Ink Bonding Properties of Electron Beam Cured Adhesive Laminates for Flexible Packaging

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Introduction

The advantages of Electron Beam laminating adhesives have been previously described.¹⁻⁸ The most significant advantage is that full bond strength is achieved immediately upon curing. This allows immediate slitting and shipping of the laminated product. Instant bonding also allows in-line integration of the laminating process with other converting steps. This may include printing, slitting, sheeting, or the addition of subsequent laminate layers.

In a previous study excellent bond performance was demonstrated with multiple flexible packaging substrates. The laminates also exhibited excellent water and food product resistance. This study was limited to unprinted substrates.⁹ In practice, most commercial packaging laminations involve the use of printed films. Since it is desirable to protect the graphics, printing is usually on the inside surface of the outermost ply of the package. In this type of construction the adhesive is in direct contact with the ink; therefore, the adhesive/ink interaction plays a key roll in the performance of the laminate. The purpose of this study was to evaluate the performance of current commercial ink systems with EB curable laminating adhesives.

Experimental

Four different solvent-based ink systems (<u>A</u> to <u>D</u>) were supplied by Siegwerk USA Inc.¹⁰:

- A. Polyurethane base
- B. Polyamide base
- C. Polyamide base with crosslinker
- D. Nitrocellulose/polyurethane base

A blue and back-up white version of each system was used. These inks were printed using a PCMC Vision flexographic printing press on an acrylic treated polyester film (48 gauge HostaphanTM 2CSR). Stripes of the different blue inks were printed in the web direction with strips of the different whites in the cross direction. The net result was a printed grid of single ink layers along with two layers the different blue and back-up white combinations.

The printed film was laminated off-line on the pilot line at the Faustel Technology Center¹¹. An EB laminating adhesive (Northwest Coatings 52100#) was applied to a 50 micron (2 mil) LLDPE film (Pliant Max200) at 2.3 g/m2 (1.4 lbs/3000 ft²) at room temperature with an offset gravure coater¹². The printed polyester film was nipped to the wet adhesive and the combined webs were irradiated through the PET film using an Energy Science Electocure EB processor operating at 125 kV with an applied cure dose of 3.0 MRads. Line speeds were 30.5 and 152 m/min (100 and 500 ft/min).

The instant bonding properties were confirmed by hand testing immediately after curing. T-peel testing (250 mm/min) was conducted within 24 hours. Each peel test was run in triplicate.

Heat sealing was tested by cutting, folding, and sealing the LLDPE side of the laminate to itself using a Sencorp laboratory heat sealer operating at 190°C (375°F) for 1.0 second at 275 kPa (40 psi). Seal strengths were measured in a T-peel geometry with a crosshead speed of 250 mm/min. Each seal strength test was run in triplicate.

Results and Discussion

The bond strengths for single ink layer laminations are shown in Figures 1a and 1b. Excellent bonds were achieved with the <u>A</u>, <u>B</u>, and <u>C</u> ink systems, as well as unprinted areas. In these cases the failure was a straight tear of the PET film. The maximum strength at tear was reported for these systems. In some cases the maximum strengths at tear were higher for the laminations run at 100 ft/min compared to 500 ft/min; however, all were greater than 440 g/in. The <u>D</u> (nitrocellulose/polyurethane) ink systems produced laminates that peeled without tearing the film. In these cases there was a significant decrease in the peel strength between samples run at 100 and 500 ft/min. The failure mode in all cases upon peeling the samples with <u>D</u> inks was a splitting of the ink layer.





The bond strengths for two ink layer samples are shown in Figures 2a-d. Any combination with the \underline{D} blue or white ink had relatively low bonding performance. This reflects the single ink layer performance with these nitrocellulose/polyurethane inks reported above.

When the <u>A</u> (polyurethane) back-up white ink was used (Figure 2a) excellent film destruct bond performance was achieved with the <u>A</u>, <u>B</u>, and <u>C</u> blue inks. In some cases the maximum at tear was slightly higher at 100 ft/min relative to the 500 ft/min lamination runs; however, all were greater than 450 g/in.

In some cases certain combinations of blue and back-up white exhibited a significant difference in bond performance between the 100 and 500 ft/min lamination speeds. This was most pronounced in the laminates with the <u>B</u> (non-crosslinked polyamide) back-up white ink (Figure 2b).

One potential explanation for the speed effects is the dwell time for the liquid adhesive to interact with the ink between the nip and the EB unit on the lamination line. (For this lamination line the dwell time is approximately 12 seconds at 100 ft/min and 2.4 seconds at 500 ft/min.)









The most common way to form and fill flexible packaging is by a heat sealing. Laminated packaging must withstand the applied heat without delamination. It must also develop and maintain adequate seal strength to contain the product for the desired application. It is common to seal packaging in printed areas of the package; therefore, the adhesive/ink bond must be maintained.

The EB cured laminations (100 ft/min samples) with the various ink combinations were examined upon heat sealing. No delamination was observed in any of the samples. The seal strengths of the laminates are shown in Figures 3a-f. The average and maximum seal strengths were recorded. In general, the seal strengths were good with most of the ink combinations. With the single ink layers (Figures 3a and 3b) the seal strength was good for all of the ink systems. When two ink layers were used (Figures 3c-f) the seal strength was somewhat lower with laminates containing the blue \underline{D} (nitrocellulose/polyurethane) ink. This is consistent with lower PET/LLDPE bond strength with this ink system. It is expected that lower laminate



bond strengths would result in lower seal strengths due to an overall reduction in the strength of the composite structure. The failure mode of the laminate was observed while performing the seal strength testing and included both tearing of the PET and delamination of the PET from the LLDPE. There was no apparent correlation between the seal strength and the mode of failure of the laminate.

Conclusions

The bond strengths of EB laminations were dependant on the types of inks that were used. Excellent bond performance was obtained with current commercial lamination ink systems. Overall, a polyurethane based ink system (<u>A</u>) gave the best performance in this study. Good bonds were obtained with this ink system with multiple ink layers and also with lamination speeds up to at least 500 ft/min.

Heat sealing of the EB laminates did not show any concerns associated with this combination of adhesive and ink technologies.

The overall performance of these EB adhesive/ink combinations further demonstrates the commercial technical viability of EB laminating technology.

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Laminating Technologies for Flexible Packaging

- Extrusion lamination
- Solvent base adhesives
- One-component water base
- Two-component water base
- Two-component solventless adhesives
- UV Curable adhesives
- EB Curable adhesives





Adhesive Laminating Technology Comparison

2-Component Solventless Laminating Adhesives	EB Curable Laminating Adhesives
Requires accurate mixing of two components	One component, no mixing, remains unchanged until EB exposure
Heated application equipment	Room temperature application
Toxicity concerns with residual aromatic amines	No aromatic amines; high conversions minimize uncured material
Typical application weight 1.0 lb/3000 ft ² (1.6 g/m ²)	Typical application weight 1.0 lb/3000 ft ² (1.6 g/m ²)



Adhesive Laminating Technology Comparison (continued)

2-Component Solventless Laminating Adhesives	EB Curable Laminating Adhesives
Requires several days to cure; delayed QC, slitting, shipping, and filling	Instant cure; immediate QC, slitting, shipping, and filling
Multilayer structures requires multiple lamination steps	In-line multilayer lamination possible
Laminate appearance <u>may</u> continue to improve in the roll	Laminate appearance is established immediately upon cure
Performance with many ink systems is known	Performance with ink systems needs to be established

Commercial Status of EB Laminating Technology

- Fully commercial with at least 7 different converters in 8 locations
- Largest application film to paperboard lamination
- Established commercial non-food flexible packaging applications
- Commercial food-packaging application
 with recognized barrier materials

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• Line speeds run over 1000 ft/min

Keys to Broad Commercial Acceptance of EB Laminating Technology

- Meet required bond performance with suitable substrates
- Suitable for food packaging FDA compliance, no taint or odor
- Applied cost competitive with other adhesive technologies
- Consistent good quality laminate appearance
- No adverse effects of EB on substrates
- Compatibility with available ink systems

Summary of Previous Studies

- Excellent film destruct bonding properties achieved with multiple substrates including BOPP, PET, LDPE based sealant films, and aluminum foil (PLACE 04)
- Adhesives optimized for various substrate compositions (PLACE 03, 04)
- Laminates exhibited good water and food product resistance (PLACE 04)
- Adhesive application properties optimized to produce desirable laminate appearance (PLACE 05)

Ink Bond Performance Study

- Four different ink systems supplied by Siegwerk USA, Inc.
- Blue and back-up white for each system
- Flexographic printing of a "grid" of single ink layers and blue/white combinations on chemically treated PET film (Mitsubishi 2CSR)
- EB adhesive (Northwest 52100) applied at 1.4 lbs/3000 ft² (2.3 g/m²) to 2 mil (50 micron) LLDPE (Pliant Lmax200) and nipped to the printed PET
- EB cure through the PET (3.0 Mrads @ 125 kV)
- Test bond strength and seal strength of the laminates for all ink combinations

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Flexographic Ink Systems

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- A = Polyurethane base
- B = Polyamide base
- C = Polyamide base with crosslinker
- D = Nitrocellulose/polyurethane base

































Summary of Bond Strength Results

- All single layer ink systems except <u>D</u> (nitrocellulose/polyurethane) gave film excellent film tear bonds at 100 and 500 ft/min
- Two-layer systems with A (polyurethane) backup white gave excellent film tear bonds except when <u>D</u> blue ink was used
- Two-layer systems with <u>B</u>, <u>C</u>, or <u>D</u> back-up white were strongly affected by the lamination line speed

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 Overall, laminates produced using <u>A</u> (polyurethane) inks gave excellent bond performance results

















Summary of Seal Strength Results

- No delamination was observed upon heat sealing with any of the ink systems
- Seal strength was good in all single layer ink areas
- Seal strength was good in areas with two inks layer expect in cases where one of the ink layers was the <u>D</u> (nitrocellulose/polyurethane) ink

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Conclusions

- Good bond and seal strength was achieved with current commercial inks systems
- Overall, a polyurethane ink system gave the best results
- Overall, a nitrocellulose/polyurethane ink system gave the poorest results
- These results along with the results of previous studies shows the commercial technical viability of EB laminating technology

Future Work: Keys to Broad Commercial Acceptance of EB Laminating Technology

- Meet required bond performance with suitable substrates
- Suitable for food packaging FDA compliance, no taint or odor
- Applied cost competitive with other adhesive technologies
- Consistent good quality laminate appearance
- No adverse effects of EB on substrates
- Compatibility with available ink systems

Four Ways To Establish FDA Compliance

- No migration/no food additive position below 50 ppb detection limit for most materials and applications – Current EB food applications
- Threshold of Regulation Listing requires nontoxic/non-carcinogen materials with dietary intake less than 0.5 ppb
- Use materials cleared under existing regulations (Prior Sanction letter, GRAS position, prior Food Additive Petition) – few UV/EB materials have appropriate FDA status
- 4. Register new materials with FDA through a Food Additive Petition or Food Contact Notification (FCN) application – In progress 28

