A METHOD FOR SPECIFYING CONSISTENT RADIATION FOR THE PROCESSING OF ELECTRON BEAM CURED COMPOSITES[1]

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ABSTRACT

Electron beam (E-beam) curing of composites is being developed for numerous aerospace applications. For implementation on these programs, a method for specifying equivalent radiation conditions at a number of facilities using a variety of different electron accelerators must be implemented. This paper describes the critical machine parameters that must be controlled, and a method for specifying them. A software program that predicts radiation rates based on these parameters is described. Radiation experiments were performed at four different companies to calibrate the software. Future efforts for determining the effect of radiation parameters on material performance are briefly described.

KEY WORDS: E-beam curing, composites, radiation processing

1. INTRODUCTION

Numerous government and industrial partners have investigated E-beam curing of composites for several years as a method of curing composites that offers a wide array of benefits over standard autoclave curing. These benefits range from manufacturing cost savings to composite performance improvements and have previously been summarized (1).

While great advances have been made in the technology during the past few years, several barriers remain before the technology can be used widely in an industrial setting. From a materials perspective, there is a low level of adhesion between the carbon fibers and epoxy matrix (2-3), and the matrix resins may need improved toughness and strength for use in many aerospace applications. The adhesion issue is being investigated by a Cooperative Research and Development Agreement (CRADA) team led by Oak Ridge National Laboratory (ORNL) and other government and industry partners. Various resin suppliers and the CRADA team are investigating the improvement of matrix resins. Considerable advances in prepreg layup and vacuum assisted resin transfer molding processing of these

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materials have been made by the Composites Affordability Initiative (CAI). These processing advances continue both within the CAI program and external to it.

In addition to these improvements, a robust, repeatable method for delivering radiation to components of varied size and geometry must be implemented. This paper presents some initial work performed by the CRADA team to develop consistent irradiation procedures for parts of varied size and geometry regardless of the curing facilities or type of electron accelerators used.

2. CURING PROFILES

For E-beam curing of composites to become widely used, the process of curing parts must become at least as well controlled as the state-of-the-art in autoclave curing. Autoclave curing requires strict adherence to a specific cure profile such as the one shown in Figure 1. Both suppliers and end-users have performed extensive testing to ensure that if components are cured within the specified limits, the resultant composites will exhibit consistent material performance. The cure profile is made to be sufficiently robust that all areas on even a complex tool will receive the required time/temperature/pressure profile. Different areas on a part (A, B, C in Figure 2) may receive slightly varied profiles as seen in Figure 1 due to tool thermal mass and spatial conductivity/heat transfer variations within the autoclave.

To achieve this uniformity, the ramped segments of cure cycles are often quite slow, allowing the temperature differential across the part to be minimized. The exact science of this is of such importance that an Air Force funded program at Boeing is developing sophisticated modeling methods to accurately predict both the cure profile experienced by any region of the part and its effect on final part geometry (4).

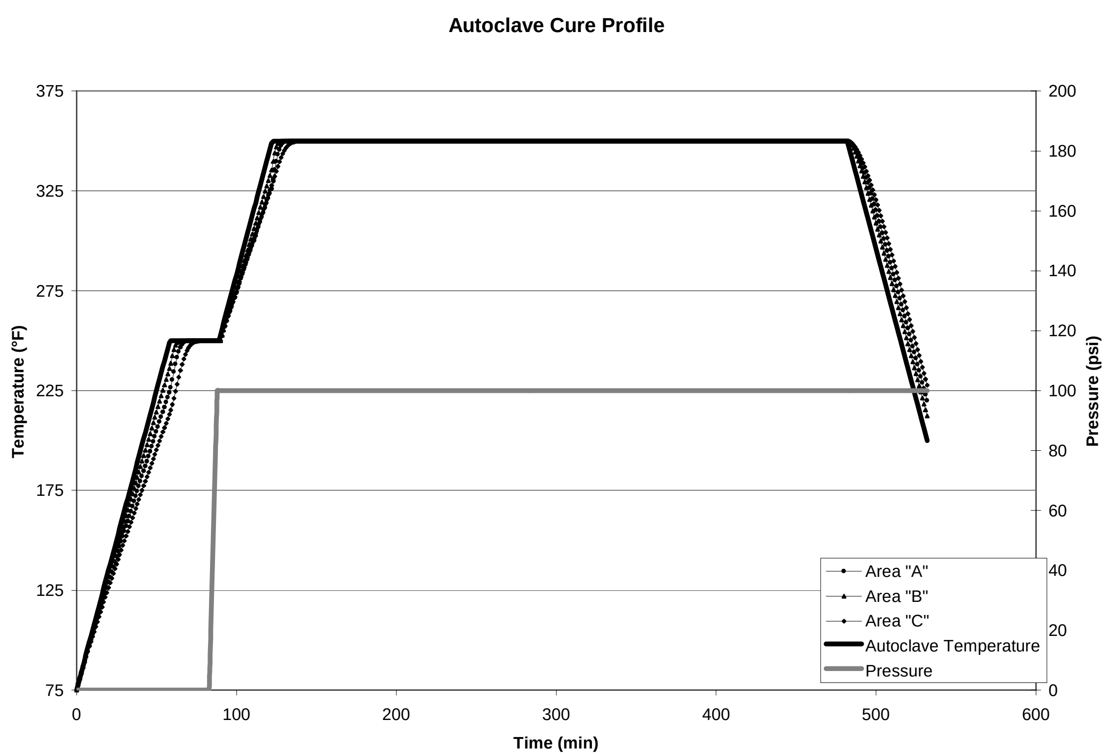

Figure 1 – Representative Cure Profile for an Autoclave Cured Component

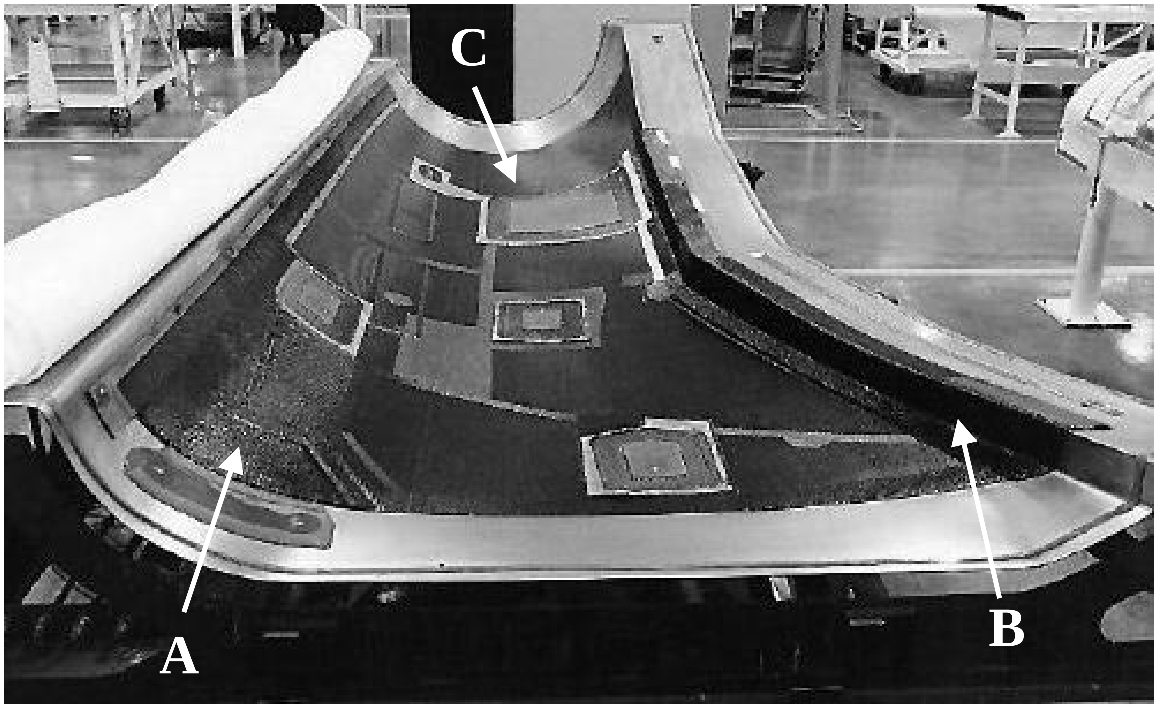

Figure 2 – Autoclave Cured Component Showing Areas with Different Thermal Profiles During Cure

It is the temperature profile experienced by each infinitesimal point in the part that must be within the tolerances of the specification for success, not just the profile of the autoclave. For instance, if we were to cure 1/8th-inch thick components of 1000ft^2 and 75ft^2 in an autoclave, they would receive a cure profile within close tolerances, and thus their composite materials would exhibit known thermal and mechanical performance.

In E-beam curing, unlike in autoclave curing, we can control both the amount of energy being applied to the part, and the location to which it is applied. This additional flexibility in controlling the delivery of energy has some major manufacturing advantages, but at the same time makes it necessary to ensure that the energy profile applied to all areas of the part lies within acceptable parameters. Following the previous example of a 1000ft^2 and 75ft^2 component, the required total dose of radiation can be delivered to the 75ft^2 part in 1/13th the time required for the 1000ft^2 component if average beam current is unchanged. Representative cure profiles for these two cases are shown in Figure 3, and it is easy to imagine that the composite properties achieved from the two profiles could be dramatically different.

While the desired profiles for most E-beam cured materials are not yet fully understood, an important step in understanding what these profiles should be is to be able to accurately deliver a specific profile to all areas of a given part despite its geometry, its size, the type and power of accelerator, or the type of transport system used to move the part relative to the beam.

3. EQUIPMENT PARAMETERS

There are a number of different types of accelerators in common use today. There are pulsed machines, direct current (DC) machines, and continuous wave (CW) machines. Each produces radiation in a different manner. Pulsed machines deliver bunches, or pulses, of electrons, usually at tens or hundreds of Hz (pulses per second). The pulses are typically microseconds in duration. Electron currents are quite high during the pulses, and null between pulses. The pulse period, or elapsed time between the beginning of consecutive pulses, is many times longer than the pulse duration. DC machines produce a continuous stream of electrons. CW machines produce a semi-continuous stream of electrons that is delivered in buckets (micro-pulses) at the microwave power frequency. The resultant beam of electrons has the appearance of a continuous wave. The characteristics of the different beams are depicted in Figure 4.

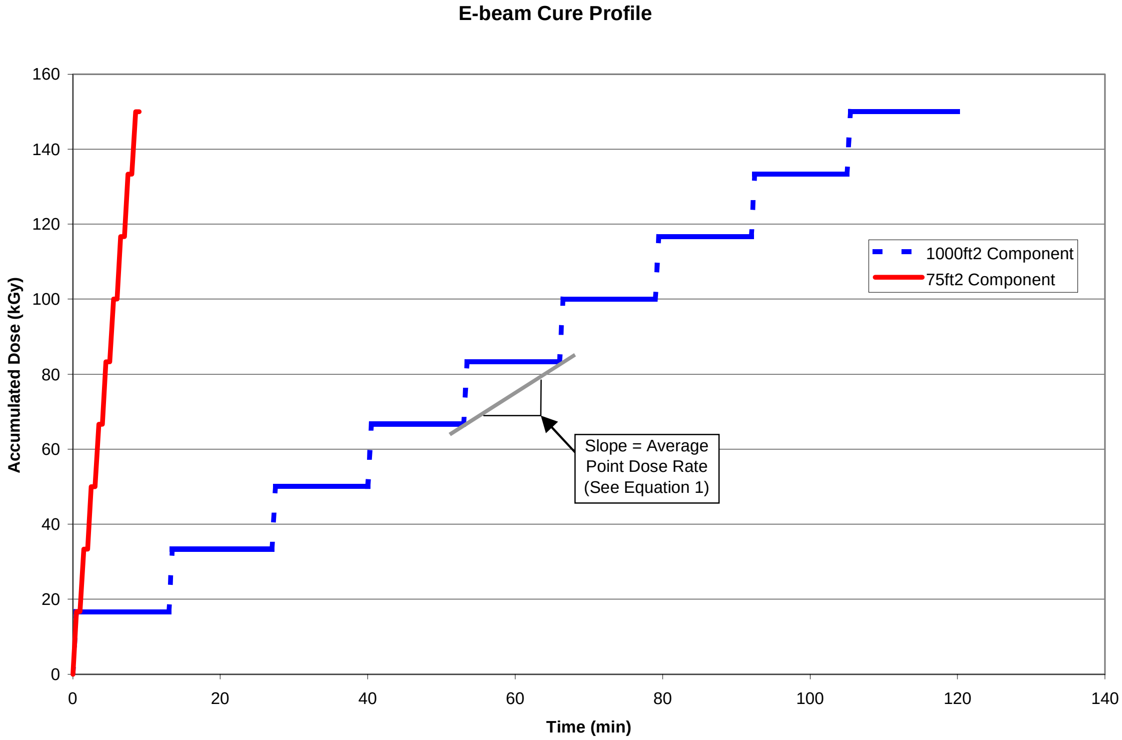

Figure 3 – Representative E-Beam Cure Profile Showing the Potential Effect of Component Surface Area on Cure Time with Constant Beam Current

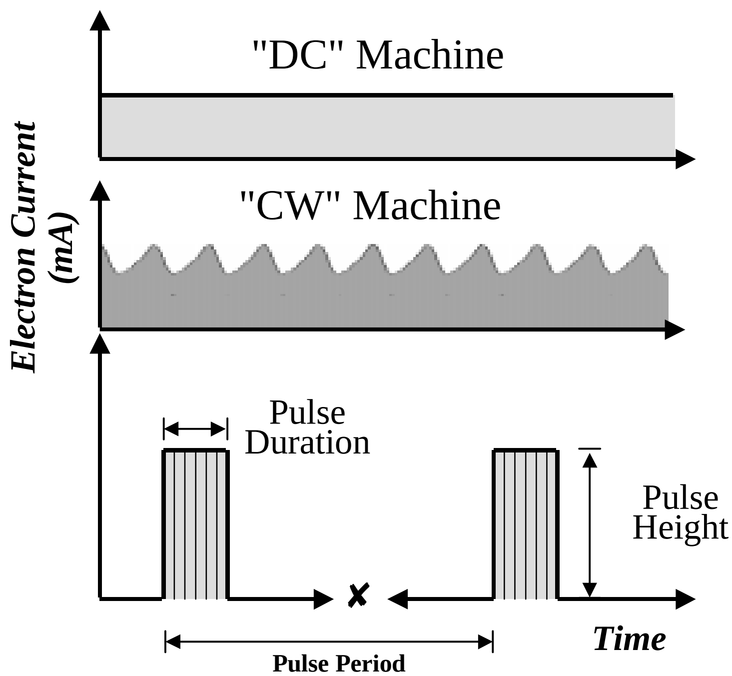

Figure 4 – Time Structure of the Beam in Different Types of Accelerators

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In addition to electron beams, x-rays produced by colliding energetic electrons into a dense metal target, gamma rays from radioisotope sources, and ultraviolet (UV) rays can be used to cure cationic and free-radical resin systems. This variation in types of radiation devices makes it essential to understand which of the cure parameters must be controlled, and to what level. Most electron beams are steered through an arc by a scanning magnet while the part is being translated on a conveyor system. The achievable width and frequency of scan, rate of translation, beam current, beam energy, and distance between the scan horn and part are different for each facility. Figure 5 shows the scan horn, conveyor system and a component being cured using the accelerator at Acsion Industries.

Figure 5 – Accelerator System Curing a Component at Acsion Industries

It is important at this point to define a few terms for the sake of clarity. The terms Average Point Dose Rate and Beam Current will be used here and are defined as follows:

Average Point Dose Rate (kGy/s) – The dose rate experienced by a small point on a part during cure. This is calculated from the electron flux at the point as follows.

$$Point\ Dose\ Rate\ (kGy/\sec) = A\Phi \frac{dE}{dx} \quad \textbf{[1]}$$

Where: A is a constant; $\Phi\left(\frac{electrons}{cm^2 \sec}\right)$ is the electron flux averaged over an integer number of pulse periods; and $\frac{dE}{dx}\left(\frac{MeV}{cm^2 g}\right)$ is the stopping power of the material being irradiated and is experimentally determined.

Beam Current - Amount of current (mA) being emitted by the accelerator. Choice of time scale affects the value of beam current for both pulsed and CW machines. For a pulsed machine, the beam current varies between a maximum equal to the pulse current, and a minimum of zero. For a CW machine, the beam current varies in a sinusoid as shown in Figure 4.

Average Beam Current - beam current averaged over an integer number of pulse periods

Table 1 shows the parameters that can commonly be adjusted during E-beam curing, and whether they affect the average point dose rate.

Table 1 – E-beam Curing Adjustable Parameters

Cure Parameter (All Others Fixed)	Machine Type	Affects Beam Current?	Affects Point Dose Rate?	Comment
Scan Width	All	No	Yes	
Scan Frequency	All	No	No	
Part Height (distance from scan horn)	All	No	Yes	Effect on dose rate depends on beam delivery system
Translation speed	All	No	Yes	Frequently used to adjust Point Dose Rate
Translation Distance	All	No	Yes	Affects average part temperature during cure
Pulse Frequency	Pulsed	Yes	Yes	
Charge/Pulse	Pulsed	Yes	Yes	
Beam Energy	All	No	No	Affects depth of penetration

5. DOSE RATE CALCULATION

Since many of these parameters can affect the dose rate in a complex manner, a software program has been created to predict the dose profile delivered to any spot on the surface of a part for a given accelerator and cure condition. The code is theoretically capable of predicting the effect of each of these parameters on the delivered energy profile at any given position on a part. This predictive capability is critical, because it enables reliable analysis of the effects of these cure parameters on final composite material properties. Without this predictive capability, variations in dose rate between machines may confound the results, making interpretation of experiments difficult.

The areal dose profile in the beam (Figure 6) can be described using a simple function such as the one shown in Equation 2. The beam may vary for different machines, so alternate equations can be used as required. The code predicts delivered radiation for pulsed machines by predicting the position of each pulse based on the machine parameters and calculating the dose delivered to the point of interest on the part.

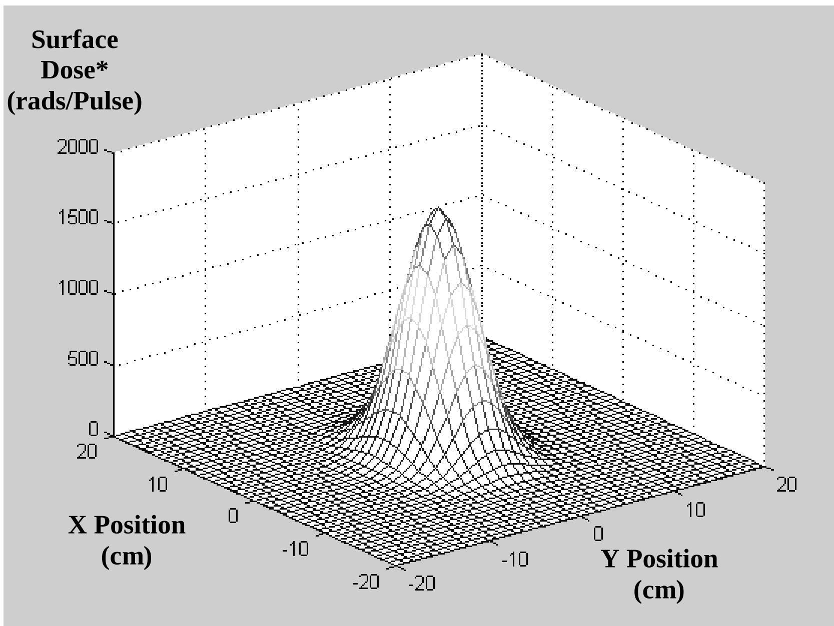

Figure 6 – Areal Dose Map of a Generic Gaussian Beam

* Surface absorbed dose as measured using film dosimeters.

$$Dose = A * e^{-\left[\frac{(x-\mu_x)^2}{2\sigma_x^2} + \frac{(y-\mu_y)^2}{2\sigma_y^2}\right]} \qquad \textbf{[2]}$$

By knowing the distance from the point of interest to the center of the beam spot, the dose delivered to that point is predicted by simply evaluating the function. The delivered dose as a function of time is obtained by predicting the position of each pulse and storing the delivered energy as a function of elapsed time. The scanning and translation between pulses are predicted using the simple calculations shown in Equation 3:

$$\alpha = \alpha + \frac{d\alpha}{dt}dt$$
$$x = x + \frac{dx}{dt}dt \qquad \textbf{[3]}$$

where α = scan angle; x = translation position; and dt = time step (pulse frequency^{-1}, sec).

CW machines can be viewed as pulsed machines with a very high pulse frequency, and thus can be predicted as such. For DC machines, however, there is no easily defined time step. The delivered dose for each time step is predicted by evaluating the dose at the current time step, D_t, averaging it with the value for the previous time step, $D_{(t-1)}$, and multiplying the result by the length of the time step, dt as shown in Equation 4.

$$D_{(t-1)\to t} = \frac{D_t + D_{t-1}}{2} dt$$
$$Error = \frac{D_t - D_{t-1}}{dt} \quad \textbf{[4]}$$

To control the accuracy of the result, an error is calculated and compared to a user defined level. If this level is exceeded, the program notifies the user and terminates. The accuracy of a result can be ensured by plotting the error as a function of the time step until convergence is seen. This validation method is commonly used in finite element analysis.

6. EXPERIMENTAL CALIBRATION OF PREDICTIVE CODE

Before any predictive method can be used, two things must occur. First, the effect being predicted must be understood and quantified so that the method can be calibrated. Second, the method itself must be verified. To accomplish this, the CRADA team performed a number of simple experiments to quantify each machine's capabilities. A dosimetry grid, consisting of sheets of dosimetry film stretched on a frame at three vertical locations, as shown in Figure 7, was used to map the spatial dose distribution on different accelerators. The measured dose was then used to validate code predictions.

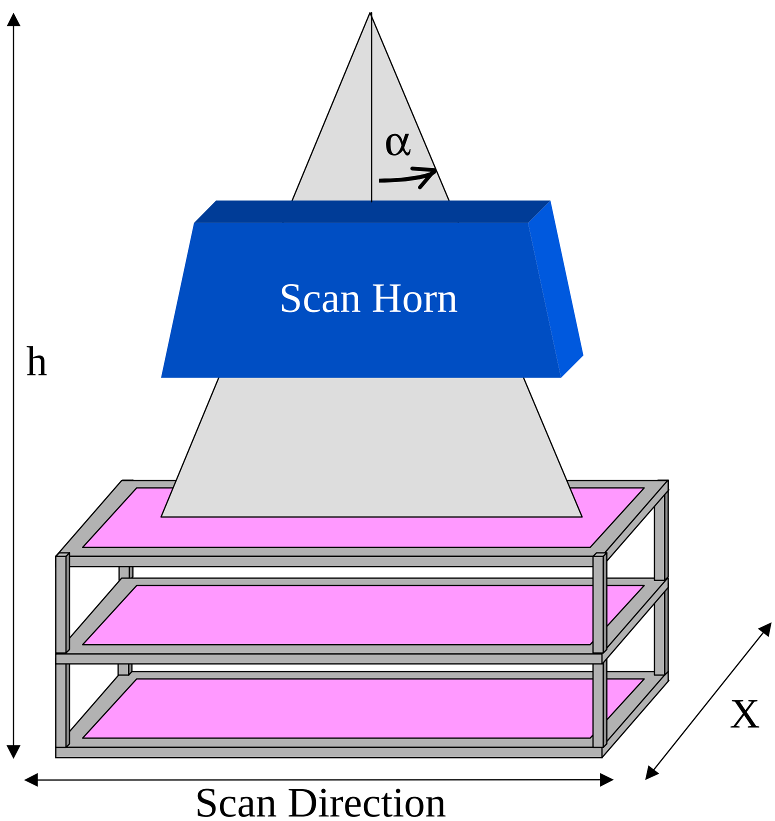

Figure 7 – Simplistic Depiction of E-beam Curing Experiments

The first measurements were "static" measurements, made with the dosimetry grid in a fixed position, i.e. with translation speed set to zero. Functionally, the objective of these experiments is to determine the constants used in Equation 2. Unfortunately, most accelerators cannot be operated without scanning their beams, and thus, the full shape of a beam cannot be measured directly. It was, therefore, necessary to assume that the beam is circular in shape as shown in Figure 6. Given this assumption, the constants can be determined by measuring the dose distribution on a sheet of dosimetry film during the "static" experiment. The resultant profile from such an experiment on the STERIS Isomedix accelerator is shown in Figure 8. This experiment provides not only the width and shape of the scan, but will also show any abnormalities in the scan path. The results of this "static" experiment were used to determine the constants in Equation 2.

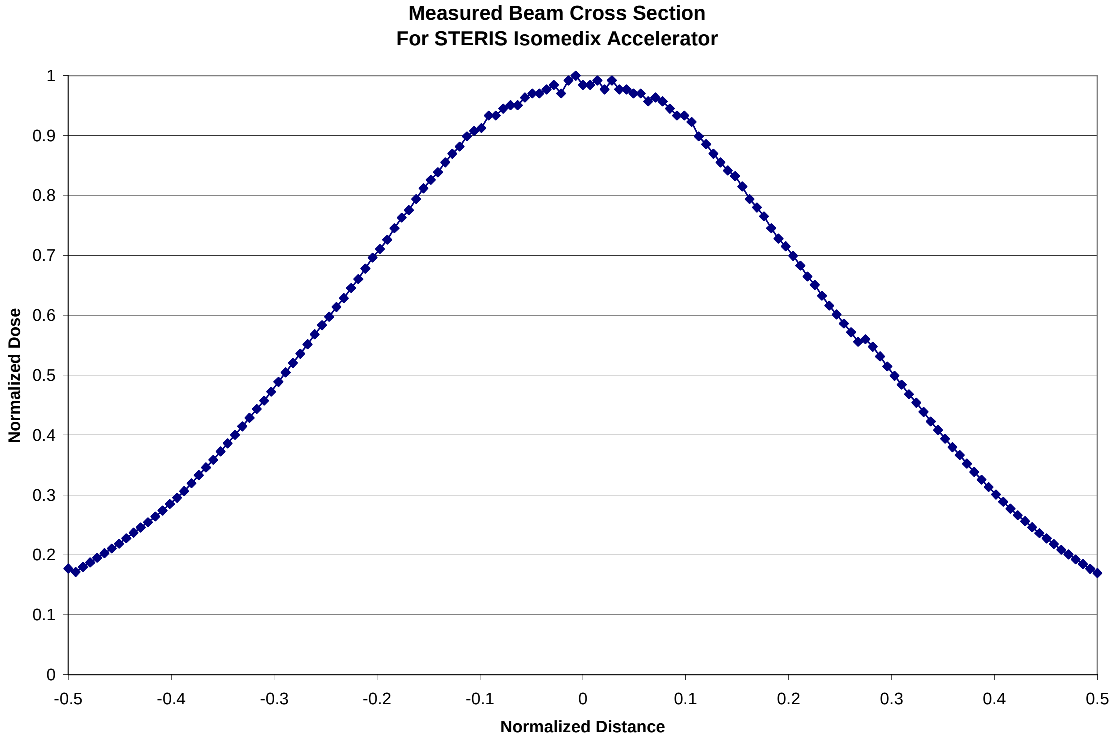

Figure 8 – Cross-Sectional Dose Profile From a "Static" Experiment

Once the width and basic shape of the equation were determined with the "static" experiment, translating experiments were performed to provide data on the effect of the other parameters. The experimental plan for these translating experiments included variations in scan width, beam current, translation speed, and part height. The results from these translating experiments were used to adjust the constants in Equation 2, and to identify the accuracy of this predictive technique.

7. RESULTS AND DISCUSSION

Four CRADA members have electron accelerators and each contributed to this effort. The basic specifications of these machines are shown in Table 2. The code was found to be in agreement with actual measured response within standard dosimetry error for the Acsion accelerator at a specified height. Full validation of all accelerators will be available for the conference presentation.

Table 2 – CRADA Electron Accelerators and Code Accuracy

Electron Accelerator Parameters
Interface CRADA

Company / **Designation**	**Acsion** *I10/1*	**Isomedix** *Rhodotron TT200*	**EB Services** *CBS*	**EB Services** *CBN*	**Boeing** *Linac*	**Boeing** *Dynamitron*
Accelerator Parameters						
Voltage, MeV (max.)	9.7	5	4.5	10	10	2
Voltage, MeV (min.)			2.5		5	0.2
Power, kW (max.)	0.75	80	150	50	1	
Current, ma (max.)	0.077	16.0	33.3	5.0	0.15	10
Rf frequency, GHz	3	107.5 MHz	DC	1.3	3	
Pulse repetition rate, Hz	0 - 300			250 - 280	0 - 15	
Scan rate, Hz	2		100	6	0.33	
Beam Parameters						
Beam delivery direction	vertical	vertical	vertical	vertical	vert. or horiz.	
Width per scan, meters (max.)	0.1 - 0.6	0.3 - 1	0.1 - 1.73	0.79 - 1.41	0 - 0.71	
Height from window, cm	60		127	127		
Conveyor Belt Parameters						
Height from window, cm	60	25	127	127		
Maximum speed, cm/s	0.05 - 13	2.54 - 25.4	2.54 - 25.4	0.04 - 2	0 - 12	
Reversible or Non-reversible	Reversible		Non-reversible	Non-reversibe	Reversible	
Return time			3 - 6 min	17 min		

8. CONCLUSIONS

This effort represents an important step toward the definition of a curing specification for E-beam cured composites and adhesives. With the ability to predict the dose delivered to a part on any of the CRADA accelerators, testing has begun to determine which radiation parameters must be controlled to guarantee the robust curing of high performance composite parts in a production environment. The parameters being investigated include the dose, dose rate, rest time between radiation passes, initial cure temperature, and resin temperature achieved during cure.

9. REFERENCES

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