Dosimetry Challenges in Low-energy Applications

Hans J. Wiegert
Cryovac, Sealed Air Corporation
Duncan, South Carolina, USA

Abstract

A power point presentation highlighting the challenges of measuring dose in industrial applications using low energy electron beams (70 to 300 keV) is presented, including:

- History of Technology and Equipment
- Overview of available Dosimetry Systems
- Practical use of ASTM 51818
- Equipment Performance and K-Factor
- Process Variables

History of Technology and Equipment:

From the discovery of the crosslinking of polyethylene with ionizing radiation by Malcolm Dole and elaborated upon by Arthur Charlesby in early 1950's, the practical application of this technology has continuously changed and equipment development has opened the door to new applications. The development of high current, low-energy electron beam (EB) accelerators allows surface treatment, for the curing inks and coatings in an environmental friendly process, for the curing of adhesives in laminating applications and for the crosslinking of plastic film for property modifications.

Manufacturers of commercial low-energy electron beam accelerators are: Advanced Electron Beams (AEB), Energy Sciences Incorporated (ESI) and PCT Engineered Systems (PCT), in the United States, Nissan-High Voltage (NHV) in Japan and Crosslinking AB in Europe. PCT is the successor of RPC Industries, having acquired the rights and trademarks of RPC's BroadBeam® equipment. ESI, PCT and NHV manufacture low-energy EB equipment that rely upon multiple filaments spaced evenly to cover the width of the product; AEB uses multiple emitters for larger width; while Crosslinking AB utilizes a "scanned" beam, which are more common in higher-energy EB equipment.

Overview of available Dosimetry Systems:

Dosimetry is a means by which one can determine the energy imparted per unit mass, or the "absorbed dose". "Dose" is expressed in Grays, where 1 Gray (Gy) = 1 Joule per kilogram (J/kg) = 100 rads, an older expression for "dose." In industrial applications, the expression is in kilograys (kGy) where 10 kGy = 1 megarad (MR). In using electron beam equipment, it is important to know that the equipment is operating correctly and that it delivers the correct exposure or dose to the product.

Radiochromic film dosimeters have been routinely used to monitor the performance of electron beam equipment in crosslinking and curing applications. These films are relatively inexpensive to use. The two most widely used radiochromic dosimeter systems are provided by: Far West Technologies (FWT) and by GEX. Both systems read the changes in the optical absorbance of dosimeters as a function of the dose received. To read the absorbance of these radiochromic dosimeters special instruments are required. GEX uses a Spectronics Genesys 20 WINdose Spectrophotometer and FWT a Digital Radiachromic Reader. FWT dosimeters are available in two thicknesses, 8 - 10 microns and 42 – 52 microns, the GEX dosimeters are about 17 microns in thickness. With both companies the thickness varies slightly from batch to batch and each batch has its own calibration curve.

GEX offers pre-assembled strips for dose uniformity responses and in stacks for depth-dose measurements together with a WINdose for EXCEL software program to calculate the respective dose based on the dosimeters absorbance reading. A computer interface is available to read the data direct from the optical reader into the spreadsheet. The FWT dosimeters are available in 1 centimeter squares (42 – 52 microns) or in sheets (8 – 10 microns). Uniformity strips and depth-dose stacks have to be assembled by the user. A calibration curve is provided, but it is up to the user to determine the necessary calculations for the dose. Again, a computer interface allows the direct reading of the absorbance values into a spreadsheet.

With both systems, the dosimeters have to be arranged or assembled in a way that will allow them to travel through the electron beam unit without getting damaged. Once removed from their protective packaging, the dosimeters are sensitive to ultraviolet light (as from common fluorescent lighting) as well as temperature and humidity during the process. It is good practice to use some "control dosimeters" to verify or establish the initial optical density.

While both dosimetry systems perform well for verifying and monitoring equipment performance, they do not easily lend themselves to determining the dose received by the product.

For crosslinking applications the dosimeters are usually too thin and several have to be stacked on top of each other to be representative of the product thickness (based on dosimeter and product density).

In curing and laminating applications, in which liquid applied formulations based on monomers and oligomers, the problem is worse, because even the thinnest dosimeter is too thick compared to the few microns of the coating or adhesive. In these cases, a correlation between the dose received by the dosimeter and product performance has to be established. This could be a "rub test" for inks and coatings, a "bond strength test" for laminations or any other test that confirms the desired results of the EB process.

Practical Use of ASTM 51818 [1]:

ASTM International's 51818 (Standard Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 80 and 300 keV) provides dosimetric procedures to be followed to determine the performance of low-energy single-gap electron beam radiation processing facilities. Important sections of this practice are:

6. Installation Qualification and Testing
6.1 Equipment Testing
This includes the necessary testing to determine whether the process equipment performs in accordance with design specifications.
6.2 Characterize the performance of the equipment using dosimetry
6.2.1 Surface Area Rate Measurements
6.2.2 Beam Uniformity Measurements
6.2.3 Depth-dose Measurements

These measurements are an essential part in the qualification and operation of any electron beam equipment.

7. Frequency of Dosimetric Measurements
7.1 Initial facility performance evaluation dosimetry should be conducted in accordance with Section 6.
7.2. Product Validation
7.3 After Routine Maintenance
7.4 After Major System Maintenance
7.5 Routine Process Control

8. Throughput Calculations
8.1 Mass Processing Rate
8.2 Area Processing Rate

ANNEX
(informative)
A1. METHOD FOR MEASURING SURFACE AREA RATE COEFFIENT (K), DOSE DEPTH AND DOSE UNIFORMITY

A1.1 This annex describes methods for measuring surface area rate coefficient (K), dose depth, and dose uniformity.
A1.2 Method for Measuring the Surface Area Rate Coefficient (K)
A1.3 Method for Measuring Depth-Dose Distribution
A1.4.Method of Measuring Beam Dose Uniformity across the Width of the Electron Beam

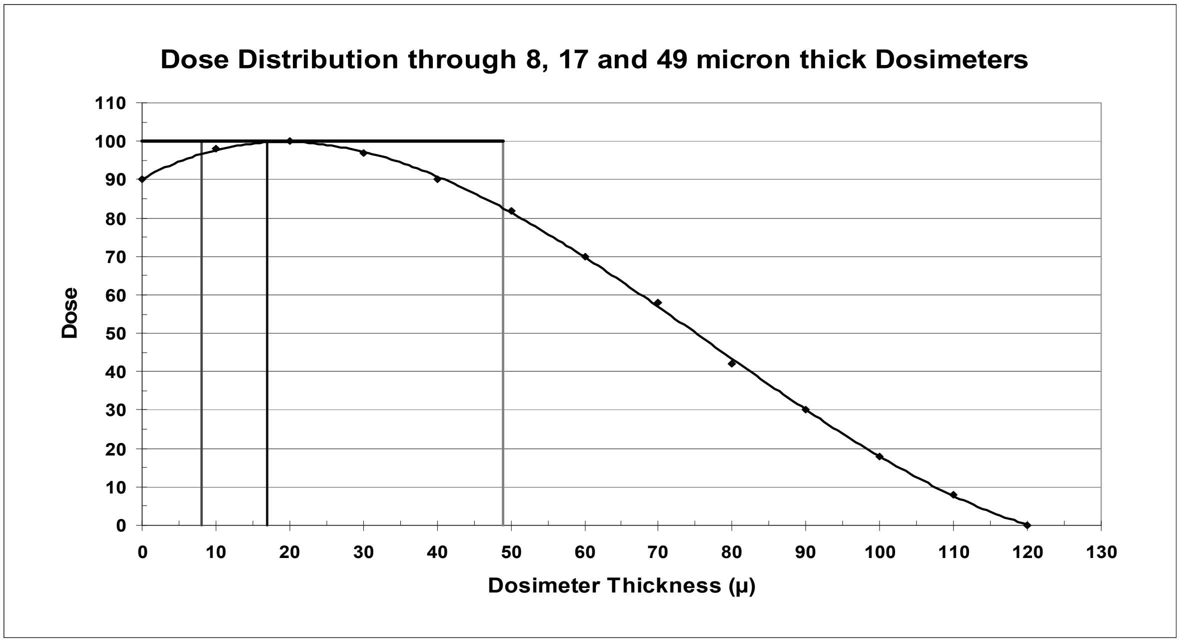
This annex is especially helpful, because it provides detailed instruction on how the different measurements are to be performed.

However, as is often the case with detailed instructions, some are too detailed. For example, in A1.2.4 the procedure refers to “index cards” as backing material for the dosimeters. Depending on product path and equipment, it may be more practical to mount the dosimeters on plastic film for greater flexibility.

While A1.2.2 points out that the value of “K”, a surface area coefficient factor, can vary widely over a range of voltages, it does not address the fact that “K” also varies with the thickness of the dosimeter being used. Using 8 micron dosimeters will result in a different "K" than one obtained with 17 or 49 micron thick dosimeters.

The graph below (Figure 1) shows the differences in dose received by dosimeters of various thicknesses, resulting in <u>different</u> "K" values at the <u>same energy</u>.

Figure 1:



Dosimeter stacks for depth-dose measurements can be quite bulky, especially at higher beam energies. It may be necessary to modify a pre-assembled stack in order to allow it to pass through processing equipment undamaged. In some cases it may even be necessary to build a stack with higher density absorbers (i.e. aluminum foil) inserted between the dosimeters to reduce the height.

Equipment Performance and K-Factor:

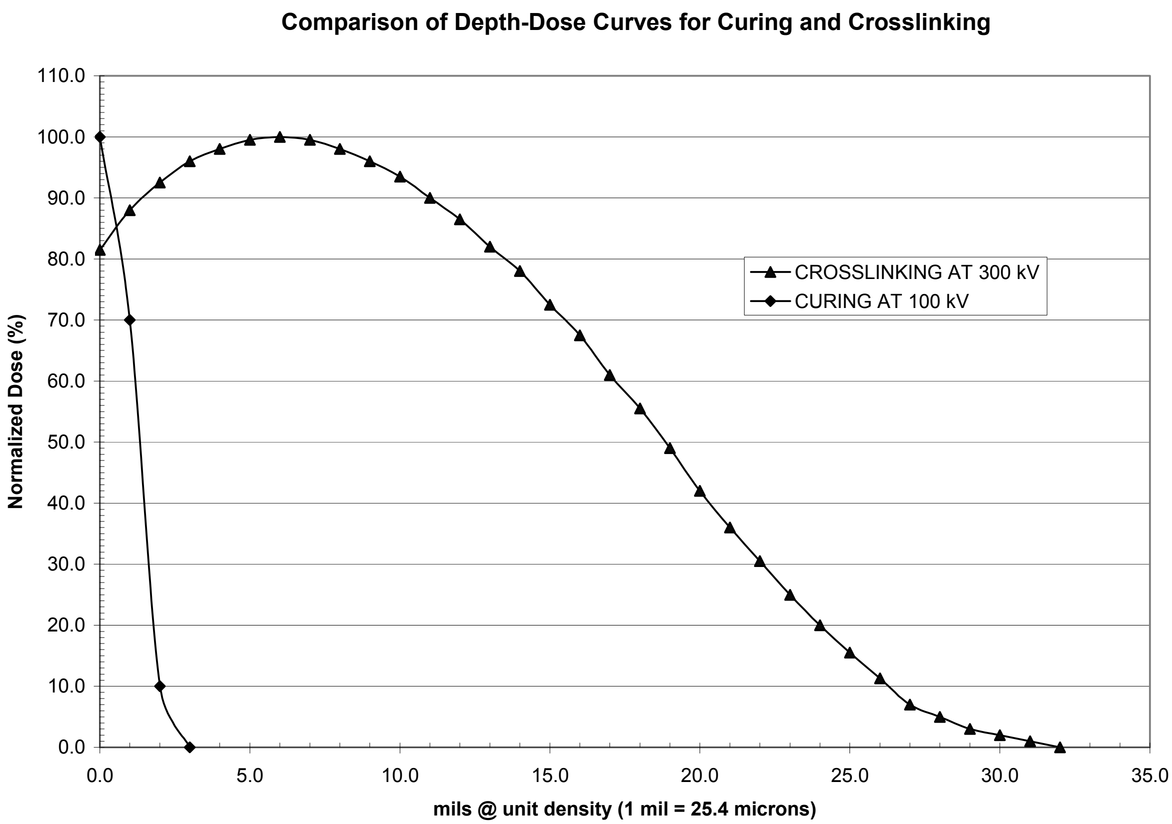
The manufacturer of an electron beam develops the equipment specifications based upon the customer's process requirements. To make sure that the equipment performs as intended, it is imperative that both the customer and the manufacturer understand each other clearly. Key points, which must be clearly understood, are: the "Operating Voltage" and the "Dose-Speed Capability" of a unit. For curing applications the voltage range is typically from 70 to 110 kV, crosslinking is done in the 150 to 300 kV range or higher.

Operating Voltage:

For curing use, the energy of the electron beam is ideally deposited only within the thickness of the coating (a few microns) without significantly effecting the rest of the structure. In a crosslinking application the electrons should penetrate the product completely and, if possible with the rear surface dose equal to front surface dose.

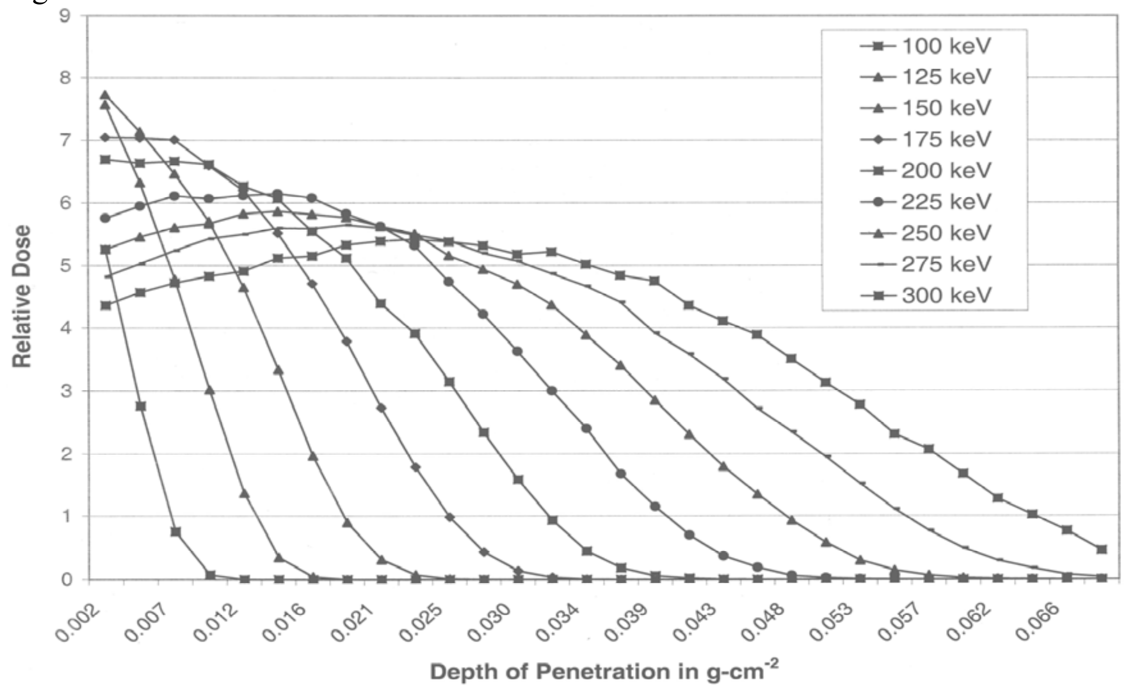
The depth-dose curves in the graph below (Figure 2) show that a 100 kV beam will effectively penetrate only about 50.8 microns (2 mils) of a material; while a 300 kV beam would be able to penetrate about 762 microns (30 mils). However, the practical maximum thickness of plastic film for crosslinking at this voltage would be 380 to 510 microns (15 to 20 mils) depending on what dose profile through the product is acceptable.

Figure 2:



The following chart is a summary of depth-dose curves from 100 kV to 300 kV.

Figure 3 as shown in ASTM 51818 [1]:



Many graphs show the depth of penetration in g/cm². While this may appear to be a confusing unit, simply by using a density of 1g/cm³ (unit density), the numbers become the penetration depth in centimeters (cm).

The graph also shows, that at voltages below 100 kV, it is becoming increasingly difficult to actually run a depth-dose test, because at least three points are needed to get a good approximation of the voltage.

Dose-Speed Capability:

The "Dose-Speed Capability" of the equipment is usually expressed in kGym/min (MRft/min) at a specified voltage. This is directly related to the "K" factor discussed earlier. Because not all manufacturers of electron beam equipment are using the same dosimetry system, it is important to know up front what dosimetry system the manufacturer uses to base the performance data of the equipment on. Using a 17 micron dosimeter will result at a higher value for the Dose-Speed Capability (and a higher "K" value) than using an 8 micron thick dosimeter. Only at very low voltages, when the electrons are completely stopped within the thinnest dosimeter, i.e. 8 microns will there be no difference since the total absorbed dose is the same in all dosimeters.

Also, not everyone in the industry is following the definition of K as per ASTM 51818, Section 8.2, when calculating the Area Processing Rate [1].

Area processing rate = $W_b \times V_1 = \frac{K \times I}{D}$

Where:

K = surface area rate in kGy m²/mA min or Mrad ft²/mA min
D = dose in kGy or Mrad
W_b = beam width in m or ft,
V_1 = line speed in m/min of ft/min, and
I = beam current in mA.

Therefore, $K = \frac{W_b \times V_1 \times D}{I}$

Some manufacturers of electron beam equipment do not include the beam width in the calculation of the K value. Again, that is something the customer needs to understand and be aware of.

It is strongly recommended, that from the beginning of a project, the equipment specifications require conforming to ASTM 51818.

Process Variables:

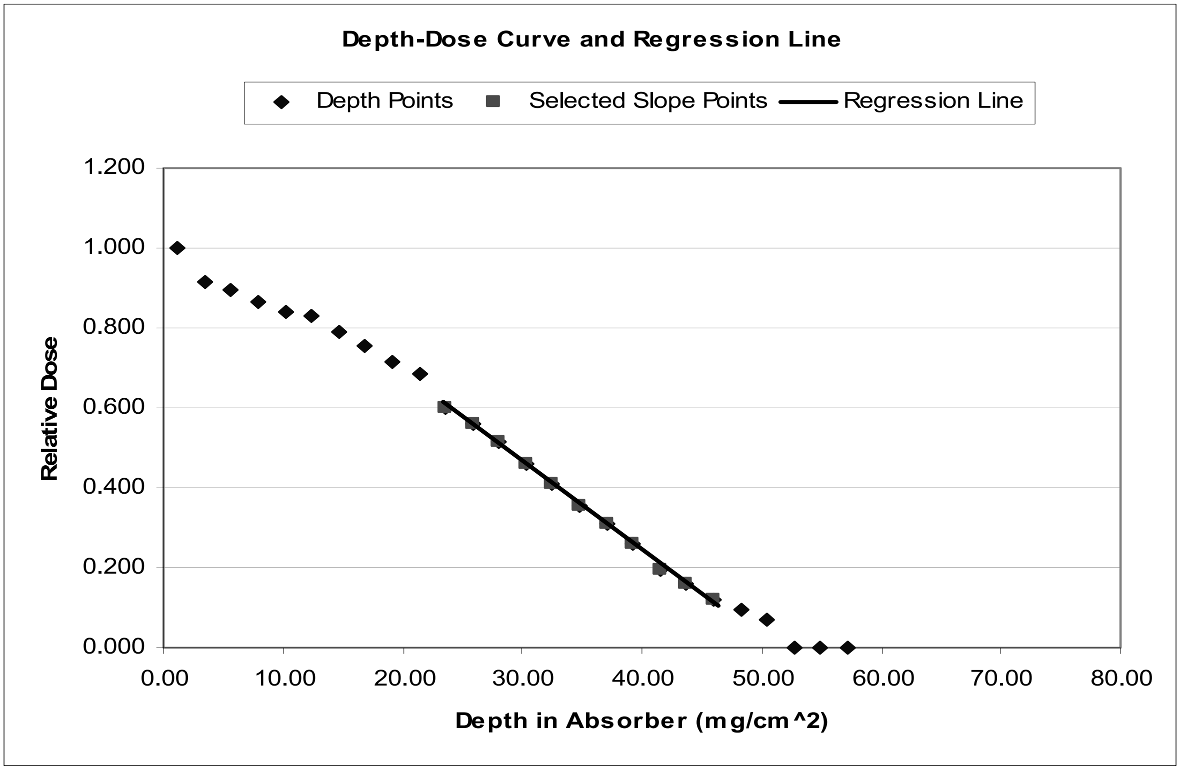
After the "how things are supposed to work", what are the challenges on the plant floor? Assuming mechanical alignment and good web tracking through the unit, there are still some surprises laying in ambush.

Depth-Dose and Scattering:

When electrons exit through the thin foil window, secondary and tertiary electrons at lower energy levels are created. Running a Depth-Dose stack through a unit with a drum (for cooling) these lower energy electrons artificially raise the dose in the top layers of a depth dose stack resulting in distorted Depth-Dose curves like the one below. Also, because of the curved surface of the drum, the distance from the window to dosimeter initially decreases as the dosimeter enters the beam area and then increases again as the dosimeter moves away from it. The electrons lose energy in air and a larger gap will result in lower energy electrons impacting the upper dosimeters in the stack, creating an artificially high reading.

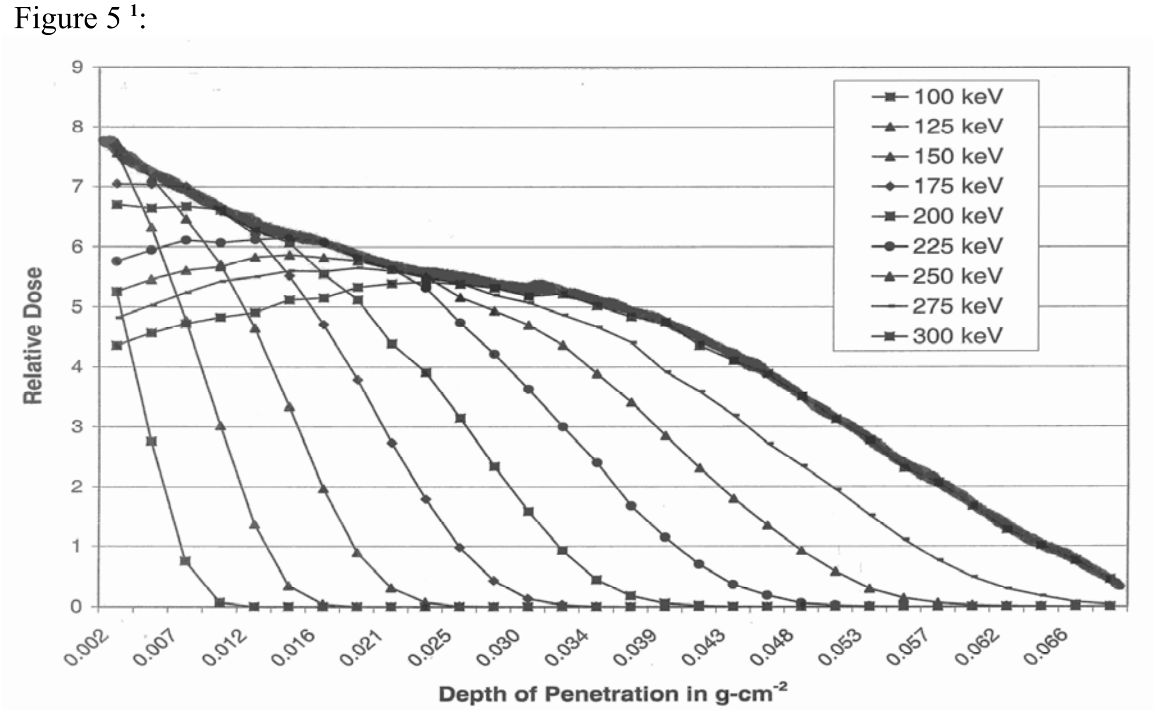
Figure 4:

BroadBeam® Unit with Chill Drum, 300 kV, 52 mA, 50 ft/min



Looking at the range of Depth-Dose curves shown in Figure 3, it looks like all energies are represented in the data; just as if a line was drawn as shown below (Figure 5).

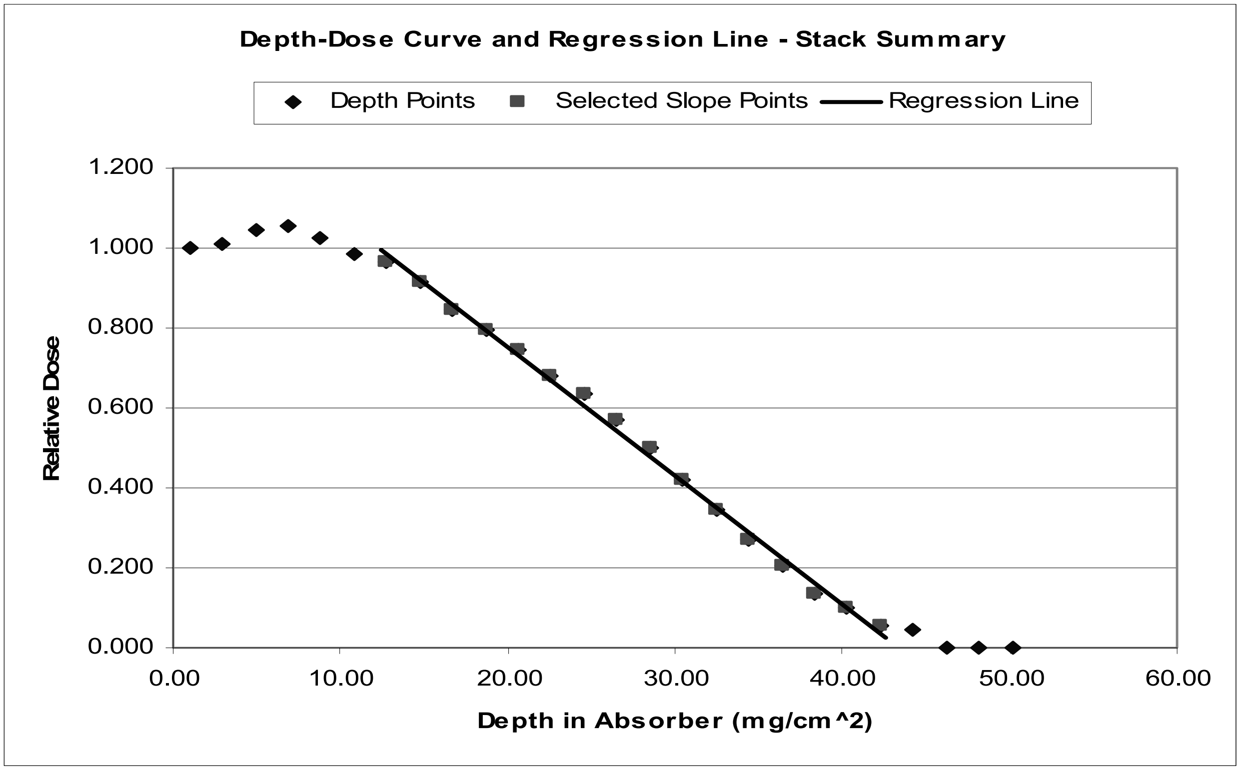
Figure 5 [1]:



Running a Depth-Dose stack through another unit without a chill drum resulted in getting a more “typical” curve.

Figure 6:

BroadBeam® Unit, 225 kV 42 mA, 50 ft/min



When comparing Depth-Dose curves for a given Voltage, one has to keep in mind that the curves will vary with window foil thickness and the size of the air gap.

Uniformity and Web Flutter

When the product is not supported in the beam area like with a drum, the web may flutter as it passes under the beam. This is especially a possibility if the equipment is not Nitrogen inerted and an exhaust system is used to remove the Ozone, causing a significant amount of air to flow through the unit. For uniformity and area processing rate measurements at least three strips should be run behind each other. The strips should be spaced at a sufficient distance to reduce the variability caused by web flutter. Wrinkles in the product – even when going over a chill roll - can have the same effect. Good control of web speed, tension and roll alignment are critical to obtaining good data.

It is important to keep in mind that at higher operating voltages those variables will have less effect on the dosimetry than at lower energies. At low voltages, small variations in the distance of the product from the window (flutter, wrinkles) will result in a larger percentage of variation in the dose measurements.

Environmental conditions (temperature, humidity) can also affect the dosimetry results, especially, if strips have to be prepared, transported to the equipment, run through the unit and transported back for processing. It is important to understand the limitations of the dosimeters used and follow the manufacturer's instructions for handling and processing as close as possible.

Dosimetry is an essential tool for monitoring equipment performance in crosslinking, laminating and curing applications. It requires a good understanding of the equipment and the process, as well as an understanding of dosimetry and its limitations.

Dosimetry is not failsafe and if it does not produce the expected results, one must always look at the dosimetry procedures and at the equipment before coming to a conclusion.

I would like to thank A.J. Berejka, Ionicorp, Huntington, New York for his helpful suggestions during the preparation of this paper.

End Notes:

1. Standards on Dosimetry for Radiation Processing, 2nd Edition, September 2004, ASTM International, West Conshohocken, PA.