Real-Time Terahertz Diagnostics for Detecting Microleak Defects in the Seals of Flexible Plastic Packaging*

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Abstract
We present a method of detecting production faults in flexible plastic packages using terahertz (THz) radiation. A focused THz beam is scanned along the sealed area of a package, and the transmitted signal is collected. Defect detection is effected through the large difference between the absorption coefficients of plastic and water for water-filled channel defects, and on the refraction index difference between plastic and air for air-filled channel defects owing to reduction of the incident signals into the detector. Compared to previous methods, such as visual and ultrasound inspection, our technique can be applied to optically opaque packages and does not require immersion in a matching liquid. The method was tested on fabricated 10~100-µm diameter water-filled and air-filled channel defects imbedded in polyethylene films. The detection limit (the minimum size of a detectable defect), which depends on the conveying speed, was determined and analyzed. The results show that our system has potential for application in actual-production, real-time, inspection.

Key words: Terahertz Radiation, Nondestructive Inspection, Flexible Plastic Package, Microleak, Defect, Detection, Heat Seal, Diagnostics, Industrial Machine, Electromagnetic Measurement

1. Introduction
Lying between millimeter and far infrared waves, terahertz (THz) radiation has properties of both sides of the electromagnetic spectrum. Like radio waves, it penetrates a wide variety of substances, such as plastics, dried food, various powders, paper, cloth, ceramics, wood, bone, and fat. Like light waves, it is easily propagated through space and reflected, focused, and refracted using special optics. Further, its sub-millimeter wavelength is much shorter than that of usual radio waves, yielding a spatial resolution sufficient for many applications.

This article focuses on detecting sealing flaws in plastic packages. To prevent post-process contamination, flexible plastic packages for food and pharmaceuticals are produced by fusing two opposing sealing surfaces hermetically. Besides preserving product quality, these packages are durable, lightweight, easy opening, and cost effective.
Obviously, there should be no flaws in the seal, no wrinkles or bond failures, and the seal must not fill with water, air, food material, or other contaminants, as they can induce a pathway for microbial penetration, resulting in spoilage or pathogen growth. Channel leaks are a particular problem. Currently, seal region defects are detected through visual inspection or destructive testing. However, channels smaller than 50 µm in diameter are not clearly visible to the naked eye, and destructive testing is only valuable statistically(9). Further, there is no standard for the minimum detected defect size; human visual inspections are limited by the opacity of the materials used in these types of packages and the resolution limitation of the human eye. Due to these traditional testing methods, shelf-stable food packages are generally re-inspected for spoilage after storage, increasing product costs and reducing production speed.

An ultrasonic-based inspection technique for examining seal integrity that can examine opaque materials and has a higher resolution than visual inspection has been developed(10)~(14); it accurately detects and images seal inclusions. However, it requires immersing the packages in water, a likely issue in real-world application.

In this study, we propose a new nondestructive inspection method that uses THz waves as the probing radiation. THz waves transmit well through various plastic films, and are highly sensitive to even small amounts of water or aqueous solutions, due to the large absorption coefficient of water (176 cm\(^{-1}\) at 0.6 THz). To confirm this theoretically plausible advantage, we developed a system to measure the technique on artificial defects of controlled size. The defects were fabricated in flexible plastic packages as channel-shaped voids with diameters in the range of 10 to 100 µm. The applicability of this system to real-world testing was evaluated by studying the relationship between the scanning speed, which corresponds to the conveying speed in an actual production line, and the minimum detectable size of the channel defects. Our results prove that the system is applicable to real-time inspection.

2. Materials and methods

2.1. Sample preparation

Two packaging materials were used for the samples. One was a two-layer transparent composite film made of nylon (N) and polyethylene (PE) with a total film thickness of 70 µm (N 23 µm, PE 47 µm), as shown in Fig. 1 (a). The PE layer is the inside heat-sealing component of the package; the outer N layer gives strength, including resistance to shear, chemicals, and scuffs to the printed surface, and presents a barrier to gases, oils and fats. The second material was an 80-µm-thick monolayer opaque film of low-density polyethylene (LDPE) film impregnated with white pigment.

To fabricate the different size channel defects, we used various diameter tungsten wires.
(from 10 to 100 µm, in 10-µm steps). Each wire was placed between two layers of the packaging materials, transverse to the sealing direction, and then sealed using an automatic heat-sealer. The sealer applied 0.2 MPa pressure between its jaws (one made of nickel-chromium alloy covered with Teflon and the other of silicon rubber) set at 115°C for 0.6 seconds. The seals were left to cool in air for three minutes before the wires were pulled out. To produce water-filled channel defects, water was forced through the channel after removing the wire. Figure 1(b) shows the final stage of sample preparation, with all edges sealed to keep the water inside. Air-filled channels were produced the same way, just without the water insertion. Transmission optical microscopy was used to check the channel contents, especially for the channels smaller than 50 µm in diameter. The transmittance of both the transparent and opaque packages in the heat-sealed area is around 90% at 0.6 THz.

2.2. Inspection system and measurement procedure

As shown in Fig. 2, the THz-based detection system consists of a backward wave oscillator (BWO) as the continuous wave source, parabolic mirrors, a linear scanning stage, a pyroelectric sensor, a low noise preamplifier, and a personal computer. The output frequency of the BWO was set to 0.6 THz (wavelength \( \lambda = 500 \) µm). The THz wave generated from the BWO was first reflected on a glass plate with an indium tin oxide (ITO) layer deposited on its surface; this is highly reflective in the THz range and transparent in the visible. This facilitated visible light alignment with the parabolic mirrors. After reflection from the ITO layer, the divergent THz beam was collimated by parabolic mirror PM1 (focal length \( f = 150 \) mm) and focused on the sample by PM2 (\( f = 75 \) mm). The beam spot size measured in the scan direction is about 0.55 mm (full width at half maximum). The linear stage holds and moves the sample in the same way a conveyor belt would in an actual production line. The beam strikes perpendicular to the sample plane, and the sample moves along the sealing line, as seen in Fig. 1(b). The speed of this linear stage is arbitrarily selectable from 1 to 800 mm/s, with 800 mm/s being fast enough to match real-world applications. This variability allowed us to investigate the important relationship between linear stage speed and the minimum detectable channel defect size.

The THz intensity transmitted through the sample was projected onto the pyroelectric sensor using PM3 (\( f = 75 \) mm) and PM4 (\( f = 100 \) mm). The sensor signal was filtered and amplified with the low-noise preamplifier and sampled at 10 kHz with an analog-to-digital converter.

![Fig. 2. Defect inspection system using THz radiation. BWO: Backward wave oscillator, ITO: glass plate with an indium/tin oxide layer; PM1, 2, 3, and 4: off-axis parabolic mirrors (focal lengths 150, 75, 75, and 100 mm, respectively); PS: pyroelectric sensor.](image-url)
Procedurally, a channel defect 100 µm in diameter was first tested at the maximum speed of 800 mm/s. If a defect was detected, the next sample, with a 90-µm defect, was evaluated at the same 800 mm/s speed. The defect size was then decreased in 10-µm steps to find the minimum detectable size at this speed. When the defect became undetectable, the stage speed was reduced in 50-mm/s steps until the defect reappeared. We fabricated at least two water-filled and two air-filled samples for each channel size with each sample being scanned several times while shifting the scanning line between measurements. In all, 234 sets of experimental data were acquired and processed to insure repeatable, reliable results.

3. Experimental results and discussion

3.1. Water-filled channel defects in transparent samples

Figure 3 shows an example of a fabricated water-filled channel defect as seen with a transmission optical microscope. To increase the visibility of the defect profile for optical measurement, a small amount of black ink was added; to avoid data contamination, it was not added to the THz samples. The actual defect size was 10 µm, the same diameter as the

![Image of transmission optical microscope image of a fabricated water-filled channel defect](image_url)

Fig. 3. Transmission optical microscope image of a fabricated water-filled channel defect, 10 µm in diameter.

Fig. 4 shows an example of transmitted THz signals through fabricated water-filled channel defects. The respective speeds of the linear stage and channel diameter are (a) 800 mm/s and 100 µm, (b) 800 mm/s and 40 µm, (c) 800 mm/s and 20 µm, and (d) 450 mm/s and 20 µm.

![Graphs of transmitted THz signals through water-filled channel defects](image_url)

Fig. 4. Transmitted THz signals through fabricated water-filled channel defects. The respective speeds of the linear stage and channel diameter are (a) 800 mm/s and 100 µm, (b) 800 mm/s and 40 µm, (c) 800 mm/s and 20 µm, and (d) 450 mm/s and 20 µm.
tungsten wire used in its production. The microscope photographs indicate that this method produces microleaks of controlled size, with an error of less than 10%.

Figure 4 shows examples of the THz scan results for several channel diameters. Due to the detector characteristics and internal electronics, as well as to the filters applied, the acquired signal was an approximate derivative of the actual transmitted signal. Therefore, the only signal variation detected in a specified frequency window was produced by defects in the package seal. The 100-µm water-filled channel defect can be seen clearly in Fig. 4(a), as indicated by the arrow. The 40-µm water-filled channel defect can also be distinguished, albeit with a poorer signal-to-noise ratio, see Fig. 4(b). Some lower frequency wavy variation and quasi-periodic signals are also visible in the graphs. The slow variation is ascribed to slight bending of the flexible package and to non-uniform thickness; the periodic signals come from the fabric-like texture of the heat-sealed area. However, the real defects, as in (a) and (b), are easily detectable because both the amplitude and frequency of the signal differ obviously. For the 20-µm water-filled channel defect observed at 800 mm/s in Fig. 4(c), any defect signal is buried in the noise. However, when the speed is reduced to
450 mm/s, the signal is again detectable, as seen in Fig. 4(d). This scanning speed dependence property on the defect detection was attributed to the beam spot width, geometry, wavelength and the sampling frequency of the detector.

3.2. Air-filled channel defects in transparent samples

An example of a fabricated air-filled channel defect, photographed through a transmission optical microscope, is shown in Fig. 5. The sharp, straight outline of this channel demonstrates proper fabrication of the air-filled defects.

Figure 6 shows the THz inspection scan results. The diffraction and refraction of the THz wave in the defect reduced the incident signals into the detector. Therefore, these optical properties make it possible to detect not only water-filled, but also air-filled channel defects in flexible packages; the THz-based detection system can be used to detect both kinds of defects in flexible food packages.

Figure 6(a) shows the result for a 100-µm air-filled channel defect. The output signal-to-noise ratio is quite low, because the absorption of the THz wave is near zero in the air-filled defect compared to the water-filled; however, it can still be detected. Reducing the defect size to 70 µm and maintaining the speed at 800 mm/s, as shown in 6(b), leaves the defect nearly indistinguishable, as the frequencies of the signal and noise are almost equal, although the amplitude differs visibly. Although the 50-µm air-filled channel defect cannot be found at a speed of 800 mm/s, as seen in Fig. 6(c), the same defect can be detected at 600 mm/s, see Fig. 6(d).

3.3. Opaque samples

The THz transmission of polyethylene, as well as other plastic films, is generally not
related to the visible range transparency or color. Furthermore, the small amount of dye (white, black, etc.) used in the production process had an insignificant effect on the THz transmission properties. Therefore, packages made of transparent and colored plastic films behaved identically in the THz tests. Figure 7 shows an example, as seen using reflective optical microscopy, of a fabricated water-filled channel defect inside an opaque white flexible package. The water in the channel contains a small quantity of black ink, leaving a vague outline of the channel inside the white package. Of course, such a defect is difficult or impossible to detect visually, especially when the channel is filled with a transparent medium. Two demonstration scans are shown in Fig. 8, where (a) is the result for a water-filled channel defect and (b) that for an air-filled channel defect inside opaque white packages. Both channels defects were 50 µm in diameter, and were observed at a stage speed of 65 mm/s. This result proves that the THz inspection system can detect both kinds of defect, regardless of whether the package is transparent.

4. Summary

Figure 9 summarizes the results obtained in this study. The horizontal axis represents the speed of the linear stage, corresponding to the speed of a conveyor belt in an actual production line; the vertical axis is the minimum detectable size of the channel defect. It was quite hard for us to fabricate the channel defects of less than 10 µm by hand. Then, the measurements of the 10 µm water-filled and air-filled channel defects were statically conducted, and they were detectable. Therefore, the 10 µm channel defects were plotted at 0 mm/s of line speed in Fig. 9. As the graph also shows, the dataset of the air-filled defect detection was least-square fit to linear functions. The dataset of the water-filled defect detection was also the same fit to linear functions though the data amount was lacking. That is why the characteristics of the water-filled defect detection could be the same characteristics as the air-filled one. The air-filled defect detection limit is worse than that for water-filled channel defects because, at the same diameter, the water absorption combined with the diffraction has a stronger effect than the refraction and diffraction alone of an air-filled channel. The ultrasound-based inspection technique(10), (13), one of the recently developed sophisticated methods, has assured 100% detection of 38-µm air-filled channel defects at a quasi-static scanning speed of 1 mm/s (see the speed of the transducer, c\textsubscript{e}, in Ref.(13)). In comparison, for the same speed range, our THz inspection technique can detect air-filled channel defects of less than 10 µm. Further, the THz method can detect 38-µm air-filled defects at scanning speeds up to 400 mm/s.

In-line detection of channel leaks as small as 50 µm in diameter is judged to provide...
adequate safety assurance\(^{(15)}\). For air-filled channel defects, it is difficult to detect channels smaller than 50 µm at speeds over 600 mm/s. However, there are not examples of detection ability on a real-world conveyor belt at such high speed. Therefore, we believe that our THz inspection system is a significant development that is qualified for real-world applications with the potential to improve product packaging safety.

References


