Developments in food packaging integrity testing*

Carol L. Harper, Barbara A. Blakistone, J. Bruce Litchfield and Scott A. Morris

The inspection of both the seal integrity and seal strength of plastic packages containing low-acid shelf-stable foods is currently performed using destructive and manual test methods. Automated non-destructive sensing technologies would allow 100% inspection to be carried out on line. As will be discussed, the evaluation and application of such technologies for package inspection is advancing as a practical solution to the current problems associated with integrity testing. This article also examines how to determine critical defect parameters to be used as design criteria for these technologies.

Foods hermetically sealed in plastic containers offer consumers lightweight, durable, microwaveable, easy-to-open packaging that maintains product quality comparable to that of the more traditional cans and glass containers. Thus, the market has seen a rapid increase in non-traditional shelf-stable packaging.

Container integrity is well understood for traditional packages, particularly cans, but the understanding of factors affecting the integrity of plastic containers is still in its infancy. Federal agencies, the food industry and university researchers are seeking an understanding of microleak size in terms of potential for microbial contamination. Hazard analysis and critical control point (HACCP) programs generally include a thorough examination of container integrity as a critical control point. HACCP may soon be mandatory for seafood[1]; an Advanced Notice of Proposed Rulemaking has been issued for foods regulated by the Food and Drug Administration[2], and the Food Safety and Inspection Service of the US Department of Agriculture is in the process of considering a proposed rule for all meat and poultry processors to develop, adopt and implement HACCP. Further information may be found in Refs 1 and 2 under 'Proposal to establish procedures for the safe processing and importing of fish and fishery products' and 'Developments of hazard analysis and critical control points for the food industry; request for comments', respectively. Given the importance of HACCP, the National Center for Food Safety and Technology (NCFST) is addressing guidelines for satisfactory seal examinations in four phases:

- the evaluation of destructive techniques for microleak detection;
- the determination of the critical defect size that permits microbial contamination;
- the evaluation of non-destructive leak-detection techniques not previously used for this application;
- the development of automated prototype units for non-destructive seal-defect detection.

Evaluation of destructive techniques for microleak detection

Current destructive test methods for determining a loss of seal strength in flexible plastic pouches include tensile testing[3] and burst testing[4]. Burst testing is accomplished by continually adding air to a pouch containing product until a specified pressure is attained. If the package bursts before this pressure is attained, the package fails and subsequent tests are performed to determine where the problem arose. The burst test is a good overall test for seal strength (especially for retortable containers) because it stresses a package uniformly in all directions and identifies the location of the weakest point and the pressure at which it fails[4]. Tensile testing involves each end of a seal being mechanically pulled until separation occurs, and the force that is required to accomplish this separation is recorded. If the force is below a previously designated range, the package is considered to lack adequate seal strength. The disadvantage of tensile testing is that it will not detect weak spots or stress points in other untested areas of the seal. The tensile test is therefore used for the surveillance of material sealability and also to spot-check equipment operations and sealing conditions[4]. When a statistically significant number of samples are tested from a production line at designated timed intervals, these methods

*Suppliers mentioned in this article are not endorsed by the National Center for Food Safety and Technology (NCFST), the National Food Processors Association (NFPA), or the University of Illinois at Urbana-Champaign (UIUC).

Carol L. Harper is at 6000 Oakwood Drive, Apt 3G, Lisle, IL 60532, USA (fax: +1-708-393-1108). Barbara A. Blakistone is with the National Food Processors Association (NFPA), 1401 New York Avenue, NW, Washington, DC 20005, USA. J. Bruce Litchfield is at the Department of Agricultural Engineering, 338 Agricultural Engineering Sciences Building, 1304 West Pennsylvania Avenue, Urbana, IL 61801, USA. Scott A. Morris is at the Department of Food Science, 382 A Agricultural Engineering Sciences Building, 1304 West Pennsylvania Avenue, Urbana, IL 61801, USA.

provide a good means of determining if the parameters ensuring good seal fusion (temperature, pressure and dwell time) are in ranges that provide adequate seal strength.

Seal integrity is defined as a seal continuum, that is complete fusion in the sealing area with no discontinuities. A seal may have a channel microleak (a continuous path through the sealing area connecting the food inside with the external environment) and still have adequate seal strength. Tests done at the NCFST have indicated that burst and tensile test methods (Tables 1 and 2) do not indicate the presence of channel leaks that are 250 μm and 200 μm in diameter, respectively. *F*-test results (from analysis of variance, one way) showed no statistically significant differences between averages at the 99% probability levels[6]. Microleaks of this size are visible. Thus, it may be concluded that microleaks that are not clearly visible, <50 μm in diameter, would similarly not be revealed by burst or tensile test methods. As random splattering of food into the seal area during filling of the packages is one cause of these channel microleaks, removing packages from the processing line, at timed intervals that are statistically adequate for destructive (burst and tensile) testing, will not guarantee the detection of this type of defect because such defects occur sporadically rather than continuously. Destructive methods, if used on a statistically significant number of samples, are excellent indicators of seal strength. They cannot, however, be used to indicate reliably a loss in seal integrity.

Currently, on-line visual manual inspection is being used to evaluate seal integrity, which imposes the limitations of both operator skill variability and human ocular resolution (≈50 μm). It is also costly, having recently been estimated at $10 000 per million packages (C. Sizer, Tetra Pak Research Center, Buffalo Grove, IL, USA, pers. commun., 28 September 1994). To summarize briefly, existing destructive test methods are inadequate for indicating hard-to-see microleaks, regularly timed samplings do not indicate randomly occurring defects, and current inspection methods are very costly. There is clearly justification for increased research efforts to develop 100%, on-line, non-destructive integrity testing systems. Research in this area is needed in two directions: first, the determination of threshold dimensions for defects; second, the development of an on-line non-destructive package integrity evaluation system.

Table 1. Ludlow air burst test results[a]

Burst point (kPa)[b]	Sample description[c]
Average = 300.2, σ = 40.7	No defective seals
Average = 307.7, σ = 17.5	Defective seals with 250 μm channel leaks

[a] Data taken from Ref. 5
[b] Number of samples equals six
[c] Pouch material was a trilaminate of oriented nylon (0.0152 mm), poly(vinylidene chloride) (0.0152 mm) and polypropylene (0.08 mm); sealing was done at 132°C with a 'Metric Model HS-C Doboy' sealer; and 250 μm channel leaks were made by removing a 250 μm diameter wire that had been embedded in the seal
σ, Standard deviation

Table 2. Instron tensile test results[a]

Break-point load (kPa)[b]	Sample description[c]
Average = 220.5, σ = 13.2	Factory seals
Average = 204.3, σ = 14.5	Doboy seals with no defects
Average = 202.6, σ = 18.6	Doboy seals with 200 μm channel leaks

[a] Data taken from Ref. 5
[b] Number of samples equals six
[c] Pouch material was a trilaminate of oriented nylon (0.0152 mm), poly(vinylidene chloride) (0.0152 mm) and polypropylene (0.08 mm); sealing was done at 132°C with a 'Metric Model HS-C Doboy' sealer; and 200 μm channel leaks were made by removing a 200 μm diameter wire that had been embedded in the seal
σ, Standard deviation

Determination of the defect size that permits microbial contamination

To develop an on-line non-destructive package evaluation system, it is imperative that evaluation guidelines are determined first. In other words, what are the minimum defect dimensions that permit microbial contamination?

The problem with determining defect dimensions is that other variables, such as pressure differential, fluid viscosity, temperature, surface activity, microorganism type, channel depth and microbial contaminant concentration, affect the microbial contamination of the food. These factors cause the experimentally determined critical defect size to vary. Table 3 shows a wide variance in the minimum defect size as determined by different experimental techniques, and demonstrates how other variables can affect the results.

Channel leaks are not the only type of defect that can result in spoilage. Wrinkling and defect size and location are characteristics that also need to be considered when evaluating whether a seal is defective or not. Some abrasions, corner dents, blisters and delaminations affect appearance only and do not represent a food safety problem. Definitive evaluation guidelines need to be determined. This can be accomplished through a combination of statistical probability studies (how often does a 10 μm leak actually occur and, if it does, what are the chances that contamination will occur), expert system software development, and comparison of production versus experimental data.

Evaluation of non-destructive analytical leak-detection technologies

A comparison of various non-destructive analytical leak-detection techniques was funded by the NCFST (see Table 4). This should not be seen as a comprehensive list, because there are other alternative technologies, such as speckle interferometry[19] and leak testing using helium and sulfur hexafluoride[20], which, due to time and

Table 3. Minimum defect diameter size for bacterial penetration[a]

Authors	Defect diameter size (µm)	Experimental technique
J.D. Floros and V. Gnanasekharan[8]	10	Predictive equation
G. Howard and J. Duberstein[9]	0.2	Filtration of certain water-borne bacteria
R.A. Lampi[10]	11	Immersion test on pouches
S.W. Keller *et al.*[11]	≈10	Aerosol test
B.A. Blakistone *et al.*[12]	<10	Immersion test
L. Axelson *et al.*[13]	80	Electrolytic test on aseptic packages
J.E. Gilchrist *et al.*[14]	22	Immersion test on pouches
C. Chen *et al.*[15]	10 5	Immersion test on vials Spray on vials
C.L. Harper[16]	<10	Biocell test
D. Rose[17]	<7	Microperfusion test

[a]Adapted from Ref. 7

money constraints, were not included in this study. Information (resolution, cost, speed and comments/applications) was compiled from brief screening procedures.

The exact resolution of the various systems remains to be determined and, in some cases, may be a function of what type of seal defect and what type of packaging material is being examined. For example, machine vision imaging is, for the time being, limited to clear packages, although it has been demonstrated that 'star burst' defects in foil packages can be visualized using this technique. Star burst defects are created when foreign material trapped between sealing surfaces causes a raised unsealed area resembling a star. As the detection of raised star burst defects is dependent on shadow interpretation, surface effects must be present in opaque types of packaging to be visualized.

The limitation of infrared imaging is that it is very sensitive to environmental conditions and requires temperature control. Infrared imaging is most appropriate for monitoring seals as they are being heat set. The infrared laser measures the transmittance of a beam of infrared light through a transparent object; the fiber optic array does the same thing but uses the visible light spectrum. Of the two instruments tested (the infrared laser and the fiber optic array), only the infrared laser was able to identify a defect.

Capacitance is a proportionality constant that expresses the relationship of potential difference between two nearby charged conductors. The package must be passed between conducting plates and the capacitance measured in farads.

X-rays will detect a 1 µm drop of water in either opaque or clear pouches. The resolution of scanning laser acoustic microscopy is also excellent; it can detect defects in either clear or opaque pouches, and is capable of detecting defects containing solids, liquids or gases.

An eddy current probe has been routinely used to detect breaks in the coating of pipes by changes in the transmittance of the electrical current. Although this probe does not work well for clear plastic laminate pouches, it can detect defects in foil laminate pouches such as the 'Meals Ready to Eat' pouches used by the military.

Magnetic resonance imaging (MRI) is capable of producing two- and three-dimensional images using magnetic fields and radio waves. The image is obtained from the radio signals emitted by protons (hydrogen nuclei) in a sample after the nuclei have absorbed energy from an external radio signal. Magnetic resonance responses of hydrogen nuclei in and around a defect are different from those from non-defective seals, as reflected by differences in the signal intensities. However, MRI does not detect voids. The MRI signal tested was derived from protons in water; if there was no water in the defect, there was no useful signal from which to construct an image.

Infrared laser transmittance was also preliminarily examined as an indicator for seal strength[21]. Non-destructive sensing detection of seal strength would be dependent on signal variation through different seal thicknesses. An optimum seal thickness (based on the desired seal fusion) would need to be identified and a corresponding transmittance signal obtained. Preliminary results indicate that non-destructive sensing techniques are capable of evaluating not only seal integrity but also seal strength.

All of the systems had application possibilities; none were infeasible. Although there was an initial failure with the fiber optic array setup, the use of different wavelengths, transmitters and/or receivers, and improvements in the technology might result in success. Startup costs only indicate what the initial research investment will be. All systems cost much less when mass-produced and used for just a single application. Different types of packaging may require different sensors. The combination of several methods will create a more flexible system. Ultrasound and machine vision imaging systems are currently being developed for this application. X-ray on-line systems already exist but would need to be fine-tuned for this application and combined with a sensor that 'sees' air defects. MRI is also a good candidate for future development and application.

For the past six years, the Kingston Research and Development Center of Alcan International Ltd (Kingston, Ontario, Canada) together with Alcan Germany (Ohle, Germany) have been working on a heat-seal inspection system for on-line applications. After an extensive

Table 4. Evaluation of current non-destructive methods[a]

Technique[b]	Resolution data (from screening procedure)	Startup cost[c]	Speed	Comments/applications
Machine vision imaging Sony black-and-white SSC model M350 with a Computar 16 mm lens, a Javelin 25 mm lens, and a Nikon 55 mm lens	269 µm	$15 000–25 000	Real time (30 per sec)	Available; proven; flexible; inexpensive; lighting is a problem as it has to be very precise; would only work for clear packages if used on its own; probably needs to be combined with another system for opaque packages
Infrared imaging Inframetrics Model 600 infrared camera	1000 µm	$30 000–50 000	Real time (30 per sec)	Can deal with opaque packages; worked best right after sealing and is therefore inflexible in line placement; resolution can probably be improved
Infrared laser/fiber optic array Omron model Z4LA-1030/ SUNX model FX-7	Infrared laser: solids: 125 µm gas: 200 µm Fiber optic array: > 200 µm	$1000 per sensor	Real time (30 per sec)	Has so far been proven to detect macroleaks; the infrared laser detected the difference between samples with 125 µm wire channels, with 200 µm air channels and without any defects 100% of the time
Capacitance Designed and built by the Agricultural Engineering Department, University of Illinois, Urbana–Champaign, IL, USA	120 µm	$200	Real time (30 per sec)	Can detect macroleaks; perhaps it could be put right in the sealer; requires less processing than machine vision imaging
Spectrophotometer BYK Gardner Color Sphere™	125 µm	$20 000	Real time (30 per sec)	Needs to be combined with another system for opaque packages. Some spectrophotometric software has been developed and is close to application
X-ray LIXI Inc., FIS Series Inspection System, LIXI Inc., Downers Grove, IL, USA	1 µm	$100 000	Real time (30 per sec)	Good resolution; sees through opaque packages, but does not 'see' air, only denser materials, so would have to be combined with other sensors; depends on >10–20% difference in structure
Ultrasound Sonoscan SLAM system, Sonoscan, Bensenville, IL, USA	10 µm	$150 000	Real time (30 per sec)	Requires a denser medium than air (such as water) through which to transmit the sound waves which are then measured by the laser vibrometer
Eddy current probe Xacttex, Pasco, WA, USA	200 µm	$400	Real time (30 per sec)	Package needs to come in contact with metal
Magnetic resonance imaging 4.7 T SISCO imager and an 8 cm imaging probe developed at the University of Illinois, Urbana–Champaign, IL, USA	30 µm	$200 000	In the order of seconds	Detects channel leaks with water (or liquid food) in package seal

[a] Data taken from Ref. 18
[b] Machine vision imaging, infrared imaging, infrared laser/fiber optic array, magnetic resonance imaging and capacitance systems were screened under the direction of J. Bruce Litchfield (UIUC) through an NCFST grant; spectrophotometer, X-ray, ultrasound and eddy current probe systems were screened by Carol L. Harper (NCFST)
[c] Costs are for the detector technology, not for the equipment needed to interpret signals, nor for conveying systems to shuttle packages into, through and out of the testing unit or chamber

review of existing options, a novel ultrasonic technique was successfully developed and patented, which the developers claim offers reliability, robustness, rapid operation and ease of adaptability to existing filling lines (for technical details of the system, see Ref. 22). The inspection system consists of an array of through-transmission ultrasonic sensors in the configuration of the sealing die, thereby eliminating the need for mechanical scanning. The system is reported to provide 100% coverage of the seal region during an inspection cycle of ~20 ms. This is advantageous as it can be simply integrated into heat-sealing equipment. An important feature of this system is its simplicity; unlike other conventional ultrasonic devices it does not require water immersion of the container. Preliminary tests suggest that system operation is independent of the package material, with promising results obtained for both steel-laminated and plastic heat-sealed packages. An exhaustive evaluation of the prototype by the developers is presently under way, while the design of a commercial version is being considered.

Developmental guidelines for automated prototype units for non-destructive seal-defect detection

There are currently several commercial on-line packaging inspection system companies. Work is being done at the NCFST to evaluate which defects could lead to a compromise in package integrity, and to develop defect identification guidelines. Defects will be ranked in categories as to their severity (using a system similar to the system used in the National Food Processors' Association Bulletin 41-L; see Ref. 3). Once these criteria are established, then equipment manufacturers can calibrate inspection systems. The NCFST is also examining a prototype unit to help determine criteria needed in an inspection system (alarm files and operator alert signals, data files, calibration techniques, etc.) to detect these defects and thus ensure food safety.

Developers of on-line systems using these technologies should consider several options for improved efficiency and accuracy: (1) splitting the production line between more than one non-destructive sensing system could help increase the production rate; (2) several technologies could be combined in one system so that a broader spectrum of packaging materials could be examined; and (3) resolution could be improved by signal amplification and line scanning or multiple sensors, thereby decreasing the area to be examined. Line scanning would physically move one sensor over the seal, evaluating only a small area at each real-time location. Multiple sensors would each evaluate a smaller area; they would be configured in the pattern of the seal. The first and third options would also be applicable if a larger area, such as a package body, needed to be examined.

Conclusions

In summary, evaluation of destructive techniques for microleak detection (seal integrity) has determined these techniques to be inadequate. Although microbial contamination can occur through microholes as small as 0.2 μm in diameter[9], other variables need to be considered. Further work needs to be done and guidelines need to be written to define exactly which seal defects might result in bacterial infiltration. This information needs to be combined with non-destructive sensing technique prototypes for seal evaluation. The development of these systems would make these alternative types of packaging more financially competitive and expand their applicability beyond a niche market.

References

1 *Fed. Regist.* (1994) 59, 4142
2 *Fed. Regist.* (1994) 59, 39888
3 Flexible Package Integrity Committee of the National Food Processors' Association (1989) in *Flexible Package Integrity Bulletin, NFPA Bulletin 41-L*, pp. 18–19, National Food Processors' Association, Washington, DC, USA
4 Hendrickson, B.P. (1989) in *Special Project (completed as part of the long term Food Technology Training Program, 1988–1989)*, pp. 32–39, Department of Health and Human Services, Public Health Service, Food and Drug Administration, Washington, DC, USA
5 Harper, C.L. (1993) *Annual Report of the Collaborative Research Programs*, The National Center for Food Safety and Technology, Summit-Argo, IL, USA
6 Ott, L. (1988) in *An Introduction to Statistical Methods and Data Analysis*, pp. A8–A19, PWS-Kent Publishing Co., Boston, MA, USA
7 Blakistone, B.A. (1994) *Proceedings of the IoPP Packaging Technology Conference, November 11–12, 1994, Chicago, Illinois*, Institute of Packaging Professionals, Herndon, VA, USA
8 Floros, J.D. and Gnanasekharan, V. (1992) in *Advances in Aseptic Processing Technology*, pp. 157–188, Elsevier
9 Howard, G. and Duberstein, J. (1980) *J. Parenter. Drug Assoc.* 34(2), 95–102
10 Lampi, R.A. (1980) *J. Food Process. Eng.* 4, 1–18
11 Keller, S.W., Marcy, J.E., Blakistone, B.A. and Lacy, G.H. *J. Food Protect.* (in press)
12 Blakistone, B.A., Keller, S.W. and Marcy, J.E. *J. Food Protect.* (in press)
13 Axelson, L., Cavlin, S. and Nordstrom, J. (1990) *Packag. Technol. Sci.* 3, 141–162
14 Gilchrist, J.E., Shah, D.B., Radle, D.C. and Dickerson, R.W. (1989) *J. Food Protect.* 52(6), 412–415
15 Chen, C., Harte, B., Lai, C., Pestka, J. and Henyon, D. (1991) *J. Food Protect.* 54(8), 643–647
16 Harper, C.L. (1995) in *Plastic Package Integrity Testing Assuring Seal Quality* (Blakistone, B.A. and Harper, C.L., eds), pp. 79–82, Institute of Packaging Professionals, Herndon, VA, USA
17 Rose, D. (1994) *Technical Memorandum No. 708, Risk Factors Associated with Post Process Contamination of Heat Sealed Semi-rigid Packaging*, Campden & Chorleywood Food Research Association, Chipping Campden, UK
18 Harper, C.L. and Litchfield, J.B. (1994) *Annual Report of the Collaborative Research Programs*, The National Center for Food Safety and Technology, Summit-Argo, IL, USA
19 Allemeir, R.T. (1995) in *Plastic Package Integrity Testing Assuring Seal Quality* (Blakistone, B.A. and Harper, C.L., eds), pp. 113–118, Institute of Packaging Professionals, Herndon, VA, USA
20 Kronberg, J.W. (1995) in *Plastic Package Integrity Testing Assuring Seal Quality* (Blakistone, B.A. and Harper, C.L., eds), pp. 95–106, Institute of Packaging Professionals, Herndon, VA, USA
21 Severino, T. (1993) *Senior Report* (with J.B. Litchfield as Advisor), Department of Agricultural Engineering, University of Illinois at Urbana-Champaign, IL, USA
22 Alcan International Ltd (1994) US Patent US 5 372 042/European Patent EP 0 493 457