

**TESTING THE INTEGRITY OF FLEXIBLE BARRIER
MATERIALS UTILIZING A UNIQUE CORONA BEAM
TECHNOLOGY**

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ABSTRACT

The porosity, permeability or integrity of a non-conductive medical and consumer flexible barrier packaging material is determined utilizing a novel electron beam technology and electronic instrumentation in an open atmosphere. The electron beam developed in an open atmosphere maintains its prescribed frequency through the use of a nitrogen cover gas, ionizing the gas to create a corona beam. The corona beam discharge, maintained at a high positive voltage, forms from the holes or anomalies in the flexible non-conductive barrier material. The anomaly is detected and analyzed in order to determine the presence of viral and sub-viral sized voids or holes, as well as other anomalies such as blisters and bubbles. This process can be performed by the flexible barrier film manufacturer to certify a quality level. It can be performed by the material fabricator to ensure quality standards. It can also be performed by the product manufacturer that uses the packaging material to wrap their products and confirm the integrity of the sealed package by measuring the atmosphere inside the finished package. There are many other packaging applications that can utilize this technology for validation and integrity testing within the medical device, pharmaceutical and food industries.

INTRODUCTION

This technology relates to a method and apparatus for the on-line, real-time, non-destructive 100% testing and measuring of porosity and anomalies of non-conductive thin flexible barrier materials and products made from these types of materials. These products include flexible barrier materials for pharmaceutical, medical, and food packaging. This also includes laminated adhesive materials to foils, paper and plastic film. Another area of compliance testing includes; surgical grade gloves, protective barrier gowns, condoms, encapsulation devices, commercial packaging, and filtration media.

The detection of viral and sub-viral sized apertures, voids, holes, blisters, contaminants, stress fractures, overlapped material, formulation defects and other anomalies are critical to the product's protective or performance function. More particularly, this technology relates to an electronic measuring method and apparatus which utilizes electron beams in an open atmosphere (i.e.; Corona Beams) and electronic instrumentation to measure the constructive and destructive harmonics of the frequency loaded electronic corona discharge from the holes or anomalies in order to qualify and quantify their integrity or performance criteria.

BACKGROUND

There is much concern among world health organizations and regulatory agencies regarding the quality of non-conductive materials, including protective flexible barrier materials or products, such as pouches, thermoformed trays, blister packages, etc. The concern is due to the fact that disease-causing bacteria and viruses can pass through small holes or voids present in these materials or goods, thus infecting or contaminating the end user with a hazardous or contagious material. These holes or voids may be formed during the manufacturing of the non-conductive products or pursuant to anomalies present in the non-conductive materials. Accordingly, the present technology has been developed for the testing and measuring of the porosity and anomalies of these non-conductive materials. In particular, the present technology has been developed for the on-line, real-time, non-destructive, non-contact, non-abrasive, 100% dry testing and measuring of non-conductive materials, including thin film protective barrier materials, for voids, holes or anomalies having a diameter as little as one nanometer. Moreover, the present corona beam technology can detect anomalies in the material such as contamination, blisters, bubbles, un-catalyzed or unblended resin, low density material (e.g. weak molecular cross linking strength), high density material, overlapping material, stress fractures, formulation defects and other structural and non-void anomalies in the material.

Products and materials used as barriers for viruses and bacteria must and should have their porosity and anomaly presence determined in order to insure that no imperfections are present or may be formed by processing which would permit the passage of a virus. These bacteria and viruses may be as small as twenty nanometers in diameter.

One primary test for determining the porosity of nonconductive thin film materials is the water or electrical hydraulic test. Using this test, the product or material to be tested, such as a glove, is placed on a conformal shaped electrode or mandrel and is submersed in a water bath or electrolyte solution. An electrical potential is applied between the mandrel and the water solution. If there is a void of material in the glove, the water will pass from the charged water bath to the electrode, causing a short circuit. A current reading will be displayed on a connected ammeter indicating that a defective void exists in the material, and the material is rejected. However, this test can only determine if there is a sizable hole in the material. It cannot reveal the presence of an anomaly, such as a blister or a bubble, in the material because the blister or the bubble will not break in the water test, thus not permitting the water to pass from the water bath to the ground. However, a blister or bubble could very easily break in the use of a glove and thus the glove would fail during use. Therefore, this test would not find the defect in the glove. Another drawback of the water test is that only holes of about fifty microns or greater will be detected, due to the surface tension of the water. It has been proposed to add soap or alcohol to the water in order to decrease the surface tension. However, even with a lower surface tension, this test will not detect viral size holes, contaminates the product and produces a gray measurement with out the exact location identified.

A second primary test which is used to determine the porosity of materials is called a dry test or a spark test. This method involves an electrically charged brush, charged with 1300 to 1500 volts A.C., 60Hz. The brush is conductive and it brushes against the mandrel, which the material (e.g. the glove) is placed on. Both the brush and the mandrel rotate. When there is a large void, about 50 to 100 microns, the voltage from the brush will spark through as a straight forward, very thick spark. Because it is a strong discharge, it creates large holes. This method is destructive because the brush touches and breaks the glove.

A third test is disclosed in U.S. Pat. No. 5,196,799 entitled METHOD AND APPARATUS FOR TESTING A PROTECTIVE BARRIER MATERIAL FOR PINHOLES AND TEAR STRENGTH and issued to Beard et al. This test is basically a water test that is conducted at different frequencies, not just D.C. or 60 Hz. This method permits the discovery of holes, bubbles and blisters in the material being tested. This is a capacitive test in which distance, environment and thickness of the product are critical to the repeatability and calibration of the test. It is an integral measurement meaning it measures relatively large areas as one "gray" measurement. It is also a wet tester. One of the inherent problems with wet testing is that after the material has been wet then it must be dried, and usually dried with hot air. The hot air, containing ozone, can weaken the nonconductive material and thus increases or enhances the number of voids that might be present in the final product.

In addition, the above tests are used in conjunction with more extensive destructive test methods to statistically sample lots of finished product. Laboratory testing has shown that a given finished production lot, there may be a few, if any, defective product. Thus more accurate and reliable process testing of each manufactured product is needed.

The foregoing illustrates the limitations known to exist in present porosity testing methods and apparatuses. Thus, it is apparent that it would be advantageous to provide an alternative porosity testing method and apparatus which will non-destructively detect viral size voids, blisters, bubbles, and other anomalies on the production line in fluctuating environments and in real-time for non-conductive materials.

TECHNOLOGY

In general terms, the porosity or presence of anomalies of a non-conductive material is determined by using an electron sensor in an open atmosphere under a fluid cover gas, or a flow of a cover gas. The cover gas is directed on the material and, if there is a small aperture, hole or anomaly in the material, a change in the electric discharge or "corona" (also known as an electron beam, an electrostatic corona or a corona discharge) occurs, which is measured by a sensor. The electron sensor comprises an electrode and a sensing mechanism which records electrons that are drawn through the hole or anomaly in the nonconductive material. The sensor also contains a series of focusing resistors for attenuating the beam. The occurrence of this change in discharge is due to the below-described Griebel-Gormley Aperture Effect (sometimes referred to herein as "the Aperture Effect").

It should be noted that anomalies in the material include, but are not limited to, contamination, blisters, bubbles, un-catalyzed or unblended resin, low density material (e.g. weak molecular cross linking strength), high density material, overlapping material, stress fractures, formulation defects, and other structural and non-void anomalies in the material.

It should also be noted that the diameter of the anode tip in the electron sensor, the quality of the plating material (e.g., barium, platinum, gold, silver), and the heating of the anode and cathode tips are factors that relate to the quality and length of the electric discharge (i.e., the corona beam) that is detected and quantified. Other important factors are the dielectric quality of the material being tested, the type of defect that is being tested, and the operating parameters of the testing equipment, such as the frequency, the amplitude, the wave shape and the voltage. The proper combination of these factors leads to the ability to detect sub-nanometer size apertures, holes or anomalies in the material being tested.

The Griebel-Gormley Aperture Effect is based on the point-to-point effect, a known effect, which is described in a text by Sir Thomas Moore, "Electrostatics" (Doubleday & Company, Inc.1968), which is incorporated herein by reference. That in practical terms is the passage of a static electrical charge from a cathode to an anode in an open atmosphere, (i.e.; like static electric from a carpet in a dry room and collected by your shoes and then discharged when near a ground). The Griebel-Gormley Aperture Effect is shown by the use of a smooth, rounded grounded cathode (i.e., approximately cylindrical) in proximity to a tip of an anode (a point). Very few electrons (or corona) are discharged if the voltage is too low. But when the cathode is masked with a dielectric material containing a very small void of material (a hole or anomaly), an electrical point is masked out on the grounded cathode. A point-to-point effect is created and electrons flow from the cathodic roller through the hole or anomaly in the dielectric material to the anodic tip of the gun without increasing the applied voltage. This flow of electrons (the corona beam) carries a prescribed frequency that is measured to determine the amount of constructive and destructive changes that occur. What electrons flow through the material at a location that is defined as "good" material creates the material's signature. It is from this baseline signature that the range of acceptability is established. The amount of change can then be compared to a library of readings contained in the system for comparative identification. The system can also be set to a range of acceptability for a pass or fail response.

A cover gas is also important in achieving the Aperture Effect. Typical cover gases include nitrogen, noncombustible gases, noble gases, and dehydrated air. The results vary with the particular cover gases used. It makes a dramatic difference whether nitrogen is used opposed to air or neon or other noble gases. The flow rate and gas pressure are also important factors; the higher the gas pressure, the more gas flows and the beam lengthens. As the pressure increases, the gas becomes denser, and the electrons flowing from the cathode to the anode become slower moving. It should be noted that the beam may move and wander in the cover gas environment. The beam is self-seeking within the material focus area that is created by the fluid cover gas. Thus, the beam moves in the

area of the material bounded by the fluid cover gas in order to locate properly sized aperture or anomaly and creates a focal area on the surface of the test material that can vary based on the setting's parameters.

Other important factors in the creation of the Griebel-Gormley Aperture Effect are the power supply voltage, the frequency of the pulsed D.C., and the distance from the cathode to the anode. Moreover, the distance between the cathode and the material being tested is an important factor in obtaining the Aperture Effect. If the material being tested is too far from the cathode, the Aperture Effect will be lost. However, this can be alleviated when a conductive noble gas is grounded and used to supplement the difference in the conformal space required between the material and the mandrel.

APPLICATION

The U.S. NIH funded the original proof of concept model that was presented to a group of NIH and the FDA scientists for comments on practical application. The U.S. FDA purchased the first prototype that was used in a study that conclusively (100%) discovered holes and anomalies in condoms from 1 micron and above (Reference: Journal of Testing and Evaluation, ASTM Publisher, January 1999). This accuracy assures the barrier against body fluid transmission, thus, viral transmission. The actual capable accuracy is to 0.5 nanometers. This puts real-time measured integrity testing on an atomic level. The FDA sponsored the ConverTec Corporation in the 1997 R&D 100 Awards Competition. ConverTec was Selected by R&D Magazine as "One the 100 Most Technologically Significant New Products of the Year".

Simply stated; the truly unique aspect of this technology is that the corona beam is drawn and not projected. The beam will follow a tortuous path through material and can curve to follow its anodic high voltage, milliamp potential in the created cover gas carder while in an open atmosphere.

The application of this technology as it relates to flexible barrier material can be applied in numerous ways and at several process levels. Some of these approaches have been mentioned in the previous text. The first format would be to test and certify the raw or initial processed barrier film. The manufacturing facility has the opportunity to certify that the manufactured film meets a prescribed and now certified quality barrier level. The manufacturer has an opportunity to analyze the quality right after the aggregate has been converted. The test could be performed as a secondary operation if the material has to be cured in a secondary process. However, if the material is fully cured at the end of the process line, the product could be tested with a series of sensors that would account for the entire area of the product's surface (100%) by a moving or stationary gantry with an array of beams each averaging a focal area of an eighth of an inch to a full half inch wide. The method of removal from the process is critical. The basic idea is to eliminate the labor factors of re-handling the product to keep the cost as low as possible and test on-line.

Another format is to test for the consistency of permeability. This would create a range requirement for air and gas permeability. This would accommodate the requirements for the different types of sterilization that are currently being used. It would also have the ability to facilitate other newer methods. This permeability level could also be calibrated and certified so that consistency is maintained. This would prevent some material from not seeking through tests on a gray level. The third integrity test of barrier applications for this technology is to certify a range of acceptability for filtration material. A sieve requirement or strainer level can be well defined on a very stringent level with a close level of tolerance.

The technology also has other unique applications that can be “food for thought” for this committee. Since we can test for blisters and bubbles in flat material, a similar type of test, for validation, has evolved that provides for testing the sealed material. After the product has been sealed in the pouch or the blister (etc.), if there has been an atmosphere change due to the addition of a head gas or a vacuum drawn, the atmosphere in the pouch (etc.) has been altered. The corona beam passing over the sealed container will provide a signal that validates that the gas has not escaped and that the at that point in time is solid.

Conclusion

This corona beam technology can be validated by the current standard mechanical tests that are used on a destructive statistical basis to confirm a small percentage of a total lot of goods. Now, the technology is available to test flexible barrier material 100% to application standards that can be precise, certified and validated, while at the same time saving money and time to each processor in the manufacturing chain.

Publications & Awards

U.S. Patents Granted: #5,824,882 October 20, 1998, #5,844,406 December 1, 1998

Journal of Testing and Evaluation, Validation of a Corona Discharge Technique to Test Male Latex Condoms for Pinhole Defects, By: Bruce A. Herman and Ronald F. Carey; ASTM January 1999, Vol. 27, Number 1

AGS Glovebox Magazine, Testing the Integrity of Glovebox Gloves for Sub-Viral Size Defects Utilizing Electron Beams, By: Gregory J. Gormley, American Glovebox Society, August 2000

Vacuum Technology & Coating Magazine, Testing the Integrity of Thin Film Material for Sub-Micron and Nano-Sized Anomalies, By: Gregory J. Gormley, December 2003

R&D Magazine, R&D 100 Awards, 1997, VirusShield Test System, “Selected by R&D Magazine as One of the 100 Most Technologically Significant New Products of the Year”