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# Biodegradable Corrosion Inhibitor Packaging for Electronic Equipment

BORIS A. MIKSIC, FNACE, BOB BERG, AND BOB BOYLE, Cortec Corp., St. Paul, Minnesota

Biodegradable products are necessary because of swelling landfills, and there is a demand for electrical equipment packaging that uses a biodegradable film with similar properties and price to a nonbiodegradable film. This article describes a biodegradable film that provides excellent contact, barrier, and vapor phase corrosion inhibition, while qualifying as an anti-stat biodegradable corrosion inhibiting film.

he use of polymer films for thorough protection of electronic equipment during shipment or storage should take into account the prevention of electrostatic discharge (ESD), corrosion, and the disposal of the film after use. There is an industrial need for a biodegradable film with similar properties and price as a nonbiodegradable film.1 An anti-stat biodegradable corrosion (ABC) film is a polymer film that not only provides thorough protection, but offers anti-stat ESD protection, corrosion inhibition, and biodegradation. These film properties facilitate a modern packaging material for electronic equipment that is truly complete. The most recent property addition to this film is biodegradability.

Biodegradation is a term used for materials that convert to another species, within a reasonable amount of time, such that the original material is no longer present. To define this time factor, there are several organizations, such as the Biodegradable Products Institute (BPI), that require specific test procedures and biodegradation rate requirements for a film to be certified biodegradable under its trade name. The BPI uses the test method ASTM D64002 to test for biodegradability.

A second property that makes an ABC film a viable packaging material for electronic equipment is the film's ability to eliminate electrostatic discharge. Electrostatic discharge is the transfer of electric charge between two or more objects with different electrical potentials that come into contact. This flow of electrical current is often irrelevant, but in the case of electrical equipment, serious damage can occur during storage and shipping if the proper precautions are not met. Damage such as tribocharging can occur when friction generated by the rubbing of materials leads to large differences in electrical potential. Even more damaging are the effects of electrostatic induction,

TABLE 1	
Surface resist	ivity of
common mate	rials4

Material	Surface Resistivity (Ω/square)	
Plastics	>1012	
Anti-static	1010-1012	
Static dissipative	106-1012	
Conductive	10-10 <sup>6</sup>	
EMI products	10-10 <sup>4</sup>	
Metals	10-1-10-5	

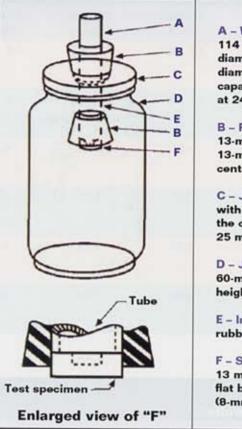
which happens when the electrically charged object is placed near a conductive object isolated from ground. The presence of the charged object creates an electrostatic field that causes electrical charges on the surface of the other object to redistribute. Even though the net electrostatic charge of the object has not changed, it now has regions of excess positive and negative charges. This can cause irreversible damage to several types of electrical equipment, most notably in microelectronic components.<sup>3</sup>

Electrostatic discharge prevention through the use of film packaging must be done using a conductive material that can be incorporated into a film. Three types of materials are available for incorporation into a film: anti-stat, static dissipative, and conductive materials (Table 1).<sup>4</sup>

For static dissipative and conductive materials, a significant amount is incorporated into an entire layer of the film, which causes the film to become static dissipative or conductive. Since the film contains a significant amount of static dissipative or conductive material, it will not biodegrade within a reasonable amount of time.

Anti-stat materials have a lesser degree of conductivity, and the loading amount in a film is very little. Therefore, anti-stat materials are a prime candidate to use for the prevention of ESD damage. An ABC film contains an internal anti-stat, as opposed to an external topographical anti-stat, which is incorporated into the film at a 1% or less value. Initially, this internal anti-stat additive has a homogenous

## FIGURE 1



A - Water retainer—Aluminum tube 114 mm in length, 16-mm outside diameter (OD), and 13-mm inside diameter (ID). The tube shall have a capacity of 16-mL distilled water at 24 ±2 °C

B - Rubber stoppers—Two no. 6 13-mm rubber stoppers with a 13-mm hole bored through centers

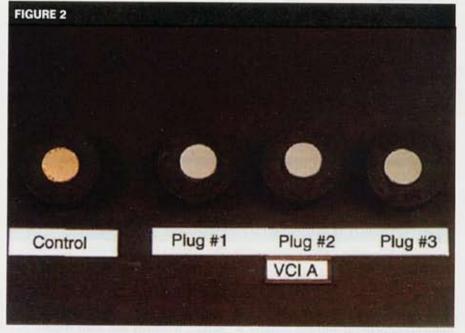
C – Jar lids—Plastic screw-type lid, with 30-mm holes drilled through the center with two 6-mm holes 25 mm apart near the edge

D - Jars—1-L size, mouth size 60-mm diameter, 177 mm in height, 136-mm ID

E - Insulating sleeve—13-mm ID rubber tubing, length 38 mm

F - Steel specimen—16-mm OD, 13 mm long with 9-mm-deep flat bottom hole drilled in center (8-mm wall cup)

VIA test.5



VIA test results for ABC film.5

Results of ABC film surface resistivity and anti-stat testing						
	Position No. 1	Position No. 2	Position No. 3	Position No. 4	Position No. 5	Position No. 6
(a) Outside Film Surface Resistivity (Ω/Square)	4.6 1010	2.8 1010	2.3 1010	1.6 1010	1.6 1010	1.4 1010
(b) Inside Film Surface Resistivity (Ω/Square)	1.2 1010	1.7 1010	1.2 1010	1.2 1010	1.1 1010	1.1 1010
(c) Static Decay Outside Surface +5,000 V	0.86	0.91	0.92	0.93	0.93	0.93
(d) Static Decay Inside Surface +5,000 V	0.88	0.92	0.93	0.93	0.95	0.96
(e) Static Decay Outside Surface -5,000 V	0.86	0.91	0.89	0.92	0.92	0.94
(f) Static Decay Inside Surface -5,000 V	0.82	0.86	0.86	0.87	0.87	0.89

TABLE 3		O THE PURE CE	Commission
Razor blade a	nd SO <sub>2</sub> test resul	ts	
(a) Razor Blade Te	st Results for CS		
Material	Panel No. 1	Panel No. 2	Panel No. 3
ABC film	Pass	Pass	Pass
Control film	Fail	Fail	Fail
Note: Razor blade tes	et is graded on a pass or fa	all scale	
(b) Razor Blade Te	st Results for Copper		
Material	Panel No. 1	Panel No. 2	Panel No. 3
ABC film	Pass	Pass	Pass
Control film	Fail	Fail	Fail
Note: Razor blade tes	t is graded on a pass or fa	ail scale	
(c) SO, Test Resul	ts		
Material	Panel No. 1	Panel No. 2	Panel No. 3
ABC film	Grade 4	Grade 4	Grade 4
Control film	Fail	Fail	Fail

distribution throughout the film, but eventually it diffuses to the surface of the film. This buildup on the surface of the humidity-dependent anti-stat additive is hydrophilic and attracts humidity to the film's surface. Any buildup of charge on the package's enclosed item is pulled away from the item and toward the film surface. Two distinct tests that measure the film's ability to perform this task are surface resistivity and static decay.<sup>6</sup>

Corrosion inhibiting properties can prolong the lifetime of a wrapped item. Often corrosion is unseen within an item and premature failure of the item is the result. A manufacturer of electrical equipment could possibly recvaluate the expected lifetime of a product, not knowing that an ongoing corrosion problem is the culprit of the failure. Several types of corrosion inhibition protection are available, such as contact, vapor, and barrier phase corrosion inhibition. Contact phase corrosion inhibition occurs when the corrosive solution is in direct contact with the metal. The next type of corrosion inhibiting protection is barrier phase corrosion inhibitor, where the corrosive species has to penetrate through the corrosion inhibiting material to reach the surface of the metal. A third type of corrosion inhibiting protection is the vapor phase corrosion inhibitor, where the inhibitor has to act at a distance.

# **Experimental Procedures**

Surface resistivity measures the resistance of electrical current flow across the surface of the film. This property is measured in accordance with ASTM D257,4 where a film sample is exposed to a conditioning period of 48 h at a temperature of 73 °F (23 °C) ±5 °F, and a relative

humidity of 50%  $\pm 2\%$ . Another 48 h of conditioning is done in the test chamber where the temperature is 73  $\pm 5$  °F and relative humidity is 12%  $\pm 3\%$ . After conditioning, the film sample is tested at six points on one face of the film, and tested at six points on the opposite side of the film, using a Monroe Electronic Surface<sup>†</sup> resistivity meter. The recorded value for surface resistivity must fall in the range of  $10^{10}$ - $10^{12}$   $\Omega$ /square.<sup>6</sup>

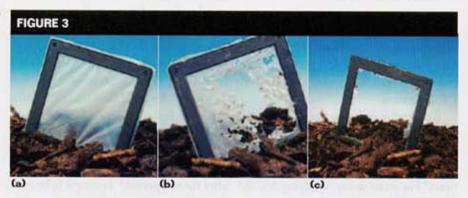
Static decay is an extremely important test because it allows the user to know how fast a material will dissipate or remove an applied charge. For the ABC film, the test procedure MIL-STD-3010 test method 40467 was used to test for static decay because it is a U.S. Department of Defense test method standard for packaging materials. In addition, the performance requirement MIL-PRF-81705D8 is the industry standard for static decay. During research and development of this product, any product that did not have a static decay of 2 s or less was eliminated.

The test procedure calls for the film sample to be exposed for 48 h of conditioning at a temperature of 73 ±5 °F, and a relative humidity of 12% ±3%. After the conditioning period, a Faraday cage is charged to +5,000 V and the film is tested for static decay on both faces of the film. Immediately after, the film is charged to -5,000 V and the film is tested for static decay on both faces of the film again. Four test scores are tabulated for each film sample. Six film samples are tested for each lot of produced film.

Corrosion inhibiting properties should be considered when allowing a product to be used in a modern packaging material for the protection of electronic equipment. Contact, barrier, and especially vapor phase corrosion inhibition are properties that allow the material to have a longer useful life and prevent the mate-

TABLE 4 VIA test results				
ABC film	Grade 3	Grade 3	Grade 3	
Control film	Fail	Fail	Fail	

Note: The VIA test is graded on a Grade 0, Grade 1, Grade 2, or Grade 3 scale. A Grade 3 signifies no corrosion, whereas, Grade 0 is extreme corrosion.



Lifecycle biodegradation process. (a) Prior to insertion into compost pile, (b) after three weeks in compost pile, (c) after six weeks in compost pile. Less than 10% of the original weight of the film is remaining after six weeks in compost pile. 10

rial from corroding and causing product failure. Contact phase corrosion inhibition testing is performed by what is called the razor blade test. Carbon steel (CS) panels composed of 1010 CS and 100% copper panels measuring 1 by 4 in (25 by 102 mm) are cleaned in methanol. Two drops of deionized water are then placed onto the surface of the metal and the film is placed onto the water (thus forming a sandwich of metal, water, and film). After 2 h, the film is removed, water is wiped away, and the surface is observed for any signs of corrosion.<sup>7</sup>

Barrier phase corrosion inhibition testing is performed using the sulfur dioxide (SO<sub>2</sub>) test. This test is performed by creating a SO<sub>2</sub> gas environment in a 1-gal (3.785-L) jar where the film is wrapped around CS 1010 panels. After a 20-h conditioning period, the SO<sub>2</sub> gas is created and a 24-h test period is started. At the end of the 24-h test period, the panels are observed for any signs of corrosion. Testing is done in triplicate, with one control panel wrapped in a noncorrosion inhibiting film.<sup>8</sup>

Vapor phase corrosion inhibition ability (VIA) testing is performed by sanding CS plugs with 120- and 240-grit sandpapers, such that all lines on the plugs are in straight lines. The plugs are then inserted into rubber stoppers attached to 1-in diameter aluminum pipes that are fastened to plastic lids. These lids are 1-qt (0.95-L) glass jar size. Then 1 by 6-in (152 mm) strips of film are hung from the underside of these lids (Figure 1). The test jars are placed onto a laboratory bench for a period of 20 h. After 20 h, a glycerol/water solution is added to the jars. The jars are again placed on a laboratory bench for an additional 2 h before being placed into a 40 °C oven for 2 h, After the test period, the plugs are observed for

The test is performed in triplicate along with a control plug. The control plug corrodes heavily at the end of the test period, and the ABC film plugs are unchanged from their original appearance (Figure 2<sup>5</sup>).

Biodegradability testing was conducted in accordance with ASTM D6400

Trade name.

Section 6.1 with requirements listed in Sections 6.2, 6.3, and 6.4. These requirements state that no more than 10% of the film's original dry weight will remain after sieving on a 2.0-mm sieve after exposing to laboratory-scale composting found in section 7.2.1 of ASTM D6002.

# **Results & Discussion**

Laboratory tests performed were surface resistivity, static decay, razor blade test, VIA test, SO<sub>2</sub> test, and ASTM D6400 testing.

Results for anti-stat testing (Table 2) clearly show that an ABC film is a product that will provide static decay and surface resistivity when exposed to extreme conditions. For static decay, applying ±5,000 V to the film has been determined as a sufficient way to test a film for surface resistivity, and test results show that an ABC film can clearly eliminate this charge quickly—the charge was removed in less than 1 s, well below the requirement of 2 s. For surface resistivity, the resistance to electric current flow across the film's surface falls within the acceptable range for an anti-stat material. 11

For contact phase corrosion inhibition, a razor blade test<sup>7</sup> indicated that the film can prevent the item from corroding if moisture becomes trapped between the item and the film. A control film with no corrosion inhibitors is used as a control (Table 3).

Barrier phase corrosion testing, as already mentioned, is conducted in accordance with a SO<sub>2</sub> test.<sup>8</sup> This test determines if a corrosive gas can penetrate through the film and then displace the corrosion inhibitors that have deposited on the surface of the metal (Table 2).

Vapor phase corrosion testing is arguably the most significant of the corrosion testing because of the level of sophistication involved in developing a product with this attribute. Often a product will have contact phase corrosion inhibition and also barrier phase corrosion inhibition, but more than likely, vapor phase corrosion inhibition will be absent. Vapor phase corrosion is a property where the inhibitor sublimates from the solid phase into the gas phase, and then condenses onto the metal surface. This condensation forms a layer of corrosion inhibitor on all metal surfaces of the part, thus preventing contaminants from reaching the surface and corroding it. The VIA test<sup>3</sup> as mentioned previously was performed on ABC film. Table 4 shows test results.

For biodegradability testing, results provided by an independent lab must show that ASTM D6400 requirements will be met, with a weight of no more than 10% of the original dry weight remaining after the test period.<sup>2</sup> Testing is to be conducted according to laboratory-scale composting as found in ASTM D6002.<sup>11</sup> Figure 3 shows a life cycle of the biodegradation process, starting with film prior to insertion into compost pile (a), three-week midterm (b), to the six-week end time (c).<sup>10</sup> The ABC film is stretched across the frame and placed into the compost.

### Conclusions

Modern packaging materials for electronic equipment are becoming more and more important in an age where highly sophisticated electronic equipment is more readily available. Packaging with polymer films must take into consideration everything that might damage this type of equipment, such as electrostatic dissipation and corrosion, and disposal concerns of the package after use. A product such as an ABC film will provide static dissipation, prevent corrosion, and biodegrade when exposed to compost conditions.

#### References

- S. Berkesch, "Biodegradable Polymers; A Rebirth of Plastic," March 2005, pp. 1-14.
- 2 ASTM D6400, "Standard Specification for Compostable Plastics" (West Conshohocken, PA: ASTM, 1999).

- 3 G. Theodore Danglemayer, "ESD Program Management: A Realistic Approach to Continuous Measurable Improvement in Static Control," pp. 1-20.
- 4 ASTM D-257, "Standard Test Methods for DC Resistance or Conductance on Insulating Materials" (West Conshohocken, PA: ASTM, 2007).
- D. Hannan, "VIA Test" (Cortec Corp. work instruction), May 2000, pp. 1-4.
- 6 MIL-STD-3010, Test Method 4046, "Electrostatic Properties" (Washington, DC: DoD, 2002).
- D. Hannan, "Razor Blade Test" (Cortec Corp. work instruction), May 2000, pp. 1-3.
- 8 D. Hannan, "SO<sub>2</sub> Test" (Cortec Corp. work instruction), May 2000, pp. 1-3.
- 9 MIL-PRF-81705D, "Barrier Materials, Flexible, Electrostatic Protective, Heat Sealable" (Washington, DC: DoD, 1998), p. 7.
- 10 ASTM D6002, "Guide for Assessing the Compostability of Environmentally Degradable Plastics" (West Conshohocken, PA: ASTM).
- 11 RTP Co., "Conductive & Anti-Stat Plastic Compounds," Oct. 2007.

This article is based on CORROSION 2008 paper no. 674, presented in New Orleans, Louisiana.

BORIS A. MIKSIC, FNACE, is president and chief executive officer of Cortec Corp., 4119 White Bear Pkwy., St. Paul, MN 55110. He has served in this capacity for 32 years. Cortec is a world leader in the manufacture of corrosion inhibitors in several industries, including modern plastic products. Miksic holds more than 43 U.S. and foreign patents and patent applications and has presented papers around the world. A NACE International Fellow, he has been a NACE member for more than 30 years.

BOB BOYLE is technical sales manager at Cortec Corp., e-mail: bboyle@cortecvci.com. He has worked for the company for seven years. He has a Bachelors degree from Hamline University (St. Paul) and a Masters degree from Concordia (St. Paul). He is focused on expanding the research and practical application of vapor phase corrosion inhibitor technology related to the environment, health, and safety.

BOB BERG is a former employee of Cortec Corp. IVIP