Lamination Scheme of Curing Degree at Multiple Levels of Temperature With Location-Scale Regression

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ABSTRACT Solar power has become a key green source of energy. An important factor that affects the reliability and lifetime of solar modules is the quality of encapsulation through the lamination process, which melts the ethylene vinyl acetate (EVA) to make the solar cells combine with the front glass side and the rear side units. The degree of crosslinking or curing degree for EVA sheets, when the EVA sheet gets heated, can affect the efficiency of the performance and power conversion of solar modules. For this reason, motivated by a lamination data, we construct here a statistical model for describing the relationship between the curing degree and the lamination factors (temperature and time). Then, based on some specification limits on the curing degree, the optimal lamination time for solar modules can be determined at different temperatures. Moreover, the optimal sample size allocation in a test for measuring EVA sheets can also be determined. A simulation study is finally carried out to show the closeness of simulation results to the asymptotic results.

INDEX TERMS Confidence bands, curing degree, degree of crosslinking, sample size allocation, specification limits.

I. INTRODUCTION

The energy crisis is of at most importance due to dwindling reserves of fossil fuels. The ever increasing use of these non-renewable sources results in an increase in the emission of greenhouse gases, thus jeopardizing our environment considerably. For this reason, considerable attention is now given to new sustainable and renewable energy solutions. Of these, solar energy has become the fastest-growing green source of energy.

The solar cells are made of N-type and P-type semiconductor materials which convert sunlight directly into electricity. A pictorial representation of this generating process of electricity, known as photovoltaic effect, is shown in Figure 1. For the reason that solar cells can easily get damaged, corroded and fail, they need to be encapsulated as a solar module in order to meet the load requirements. The most common structure of a solar module is that the solar cells encapsulated by a polymer encapsulant, EVA (ethylene vinyl acetate), adhere to a tempered glass on the top and a back sheet on the bottom, as shown in Figure 2.

Because solar modules get exposed to various climatic conditions, the quality of encapsulation through lamination can affect the reliability and lifetime of solar modules. When the EVA sheet gets heated in a vacuum chamber, the degree of crosslinking, which is mainly controlled by lamination temperature and lamination time (see Lange et al. [9]), impacts the efficiency of the performance and power conversion of...
solar module. High lamination temperature or longer lamination time can increase the degree of crosslinking, resulting in EVA sheets becoming crisp, and then failing to remain usual quality to protect the solar cell. On the other hand, when the lamination temperature is too low or lamination time is not long enough, the EVA will have a lower degree of crosslinking, resulting in a reduction of both aging resistance and creep resistance. More discussions on the crosslinking behavior and properties of EVA in solar panels can be found in Wang et al. [15], [16], Oreski et al. [12], Choi and Chung [5], and the references contained therein.

In industrial practice, several methods have been suggested for determining the degree of crosslinking on EVA. Differential scanning calorimetry (DSC) is an effective tool for this purpose as it possesses many powerful techniques to study the thermal properties of EVA. With DSC, the heat flow of both the cured EVA sample and uncured EVA reference are monitored as a function of time and temperature, as displayed in Figure 3. Thus, the curing degree given by

\[
\text{curing degree} = \frac{\Delta H_1 - \Delta H_2}{\Delta H_1} \times 100%,
\]

where \(\Delta H_1\) and \(\Delta H_2\) are the reaction enthalpy of the uncured EVA reference and cured sample, respectively, can be used as an indicator to determine the degree of crosslinking on EVA. Much literature exists on methods for determining the degree of crosslinking by DSC and its applications (e.g., Bruckman et al. [2]; Jaunich et al. [7]; Ogier et al. [11]); but, previous work has not dealt with optimal lamination time at different temperatures for the curing degree on EVA in a natural way, and this provides the main motivation for the present article.

Optimal designs have many diverse applications in experimental designs (Cheng [3]; Jones and Majumdar [8]), in accelerated life and degradation tests (Tsai et al. [14]; Hu et al. [6]), and in clinical trials (Cheung et al. [4]; Atkinson and Biswas [1]; Zang and Lee [17]). Though optimal designs have been utilized in many applied problems, its application has not been explored in solar lamination tests. Based on a location-scale family of distributions and prefixed specification limits of the gel content, Tsai [13] discussed the optimal lamination time at a specific lamination temperature by conventional chemical extraction method, and then determined the corresponding specification limits of the curing degree at this obtained optimal lamination time by DSC. However, the degree of crosslinking or curing degree is controlled by both lamination time and temperature. Hence, improvements on the determination of the curing degree for EVA sheets at different lamination times and temperatures by DSC method are presented here. Specifically, under pre-fixed specification limits of the curing degree, the optimal lamination time at different lamination temperatures are determined here.

The rest of this paper is organized as follows. Section II presents a real example that motivates the present work, and it is also used later for illustrating the methodology developed here. Section III describes the lamination model of curing degree and derives the optimal lamination time and the optimal sample size allocation for measuring curing degree. Section IV presents analytical results with regard to fitting of the model for the motivating example. Finally, Section V makes some remarks on this study and also points out some possible topics for future research.

II. MOTIVATING EXAMPLE AND OPTIMIZATION PROBLEMS

We motivate this study by a lamination data of a solar company in Taiwan. The lamination process was done as follows. A solar module with nine EVA sheets (105cm wide \times 165cm length) were heated at three lamination times \(t_1 = 5.5\), \(t_2 = 6.5\), and \(t_3 = 8.5\) minutes with three different temperatures at \(S_1 = 140^\circ\text{C}\), \(S_2 = 150^\circ\text{C}\), and \(S_3 = 160^\circ\text{C}\), respectively, in the chamber. These sheets were then cut into small specimens at the end of process. Figure 4 presents the curing degree of 6 randomly selected specimens at each lamination temperature and time.

Our aim now is to determine the optimal lamination time at different temperatures in such a way that the value of curing degree will fall within the specification limits with high probability. Furthermore, the determination of optimal allocation of sample sizes for the nine lamination tests, subject to a fixed sample size, is also discussed in the context of optimal lamination tests.
III. LAMINATION MODEL OF CURING DEGREE

Figure 4 suggests that there may be a linear relationship between the curing degree of specimens and the lamination time at three different temperatures. The values of coefficient of determinant ($R^2 = 0.86, 0.84, 0.93$) of the three linear regression models reveal readily that a location-scale-distribution-based model would be suitable for this lamination data. The Brown-Forsythe test, with $p$-value of 0.42, suggests that the null hypothesis of variances of curing degree being equal across different lamination temperatures and times cannot be rejected. Hence, the curing degree $X_k(t)$ of the specimens at lamination temperature $S_k$ and time $t$ can be described by a location-scale family of distributions with cumulative distribution function (cdf)

$$F_{X_k(t)}(x; \mu_k(t), \sigma) = \Phi \left( \frac{x - \mu_k(t)}{\sigma} \right)$$

and probability density function (pdf)

$$f_{X_k(t)}(x; \mu_k(t), \sigma) = \frac{1}{\sigma} \phi \left( \frac{x - \mu_k(t)}{\sigma} \right),$$

where $\mu_k(t) = \alpha_{0k} + \alpha_{1k}t$ is the location parameter, $k = 1, 2, 3$, $\sigma > 0$ is a scale parameter, and $\phi(\cdot)$ and $\Phi(\cdot)$ are the standard pdf and cdf (with $\mu = 0$ and $\sigma = 1$), respectively.

A. DETERMINATION OF OPTIMAL LAMINATION TIME

From (1) and (3), we assume that the curing degree $X_k(t)$, at a specified lamination temperature $s$ and lamination time $t$, follows a location-scale family of distributions with location parameter $\mu_s(t) = \alpha_{0s} + \alpha_{1s}t$ and scale parameter $\sigma$, where

$$\alpha_{it} = r_{i0} + \frac{r_{i1}}{273.15 + s}, \quad i = 0, 1.$$  

Let LSL and USL denote the prefixed lower and upper specification limits for the curing degree, respectively. Then, the optimal lamination time $t^*_0(s)$ can be determined as

$$t^*_0(s) = \arg \max_t P(LSL \leq X_t(s) \leq USL)$$

where $P$ denotes the probability. The optimal lamination time $t^*_0(s)$ can be determined by maximizing the function

$$g(t) = \Phi \left( \frac{USL - \mu_s(t)}{\sigma} \right) - \Phi \left( \frac{LSL - \mu_s(t)}{\sigma} \right);$$

that is, the optimal lamination time $t^*_0(s)$ can be determined from the equation (obtained by differentiating $g(t)$ and equating it to 0)

$$\phi \left( \frac{USL - \mu_s(t^*_0(s))}{\sigma} \right) - \phi \left( \frac{LSL - \mu_s(t^*_0(s))}{\sigma} \right) = 0.$$  

The following result then presents the optimal lamination time $t^*_0(s)$ for two special cases.

Result 1: Let LSL and USL be the prefixed lower and upper specification limits for the curing degree, respectively. Suppose the curing degree $X_k(t)$ follows a location-scale family of distributions with location parameter $\mu_s(t) = \alpha_{0s} + \alpha_{1s}t$ and scale parameter $\sigma$. Then, the optimal lamination time is

(i)  

$$t^*_0(s) = \frac{LSL + USL - 2\alpha_{0s}}{2\alpha_{1s}}$$  

if the standard pdf of the curing degree $X_k(t)$ is symmetric about 0, such as normal and logistic distributions;

(ii)  

$$t^*_0(s) = \frac{\sigma \ln \left( \frac{USL - LSL}{\sigma} \right) - \alpha_{0s} - LSL}{\alpha_{1s}}$$  

if the standard pdf is that of the smallest extreme value distribution.
B. ESTIMATION AND INFERENCE

Some conditions for conducting this lamination experiment are as follows:

(1) Under three different lamination temperatures, \( S_1 < S_2 < S_3 \), nine EVA sheets were laminated at three different lamination times, \( t_1 < t_2 < t_3 \). Then, they were cut into small specimens at the end of the process;

(2) For the sheet at the lamination temperature \( S_k \) and lamination time \( t_j \), \( n_{kj} \) specimens are picked randomly for measuring the curing degree, for \( j, k = 1, 2, 3 \). Consequently, the available total number of measurements for the curing degree is \( N = \sum_{k=1}^{3} \sum_{j=1}^{n_{kj}} n_{kj} \);

(3) Let \( X_k^{(i)}(t_j) \) denote the measured curing degree of the \( h \)th specimen at lamination temperature \( S_k \) and time \( t_j \). They are assumed to be independent random variables drawn from a location-scale family of distributions with location parameter \( \mu_k(t_j) = \alpha_k + \sigma_k t_j \) and scale parameter \( \sigma \), for \( j, k = 1, 2, 3, \ h = 1, \cdots, n_{kj} \), with the relationships of \( \alpha_k, i = 0, 1 \), to stress \( S_k \) being as given in (3).

Then, based on the observations \( \{X_k^{(i)}(t_j)\}_{i=1}^{n_{kj}} \), where \( j, k = 1, 2, 3 \), the log-likelihood function is given by

\[
\ell(\theta) = -N \ln(\sigma) + \sum_{k=1}^{3} \sum_{j=1}^{n_{kj}} \ln(\phi(z_{kjh})) ,
\]

where

\[
z_{kjh} = \frac{X_k^{(i)}(t_j) - \mu_k(t_j)}{\sigma}
\]

and \( \theta = (\alpha_0, \alpha_1, \sigma_0, \sigma_1) \). Because this log-likelihood cannot be maximized analytically, a numerical optimization method needs to be employed for obtaining the maximum likelihood estimates (MLEs), \( \hat{\theta} = (\hat{\alpha}_0, \hat{\alpha}_1, \hat{\sigma}_0, \hat{\sigma}_1) \), of \( \theta \).

Then, upon plugging \( \hat{\theta} \) into \( t_k^*(s) \) in (6), and applying the best asymptotic normality (BAN) property of the MLE, the asymptotic variance of \( t_k^*(s) \) can be obtained as

\[
AVar(\tilde{t}_k^*(s)) = (\nabla t_k^*(s))^T I(\hat{\theta})^{-1} (\nabla t_k^*(s)),
\]

where \( (\nabla t_k^*(s)) \) is the gradient of \( t_k^*(s) \), \( (\nabla t_k^*(s))^T \) is the transpose of \( (\nabla t_k^*(s)) \), and \( I(\hat{\theta}) \) is the Fisher information matrix. Relevant details are presented in the Appendix. Then, a two-sided approximate 100(1 - \( \alpha \))% confidence interval (CI) (Meeker and Escobar [10]) for \( t_k^*(s) \) can be given as

\[
\tilde{t}_k^*(s) \pm z_{1-\alpha/2} \sqrt{AVar(\tilde{t}_k^*(s))},
\]

where \( z_{1-\alpha/2} \) is the 100(1 - \( \alpha \)/2)th percentile of the standard normal distribution.

At a specific lamination temperature \( s \), the sample size allocation \( (n_{11}, n_{12}, \cdots, n_{32}, n_{33}) \) affects the variance of the estimate of the optimal lamination time given in (11). So, it would be of interest to obtain the optimal sample size allocation in such an experiment. One may consider determining the optimal solution by searching all possible allocations with an integer restriction on these decision variables. But, there will still be about 3.38 \times 10^9 possible solutions when \( N = 54 \). Note that for many practical situations in test plans, an optimal plan often has test units allocated at the boundaries of the design. This suggests that we may find an optimal solution through a complete search at the corners of the design; that is, under the constraints \( n_{12} = n_{21} = n_{22} = n_{23} = n_{32} = 0 \) and \( n_{11} + n_{13} + n_{31} + n_{33} = N \), the optimal decision-making model can be expressed as

\[
(n_{11}^*, n_{12}^*, n_{31}^*, n_{33}^*) = \arg\min_{n_{11}, n_{13}, n_{31}, n_{33}} AVar(\tilde{t}_k^*(s))(n_{11}, n_{13}, n_{31}, n_{33})
\]

for \( 1 \leq n_{kj} \leq N, j, k = 1, 3 \). Note that the determinant of the Fisher information matrix in (11) will be zero whenever one of the four-corner configurations is zero. In other words, with the positive integer restriction on the allocation variables, the optimal solution \( (n_{11}^*, n_{12}^*, n_{31}^*, n_{33}^*) \) can be easily evaluated by the complete enumeration algorithm as described below:

Step 1. Set \( n_{11} = 1 \);
Step 2. Set \( n_{13} = 1 \);
Step 3. Set \( n_{31} = 1 \);
Step 4. We have \( n_{33} = N - n_{11} - n_{13} - n_{31} \);
Step 5. Calculate \( AVar(\tilde{t}_k^*(s))(n) \) by \( n = (n_{11}, n_{13}, n_{31}, n_{33}) \);
Step 6. Set \( n_{31} = n_{31} + 1 \), and repeat Steps 4 and 5 until \( N - n_{11} - n_{13} - 1 \);
Step 7. Set \( n_{13} = n_{13} + 1 \), and repeat Steps 3 and 6 until \( N - n_{11} - 2 \);
Step 8. Set \( n_{11} = n_{11} + 1 \), and repeat Steps 2 and 7 until \( N - 3 \);
Step 9. The optimal solution \( n^* = (n_{11}^*, n_{12}^*, n_{31}^*, n_{33}^*) \) can then be obtained as \( AVar(\tilde{t}_k^*(s))(n^*) = \min_n AVar(\tilde{t}_k^*(s))(n) \).

IV. MOTIVATING EXAMPLE REVISITED

We now revisit the motivating data described earlier in Section II and use it to illustrate the determination of optimal lamination design for the EVA sheets by DSC method proposed in the last section.

A. MODEL VALIDATION AND PARAMETER ESTIMATION

First, the \( p \)-value of 0.42 in Brown-Forsythe test in Section III reveals that the homogeneity of variances for the nine lamination tests is quite reasonable. Next, the \( p \)-values (being all greater than 0.05) of the Anderson-Darling test show that normal, logistc, or smallest extreme value distributions can be appropriate to describe these measured curing degree of specimens. Finally, we use the Akaike information criterion (AIC).

\[
AIC = 2s - 2\ell_{\text{model}}
\]

where \( s \) is the number of estimated parameters in the model and \( \ell_{\text{model}} \) is the maximized log-likelihood value under that model, to select the most suitable model. The MLEs of parameters and the AIC values of the three models are
B. OPTIMAL LAMINATION DESIGN
Suppose the pre-fixed specifications for the curing degree are LSL = 65 and USL = 80. When the lamination process is set at temperature \( s = 147^\circ\text{C} \), the estimated optimal lamination time and the corresponding estimated asymptotic variance are obtained as \( 12.7908 \) and \( 0.0815 \), respectively, upon substituting \( \hat{\theta} \) into (8) and (11). These readily yield a 95% normal-approximate confidence interval (CI) as \( 12.7908 \pm 1.96\sqrt{0.0815} = [12.2312, 13.3505] \). In a similar manner, we can construct 95% confidence intervals at each temperature setting. Figure 7 presents, for the solar lamination test data, a 95% pointwise normal-approximate confidence band for the optimal lamination time. These results show that when higher lamination temperature is chosen for the lamination process, shorter estimated optimal lamination time will be required. Moreover, confidence interval bands become narrower when the lamination temperature gets higher, which would result in more precise estimates of optimal lamination times.

Clearly, for the lamination temperature \( s = 147^\circ\text{C} \), \( \text{AVar}(\hat{t}^*_n(s)) \) in (13) is a function of \( \theta \) when the values of \( n_{11}, n_{12}, \ldots, n_{32}, n_{33} \) are all given. Thus, we must have some reasonable idea about \( \theta \) for the determination of optimal sample size allocation; for this purpose, therefore, we use the MLE of \( \theta \) as an apriori information for the true model parameter \( \theta \). Setting \( n_{12} = n_{21} = n_{22} = n_{23} = n_{32} = 0 \), the optimal allocation of sample size is then determined by a complete discrete search as

\[
(n_{11}^*, n_{13}^*, n_{31}^*, n_{33}^*) = (13, 22, 7, 12).
\]

Now, for assessing the effectiveness of this allocation, the relative efficiency (RE) index, which measures the performance of the optimal allocation of specimens with respect to that of equal sample size allocation given by

\[
\text{RE} = \frac{\text{AVar}(\hat{t}^*_n(s)|\text{optimal allocation})}{\text{AVar}(\hat{t}^*_n(s)|\text{equal allocation})}.
\]

is computed. The RE index of 0.0461/0.0815 = 0.5652983 shows clearly that the optimal allocation results in a much smaller asymptotic variance than the equal sample size allocation, and thus would result in more efficient inference.

C. SIMULATION STUDY
The results in Section IV-B were all based on the asymptotic normality of the MLEs of model parameters. It would, therefore, be good to conduct a Monte Carlo simulation study to verify that the empirical results are indeed close to the asymptotic results. Consider a lamination process in which we have three lamination temperatures and times, as described in Section II, and the true parameters of the curing degree of specimens as the MLEs of parameters of the smallest extreme value model reported in Table 1. Let LSL = 65 and USL = 80 be the specifications for the curing degree. We then simulated 1000 times from the above described process. Table 2 presents the true and simulated values of the parameters, and optimal lamination time at lamination temperature \( s = 147^\circ\text{C} \), with the corresponding asymptotic and simulated standard deviations reported within parentheses. From these results, we can conclude that the proposed method works very well.

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**TABLE 1.** The estimated parameters and AIC values for the three fitted models.

| Distributions         | \( \hat{\theta} = (\hat{\theta}_0, \hat{\theta}_{10}, \hat{\theta}_{11}, \hat{\sigma}) \) | \( \ell_{\text{MLE}, n} \) | AIC  |
|----------------------|-------------------------------|-------------------|-----|
| Normal               | (356.03, -1369963.5, 52.39 - 20542.92, 1.78) | -107.64           | 225.28 |
| Logistic             | (345.11, -132382.82, 54.04, -21236.09, 1.01) | -107.77           | 225.54 |
| Smallest extreme value | (410.99, -160283.95, 45.47, -17556.72, 1.48) | -105.91           | 221.83 |

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**FIGURE 6.** Estimated mean lines for curing degree under the smallest extreme value model.

**FIGURE 7.** Estimated optimal lamination time and pointwise 95% confidence bands.
model in this context. We are currently working on these problems and hope to report the findings in a future paper.

### APPENDIX A

#### PROOF OF RESULT 1

Proof: (i) As the density $\phi(\cdot)$ is symmetric about 0, i.e., $\phi(z) = \phi(-z)$, we see from (6) that

$$\frac{USL - \mu_s(t^*_n(s))}{\sigma} = -\frac{LSL - \mu_s(t^*_n(s))}{\sigma},$$

where $\mu_s(t^*_n(s)) = \alpha_0 + \alpha_1t^*_n(s)$. So, the required result follows.

(ii) As $X_{j,t}$ follows the smallest extreme value distribution with standard pdf $\phi(z) = \exp(-z - \exp(z))$, we obtain from (6) that

$$\exp\left(\frac{USL - \mu_s(t^*_n(s))}{\sigma}\right) - \exp\left(\frac{LSL - \mu_s(t^*_n(s))}{\sigma}\right) = \frac{USL - LSL}{\sigma},$$

where $\mu_s(t^*_n(s)) = \alpha_0 + \alpha_1t^*_n(s)$. The required result then follows after some algebraic calculations.

### APPENDIX B

#### DETAILED EXPRESSIONS OF $\left(\nabla t^*_{i,n}(s)\right)^T$ AND $I(\theta)$ IN (10)

Proof: First, we have

$$\left(\nabla t^*_{i,n}(s)\right)^T = \left(\frac{\partial t^*_{i,n}(s)}{\partial r_{00}}, \frac{\partial t^*_{i,n}(s)}{\partial r_{01}}, \frac{\partial t^*_{i,n}(s)}{\partial r_{10}}, \frac{\partial t^*_{i,n}(s)}{\partial r_{11}}, \frac{\partial t^*_{i,n}(s)}{\partial \sigma}\right).$$

Next, from (10), we see that

$$\begin{align*}
\frac{\partial z_{kjh}}{\partial r_{00}} &= -\frac{1}{\sigma}, \\
\frac{\partial z_{kjh}}{\partial r_{01}} &= -\frac{1}{\sigma} \left(273.15 + S_k\right), \\
\frac{\partial z_{kjh}}{\partial r_{10}} &= -\frac{f_j}{\sigma}, \\
\frac{\partial z_{kjh}}{\partial r_{11}} &= -\frac{f_j}{\sigma} \left(273.15 + S_k\right), \\
\frac{\partial z_{kjh}}{\partial \sigma} &= -\frac{z_{kjh}}{\sigma}.
\end{align*}$$

### TABLE 2. Results from Monte Carlo simulation study.

| Parameter | True value | Simulated value |
|-----------|------------|-----------------|
| $r_{00}$  | 410.99 (58.1252) | 413.08 (62.3080) |
| $r_{01}$  | -160283.95 (24581.91) | -161174.23 (26363.36) |
| $r_{10}$  | 45.47 (8.3679) | 45.11 (9.0451) |
| $r_{11}$  | -17556.72 (35388.89) | -17409.24 (38288.38) |
| $\sigma$  | 1.48 (0.1570) | 1.40 (0.1621) |
| $t^*_n(s)$ | 12.7968 (0.2865) | 12.8519 (0.3118) |

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Then, each element of the Fisher information matrix $\mathbf{I}(\theta)$ is negative of the expected value of the second partial derivative of $\ell(\theta)$ in (9) with respect to the elements of $\theta$, and they are presented in the bottom of the previous page.

\[
\begin{align*}
\text{ACKNOWLEDGMENT} \\
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\text{Sections II and IV.}\nonumber
\end{align*}
\]

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