

Evaluation of Low Voltage Electron Beam Processors Using Thin Film Dosimetry Techniques

By Im Rangwala and Mike Swain

Radiation-induced in situ polymerization reactions offer significant advantages over conventional thermal processes. The biggest advantage is usage of 100% reactive and compliant chemistry and thus no thermal drying required at all.

Since the introduction of electron beam equipment in the early 1960s, polymer chemists were intrigued by the ability of the electrons to initiate free-radical polymerization reaction without the addition of any

initiated substantial interest in food packaging applications. These applications are curing of coatings, laminating adhesives and inks. For each of these applications, the development cycle begins at the chemistry supplier's laboratory, most of them are equipped with laboratory EB equipment. Once the formulation meets the desired specifications for each of these applications, the next phase in development is scale-up to pilot or continuous roll-to-roll process using pilot EB equipment. This is the pre-commercialization phase, and it is at this phase that the chemistry supplier will produce enough material to do migration studies to comply with food law regulations. It is very important that the dose used to do early development using laboratory EB equipment is appropriately transferred to pilot EB equipment and finally to commercialization (commercial EB equipment).

For example, an adhesive chemist develops an EB-laminating adhesive to laminate two dissimilar plastic films and uses a dose to cure the adhesive of 3 Mrads. Suppose this is a food application and requires FDA compliance. To obtain FDA compliance, migration studies are completed using pilot EB equipment. The migration study involves producing laminates on

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photoinitiators or photosensitizers.¹ Immediate applications were sought in packaging since electron processing offered high speed curing. In particular, food packaging because electron beam processing results in:

- High degree of conversion. (Low migration)
- No photo-initiator or other additives like peroxides.
- Good quality control through NIST traceable dosimetry techniques and closed loop control electronics.

The development of low voltage electron beam equipment² has

the pilot then EB cured at a dose of 3 Mrads as used during laboratory work. These laminates are then used for extraction work, using appropriate food stimulants as stipulated by the food and drug guidelines. Upon obtaining FDA compliance, the product is commercial and is manufactured using commercial EB equipment; again, 3 Mrads dose is used to cure the adhesive.

The question is how would one relate the 3 Mrads dose to cure the adhesive using three different electron beam sources, lab EB equipment, pilot EB equipment and then finally commercial EB equipment.

In addition, a further question remains "how would one ensure that the EB equipment is delivering the required 3 Mrad dose on a regular basis."

The answer to these questions is thin film dosimetry techniques. Using thin film dosimetry, one can measure the entire output of the electron beam processors with good accuracy, providing required quality control during both development and ongoing processing.

Thin Film Nylon Dosimetry

A dosimeter is anything that undergoes an observable and consistent physical change that can be correlated with the dose of radiation it has received. For example, some dosimeters change color when irradiated. The bigger the dose, the more pronounced the color change. The color change is due to a presence of a radiochromic dye that is blended in a polyamide matrix. The polyamide film is then cast out of solution; cut to 1 cm x 1 cm then is called radiochromic film dosimeters.³

The very high stopping powers of matter for low energy electrons (<125kV) pose unusual difficulties in the direct mapping of the output of such a processor due to the very

limited range of its primary beam.

Here's why. Voltage determines the penetration—the higher the voltage, the deeper the penetration. One of the advantages of low voltage is that the beam can be tuned to only penetrate into the inks and coating on top of a substrate. The process is efficient. Instead of dumping waste energy into

Reading Dosimetry

The naked eye can easily determine if a film has been irradiated, but to actually measure a dose requires instruments. The films start out transparent with a pale blue color. When irradiated, they turn a dark blue and pass far less light. The dose that they have received can then be

FIGURE 1

Equation for the change in optical density of film dosimeters. Optical density equals the change in absorbance divided by the film thickness.

$$\text{Optical Density} = \frac{\text{Absorbance After} - \text{Absorbance Before}}{\text{Thickness}}$$

the paper below, the radiation only reaches the surface coatings.

While shallow penetration is good for the product, it complicates dosimetry. For accuracy, the entire dosimeter should receive the same dose. If a thick dosimeter is used, the top surface may receive a different dose than the center or bottom surface. Radiochromic film dosimeters come in a variety of thickness from 50-microns down to eight-microns. Low voltage EB processors (<125kV) demand a thin dosimeter eight-microns thick because of its lower penetration. In general, thicker films are more robust and accurate when they can be used. For thinner dosimeters, great care must be exercised handling the films. Also, the inherent accuracy of the measurement tends to be lower.

correlated to their change in optical density. The optical density of the film is calculated from three measurements. The amount of light blocked by the film is measured before and after irradiation. This measurement is completed by a densitometer or a spectrophotometer. Only light of a specific wavelength is used; this wavelength is 600 nm. The pre-irradiation measurement is subtracted from the post-irradiation measurement. This difference is the change in absorbance. The change in optical density is the change in absorbance divided by the film thickness (Figure 1). To convert this to dose, one will need a valid calibration curve.

The good news is that it may not be necessary to perform all three measurements for each dosimeter. If the dosimeters are sufficiently uniform,

it may be possible to use an average value for the pre-irradiation absorbance and/or for the thickness. Post-irradiation absorbance is always measured.

Calibration is the process of correlating changes in optical density with known doses. NIST in the United States and NPL in the United Kingdom are the two primary sources of calibrated doses. These national laboratories will subject films to a well-characterized gamma source (Co^{60}) of radiation. The sources themselves have been characterized with calorimetry.

In order to calibrate dosimeters, it is first necessary to select calibration doses. Once the range has been selected, the number of dose points within the range should be determined. Five points per decade is a good rule of thumb. A statistically significant number of dosimeters are selected for each dose point, as well as for undosed control dosimeters. The resulting data can be interpolated to provide a lookup table, or multiple regression analysis can be performed to generate a function.⁴

EB Processor Characterization

The whole point of doing dosimetry is to determine if a given EB processor is performing within the required process parameters. These processors are characterized in three aspects:

- Yield measurements
- Beam uniformity
- Depth dose

Yield Measurements

The EB processor has no direct knowledge of the dose being delivered to the product. It can, however, regulate the beam current. By maintaining the beam current proportional to the line speed, it is possible to deliver a consistent dose to the product. The constant of proportionality is called "k." This "k" constant is measured for every EB unit and is

dependent on the width of the EB unit and the operating voltage. During a yield measurement, the "k" is determined. Thereafter, the "k" is used to compute the amount of beam current required to deliver a given dose at a given line speed. The equation governing the relationship is:

$$k = \frac{D \cdot S}{I}$$

Here, "I" stands for beam current measured in mA, "D" for dose measured in Mrads obtained by dosimetry and "S" for speed in feet/min. Therefore, the units of "k" are Mrads/fpm/mA

Dose is measured by dosimetry and empirically "k" the yield constant is calculated for the various EB units at fixed operating voltages. In this manner, the absorbed dose used to cure the product is correlated for various EB units. In addition, for the same EB unit an ongoing periodic determination of "k" keeps track of electron beam processor output with time and thus the absorbed dose.

Uniformity

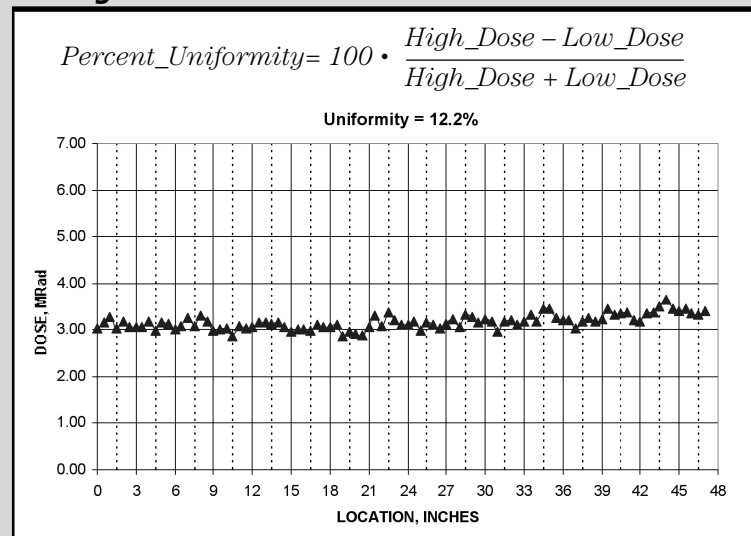
Regardless of how uniform most of the beam is, any high or low point can result in a product of unacceptable quality. For this reason, the uniformity statistic is determined by the highest and lowest doses seen across the width of the product.

Columns of films are placed across the width of the product at regular intervals. Then by averaging the dose from each dosimeter in the column, it is possible to reduce the uncertainty of the measurement. Also, if all columns display an increasing or decreasing trend, this would indicate that some sort of transient had occurred, and that the test should be re-run. Three is a good column depth.

As for the spacing between the columns, the Nyquist Sampling Theorem suggests that you should sample at least twice as frequently as any source of variation. For example, if your beam sources are placed every three inches, then you should have at least one

FIGURE 2

Formula for percent uniformity and chart plotting the highest and lowest doses of the electron beam



column for each inch and a half. One inch spacing is even more reasonable.

Once the average dose of each column has been computed, it is possible to plot the doses to obtain a profile of the beam. In addition, as a summary statistic, the uniformity can be expressed as a ratio of the highest or lowest point to the midpoint between the highest and lowest points. The formula for percent uniformity is shown in Figure 2.

Depth Dose

The simplest way to understand the penetration profile of the dose is with a depth dose chart. Film dosimeters are stacked on top of each other, and then irradiated. The dose absorbed by each layer is recorded and divided by the dose of the top film. The mass density of the dosimeters is recorded in grams per square centimeter. Finally, the normalized dose is graphed as a function of the total number of grams per square

centimeter required reaching the middle of the dosimeter. Figure 3 shows the depth-dose profiles from 70-125 kV.

The resulting curve depends on the high voltage level, as well as the thickness and density of any intervening air and window foil.

For each EB unit using stacks of dosimeters, such profiles are generated at each voltage level. These profiles are then compared with the expected profiles to ensure that the EB unit high voltage is in calibration.

Future Developments

Despite their utility, radiochromic film dosimeters leave much to be desired. For example, the absorbance of the film changes with time after irradiation. In addition, changes in humidity can introduce large measurement errors.⁵ These thin films are also cumbersome to handle, leading to human errors. While other technologies, such as alanine, promise to

someday provide more accurate results; none has surmounted the technical and economic barriers to routine industry use. For the time being, radiochromic film dosimeters remain the state of the art.

Conclusion

The use of radiochromic thin film nylon dosimeters is an accurate method of measuring the performance characteristics of low voltage EB processors. Challenges do remain in maintaining the accuracy and the handling of the eight-micron nylon films. Development work is ongoing to reduce these challenges and improve the accuracy of these dosimeters. ▀

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FIGURE 3

Low voltage EB equipment depth dose profiles

