seal. This also implies that the seal should have a constant thickness to facilitate inspection.

1.5. Machine Specifications for an On-Line Testing Device

It is envisioned at this stage that the on-line ultrasonic testing device will consist of a series of focussed transducers of frequency ranging from 30 to 100 MHz depending on the seal thickness and packaging material composition. Water will be used as a coupling fluid to guide the acoustic signal to the surface of the seal. Because of the variation in thickness occurring at the edges of the seal, these will probably not be scanned to prevent any variation in the acoustic signal that might interfere with defect detection.

2. Bio-Testing

The results of the bio-test of the four sets of MRE pouches and controls are recorded in Table 5. The percentages recorded in this table referred to the pouches showing positive swelling, turbidity and presence of CO_2.

All the pouches submitted to the bio-test were submitted to a dye test in order to determine both the presence of leaks other than that manufactured that might lead to false positive, and if the manufactured leak was actually present. Among the 50 pouches having a 210 μm leak, only one did not show swelling, however this same pouch showed positive results for turbidity and headspace analysis. The absence of swelling was really a loss of swelling due to leakage of the broth during incubation.

Out of the 150 pouches having a 20 μm channel leaker, only 6 showed negative results for the bio-test. After the dye test, it appeared that 2 of these were negative because the manufactured leak was incomplete or absent, and that one pouch out of the 144 showing a positive result had done so because it had a leak in addition to that manufactured.

These results showed that a 20 μm channel leaker located at the interface of a seal 5 mm in width is sufficient in size to cause post process contamination. Although further research needs to be done on the effect of channel leaker length on post-process contamination, the results obtained in this study tend to confirm those obtained by Gilchrist et al. (1985), which indicated that bacteria passed through holes down to about 20 μm in diameter located in the wall of retortable pouches (trilaminate of PE, aluminum, and PP of 127

TABLE 5: PERCENTAGE OF POUCHES SHOWING POSITIVE SWELLING, TURBIDITY AND PRESENCE OF CO_2 AFTER THE BIO-TEST.

LEAK DIAMETER	CONTROL	210 μm	130 μm	100 μm	20 μm
SWELLING	0%	98%	100%	100%	96%
TURBIDITY	0%	100%	100%	100%	96%
HEADSPACE[a]	0%	100%	100%	100%	100%

[a] Headspace analysis was done for 20% of the pouches of each set.

μm thickness). Based on these results, it appears that 20 μm leak are critical to package integrity, and that additional research is needed to determine the smallest channel leaker diameter at which post-process contamination becomes possible. Furthermore, channel leaker length does not appears to affect the likelihood of post-process contamination since identical results were obtained with 20 μm channel leakers of 5 mm and 127 μm in length.

3. Channel Leaker Size Determination

The confirmation of the channel leaker diameter was done before and after the bio-test using both optical microscopy and electrolytic testing.

3.1. Optical Microscopy

The bio-test showed that a 20 μm channel leaker was sufficient to cause post-process contamination, consequently, it was not of any interest to confirm the diameter of the other channel leakers. After delaminating 20% of the pouches containing a 20 μm channel leaker used in the bio-test, and measuring their respective diameter, it was determined that the diameter of the channel leakers were of 20±1 μm in diameter.

3.2. Electrolytic Testing

The attempt to correlate the current intensity with the diameter of a leak present in a 5 mm wide seal was successful when using the retortable plastic pouches provided by Cryovac Inc. Electrolytic testing was done using 1% and 2% salt solutions and using alternating current of 9 and 18 volts. Figure 12 shows the correlation between channel leaker diameter and current intensity as well as a regression for each salt solution and voltage. The regression data and equation for each salt concentration and voltage levels are listed in Table 6.

The R-square calculated for any salt solution and voltage used were all above 0.998, showing a good correlation between the current intensity and the diameter of the channel leaker. These results confirm the work done by Axelson et al. (1990) which showed that the size of microholes can be quantified by measuring electrolyte conductance. However, it was found impossible to repeat this work when working with plastic/aluminum pouches. This is believed to be due to the presence of the aluminum layer which is exposed during leak manufacture. According to Axelson et al. (1990), this correlation between electrolyte conductance and microhole cross section remains true only when working with identical packaging material and test conditions (voltage, alternating

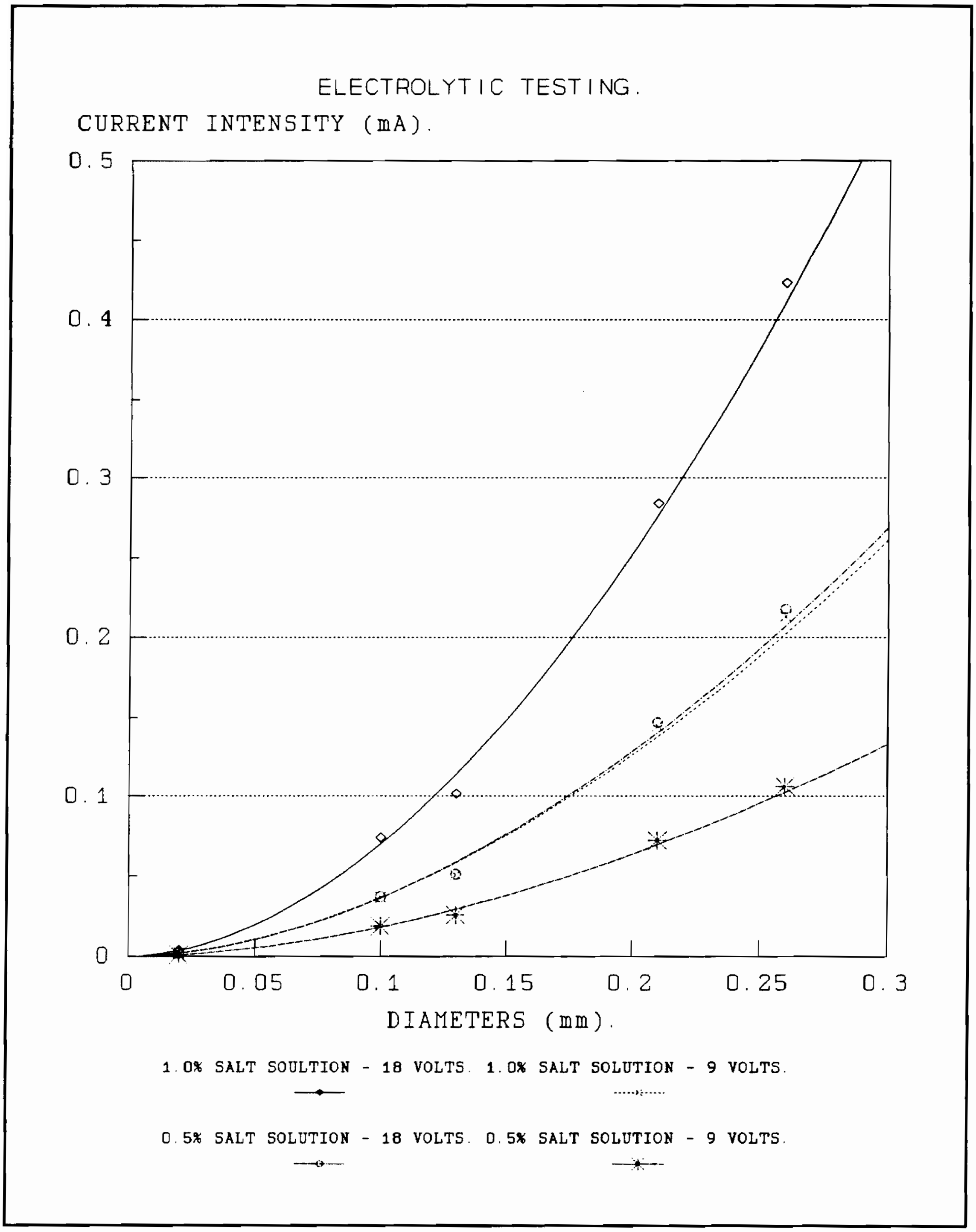

FIGURE 12: ELECTROLYTIC TESTING CURRENT INTENSITY OVER CHANNEL LEAKER DIAMETER.

TABLE 6: REGRESSION DATA AND EQUATION CALCULATED FROM DATA MEASURED AT VARIOUS VOLTAGES AND SALT CONCENTRATIONS.

REGRESSION DATA $Y = A \cdot X^B$	0.5% SALT SOLUTION		1.0% SALT SOLUTION	
	9 VOLTS	18 VOLTS	9 VOLTS	18 VOLTS
R SQUARE	0.99848	0.99818	0.99803	0.99876
B	1.81292	1.82020	1.79266	1.85117
A	1.17903	2.40679	2.26252	4.98193

or continuous current, electrode material, and salt concentration), and with channel leakers of constant length.

VI. SUMMARY AND CONCLUSIONS

Ultrasonic inspection for on-line testing of semi-rigid and flexible container was proven to be possible. The acoustic operational parameters for detection of flaws located at the seal interface were determined for four of the five types of package studied in this research.

Detection of a 20 μm channel leaker located at the seal interface of a paper/aluminum laminate was unsuccessful due to scattering of the acoustic wave by air entrapped in between the paper fibers. The frequencies used for inspection of the other four types of packages ranged from 100 MHz to 30 MHz, the lower limit being determined not by the application constraints, but by the operating frequencies of the scanning acoustic microscope.

The attenuation settings were determined for all four package types, however the actual values are dependent on the electronics being used. As a general rule, higher attenuation settings were required when working at lower frequency and/or with thin and homogeneous materials. The Z-value is also dependent on the transducers used for inspection, nevertheless, the focal point of the transducer should be located on the bottom of the seal or on any interface located below the seal interface and providing sufficient reflection to allow defect detection.

It is the opinion of the author that identification of seal defects should be possible by using several transducers to differentiate between channel leakers and blisters, and by gating both the top and bottom surface echo of the seal to differentiate between wrinkles and channel leakers. It is envisioned at this time that an on-line, ultrasonic inspection device might consist of several focussed transducers using water as coupling fluid. Even though the speed of inspection will be limited by the scanning mechanism, data interpretation and physical dimensions of the seal to be inspected, ultrasonic inspection should be fast enough to accommodate most food processing line.

The main limiting factors for ultrasonic inspection will be in the seal topography. To facilitate inspection this one should be flat, smooth and parallel to the surface of the transducers.

The bio-test performed in this research showed that the ultrasonic inspection needs to be able to detect at least channel leaker 20 μm in diameter since it was found sufficient to cause post-process contamination.

VII. SUGGESTIONS FOR FURTHER RESEARCH

Before ultrasonic inspection can be used as an on-line inspection system, it will be necessary to built a prototype for a specific application to solve problems such as design of the transducers and scanning mechanisms, as well as writing of a computer program to analyze the acoustic signals received by the transducers. This prototype should be used to determine the feasibility of defect identification as described in this research and examine other possibilities to do identify defects.

Further research is needed on the effect of channel leaker diameter and length on post-process contamination. It would be desirable to determine a way to manufacture channel leakers of a diameter smaller than 20 μm in order to determine the diameter at which a channel leaker becomes critical to the integrity of a container. Other factors such as the surface properties of the sealing materials should also be considered in this study.

VI. REFERENCES

Amini, M.A. and Morrow D.R. 1979. Leakage and permeation: theory and practical applications. Package Development & Systems, 9(3):20-27.

Anderson, G.L. 1989. Leak testing. Volume 17: Non-destructive Evaluation and Quality Control. In *Metals Handbook Ninth Edition*, ASM International Handbook Committee (Ed.), p. 57. ASM International, Metals Park, OH.

Andres, C. 1982. Non-destructive inspection of aseptic packaging. Food Processing, 43(1):166-167.

Anonymous, 1991. Safeseal Sales Literature. Alfastar, Box 198, S-22100 Lund, Sweden.

AOAC. 1984. Examination of flexible package for integrity. Ch. 24. In *Bacteriological Analytical Manual 6th Ed.*, p.24-37. Association of Analytical Chemists, Arlington, VA.

ASTM. 1989. Standard definitions of terms relating to aseptic packaging. ASTM F-1167. In *Annual Book of ASTM Standards,* Section 15, Volume 9, *General Products, Chemical Specialties, and End Use Products.* American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA.

Axelson, L., Calvin S. and Nordström J. 1990. Aseptic integrity and microhole determination of packages by electrolytic conductance measurement. Packaging Technology & Science, 3(3):141-162.

Axelson, L. and Calvin S. 1991. Aseptic integrity and microhole determination of packages by gas leakage detection. Packaging Technology & Science, 4(1):9-20.

Bar-Cohen, Y., Mal, A. K., and the ASM Committee on Ultrasonic Inspection. 1989. Ultrasonic Inspection. Volume 17: Non-destructive Evaluation and Quality Control. In *Metals Handbook Ninth Edition*, ASM International Handbook Committee, p. 465. ASM International, Metals Park, OH.

Bongo, R.L. 1978. Ultrasonic sealing of thermoplastic films. Session 11: Manufacturing processes & techniques. In *Plastic in Packaging*. National Technical Conference, p. 170. Society of Plastic Engineers, Inc., Chicago, IL.

Briston, J.H. 1986. Film, plastic. In *The Wiley Encyclopedia of Packaging Technology*, M. Bakker and D. Eckroth (Ed.), p. 329-335. John Wiley & Sons, New York, NY.

Brody, A.L. 1989. High-powered market for high-acid aseptics. Prepared Foods, 158(11):166-168.

Denny, C.B. 1989. Industry action to ensure plastic package integrity. Food Technology, 43(12):88.

Downes, T.W., Arndt, G., Goff, J.W. and Twede D. 1985. Factors affecting seal integrity of aseptic paperboard/foil packages. Proceeding of the Third International Conference & Exhibition on Aseptic Packaging. Aseptipack, 363-401.

Downes, T.W. 1989. Food packaging in the IFT era: five decades of unprecedented growth and change. Food Technology, 43(9):228-240.

Drimmer, V. M. 1985. Plastic package doesn't mean plastic food. Proceedings of National Food Processors Association Conference. Capitalizing on Aseptics II, p.21. Washington, D.C.

Dunkelberger, H. E. 1985. Regulatory policy and status of aseptic packaging materials. Proceedings of National Food Processors Association Conference. Capitalizing on Aseptics II, p.67. Washington, D.C.

Fox, R.A. 1989. Plastic packaging-The consumer preference of tomorrow. Food Technology, 43(12):84-85.

Garret, K. M. 1987. Laboratory testing procedures: Heat sealed package. Proceeding of National Food Processors Association Conference. Plastic Package Integrity: Successfully Meeting the Challenge, p.39-40. Washington, D.C.

Giacin, J.R., Harte, B., Lockhard, H.E. and Richmond, M. 1984. Package testing, present and future. The 1984 Packaging Encyclopedia, 35-44.

Gilchrist, J.E., Rhea, U.S., Dickerson, R.W. and Campbell, J.E. 1985. Helium leak test for micron-sized holes in canned foods. Journal of Food Protection, 48(10):856-860.

Gilchrist, J.E., Shah, D.B., Radle, D.C. and Dickerson, R.W.Jr. 1989. Leak detection in flexible retort pouches. Journal of Food Protection, 52(6):412-415.

Hahn, H. 1989. Plastics and plastic laminates for prefabricated cups. Ch. 5. In *Aseptic Packaging of Foods*, H. Reuter (Ed.), p. 227. The Technomic Publishing Company, Lancaster-Basel.

Halmsaw, R. 1987. *Non-destructive Testing*. Edward Arnold Ed. London, Great Britain.

Hammond, F.M. and Potts M.W. 1989. An overview of flexible packaging markets and structures. Tappi Journal, 72(1):81-88.

Hatano, Y. and Taira, K. 1991. Can a lid have both good seal integrity and easy openability? Journal of Packaging Technology, 5(1):16-20.

Kelsey, R. J. 1990a. On-line monitoring devices. Food & Drug Packaging, 54(5):10-14.

Kelsey, R. J. 1990b. Uptime: the status of leak detection. Food & Drug Packaging, 54(11):8-21.

Kessler, L.W. 1989. Acoustic Microscopy. Volume 17: Non-destructive Evaluation and Quality Control. In *Metals Handbook Ninth Edition*, ASM International Handbook Committee, p. 465. ASM International, Metals Park, OH.

Kopetz, A.A. 1987. Path to package integrity-An historical perspective. Proceeding of National Food Processors Association Conference. Plastic Package Integrity: Successfully Meeting the Challenge, p.1-5. Washington, D.C.

Krautkrämer, J. and Krautkrämer, H. 1990. *Ultrasonic Testing of Materials*. Springer-Verlag Ed., New York, NY.

Lampi, R.A., Schulz, G.L., Ciavarini, T. and Burke P.T. 1976. Performance and integrity of retort pouch seals. Food Technology, 30(2):38-48.

Maros, F.G. 1985. New developments in packaging materials. Proceedings of National Food Processors Association Conference. Capitalizing on Aseptics II, p.69-71. Washington, D.C.

Martin, E.L. 1986. Multilayer flexible packaging. In *The Wiley Encyclopedia of Packaging Technology*, M. Bakker and D. Eckroth (Ed.), p. 451-464. John Wiley & Sons, New York, NY.

Maunder, D.T., Folinazzo, J. F. and Killoran, J.J. 1968. Bio-test method for determining integrity of flexible packages of shelf-stable foods. Food Technology, 22(5):81-84.

Milliner, R.A. 1985. The future of aseptic packaging. Proceedings of National Food Processors Association Conference. Capitalizing on Aseptics II, p.97-98. Washington, D.C.

Morris, C.E. 1989. Convenience foods: The second revolution. Food Engineering, 61(9):69-82.

NFPA. 1989. Flexible package integrity bulletin. NFPA Bulletin 41-L, National Food Processor Association, Washington, D.C.

Put, H., Witvoet, H.J. and Warner, W.R. 1980. Mechanism of microbiological leaker spoilage of canned foods: Biophysical aspects. Journal of Food Protection, 43(6):488-497.

Reuter, H. 1989. Aseptic packaging of foods. Ch. 1. In *Aseptic Packaging of Foods*, H. Reuter (Ed.), p. 3. The Technomic Publishing Company, Lancaster-Basel.

Rice, J. 1988. Seal integrity analyzer. Non-destructive tester handles almost any container type. Food Processing, 49(4):124.

Rice, J. 1989. Ocean spray targets HRI with aseptic 4-oz juices. Food Processing, 50(4):153-155.

Rice, J. 1990. Food processing's packaging survey. Food Processing, 51(3):70-71.

Stauffer, T. 1987. Non-destructive in-line leak testing for flexible packages. Proceeding of National Food Processors Association Conference. Plastic Package Integrity: Successfully Meeting the Challenge, p.1-5. Washington, D.C.

Stauffer, T. 1988. Non-destructive in-line detection of leaks in food and beverage packages-An analysis of methods. Journal of Packaging Technology, 2(4):147-149.

Stauffer, T. 1990. Non-destructive testing on flexible packaging. Journal of Packaging Technology, 4(4):27-29.

Swientek, R.J. 1991. Aseptic low-acid foods *inch ahead*. Food Processing, 52(5):216-220.

Toner, D.F. 1987. Perspectives on package selection-A new ballgame. Proceeding of National Food Processors Association Conference. Plastic Package Integrity: Successfully Meeting the Challenge, p.7-9. Washington, D.C.

Trager, C. 1991. Is there life after juice? Beverage World, 1492:52-54.

Wurts, C.B. 1985. In plant quality control checks for aseptic container integrity. Proceedings of National Food Processors Association Conference. Capitalizing on Aseptics II, p.79-82. Washington, D.C.

Young, W.E. 1986. Sealing, heat. In *The Wiley Encyclopedia of Packaging Technology*, M. Bakker and D. Eckroth (Ed.), p. 574-578. John Wiley & Sons, New York, NY.

VITA

The author, Bruno Jarrosson, was born on June 14, 1963 in Lille, France. In June 1982, he received his Baccalaureate degree from the Lycée Florent Schmitt in Saint Cloud, and in September of the same year, he was accepted in the Ecole Supérieure d' Ingenieurs et Téchniciens Pour l'Agriculture (E.S.I.T.P.A.) in Rouen. After studying agricultural engineering for two years, the author left for New York where he studied the english language and passed the T.O.F.E.L. In February 1985, he was accepted in an officer school in Saint Cyr Coëtquidan and, after five month, graduated as a second lieutenant. He was sent in Varce, in the French Alps, where he completed his military service as a training officer. After a year in the army, the author left for Madrid, Spain, where he worked in the marketing department of a Philips' subsidiary.

In August 1986, the author entered the University of the District of Columbia in Washington D.C. where he studied Food Science and Technology until he graduated in May 1989. Finally, in August of the same year, he began graduate study at Virginia Polytechnic Institute and State University for a Master of Science Degree in Food Science and Technology.

B. Jarrosson