Part 1 of 2

New Sensors Help Improve Heat-seal Microleak Detection

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FOCUS: A recent study demonstrates the ability of acoustic imaging to nondestructively image hidden, obscured or overprinted micrometer-scale defects as well as inclusions in the seal area in the heat-seals of film-sealed and flexible packages. The implications of this are that an acoustical method may be the best basis for developing an accurate on-line sensor capable of 100 percent inspection of heat-seals at production-line speeds. This study shows ultrasound to be an effective and precise way to image defects in food packaging materials, and to detect them to the limits of resolution of the equipment used. The study provides the basic acoustic data needed to further develop a robust, dependable on-line dedicated sensor for 100 percent package seal inspection.

Introduction

The implementation of several broad classifications of new process/package systems has begun to change the way food is packaged. These include thermally processed film-sealed shelf-stable plastic cans and trays suitable for microwave reheating and retortable pouches. These new package types are alternatives to traditional cans and jars, which use mechanically compressed gasket-seals, and instead utilize welded-plastic heat-sealing of the food contact layer of the package.

In addition to changes in the fundamental technologies of food packaging, the processing of food products is moving towards more High-Temperature, Short Time (HTST) and aseptic processing technologies, where food is processed and packaged in a sterile environment and is not subjected to post-filling thermal treatment. Although these food processing and packaging changes offer increased consumer convenience and better product quality (due to reduced overprocessing), they are potentially susceptible to microbial contamination through undetected package defects.

Editor's Note: This is Part 1 of a two-part series on the development of new on-line dedicated sensors for package seal inspection. Part 1 deals with developing and evaluating ultrasound sensing methods, while Part 2, which will run in the August issue, will discuss the development of a new Backscattered Amplitude Integral (BAI) pulse-echo sensing method.

While these new packaging and processing techniques represent advances in technology, inspection methods have lagged behind. Without adequate inspection technologies, the potential exists for pathogen growth in packaged foodstuffs as well as contamination of similarly packaged pharmaceutical products.

Overt, large-scale mechanical damage is easily detected during production, or by the consumer, but seal defects or inclusion in the micro-meter (µm) range can pose a hazard that would only become evident in retrospect. Also, the flexible nature of these package components makes the packages inherently undependable in indicating vacuum loss with low levels of gas formation, a traditional indicator of spoilage with more conventional rigid packages. A better method of inspecting these new types of packages is needed to guarantee package integrity and product safety.

The Need for Better Inspection Systems

Heat-seals in packages are usually formed by applying energy to the sealing surface of the layers of materials to be sealed. These seals are subject to the formation of defects from a variety of sources, such as wrinkles in the materials, inclusion of foreign materials (including the packaged food) and post-sealing mechanical damage. Such defects may compromise the hermeticity of the package.

Current quality control techniques for defects depend on human visual inspection (as mandated by 9CFR§381.301(d)) and hold-and-inspect methods by process batch whereby entire production lots are held and samples are inspected for evidence of microbial growth after a suitable incubation period. With global canned food production in the

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tens of billions of cans per year, neither of these inspection methods is fast enough to allow metal cans and jars to be cost-effectively replaced by the new-style packages when packaging pathogen-susceptible foodstuffs.

Current test methods for determining a loss of seal integrity and strength in flexible plastic pouches include burst testing, squeeze testing, tensile testing, trace gas "sniffers," pressure change devices and visual inspection. When a significant number of samples are tested from a production line at designated intervals and destructively tested with the former three methods, these methods provide a good means of determining if adequate macro-scale seal strength exists, though these methods are too insensitive for finding micron-scale leaks. Trace gas (usually helium) "sniffers" are quite sensitive, but have the same problem as pressure change devices in that a leak or channel in the heat-seal may be plugged with product, providing a ready-growth path for microorganisms, but not allowing gas exchange for leak indication. Of the test methods listed above, then, only visual inspection is suitable for micro-leak detection.

Currently, human on-line visual inspection is being used with these new types of packages. This imposes the limitations of both operator skill variability and human ocular resolution (ca. 50 µm). It is also costly: $10,000/million packages (*Harper et al., 1995*). Moreover, internal or seal defects that do not produce sufficiently large surface artifacts will go undetected, particularly in opaque materials.

The physical dimensions and mobility of most types of pathogenic organisms are well documented (*Harper et al., 1995*). Similarly, studies using intentional micro-defects have been done to determine the ability of microorganisms to penetrate membranes. What is not yet known is the lower limit of the ability of pathogenic microorganisms to penetrate a transverse channel formed in a heat-sealed package.

A compilation of minimum defect sizes that have resulted from the work of various researchers was presented by Harper et. al. (*1995*). The experimentally determined values for minimum defect sizes have ranged widely between 0.2-80 µm, as have experimental conditions, giving no clear-cut lower limit.

Human visual inspection suffers from the predictable effects of fatigue and error. Machine inspection is possible and practical but has inherent limitations in the ability of the sensors used to detect the sorts of hidden defects that can provide an entry pathway for pathogenic organisms. Further limitations occur when using machine-vision types of sensors to detect defects in materials that are opaque or have obstructive overprinting. The Harper et al. survey and trial of leak-detection methods shows that of the methods available, even in laboratory scale, for leak detection in food packages, only magnetic resonance imaging (MRI), acoustic imaging and x-ray imaging potentially meet the criteria of finding small internal defects in relatively thick sections of opaque materials.

X-ray systems have so far not been able to demonstrate the ability to find an unfilled leak with any useful resolution (due to the lack of differential in electron density between a whole seal and two unsealed but adjacent pieces of material). MRI has an extremely long computation and resolution time (by production line standards). This leaves acoustic imaging as the best alternative for the development of a sensor that would provide the needed sensitivity, spatial and temporal resolution (when used as a dedicated linear scanning sensor to provide high feed-through speeds).

Acoustic imaging in the frequency range that yields micrometer-scale spatial resolution is termed acoustic microscopy. Because the acoustic microscope uses the change in mechanical properties between regions to achieve image contrast, and because ultrasound can penetrate opaque materials to a limited depth, the information obtained by the acoustic microscope can offer new insights into the micromechanical behavior of thin materials and thin bonded materials, and can lead to the development of a more efficient sensor for package inspection.

The Scanning Laser Acoustic Microscope (SLAM), used in this initial study, is a transmission acoustic microscope that can image bulk properties of a thin specimen and measure the bulk wave ultrasonic propagation properties. This class of transmission acoustic microscope operates in the frequency

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range between 10 and 500 MHz and has corresponding spatial resolution (inversely proportional to frequency) in the range of 200 and 4 μm, respectively. Generally, transmission acoustic microscopes are sensitive to the bulk transmission property variations.

The SLAM uses transmitted sound waves imaged with a scanning laser on the surface of the coupling medium (usually degassed water) instead of light to produce images, and has the documented ability to detect, classify and accurately reproduce the internal structure and defects of opaque materials (*Briggs, 1985, 1992*). Acoustic imaging techniques (virtually all of which use pulse-echo techniques at much lower frequencies and, thus, with lower resolution) have successfully been used to detect voids, flaws and cracks in epoxy-fiber composites, metals and biological materials, but at the outset of this study had not been able to successfully combine penetration depth and the necessary resolution to provide a useful sensor for this type of application.

Methods and Materials

To test the hypothesis that heat-seal defects may be detected by acoustic microscopy, channel defects were prepared by sealing tungsten wires (10, 16, 25 and 37 μm dia.) between two layers of plastic material, either polyethylene Ziploc bag material or plastic microwaveable retort pouch material (Fuji Tokushu Shigyo Co. Ltd., Seto Aichi, Japan). The wire was then pulled out, leaving cylindrically shaped channel defects transverse to the heat-seal. A small region of the channel defect was carefully cut out and placed on the acoustic microscope stage for imaging. This was done without disturbing the channel region to be imaged. Some samples were also frozen and cut to preserve geometry of the channel (for independent characterization of the channel with a confocal microscope) using a cryo-microtome cutter. Operational details of the scanning laser acoustic microscope (SLAM) have been published previously (*Embree et al., 1985; Tervola et al., 1985; Tervola and O'Brien, 1985a*).

A. SLAM Operation and Measurements—Briefly, the specimen is placed on a sonically activated fused-silica stage along with a thin layer of normal saline and covered with a semi-reflective cover slip. A focused scanning laser detects a dynamic surface displacement ripple by reflection from the cover slip's lower surface (Figure 1). The reflected laser beam is processed to yield the acoustic image, from which the attenuation coefficient is determined, and the interference image, from which the speed and heterogeneity indices are determined. The laser beam also is transmitted through the specimen to yield an optical image.

All three images are displayed in real-time on a standard television monitor representing a specimen area approximately 3 mm horizontally by 2 mm vertically (typically 100X). A defect containing air should show enough of a shift in sound penetration to provide significant contrast, highlighting the defective area.

B. Image Processing—Grayscale images, representing the reflected signal's value at a point as a value between 0 (black) and 255 (white), were obtained from the SLAM and the confocal laser-scanning microscope (CLSM) in a digitized form. Image intensity was adjusted to bring out salient features, and the images were arranged by channel size and type of material used (polyethylene or plastic laminate). SLAM optical and interference images of plastic laminate and polyethylene film channels fabricated from the four different wire diame-

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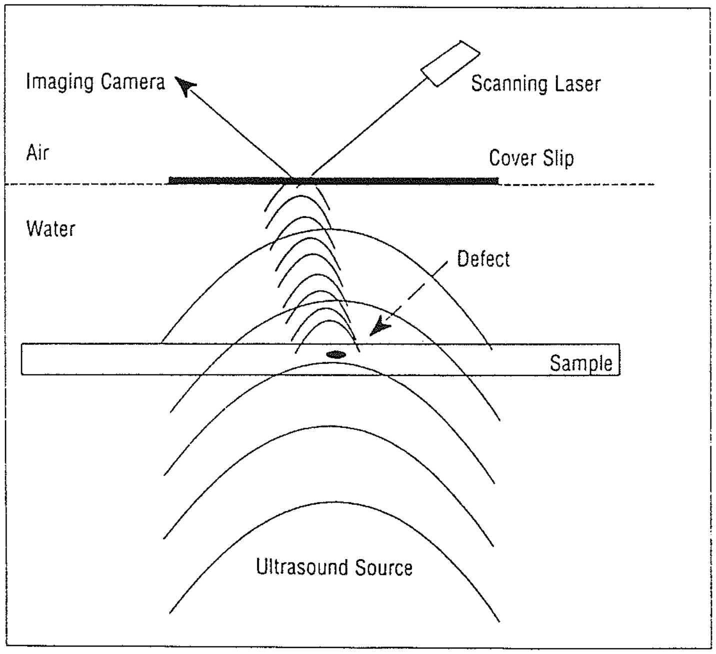
Figure 1

Operation of Scanning Laser Acoustic Microscope (SLAM)



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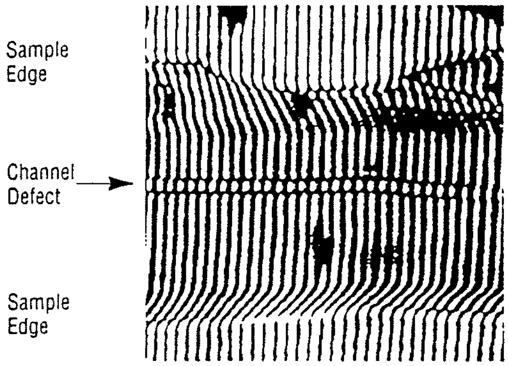
ters were obtained from the acoustic microscope. Figure 2 shows a 10 µm channel defect in opaque plastic laminate. Note that the image clearly shows the channel.

As for channel contents, sample channels were filled with saline solution, and data indicated that there was enough of a shift of the speed of sound even through the saline-filled defects to provide contrast in the images. This verified that the channels could be detected even when filled with a conductive medium and that channels could be classified as being "full" or "empty."

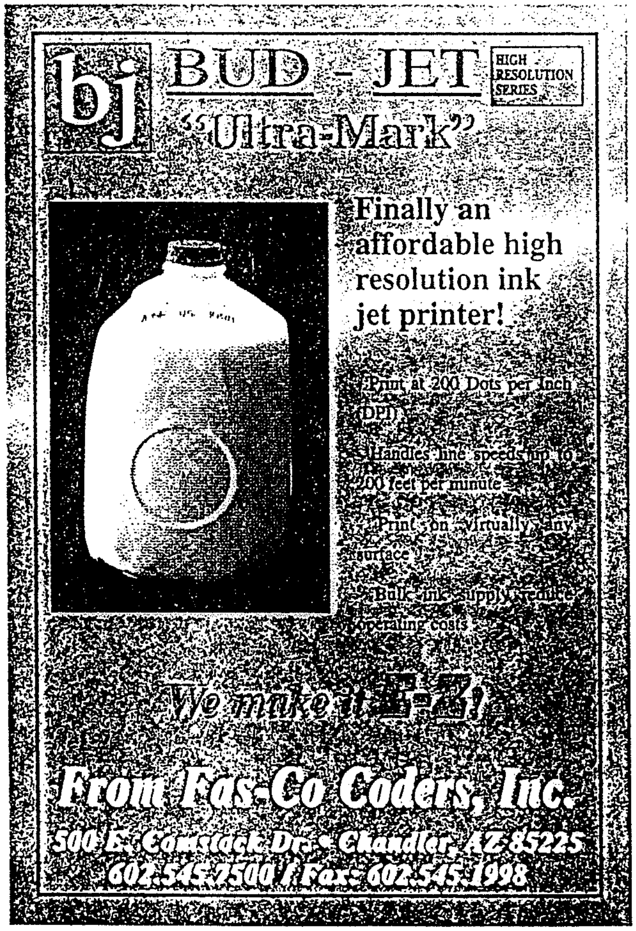
C. Confocal Microscope Verification Measurements—Characterization of the sample in terms of channel size and distinctive features was achieved using a Zeiss CLSM. The CLSM can produce topographical images and can make micron scale measurements on the image with a resolution of less than 0.5 µm. The microscope was used to measure channel diameter with an end view and side view. The side view also showed the uniformity of the channel diameter.

Figure 2

Image of 10 µm Channel Defect in Foil/plastic Retort Pouch Laminate



In confocal mode, the depth of field is in the range of a few microns so that a topographical image can be obtained. Confocal microscopy using the CLSM was used as an independent verification method to characterize the channel size and shape. Based on CLSM measurements, the four channel different diameters in the sealed plastic film corresponded well to the respective wire diameters used to create the channel. All of the channels showed a circular-shaped channel when sec-



tioned and imaged end-on with the CLSM.

Results for sealed plastic film samples having diameters of 10, 16, 25 and 37 µm were shown to be repeatable by fabricating a new set of laminate samples three times and measuring their diameters via confocal microscopy. There was a 3 percent to 5 percent difference between the channel diameters measured by the confocal microscope and that of the actual wire diameters (within the resolution limits of the confocal microscope).

Discussion

Channel defects in polyethylene film were more detectable than in plastic laminate film due to reduced thickness (136 µm for two layers of polyethylene vs. 230 µm for two layers of tri-laminate), which affected total sound transmission through the samples. This was compensated to some extent by using image processing (brightness and contrast filters) to bring out salient features. The imaged channel size in the SLAM images of smaller (10-25 µm) channels did not correspond well to the size of the wire used to make the channel.

A possible explanation for the inability of the SLAM to differentiate channel size near its resolution limit is attributed to the general diffraction-limited capability of imaging techniques. This implies that SLAM cannot measure diameters close to the resolution limit but, nevertheless, can detect the fact that a channel defect exists. As the diameter of the defect is increased, the channel size becomes more clearly and accurately defined in the acoustic image, as expected. In the saline solution-filled channels, a reduction in propagation speed occurred in the interference image for both polyethylene and plastic laminate channels, allowing the discrimination between filled and unfilled channels.

Conclusions

This preliminary study clearly demonstrates that acoustic imaging can nondestructively

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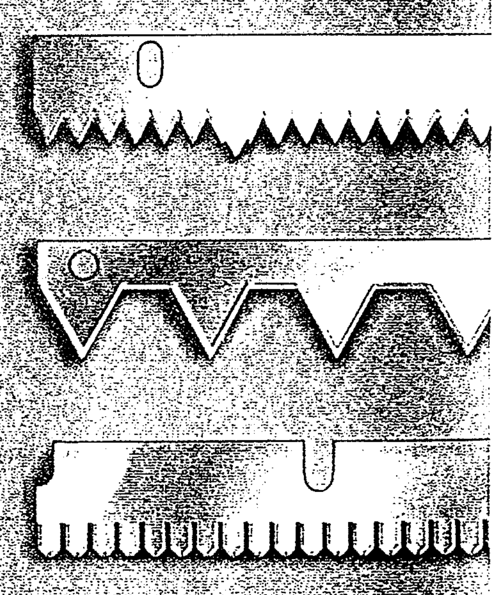

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image micrometer-scale defects as well as any sort of inclusions in the seal area in heat-seals irrespective of their optical properties. The implications are that an acoustical method may prove to be the basis for the development of an accurate on-line sensor capable of 100 percent inspection of heat-seals at production-line speeds. The results obtained significantly exceeded expected results. A 10 µm channel defect was detected using a SLAM with a 20 µm resolution. However, a 10 µm channel defect could not be measured with useful accuracy. If it were necessary to characterize channel defects in the 10 µm diameter range, higher frequency acoustic microscope technology could be used.

The spatial resolution of the SLAM is inversely proportional to frequency. Therefore, when the frequency is doubled, the resolution is halved. Based on these results, if a 500 MHz SLAM were used (that is, 4 µm resolution), channel defects in the range of smaller than 10 µm should not only be detected but also characterized. However, as ultrasound frequency is increased, penetration depth decreases since the attenuation coefficient is frequency-dependent. This tradeoff between imaging depth and resolution may limit the depth of defect detection in food packaging.

This study conclusively demonstrates the ability of ultrasound to be an effective and precise way to image defects in certain food packaging materials and to detect them to the limits of resolution of the equipment used. The on-line implementation of a SLAM-based method is impractical since the equipment is cumbersome and expensive. The data obtained using the instrument has provided the basic acoustic data needed to develop a robust and dependable on-line dedicated sensor for 100 percent package seal inspection. ☐

For more information about this topic, contact the author at (217) 333-9330 or email smorris@uiuc.edu. If this article is helpful in improving your packaging operation, **Circle 190** on Reader Service Card.

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