High Performance Materials for Laminating Adhesives

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Abstract

The market demand for laminating adhesives in electronics or solar panels has been ramping up during recent years. Key performance requirements include excellent adhesion properties on a variety of substrates such as PET, PMMA or glass, high heat stability (reliability) and good moisture resistance. In many applications, such as displays or touch panels, low-k dielectric property is also required. In this paper, several innovative materials that can meet these requirements will be discussed.

Introduction

Laminating adhesives are used for bonding two substrates together. They can be used in a variety of applications, such as food packaging, aerospace, automobiles, electronics or solar panels. The major technologies are solventborne, waterborne, 2k urethanes and UV curable resins. UV technology is rapidly gaining acceptance in a variety of industries due to its environmental and economic advantages [1].

During recent years, there has been strong market demand for liquid optically clear adhesives (LOCA) for display or touch panel applications. Key performance requirements include strong bonding strength to the substrates, excellent reliability, high optical clarity and superior moisture resistance. For high quality displays, low dielectric constant bonding adhesives are also necessary. The moisture barrier performance of UV/EB cured materials has been discussed in previous publications [2-3].

In this paper, we will discuss the durability properties of UV cured acrylates under three weathering conditions including QUV, 85°C/85% relative humidity and freeze-thaw cycles. The key properties we evaluated are optical clarity characterized by UV-Vis % transmission and T-peel strength. In the end, we will discuss the dielectric properties of different types of oligomers.

Experimental

Several new products were developed for laminating adhesive applications. Their adhesive and durability properties were evaluated using two commercial products CN966J75 and CN9018 as controls.

Reliability Property Tests

Sample Preparation

Each oligomer was formulated using Sartomer's standard adhesive formulation package. The adhesives were laminated between DuPont (XST-6578) PET films. They were cured under a Fusion 600 W/in D lamp at a speed of 15 fpm. The thickness of each adhesive sample was controlled at approximately 9 mils.

Weathering Tests

We evaluated the adhesive performance under three weathering conditions: ASTM D4329-05 cycle B QUV; 85°C/85% relative humidity; and freeze-thawn cycles (each cycle includes 20 hours at 85°C/85% relative humidity and 2 hours at -40°C).

T-Peel Test

The samples were cut at 1” width. T-peel test was performed using Instron 5543 following ASTM D1876-08 standard. The weathering samples were equilibrated at RT/50% relative humidity environment for seven days before T-peel testing.

UV-Vis spectra

UV-Vis spectra were collected for each laminating adhesive before and after weathering up to 2000 hours.

Dielectric Property Tests

Sample Preparation

Oligomers or oligomer/monomer blends were formulated with 3% Esacure KIP 150 and 1% Esacure TZT. The samples were drawn down onto aluminum, followed by cure under a Fusion 600 W/in H lamp at a speed of 25 fpm.

Dielectric Constant

The films were cut into 3”x3” squares. The thickness was measured to be 2 to 3 mils. The dielectric constant was measured following ASTM D150 method at a frequency of 1 kHz.

Results and Discussions

Table 1 introduces nine new acrylate products using two commercial products CN9018 and CN966J75 as controls. CN9018 is well accepted for pressure sensitive adhesive applications, and CN966J75 for laminating adhesive applications. Using the standard formulation package (see Experimental section), adhesive samples were prepared for each oligomer. Then the adhesives were laminated and cured between two PET films with controlled thickness at around 9 mils.

Table 1. Structures and Properties of Oligomers.

Products	Type of Oligomers	Viscosity at 60°C, cps	Color
CN9018	Polyether UA	20,000	<100 APHA
CN966J75	Polyester UA	4500	<100 APHA
PRO12184	Polyester UA, High molecular weight	16,000	<100 APHA
PRO12507	Tin-free polyester UA	15,500	<100 APHA
NTX12321	Allophonate	41,000	<120 APHA
NTX11945	Bio-based UA	12,000	<120 APHA

NTX12514	Moisture-resistant polyester UA	5600	<150 APHA
NTX12427	Polyvinyl Acetal	25,000	<150 APHA
NTX12585	Polycaprolactone UA	22,800	<120APHA
PRO12384	Polycarbonate UA	26,000	<100 APHA
PRO12546	Polycarbonate UA	22,500	<100 APHA
PRO12599	Polycarbonate UA, High Molecular weight	80,400	<100 APHA

Peel strength and optical clarity are critical properties for electronic laminating adhesives. In this study, we monitored the change of these properties with time under three weathering conditions. Due to the flexibility of the PET substrates, we chose the T-peel test to determine the strength of the adhesives. The three weathering conditions reflect different levels of temperature, moisture and UV exposure. The QUV testing condition is relatively mild in both temperature and humidity. This test indicates the material's UV resistance. The 85°C/85% relative humidity weathering test indicates the adhesive's moisture resistance property. Finally, the freeze-thaw weathering test is used to screen for adhesives that can survive very harsh conditions.

Figure 1 presents the T-peel strength of the adhesives after 500 hrs exposure to QUV, 85°C/85% relative humidity and freeze-thaw conditions. As seen from Figure 1, the commercial control, polyester urethane acrylate CN966J75, has much higher initial peel strength than polyether urethane acrylate CN9018. Similarly, the experimental oligomers with the highest initial peel strength are polyester urethane acrylates (PRO12184, PRO12507 and PRO12546) and polycarbonate urethane acrylates (PRO12384 and PRO12599). Most experimental products except for reactive polyvinyl acetal present better durability than controls. The best candidates for extreme weathering conditions (freeze-thaw) are polycarbonate urethane acrylates PRO12546 and PRO12599. Interestingly, even though the ester group was easily degraded at high humidity conditions, NTX12514, based on a moisture resistant polyester polyol, presents superior durability.

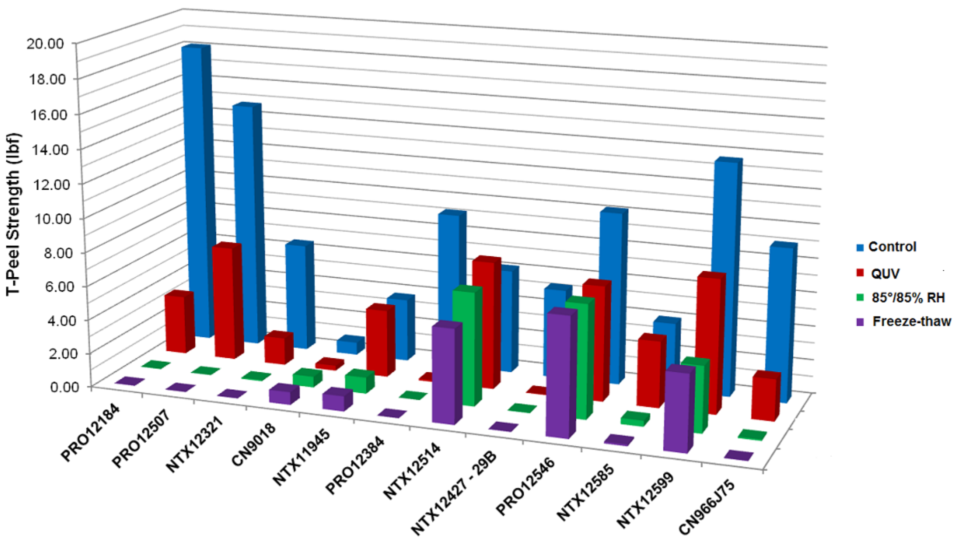
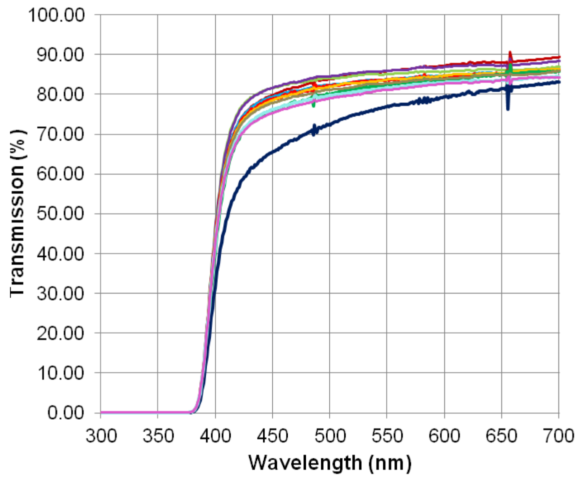
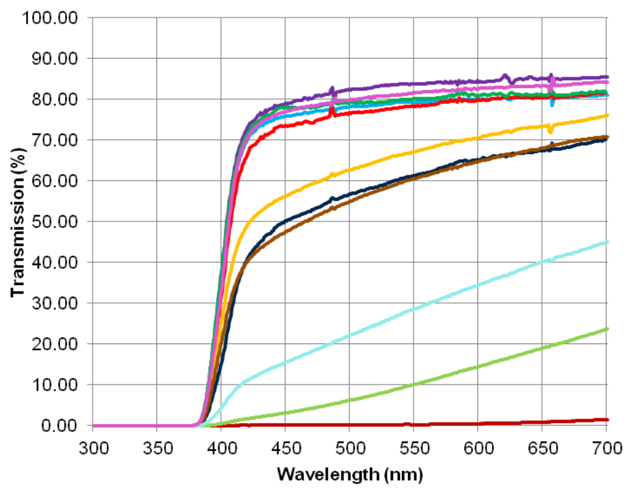


Figure 1. T-peel strength of the adhesives after 500 hour weathering tests.

Figure 2 presents the UV-vis spectra for the adhesives after 500 hour weathering tests. All of the oligomers exhibit good optical clarity after QUV aging. Three oligomers, including polyester urethane acrylates PRO12184 and PRO12507, and polycarbonate urethane acrylate PRO12599, lost clarity after 500 hrs of 85°C/85% RH aging. The common feature of these three oligomers is their high molecular weight. Lower crosslink density could allow higher moisture permeability. The low peel strength and high cloudiness of the adhesives based on PRO12184 and PRO12507 indicate that the ester group on the oligomer backbone might degrade during 85°/85% RH weathering. The high peel strength but low clarity of the adhesive based on PRO12599 indicates that the penetration of moisture into the films might also cause cloudiness. As expected, these three oligomers are also the worst performers under freeze-thaw weathering conditions. The combination of high peel strength and excellent optical clarity of NTX12514 indicates that this oligomer is the best candidate for electronic adhesive applications.



(a) QUV



(b) 85°/85% RH

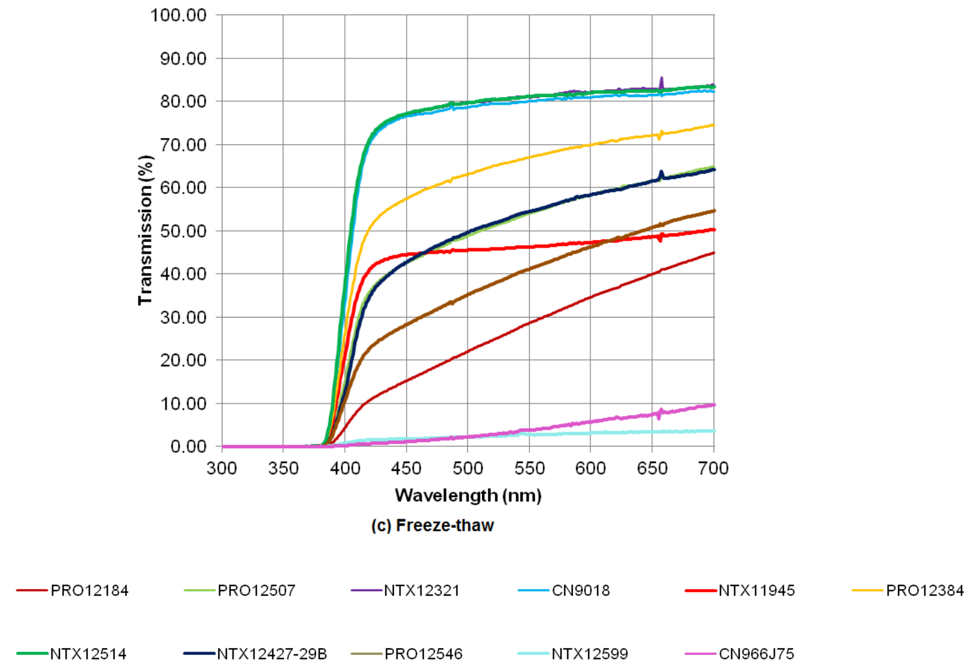


Figure 2. UV-Vis spectra for adhesives after 500 hour weathering tests.

As mentioned before, consumer electronic adhesives usually require 1000 hours durability, thus we continued the weathering tests to 1000 hours and 2000 hours. Obviously, the T-peel and clarity of the films after 1000 hours weathering are better than those after 2000 hours. So we only present the T-peel strength data after 2000 hours aging (Figure 3). The two best products were NTX12514, a urethane acrylate based on moisture resistant polyester polyol, and PRO12546, a polycarbonate urethane acrylate. Only four oligomers including NTX12321, CN9018, NTX12945 and NTX12514 could maintain optical clarity throughout 2000 hours of all the weathering tests. Again, we believe that the cloudiness of the adhesive based on PRO12546 is caused by moisture penetration. Therefore, we collected UV-vis spectra again after the adhesive was equilibrated at room temperature for seven days. Under this condition the film based on PRO12546 exhibited superior optical clarity, which supports our hypothesis.

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In conclusion, NTX12514 and PRO12546 are the best candidates for electronic or solar panel laminating adhesive applications. Other experimental oligomers PRO12184, PRO12507 or NTX12599 also present higher performance than existing commercial product CN966J75.

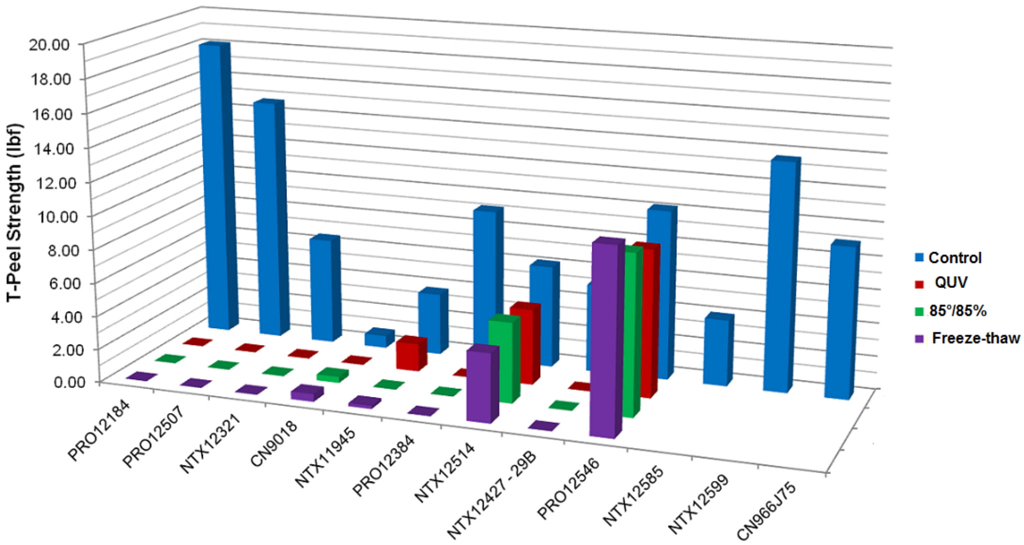

Figure 3. T-peel strength of the adhesives after 2000 hour weathering tests.

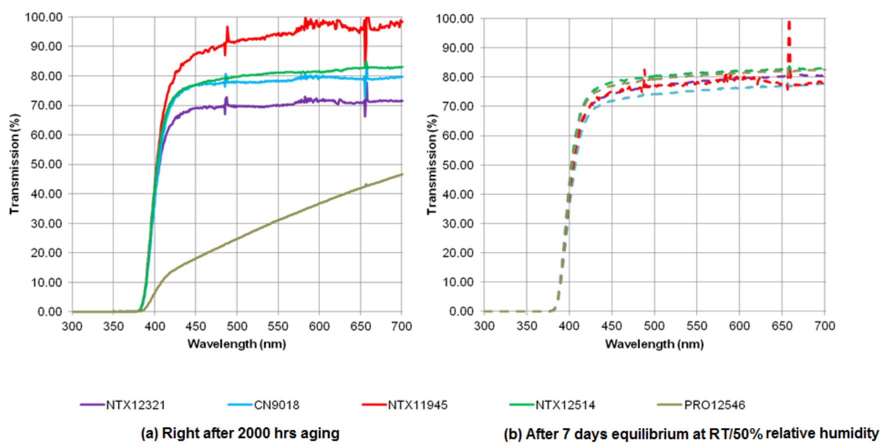

Figure 4. UV-vis spectra for the adhesives (a) immediately after 2000 hour weathering tests and (b) following equilibration at room temperature/50% relative humidity for seven days after 2000 hour weathering tests.

With the increasing popularity of smartphones and tablet PCs as enabling tools, the importance of display quality is becoming more important. In particular, the need for better outdoor readability is emphasized [4]. Filling the air gap between the touch panel and the display by directly bonding the optical clear adhesives (OCA) drastically improves both indoor and outdoor readability. The lower the dielectric constant of OCA layer, the thinner the OCA layer and the better the performance. In this study, we evaluated the dielectric constant for several commercial products. Table 2 summarized our testing results. The best products with low dielectric constant and superior adhesion properties are CN9014 and CN823. The testing on experimental products presented in earlier section is underway.

Table 2. Dielectric constant of oligomers or oligomer/monomer blends at 1 kHz.

Products	CN9014/SR238	CN9023	CN309	CN9030	CN823/CN309
Dielectric Constant at 1 KHz	1.9	4.1	2.0	2.7	2.2

Conclusion

In this study, NTX12514 and PRO12546 demonstrate high bonding strength to PET substrates and excellent durability under extreme weathering conditions. Products PRO12184, PRO12507 or NTX12599 demonstrate high bonding strength and reasonable reliability, which can be good candidates for conventional consumer or industrial electronics.

For liquid OCA application, the existing commercial products CN9014 and CN823/CN309 are the best candidates. A series of new products are under development to meet this market needs.

References

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