TA Instruments

Thermal Analysis & Rheology

Thermal Analysis Application Brief

Determination of Layer Thickness in Multilayer Packaging Films

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SUMMARY

Multilayer thin films are becoming more common as manufacturers and users of plastics for film applications such as packaging strive to develop materials with a balance of enduse properties (e.g., toughness, selective atmosphere permeability). An important quality parameter for these multilayer films is the total film thickness, as well as thickness of the individual component layers. Thermomechanical analysis (TMA), because of its ability to measure small dimensional changes, is ideal for evaluating thin films. TMA provides an alternative to spectral techniques, particularly in situations where the presence of inks, adhesives or lacquers might interfere with the measurement.

INTRODUCTION

The end-use properties of films used for packaging applications are dependent on their polymer (plastic) composition. As requirements for specific end-use properties become more demanding, the use of multilayer films, rather than single homogeneous layer films, is becoming more common. The ultimate functionality and cost of these multilayer films depend not only on chemical make-up, but also on the thickness of the total film and respective internal layers. Hence, tests usually based on spectral techniques, such as infrared spectroscopy, have become widely accepted as a means of monitoring film layer thickness (1). These tests, however, because they are often based on ATR (attenuated total reflectance) and MIR (multiple internal reflectance) measurements, can be difficult to interpret, particularly if surface treatments such as inks or lacquers are present. Thermomechanical analysis (TMA) is an alternative way of testing for film thickness. TMA is easier to use and interpret than the spectral techniques.

TMA measures dimensional change while a material is subjected to controlled heating or cooling. The most common TMA measurement modes are expansion and penetration. In expansion, a measurement probe is placed in contact with the material and bulk dimensional change with temperature (coefficient of expansion) is determined. In penetration, a weighted measurement probe is placed in contact with the material, and either the total material thickness or the thickness of individual layers is determined. Individual layers on the surface are determined directly, while internal layers are determined by difference. Despite its high sensitivity, TMA is not affected by surface inks or lacquers.

EXPERIMENTAL

In TMA penetration analysis, the sample is positioned on a quartz stage and a probe with a small diameter tip (for concentrating force) is placed on the top of the sample. A furnace surrounds the sample, stage and probe to provide accurate heating/cooling during measurement. A thermocouple located near the sample monitors sample temperature so that the dimension change can be followed as a function of temperature. The experimental data obtained is stored and subsequently analyzed using dedicated data analysis software.

In this case, several film samples were analyzed over the temperature range ambient to 280°C. The programmed heating rate was 5°C/minute, the atmosphere around the samples was nitrogen, and the applied loading was 0.1 Newton. The tests were run on a TA Instruments TMA 2940 Thermomechanical Analyzer.



RESULTS

Displayed in Figure 1 are the results for a multilayer packaging film comprised of a metallic substrate coated on both sides with polymer. The TMA curve shows two sharp negative dimensional changes (penetrations) at 103 and 258°C. These two penetrations indicate that the film is probably a three layer film containing two polymer layers and the metal foil layer. The

thicknesses of the three layers are 93 μ m (polymer 1), 15 μ m (polymer 2) and 6 μ m (foil substrate).

In an attempt to identify the nature of the individual polymer film layers, several homopolymer films were evaluated. The results for two of these, namely polyolefin (polyethylene) and polyester, are shown in Figures 2 and 3 respectively. The sharp penetrations at 105 and 255°C verify that these are the polymer layers in the original multilayer film.





Further evaluations of the original multilayer film, however, reveal that the film actually contains more than three layers. Figure 4 and 5 show the results after the film layer on one side of the metal foil is peeled away. Figure 4 shows that the film remaining on the metal foil is polyethylene, while the peeled film (Figure 5) is not only polyester, but also contains a layer of polyethylene. The thin layer of polyethylene between the foil and the polyester layer serves as a "hot melt" adhesvie to bond those layers together (2). In the TMA results for the total film, the two polyethylene layers soften at the same temperature, and the total penetration represents the combined layer thicknesses.

Other thermal analysis techniques can be used to measure additional multilayer film properties. These include compositional analysis by thermogravimetric analysis (TGA) and softening and crystallinity by differential scanning calorimetry (DSC).

REFERENCES

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