

# Advanced Aluminum Foil Products for Packaging Applications

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## Abstract

Aluminum foil is widely used in flexible packaging for food, beverage, pharmaceutical and other products because it offers outstanding barrier properties (for moisture, oxygen, carbon dioxide and other gases) and also provides protection for UV sensitive products. This paper will describe some advanced foil technologies, which offer additional benefits for the packaging industry. The use of an engineered, weakened oxide interface for controlling peelability for blister packages, or for lidding applications will be described. Using this material with a suitable heat seal lacquer or laminate material, a precisely controlled and consistent peel strength can be achieved. In addition, a novel route to the production of “color shift” foil materials will also be introduced. As well as providing an interesting decorative effect, with a range of bright pastel colors, the color change or shift which occurs when the viewing angle changes, is very difficult to replicate. Consequently, this technology has potential applications for enhanced brand authentication and security.

## Introduction

The attractive metallic appearance and exceptional barrier properties have made aluminum foil a material of choice for a wide variety of packaging applications. However, recent developments based on anodizing, can produce nano-engineered surfaces which offer new and unique opportunities for some advanced packaging applications. The growth of interest in “consumer friendly” packages which are easy to open, has led to a need for consistent and controllable peel strengths. Package designers also strive for packaging features which offer differentiation and brand identity opportunities. As a result, packaging which incorporates unique and difficult to reproduce decorative colors and patterns are of growing interest.

## **Nano-engineered Surfaces on Aluminum Foil : The Basics of Anodizing**

Anodizing is an electrolytic process where aluminum sheet or foil is the anode and a suitable acid, such as dilute phosphoric acid, is the electrolyte. When a voltage is applied, the oxide on the aluminum surface grows in a very uniform and controlled way. Although a number of metals can be anodized, the structure of the anodic film that grows on aluminum has a unique nano-porous form. The structure of this porous anodic film has been well documented and is shown schematically in figure 1.

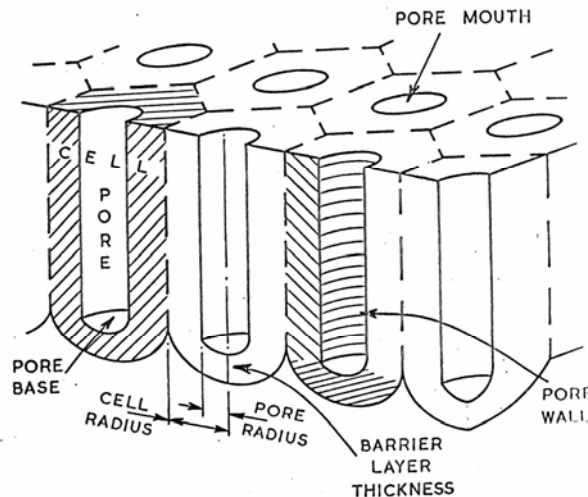


Figure 1 Schematic diagram of porous anodic film structure.

The barrier oxide layer adjacent to the metal, the pores and pore wall structures have been labeled. The detailed structure varies somewhat, depending on, for example, the acid and concentration, the temperature, and the electrical conditions used. However, some useful and simple approximations have been established which relate the pore size, barrier layer thickness and pore wall thickness to the anodizing voltage used.

For anodizing with a voltage  $V$  (dc power supply),

Pore diameter	$\sim V$ nm
Barrier thickness	$\sim V$ nm
Pore wall thickness	$\sim V$ nm

Thus for a sample anodized for a short time at say 18 volts (dc), the oxide will have pore diameters of  $\sim 18$ nm, barrier oxide thickness of  $\sim 18$ nm and pore wall thickness of  $\sim 18$  nm.

Note on units :

1,000 nm (nanometre) = 1 micron

24,500,000 nm  $\sim$  1 in.

The detailed mechanisms which give rise to this nano-pore structure will not be discussed in detail. However, a basic introduction to the principal effects is necessary, in order to introduce and describe the oxide weakening mechanism which is used to control peel strength.

## **Peelable Oxide Foil**

### **The Voltage Reduction Procedure For Weakening Anodic Films**

If an aluminum sheet is anodized at a selected voltage to form a porous anodic oxide film, the pore size and boundary layer thickness will depend on the voltage chosen as described earlier. If, after a desired thickness of anodized film has been grown, the voltage is reduced significantly, there will be an initial period when no further current flows. This is because the barrier oxide layer produced at the higher voltage is too thick to allow current to flow when the lower voltage is applied. However, since there will still be a relatively high field across the oxide, some enhanced, field assisted, dissolution will occur. This will cause the oxide to thin until it corresponds to the barrier thickness expected for the lower voltage. At this stage, current will begin to flow, and anodizing at the lower voltage will commence. The structure of the newly anodized region corresponding to this lower voltage will have a reduced pore size, pore wall thickness and barrier oxide thickness.

In the transition zone, between the porous oxide grown at the high voltage and the porous oxide corresponding to the reduced voltage, the comparatively large pores sub-divide and branch to the finer pore structure of the lower voltage region. This is shown schematically in Figure 2.

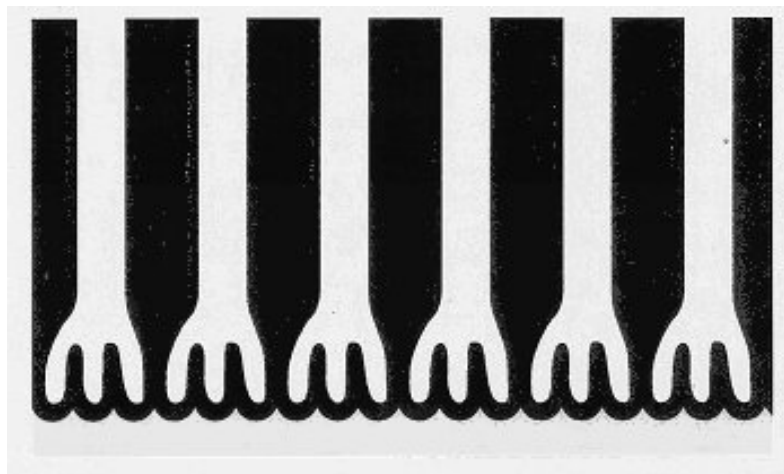


Figure 2 Schematic diagram showing the pore branching which occurs as a result of a voltage reduction.

If the voltage is further reduced, further branching of the pores to an even finer structure will result. By a series of successive voltage reduction steps until the final voltage is very small ( e.g. to ~ 1 volt ), the pore size adjacent to the aluminum substrate can be reduced to a very small diameter. The overall structure of the resulting anodized film consists of a relatively large pore size region with thick pore walls supported by a progressively finer and finer pore structure with the smallest pores and the thinnest pore wall region close to the metal substrate. A more detailed description of this process can be found in reference 1.

If the voltage is reduced to zero, no further anodizing will occur, but chemical dissolution by the acid will continue. In the absence of the electric field, this dissolution will be slow, and the pore walls in the large pore zone will be thinned slightly. However, in the region of very fine pores and thin pore walls, the dissolution will lead to a progressive weakening of the film in this region. By interrupting this dissolution stage by rinsing away the acid, the bond between the oxide and the underlying aluminum metal can be controlled.

A wide variety of packaged products, including many food products, use aluminum foil as the lidding material. Common examples include yogurt and pudding containers, individual jam portions and a variety of petfood containers. An important feature of these packages is the ease of opening, or peelability. For most containers, consumer testing has found that the peel force required to open the lid should be in the range between about 10 N (~2.2 lb force) and about 15 N (~3.3 lb force) as measured using a 90 deg. peel test. This is usually achieved by using specially formulated heat seal lacquers and carefully controlled heat sealing conditions.

Preliminary experiments in the laboratory used a programmable power supply to generate a thin anodic film and then carry out a sequence of voltage reductions. After rinsing and drying, the resulting foil was then coated with a heat sealable lacquer which had a very high inherent peel strength. When heat sealed to a container in the normal way, peeling occurred at the weakened metal-oxide interface rather than in the heat seal lacquer layer. After a series of experiments to vary the final voltage and the final soak time in the acid, it was found that the peel strength could be adjusted to fall within the target range for the foil lidding application. These “proof of concept” laboratory experiments required total processing times of several minutes and would not be cost effective for a commercial packaging application.. However subsequent work to refine the procedure and to develop a continuous coil-to-coil process has reduced the total process time to a few seconds, which allows comparatively high line speeds for production. To achieve these short processing times, the weakened oxide is extremely thin ( typically less than 0.05 microns) as shown in the TEM cross-section in Figure 3.

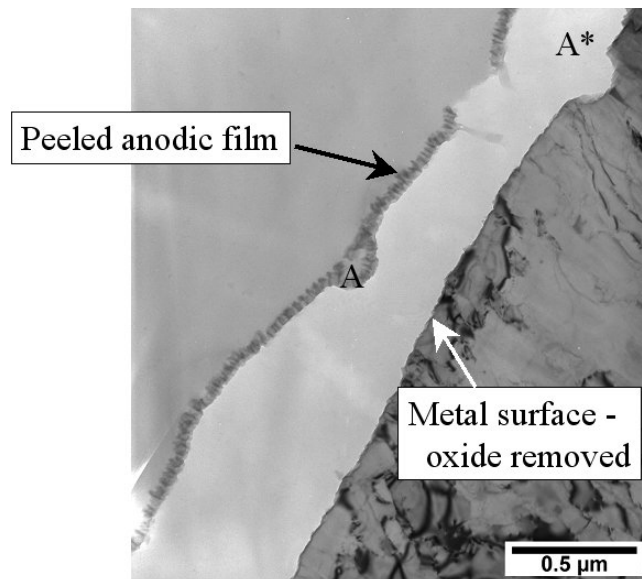


Figure 3 A transmission electron microscope image of a cross section through a partially peeled sample. Note that the extremely thin porous oxide has detached from the metal surface but remains bonded to the heat seal lacquer. The labeled features (A and A\*) show equivalent locations in the oxide and parent metal. Some displacement occurred during the peel.

## Lidding Foil Performance

For the lidding application, the foil with weakened oxide was coated with a polypropylene dispersion based heat seal lacquer (Rohm and Haas, Morprime 10B) of high inherent peel strength to a coating thickness of about 7 microns. Lids were die cut from the coated foil and heat sealed to the rim of test containers in the normal way. The structure of the resulting heat sealed container, showing the location of the weakened oxide is illustrated schematically in figure 4.

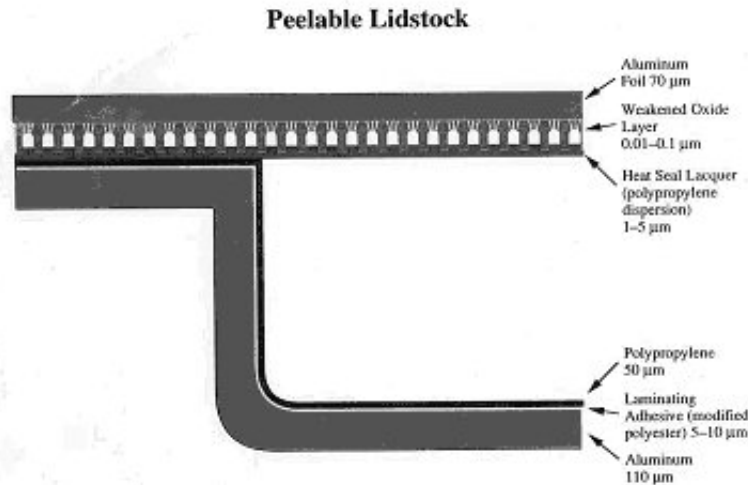


Figure 4 Schematic (not to scale) showing a foil laminated container with a peelable foil lid.



Figure 5. An example of a plastic container with a peelable lid which uses the weakened oxide to achieve the desired peel strength.

Peel tests have been carried out on the resulting containers to confirm that they meet the typical peel strength requirements. A typical set of data is shown in figure 6. This plot shows the variation in peel force required ( at a constant  $90^{\circ}$  angle) as the lid is peeled. In each test, the peel force was very uniform and consistent and well within the acceptable target range. By varying the operating conditions to grow and weaken the oxide, the peel strength can be varied to meet other target specifications as necessary and no reformulation of the heat seal film or lacquer is

necessary. Furthermore, since the peel force is controlled by the oxide, it is not affected by subsequent processing conditions or storage. Thus, for example, the peel strength is virtually unaffected by retorting the product.

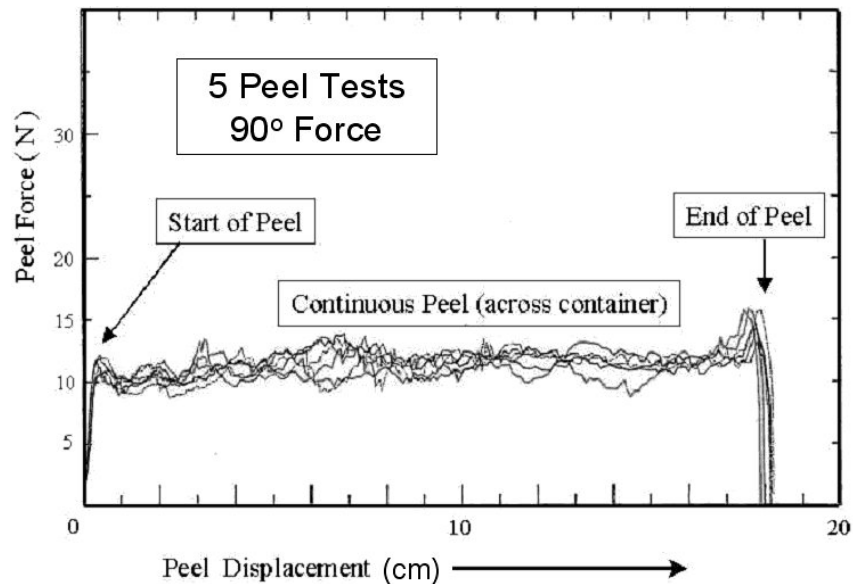


Figure 6 A series of five peel tests carried out on rectangular containers. Note the uniformity of the measured peel force during each test, and also the consistency of peel for the five samples tested.

## **Color Shift Foil**

Color shift materials are already widely used in a range of security applications. For example, the Canadian \$20 bill which has been in service for the last few years has incorporated a small “color shift” foil label as an anti-counterfeit feature. Typically these materials are produced using complicated multi-layer vacuum sputtering technology, with relatively low production rates and high capital cost. As a result, their use has generally been limited to small volume, high value, niche applications.

Aluminum foil has a highly reflective surface. By growing a thin anodized film of a suitable thickness, and then metallizing the oxide surface with a thin semi-transparent aluminum film using a standard vacuum metallizing method, a “Color Shift Foil” can be produced. The color shift effect, also referred to as “dichroism” or optically variable device technology (OVD), is based on an optical interference effect.

## **The Interference Coloring Mechanism**

A schematic diagram of the color shift foil is shown in figure 7. An incident beam of “white” light consists of all wavelengths within the visible spectrum range (from ~ 0.7 microns (red) to ~ 0.4 microns (blue/violet)). Part of this incident light is reflected from the metallized surface. The other portion passes through the transparent anodic oxide layer and is reflected from the underlying foil surface. The two reflected beams have traveled different distances (a phase shift). This phase shift will correspond to a particular wavelength (or color) of light, and light of this color will be reinforced. This is known as constructive interference. For other wavelengths, the two reflected waves will be out of phase and will be attenuated to varying extents. For wavelengths where the reflections are exactly out of phase, destructive interference occurs, and the light of these wavelengths will be attenuated the most. Since some wavelengths (colors) are reinforced and other wavelengths are attenuated, the overall result is a bright metallic coloration of the foil. It is worth noting that this coloring effect is achieved without the use of any inks or pigments and the particular color effect is controlled by the oxide thickness.

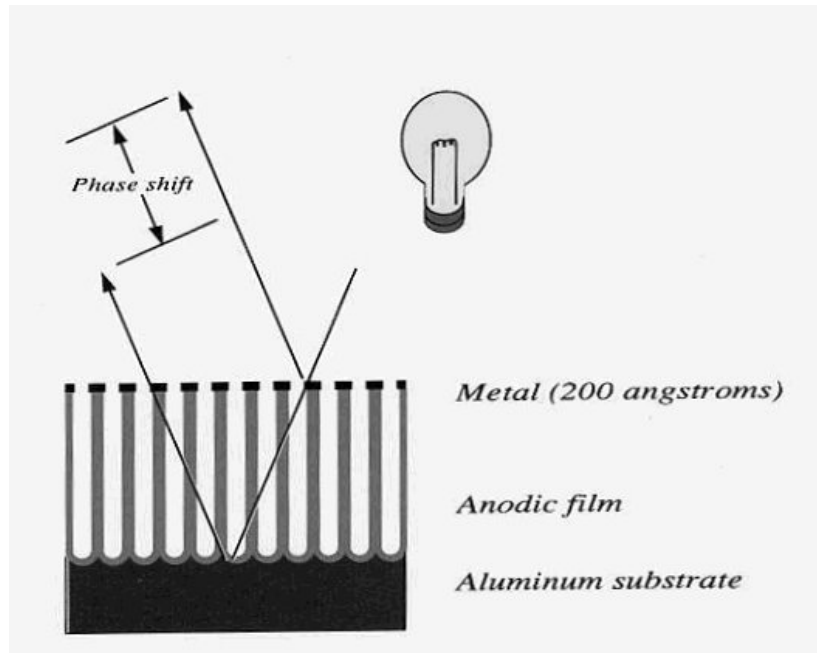


Figure 7 Schematic diagram of a “Color Shift” thin film.

Different oxide thicknesses will produce different interference colors. An example of the interference colored foil surface is shown in cross section in figure 8. In this case, the porous anodic oxide layer is ~ 0.3 microns thick. The phase change which gives rise to the colors is controlled by the thickness and refractive index of this oxide. When first produced, the anodic film consists of aluminum oxide and air filled pores. It is possible to fill the pores with another material such as a clear lacquer. Since this will generally change the overall refractive index, the color of the foil will also be changed.

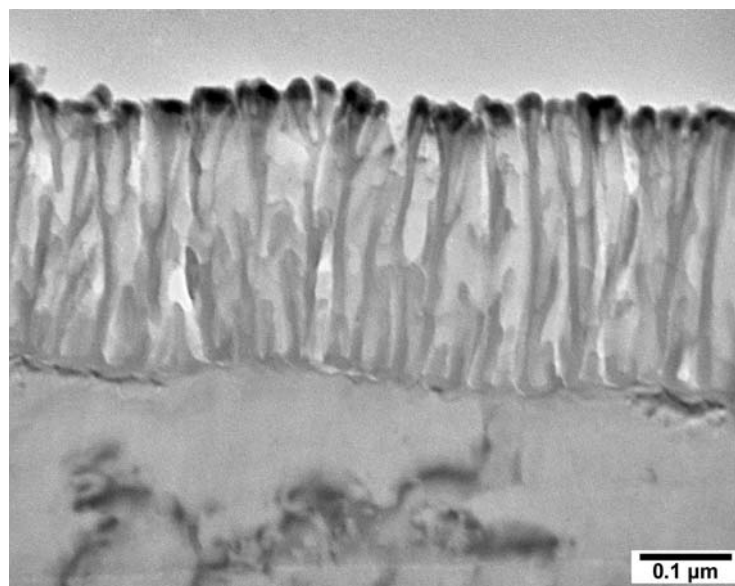


Figure 8 Cross section through a color shift foil imaged in a transmission electron microscope. The aluminum foil substrate, the porous anodic oxide film and the thin, semi-transparent metallised layer on the surface can clearly be seen. Note the oxide layer thickness is ~ 0.3 microns.

## **The Color Shift Effect**

As the viewing angle for the foil is varied, the difference in path length (which causes the phase shift) between the two reflected light components will also vary. This means that the wavelengths for the interference effects will also change. The overall result is that the color of the foil changes with viewing angle as the foil is tilted. In some cases, these color shifts can be very dramatic. Examples include :

Pink > Green  
Dark Blue > Purple  
Green > Blue

The thickness of the anodic film which is produced, depends on the anodizing conditions, and in particular the current and time (total coulombs per unit area). Again the process is very controllable and reproducible and can be carried out using a specially designed continuous processing line. Since the oxide thicknesses required are thicker than those needed for the peelable oxide, a longer time and higher operating current are required. However, for most colors of interest the thicknesses are between about 0.15 and 0.3 microns, and the time required is still quite short (seconds).

## **Peelable Color Shift Foil**

By combining the color shift and oxide weakening procedures, a peelable color shift foil can be produced. This is of interest because when the oxide is peeled away from the underlying foil, the optical interference effect is destroyed, and therefore the color is lost irreversibly. This has potential applications in “tamper evident” packaging, since once the package has been peeled open, a dramatic and irreversible loss of the color occurs.

## **Summary**

The formation and precise control of anodic films on aluminum foil can offer some unique and interesting possibilities for the package designer. A weakened oxide interface can provide controlled peelability which is independent of the heat seal film and conditions which are used. For packages sealed with this foil, the peel strength will be substantially independent of subsequent processing conditions or storage times. For this reason, it is of particular interest for the more demanding, retortable food or pharmaceutical applications.

Interference coloring and color shift foils also have wide potential in applications for brand authentication and security packaging. As well as having high visual appeal, the colors are difficult to reproduce and therefore deter counterfeiting. Combined use of the color shift and weakened oxide technologies offers an additional degree of protection and tamper evidence. Foils with these specifications can be produced economically by a continuous process.

## **Acknowledgements**

The author is grateful for the help and assistance of many members of the surface science team at Novelis Global Technology Centre who have participated in the developments described here, and to Novelis Inc. for supporting the projects and for granting permission to present this paper.

## **References**

1. US 5,582,884 “Peelable Laminated Structures and Process for Production Thereof”, M.D. Ball, L.A. Coady, W.H. Kuenzi and R.G. Jones (1996).
2. US 5,218,472 “Optical Interference Structures Incorporating Porous Films”, M.A. Jozefowicz and A.M. Rosenfeld (1993).

## Advanced aluminum foil products for packaging applications.

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### Overview

- Introduction / Background
  - Basics of anodizing aluminum
- Peelable foil lidding for retort applications
- Color Shift (Dichroic) foil

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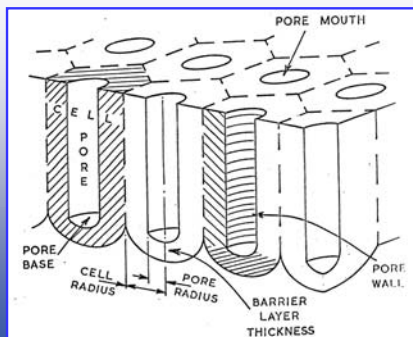
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### Schematic Diagram of Anodic Film Structure



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## Anodizing Basics

- For anodizing with a voltage  $V$  (dc power supply),
  - Pore diameter  $\sim V$  nm
  - Barrier thickness  $\sim V$  nm
  - Pore wall thickness  $\sim V$  nm
- Example :
  - at 18 volts (dc) in 0.1N phosphoric acid, the porous oxide will have pore diameters of  $\sim 18$ nm, barrier oxide thickness of  $\sim 18$ nm and pore wall thickness of  $\sim 18$  nm.

Note on units :  
1,000 nm (nanometre) = 1 micron  
24,500,000 nm  $\sim$  1 in.

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## Method of Anodic Film Weakening

- Grow anodic film to the desired thickness at voltage  $V$
- Reduce voltage
  - Current falls to a low value initially, then start to recover
  - As current recovers, anodizing at the lower voltage proceeds
    - NOTE: Pore size, etc. is now smaller
- Repeat voltage reduction procedure, allowing current recovery between successive steps
- After a final "low" voltage anodizing step, allow the sample to "soak" in the acid for a short period to produce a controlled weakening
- Rinse and dry

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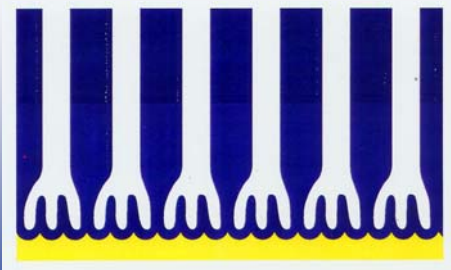
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## Schematic of Pore Branching



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Peeled Anodic Film Structure: SEM Image



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Concept for Foil Lidding

- Anodize the foil and produce a controlled, weakened oxide
  - Use high speed, low cost production line
- Apply a “high strength” heat seal lacquer to the anodized side
- Prime, print and overlacquer the “public” side
- Fill containers and apply the heat seal lidding as normal

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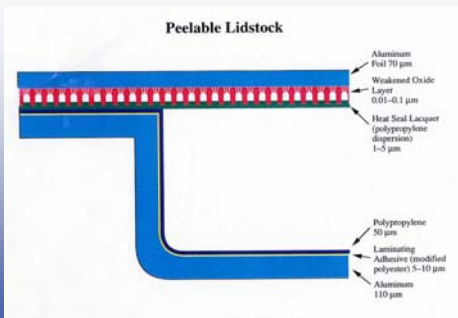
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Peelable Lidstock



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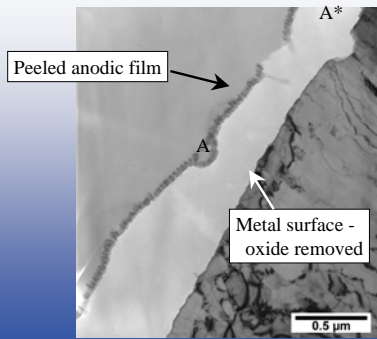
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### Ultramicrotomed Cross-section of Peeled Anodic Film (TEM Image)



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### The Challenges

- Cost must be competitive with current retortable lidding foil
  - Production speed must be high (continuous line)
  - Low cost lacquers must be used
- Must be fully compliant with FDA etc.
  - Choice of anodizing acid (phosphoric acid)
- Require good bond to heat seal lacquer
  - Also favors phosphoric acid
- Good peel strength control and consistency

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### Example of Foil Lidding with Polypropylene Containers



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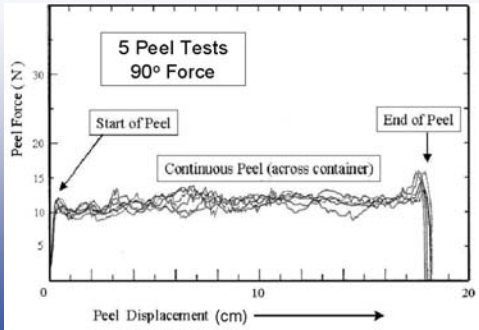
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### Peel Test Performance: Example



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### Summary: Peelable Lidding

- Peelable oxide foil meets all key performance requirements
  - Compatible with polypropylene containers
- Continuous process successfully demonstrated
- Cost competitive compared to other retortable lidding
- Some (anticipated) potential benefits
  - Process cleans the foil (eliminates foil cleaning operation)
  - Better peel control / consistency
  - Reduced dependence on heat seal coating variation and coating parameters

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### Dichroic Foil on a Curved Surface: Color Shift Effect Depends on Viewing Angle



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### Colored Dichroic Foil: What Is It ?

- Based on nano thin film technology
- A very thin controlled oxide can be produced on Aluminum foil
- When metallized, bright pastel colors are achieved by optical interference
  - the same effect that produces the colors seen in thin films of oil on water, but much brighter
- Color changes when viewed from different angles
  - color shift or dichroism
  - Similar effect is used in security applications, banknote anti-counterfeit features etc.
- A wide range of colors is available
- Many additional enhancements have been identified

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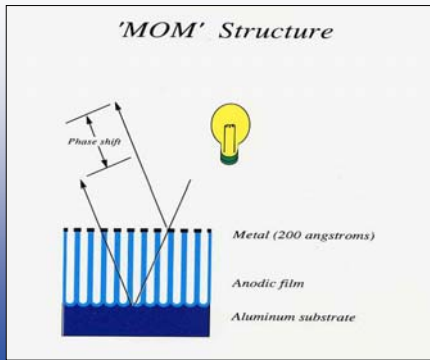
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### Schematic of Interference Coloring Mechanism



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### Dichroic Foil: Some Market Drivers

- Eye catching, decorative finishes: Shelf appeal
- Product differentiation
- Anti-counterfeiting (pharmaceutical, cosmetics, etc.)
  - Counterfeit drugs account for ~ 7% of supply (WHO)
- Brand Authentication / Protection
  - P&G estimated losses of \$225 MM per year; Global Industry losses ~ \$200 bn per year and rising!
- Security Applications

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### Continuous Anodizing Process: Dichroic Foil

- One side of the foil (or laminate) is anodised using conditions for rapid film growth
- Line speed may be reduced (compared to Peelable oxide process) since oxide thickness ( $t$ ), needs to be  $\sim 0.3 - 0.5$  microns
  - for peelable oxide,  $t \sim < 0.1$  microns
- Color depends on oxide thickness and can be controlled by line speed, voltage, current density, etc.
  - Some colors are more expensive than others!!

The resulting strip is metallized using a standard high speed vacuum metallizing line.

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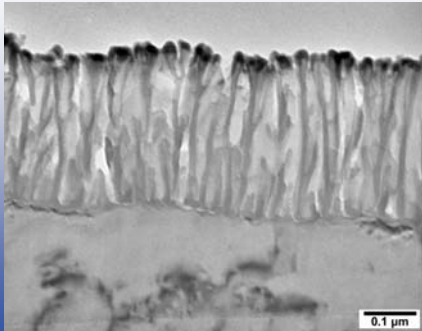
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### Cross Section Through the Interference Coloring Layer (TEM image)



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### Colored Dichroic Foil: Enhancements

- Surface can be made “touch sensitive”
  - Color changes when surface is wetted or adhesive is applied
    - The basis for “visual seal integrity” foil for blister packs
- Can be combined with “Peelable oxide” technology:
  - Colors disappear when peeled apart (*Tamper evident*)

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## Acknowledgements



The author is grateful for the help and assistance of many members of the surface science team at Novelis Global Technology Centre who have participated in the developments described here, and to Novelis Inc. for supporting the projects and for granting permission to present this paper.

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## Thank You

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*Please remember to turn  
in your evaluation sheet...*

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