

# Batch Chemical Process Quality Control Applied to Curing of Composite Materials

Matthew M. Thomas, Babu Joseph, and John L. Kardos

Dept. of Chemical Engineering, Campus Box 1198, Washington University, St. Louis, MO 63130

*Model predictive control is used extensively to control continuous-process systems. Application of a shrinking horizon model predictive control strategy is used to predict and control the end product quality of composite laminates produced by batch autoclave curing. The main contribution is the demonstration, using a laboratory-scale autoclave, of the feasibility and advantages of a control strategy that adjusts the batch recipe on-line to correct for unmeasured disturbances entering the process. Readily available, on-line secondary measurements are used in conjunction with the process model to predict (and hence control) quality-related end product properties. On-line monitoring is also used to monitor the process even after the possibility of on-line correction has passed. The results show that this approach significantly reduces end product quality variance.*

## Introduction

Two approaches to batch-control strategy categorization are taken. The first considers two strategy categories: state-estimation and knowledge-based (Nomikos and MacGregor, 1994). This approach is wider ranging than the second, which tends to be model based. This second, model-based approach considers three strategy categories (Soroush and Valluri, 1994). Of these, the first involves off-line calculation of optimal batch-recipe settings and open-loop operation (Eliçabe and Meira, 1988). This approach assumes that an accurate process model is available. It cannot cope with on-line disturbances affecting either the manipulated inputs or the quality output variables. The second involves off-line calculation of optimal profiles maintained by closed-loop regulatory control (Soroush and Kravaris, 1992). This approach also assumes the availability of an accurate process model, and can cope with on-line disturbances that alter the manipulated inputs, but cannot cope with those disturbances that affect the quality output variables. The third involves end-point optimization using state estimation and feedback (Palanki et al., 1993; Cuthrell and Biegler, 1989; Terwiesch and Agarwal, 1994).

This article presents strategies that combine the desirable aspects from all three categories. Off-line models are used to

optimize the batch recipe prior to the start of the batch. During batch processing, secondary measurements are used with on-line process models to make adjustments to the recipe, thereby permitting compensation for any process disturbances not detected at the start. These strategies perform the end-point optimization common to methods in category three, but use nonlinear optimization of linear and nonlinear statistical model components to do so; they do not use the traditional state-estimation and -feedback techniques common to category-three methods. Concepts from the latter categories, with enhancements, allow for improved batch-chemical process control.

The autoclave curing of composite laminate materials is a prototype of batch-chemical processes. In such processes: (1) significant quality variations can occur even if the batch recipe is kept constant from batch to batch; (2) the end-product quality is difficult to measure on-line; and (3) the process models are often very complex and are often not suitable for accurately predicting end-product quality during a batch run. For autoclave curing: (1) significant quality variations are caused by factors such as variable raw material ("prepreg") quality; (2) one of the end-product quality measurements (void content) cannot be made until hours after batch run completion; and (3) the available autoclave curing process models are far too complex for use in on-line model based control, and cannot account for on-line, unmeasured dis-

Correspondence concerning this article should be addressed to B. Joseph.  
Present address of M. M. Thomas: Boeing Phantom Works, P.O. Box 516 MC  
5106-7126, St. Louis, MO 63166.

turbances. Accordingly, the autoclave curing process is as challenging as any for testing a control strategy within a batch run. With composite materials costing \$130/lb (\$286/kg) of fabricated part, and with fabrication contributing to 70–75% of that cost (Thayer, 1990) in the absence of on-line quantitative process control, development of such a control strategy is warranted.

In prior work, Joseph and Wang Hanratty (1993) proposed a shrinking-horizon model predictive control (SHMPC) strategy based on the following concepts:

- Use of a data-driven on-line process model to predict end-product quality
- Use of a nonlinear optimizer to make continuous adjustments to the batch recipe, so that predicted end-product quality is within specifications
- Use of on-line corrections to the process model by incorporating available on-line secondary measurements as model input variables, to account for model errors and effects of unmeasured disturbances.

This work lends formalism to the SHMPC concept, and extends it to include postcontrol monitoring (PCM). This extension allows for monitoring of the batch process after all batch process control actions have been taken. The SHMPC and PCM strategies are then applied to the laboratory-scale autoclave curing of fiberglass-epoxy polymeric composite laminate materials. This work also considers variable settings of end-product quality parameter targets. Results indicate that these strategies significantly improve the end-product quality of the laminates.

One could pose the end-point control problem as an optimal control problem with differential equation constraints describing a dynamic process model. A completely different strategy is used in this work, based on the following restrictions:

- (1) Control actions are taken at discrete points in time
- (2) On-line measurements are also available at discrete points in time.

With these restrictions, the problem is not one of computing an optimal control move trajectory, but rather of computing the finite set of discrete control moves to be made to minimize the end-point quality deviations. This type of problem lends itself to

- (1) Use of *static* on-line processing models relating discrete control actions to end-product quality

- (2) The incorporation of discrete on-line secondary measurements as *additional inputs to the on-line model*, to detect and hence infer the effects of all other unmeasured disturbances entering the process.

This approach, while simplifying the computational problem in a very significant way, retains the advantages of on-line adaptation to unmeasured process input disturbances. Both are extremely important from the *practical* standpoint. Even when some of the measurements are monitored continuously, control action is based on derived, discrete features of such continuous variables (ramp rate at a critical point in a batch run, or the peak value attained by a variable, for example). The relevancy of such extracted features is established from prior understanding of the process and analysis of past operation data.

In this article, the SHMPC concept and PCM are formalized, and the on-line models to be used with these strategies

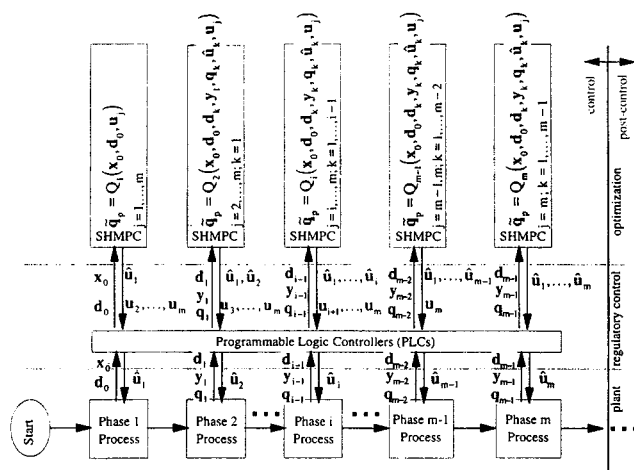
are described. The autoclave curing process and the results of SHMPC and PCM applications to experimental autoclave curing are also discussed.

## Shrinking-Horizon Model Predictive Control

This work lends formality to the SHMPC concept (Joseph and Wang Hanratty, 1993; Thomas et al., 1994; Joseph et al., 1995). In continuous model predictive control (MPC) (Garcia and Prett, 1986; Garcia et al., 1989; Ricker, 1990, 1991; Garcia et al., 1991; Morari and Lee, 1991; Richalet, 1993; Morari, 1993; Lee et al., 1994), the control horizon is said to recede. In contrast to continuous MPC, the SHMPC batch variant features a control horizon that shrinks with the approach of the well-defined batch run end. SHMPC is geared toward batch-chemical processes—processes with well-defined start and (especially) end points; processes for which the window of control opportunity increasingly closes as the batch run progresses. The SHMPC strategy accepts linear and nonlinear process models.

### SHMPC — Interphase perspective

Figure 1 illustrates SHMPC within the optimization/regulatory control hierarchy context (Jang et al., 1987). In this context, regulatory control ensures that process-manipulated inputs are at their prescribed settings with minimal error. In an autoclave, for example, if the autoclave temperature and pressure are set to 225°F (107°C) and 100 psi (690 kPa), respectively, it is the task of the programmable logic controllers (PLCs) at the regulatory control level to keep the autoclave temperature at 225°F (107°C) and the pressure at 100 psi (690 kPa), using sensors installed in the process. It is at the optimization level that the prescribed settings for the process manipulated inputs are made. In the autoclave example just cited, the decision to set autoclave temperature and pressure (respectively) to 225°F and 100 psi is made at the optimization level. These manipulated input settings are optimized, in that they are to lead to optimal end-product quality variable values (e.g., minimal void content and optimal thickness for an autoclave-cured composite laminate). The SHMPC strat-



**Figure 1. Multilevel multiphase decomposition of control tasks (shrinking-horizon model predictive control phases).**

egy, based on information received from the sensors at the plant level through the PLCs at the regulatory control level, performs this manipulated input optimization.

In the control hierarchy context, Figure 1 illustrates an increase in control sophistication from bottom to top. In terms of time, Figure 1 shows the progression toward batch run completion from left to right. Distinct SHMPC phases characterize this progression. Receipt of a new primary measurement, secondary measurement, or disturbance at the optimization level triggers the beginning of a new SHMPC phase (to last until receipt of another such measurement or disturbance). These measurements are information-rich; they are not, for example, individual temperature readings, but rather temperature ramp rates—the latter are much more useful in gauging, say, the thermal conductivity of an autoclave-cured part. This breaking down of a process into phases is much better suited for batch processes than for their continuous counterparts, given the well-defined end state for the former.

The MPC nomenclature (of  $m$  steps in the control horizon and  $p$  steps in the prediction horizon) is adapted for codifying SHMPC. In Figure 1,  $m$  denotes the number of SHMPC phases during which manipulated inputs can be altered (i.e., during which control action can be taken). In Figure 3 below,  $p$  denotes the number of SHMPC and PCM phases in the batch process. For SHMPC,  $p$  remains the prediction horizon size, as end-product values of quality variables  $q_p$  are predicted from the first phase of the batch run; likewise,  $m$  remains the control horizon size, and  $m \leq p$ . As the batch run progresses, both horizons shrink. The prediction horizon shrinks from  $p$  (initially) to  $p-i$  (after phase  $i$ ); similarly, the control horizon shrinks from  $m$  to  $m-i$ . As will be illustrated, a batch run is not necessarily completed upon shrinkage of the control horizon to zero—the inequality  $m < p$  describes those instances when the batch run continues after the control horizon shrinks to zero.

Table 1 describes the SHMPC-related variables in Figure 1. The premise of SHMPC is to use all information available, in each SHMPC phase of the batch process, to predict end-product quality variable values, and to fix manipulated inputs to maximize the likelihood of realizing those end values. During each phase  $i$ , end-product quality variable values  $\tilde{q}_p$  are predicted by linear or nonlinear model component  $Q_i$ .

An earlier approach to SHMPC (Joseph and Wang Hanratty, 1993) used one model  $Q$  for a batch process, and aver-

age previous batch measurement values represented measurements in  $Q$  that had yet to occur. The approach in this work is to use a different model component  $Q_i$  for each phase  $i$  in the batch process. The drawback to this approach is that it requires not one but  $p$  models to describe the process, where  $p$  is the total number of SHMPC and PCM phases. The advantage is that each model is especially suited to each phase (with its unique combination of available measurements and settings to be optimized.)

### SHMPC—Intraphase perspective

Figure 2 illustrates the inner workings of SHMPC during a given phase  $i$ . Newly available values of primary and secondary measurements and disturbances are combined with previously obtained values, as well as with manipulated input settings previously fixed by the SHMPC scheme. These values are then fed to the optimization component of the SHMPC scheme. That component addresses the problem

$$\text{Min } \|\tilde{q}_p - \tilde{q}_p^{\text{target}}\|_2$$

$$u_i, u_{i+1}, \dots, u_{m-1}, u_m$$

subject to

$$g(u_i, u_{i+1}, \dots, u_{m-1}, u_m) \leq 0$$

$$h(u_i, u_{i+1}, \dots, u_{m-1}, u_m) = 0$$

by interacting with the model component of SHMPC (which also represents a constraint). This model component comprises the linear or nonlinear model:

$$\tilde{q}_p = Q_i(x_0, d_0, d_1, \dots, d_{i-1}, y_1, \dots, y_{i-1}, q_1, \dots, q_{i-1}, \hat{u}_1, \dots, \hat{u}_{i-1}, u_i, \dots, u_m). \quad (1)$$

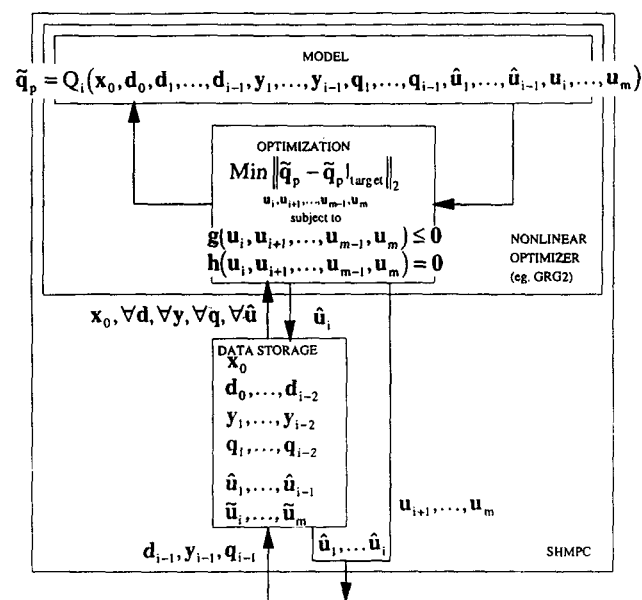


Figure 2. Shrinking-horizon model predictive control (SHMPC).

Table 1. Variable Definitions for SHMPC

Variable	Description
$x_0$	Initial-process state
$d_i$	Vector of measured disturbances first available to the SHMPC strategy after SHMPC phase $i$
$y_i$	Vector of secondary measurements first available after phase $i$
$q_i$	Vector of primary measurements (quality-variable values) first available after phase $i$
$\tilde{q}_p$	Vector of predicted end product quality-variable values
$q_p$	Vector of actual end product quality-variable values
$\hat{u}_i$	Vector of manipulated inputs first fixed in SHMPC phase $i$
$u_i$	Vector of manipulated inputs not yet fixed, going into SHMPC phase $i$
$Q_i(\cdot)$	SHMPC linear/nonlinear model component in phase $i$

A nonlinear optimizer combines the model and the optimization components of SHMPC within an algorithm (the generalized reduced gradient algorithm GRG2, for example) for solving nonlinear programming (NLP) problems (Lasdon et al., 1978). During an SHMPC phase, free manipulated input settings are chosen to minimize deviation of  $\bar{q}_p$  from its target value; previously fixed manipulated input settings—having been implemented and no longer adjustable—serve only as model inputs.

Often, only one secondary measurement or one measured disturbance is available in a given SHMPC phase. This lack of available information leads to a sparseness or “parsimony” in the SHMPC models, in that the effects of fewer variables upon  $\hat{q}_p$  need to be gauged, thereby making for a lighter model processing load. When real-time primary measurements cannot be made, then only after the batch run ends do measurements  $\mathbf{q}$  become available; vectors  $\mathbf{q}_1$  through  $\mathbf{q}_{p-1}$  cannot have been obtained.

## Postcontrol Monitoring

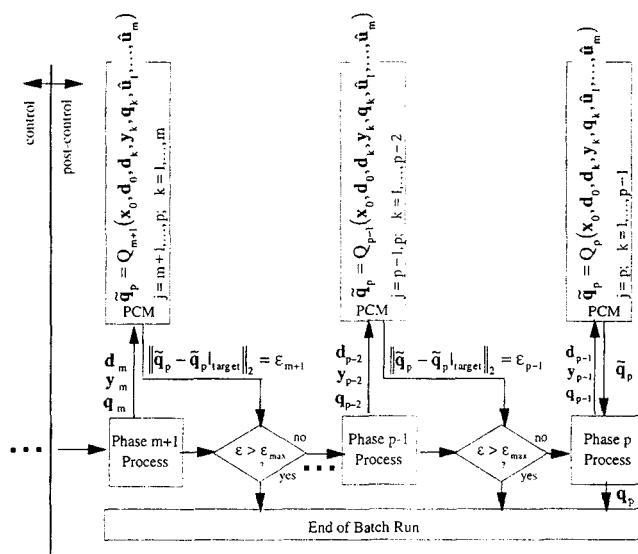
The SHMPC strategy accommodates those batch processes in which control action can be taken through the last phase in the batch run. It is the extreme case when control-horizon size is identical to prediction-horizon size ( $m = p$ ). While a batch-chemical process for which  $m = p$  is possible, those for which  $m < p$  are more common. When  $m$  is far smaller than  $p$ , control actions taken during phases 1 through  $m$  should be evaluated during phases  $m + 1$  through  $p$ . Such evaluating is the postcontrol monitoring of the SHMPC-controlled batch-chemical process.

### *Motivation for postcontrol monitoring*

If  $m \ll p$ , all control action may be completed with much of the batch run remaining. A long time exists between the end of the control horizon and the end of the run. The role of PCM is then to monitor the run during that time, allowing it to proceed if  $\tilde{q}_p$  is on target, and terminating it if  $\tilde{q}_p$  has gone off target by an unacceptably large magnitude. The SHMPC strategy is designed to minimize the deviation of  $\tilde{q}_p$  from  $q_p$ . Nevertheless, if PCM determines that  $\tilde{q}_p$  is continuously diverging from  $q_p$ , despite the best efforts of SHMPC, the batch run should be terminated. Run termination salvages time that would have been wasted completing a flawed run. Batch operating time is costly and should not be spent on an irreparably flawed run. While manipulated inputs are fixed after the control horizon shrinks,  $d$ ,  $y$ , and  $q$  process values can be gauged until the end of a batch run. With access to these values, and with PCM-strategy phase models to determine  $\tilde{q}_p$  from these values, the means as well as the motivation for postcontrol SHMPC evaluation is present.

### ***PCM—Interphase perspective***

Figure 3 provides a logical representation of PCM. Note the similarities in structure between PCM in Figure 3 and SHMPC in Figure 1: monitoring/control sophistication increases from bottom to top; batch run progression is from left to right. During PCM, values  $\hat{u}_1$  through  $\hat{u}_m$  serve as parameter inputs to PCM model components  $Q_{m+1}$  through



**Figure 3. Multilevel decomposition of control tasks (postcontrol monitoring phases).**

$Q_p$ . The decision diamonds in Figure 3 assess  $\epsilon_{m+i}$ , where Eq. 2 defines  $\epsilon_{m+i}$ :

$$\|\tilde{\mathbf{q}}_p|_{m+i} - \tilde{\mathbf{q}}_p|_{\text{target}}\|_2 = \epsilon_{m+i}. \quad (2)$$

If  $\epsilon_{m+i}$  is less than a critical value  $\epsilon_{\text{MAX}}$ , the process is considered on target and is allowed to continue; if greater, the process is terminated. Figure 3 illustrates but one process-termination criterion; other criteria might hinge upon  $\epsilon$  values showing marked increase over the past  $n$  phases.

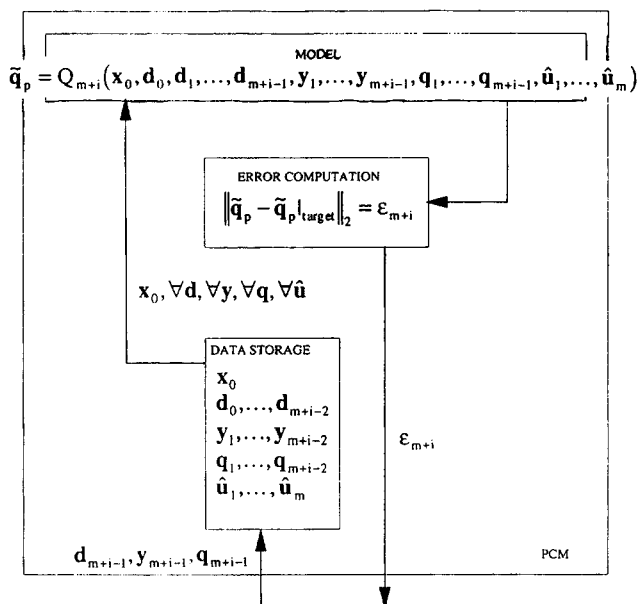
### *PCM—Intraphase perspective*

Figure 4 illustrates the inner workings of PCM, just as Figure 2 did for SHMPC. In lieu of an optimization module in Figure 4, there is one for error computation; with no optimization problem to solve, that error computation module only implements Eq. 2. Data transfer among PCM modules is identical to that among SHMPC modules: the latest measurements are combined with their previously stored counterparts; all are then sent to serve as current PCM phase-model input; model output (an error computation by PCM) is then sent to the PLCs.

## On-Line Models for Use with SHMPC and PCM Strategies

Models  $Q_1$  through  $Q_m$  used in SHMPC and models  $Q_{m+1}$  through  $Q_p$  in PCM need to be efficient, because they must provide real-time output in an on-line application. Model output must be available before the process moves into the next SHMPC or PCM phase. The requirement for real-time models in an on-line application restricts the types of models that can be used with the SHMPC and PCM strategies.

For batch processes such as the autoclave curing process considered herein, detailed physical models tend not to supply the required output rapidly enough. In time, greatly increased computer speed will allow for consideration of de-



**Figure 4. Postcontrol monitoring equivalent of SHMPC (PCM).**

tailed physical models for use. For now, however, those models cannot be used with the model-based control strategies discussed herein. But even when computers become fast enough to accommodate detailed physical models, model-based quality-control strategies still have to make use of process data. All models, even detailed physical models, are plagued by inherent inaccuracies. Process data must be used to correct for those inaccuracies. Even if detailed physical models were made less detailed through use of simplifying assumptions, the increase in model speed is compromised by a loss in model accuracy. Also, physical models cannot account for unmeasured disturbances such as the inevitable variance in raw material quality. These problems lead to a preference for data-driven (statistical) models based on actual process measurements. (A similar approach is followed in the industrial implementation of MPC: even though detailed physical models are available, control is based on simpler linear models identified from plant tests.)

Fundamental process knowledge should be used as much as possible to select model input variables. Use of this knowledge serves two purposes: it eliminates needless inputs, thus avoiding the model that is too large and thus too slow; it lends physical meaning to statistical model inputs, thus avoiding the model regression abuse that arises from spurious inputs (Box, 1966). The traditional approach to statistical models has been to develop linear input-output models from empirical plant tests. Batch chemical processes, however, tend to be nonlinear; the models needed for their quality control must be able to account for nonlinearity. Statistical model overfitting must also be avoided (Larimore and Mehra, 1985).

Linear, quadratic, and feedforward artificial neural network (ANN) models have been examined in the context of statistical models in SHMPC and PCM. ANN models were used in this work because of the inherent nonlinearities present in the batch process. A comparison of the ANN models with linear and quadratic regression models is presented elsewhere (Thomas and Joseph, 1997). Although ANN mod-

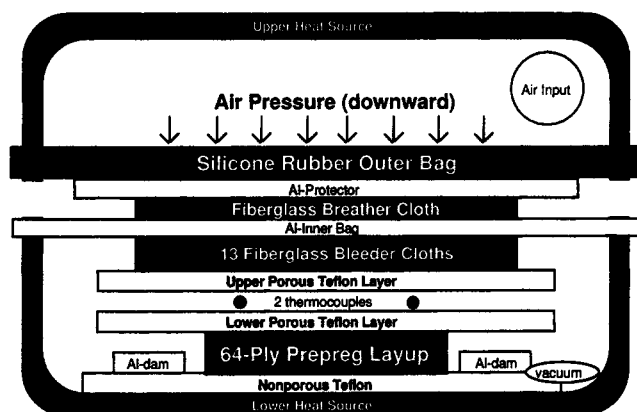
els have been selected for use with the SHMPC and PCM strategies in this application, any model type described previously can be used in conjunction with these strategies, provided that it is sufficiently fast and accurate for the given application.

## Overview of Autoclave Curing Process

The autoclave curing process produces fiber-resin composite laminate materials, the strength and utility of which can exceed those of the fiber and resin individually. As a prelude to the curing process, woven fibrous clothes preimpregnated with resin (prepreg) are cut to size and collated upon a contoured metal support surface called a "tool." Other auxiliary materials such as sheet Teflon and fiberglass cloth are also used in this collation with the tool; the resulting unit is labeled a "layup." For this work, Figure 5 illustrates the specific arrangement of prepreg and auxiliary materials for the 112 experimental autoclave curing runs performed as part of this work. Davé (1990) provides a tutorial on the autoclave curing process.

Upon collation completion, the layup is inserted into the autoclave, and is "bagged" such that a vacuum can be pulled on the layup while autoclave pressure is applied to it. For this work, the curing process entailed exposing the layup to (1) a nominal 5°F/min temperature ramp; (2) a 45–90 min hold at 165°F–255°F (74–124°C), during which the vacuum is broken, and 40–100 psi (276–690 kPa) pressure is applied; (3) a second nominal 5°F/min temperature ramp; and (4) a 3-h hold at 350°F (177°C). Figure 6 in the following section qualitatively illustrates the relevant temperature-time profile. This cure profile primarily influences resin viscosity—a function of temperature and degree of resin polymerization or "cure."

During the cure cycle, the first temperature ramp lessens viscosity and begins resin flow within the collated prepreg. Degree of cure (and thus viscosity) increase during the first temperature hold: It is during this hold that pressure is applied to thwart void nucleation and growth. Viscosity is lessened again during the second temperature ramp, but increases dramatically upon resin gelation. Upon process completion, the cured laminate is thinner and stronger than the collated prepreg. Void content, which undermines laminate strength, must be minimized, and thickness must be controlled during this process.



**Figure 5. Assembled miniclave.**

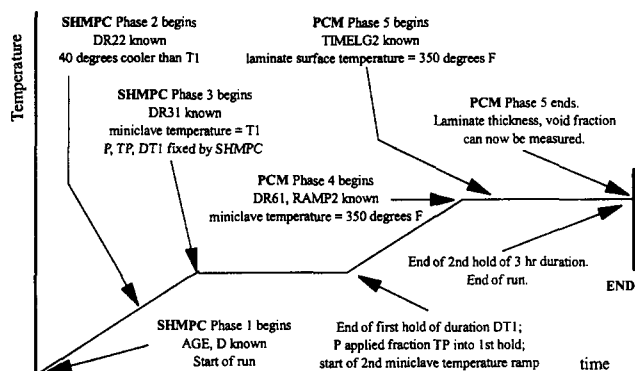


Figure 6. SHMPC/PCM phases in autoclave curing.

For this work, the prepreg comprised 120-style E-glass fiber and Hercules-brand 8551-7A thermosetting (chemical curing) epoxy resin. Seven series of sixteen cures each were performed on 64-ply, 3-in.-by-3-in. collations of this prepreg (as arranged in Figure 5). Most of these series were governed by a four-level, five-factor orthogonal array (Box et al., 1978; Taguchi, 1987). For these series, the five factors were curing commencement, *D*, applied pressure, *P*, first temperature hold duration, *DT1*, first hold temperature, *T1*, and the fraction into *DT1* that *P* is applied (*TP*)—four measured inputs; one measurable disturbance.

Two thermocouples were used to record laminate surface temperature every 10 s during the 4.5–5.5-h batch run. These data were used to calculate localized surface-temperature ramp rates, thermal delays, and other secondary measurements of greater significance than any individual temperature reading taken once every 10 s. Thomas (1995, Appendix G) has tabulated these secondary measurement values, manipulated inputs, and measured disturbances and other variables from each of the 112 batch runs. These data were used for SHMPC and PCM model development, and are available from the authors. Results from these runs have been analyzed in a composite materials context elsewhere (Thomas et al., 1997).

## Experimental Testing of the SHMPC Strategy

Table 2 summarizes the seven successful SHMPC input candidates. Eighteen SHMPC input candidates (12 secondary measurements, 4 manipulated inputs, 2 disturbances) had their impacts on thickness and void content assessed using feature selection routines such as *forward selection*, *backward elimination*, and *stepwise regression* (Draper and Smith, 1981). These linear selection methods were used in the absence of well-established nonlinear counterparts, to provide at least a linear approximation to phenomena that may (thickness) or may not (void content) vary linearly with some of the candidates. Despite the constraints of their linear basis, these methods constrained the nonlinear ANN model component sizes in the SHMPC and PCM strategies, an important step in ensuring that the ANN model components' parameters are few enough to be fitted, using data from the 112 experimental autoclave curing runs. Of the 18 SHMPC input candidates, seven had confidence levels sufficiently greater than the other 11 to warrant inclusion (Thomas and Joseph, 1997).

Table 2. Seven Input Candidates to the SHMPC Strategy

SHMPC Candidate	SHMPC Candidate Description	Variable Type
AGE	Age of prepreg (since June 29, 1993) used in curing run	Measured disturbance
<i>D</i>	Delay in beginning of miniclave curing run ("out time" of the prepreg material)	Measured disturbance
DR22	2nd laminate surface temperature ramp rate, right thermocouple <i>Tmini2</i> , measured over autoclave temperature ( <i>Tavg</i> ) range $(T1 + 70)/2^\circ\text{F}$ to $T1 - 40^\circ\text{F}$	Secondary measurement
DR31	3rd laminate surface temperature ramp rate, left thermocouple <i>Tmini1</i> , measured over $Tavg = T1 - 40^\circ\text{F}$ to <i>T1</i>	Secondary measurement
<i>DT1</i>	Duration of first temperature hold in miniclave cure cycle	Manipulated input
<i>P</i>	Pressure applied within miniclave to prepreg layup	Manipulated input
<i>TP</i>	Fraction of time into <i>DT1</i> (0 through 1) that <i>P</i> is applied	Manipulated input

SI conversion:  $^\circ\text{C} = (^\circ\text{F} - 32)/1.8$

These seven inputs—see Table 2—are used in the SHMPC strategy.

The availability of a new secondary measurement, measured disturbance, or primary measurement denotes the start of a new SHMPC or PCM phase. By definition, a primary measurement can be inferred from secondary measurements and measured disturbances, and phase *i* primary measurements along with the other measurements allow for estimation of end-product quality variable values. When such a measurement or disturbance becomes available, the current phase model treats it as an additional input, the better with which to estimate end product quality.

Three SHMPC and two PCM phases can be identified for the autoclave curing process. Phase 1 (an SHMPC phase) begins at the start of the batch run; the two measured disturbances AGE and *D* are known at that time. There are three manipulated inputs: *P*, *TP*, and *DT1*. Model Q1 has these five variables as inputs. At the start of the batch run, model Q1 is exercised to determine the initial optimum settings of manipulated inputs. The availability of secondary measurement DR22 signals the end of phase 1 (whereupon model Q2 is exercised), and the availability of secondary measurement DR31 signals the end of phase 2 (whereupon model Q3 is exercised). For this autoclave curing process, manipulated inputs *P*, *TP*, and *DT1* are fixed and applied during phase 3. These three phases are the SHMPC phases.

After phase 3, only monitoring can be done—SHMPC ends and PCM begins. Phase 4, the first PCM phase, begins upon the availability of secondary measurements DR61 and RAMP2 (see Table 3). Phase 5, the second PCM phase, begins once secondary measurement TIMELG2 (Table 3) is available. Phase 5 ends with the end of the batch run. The following section contains a more extensive discussion of the PCM phases. For both SHMPC and PCM, availability of a primary or secondary measurement (or a measured disturbance) dictates a phase change within the strategy. Such is the case not only for autoclave curing, but also for any batch chemical process to which SHMPC and PCM may be applied. The subsection titled "SHMPC–Interphase Perspective" noted that measurements, in this context, are chosen to be information-rich. As soon as such a measurement be-

comes available during a batch run, it and its predecessors should be used by the SHMPC strategy to determine future control actions, or by the PCM strategy to update estimates. Phase, in an SHMPC or PCM context, denotes occurrences of these information-rich measurements as a batch run progresses.

Figure 6 qualitatively illustrates the temperature-vs.-time aspects of the Hercules 64-ply E-glass/8551-7A collated prepreg miniclave curing cycle. Noted in Figure 6 are the times in that curing cycle when discrete SHMPC/PCM phases begin. Also noted in Figure 6 are the times when the selected SHMPC measured disturbances (AGE, D) and secondary measurements (DR22, DR31) become known, as well as when the manipulated inputs (P, TP, DT1) are fixed by SHMPC. This figure also indicates when the selected PCM secondary measurements (DR61, RAMP2, TIMELG2—see the following section) become known.

### *Nonlinear optimization within the SHMPC strategy*

At the start of each SHMPC/PCM phase as noted in Figure 6, a nonlinear optimization routine GRG2 (Lasdon et al., 1978) is invoked. The function of GRG2, given (1) known secondary measurement and measured disturbance values, (2) model components that use these values and manipulated input values to predict end-product quality variable values, and (3) target quality variable values, is to determine optimal manipulated input settings that minimize the differences between estimated and target quality variable values. The known secondary measurement and disturbance values are provided by the batch-curing process. The target quality variable values are set by technical and economic conditions whose requirements the batch process aims to meet.

SHMPC model Q1 comprises two 5-input ANNs, whose inputs are D, AGE, TP, P, and DT1; the output of one ANN is estimated final laminate average thickness, while that of the other is estimated final laminate void content. Model Q2 comprises two 6-input ANNs, whose inputs include DR22 and the aforementioned five, and whose outputs are as before. Model Q3 comprises two 7-input ANNs, whose inputs include DR31 and the aforementioned six, and whose outputs are as before. Thomas (1995) and Thomas and Joseph (1997) provide details on the development and use of these six and other ANNs.

### *SHMPC targets and operating ranges*

The final average laminate thickness values ranged from 0.194 to 0.222 in. (4.93 to 5.64 mm) for the 112 miniclave curing runs. (Results herein are presented in English units, given that both standard autoclave process equipment and gauges are graduated in these units.) Ten SHMPC test runs were made to test control strategy effectiveness. The objective was to minimize laminate void content and attain a target thickness. For the first five SHMPC test runs, the target-average laminate thickness was chosen to be 0.203 in. (5.16 mm). For the second five SHMPC test runs, that target was chosen to be 0.213 in. (5.41 mm). Achieving minimal void content for a 0.203-in.-thick laminate is clearly easier than for a 0.213-in.-thick laminate (as Figure 9 illustrates).

Within the SHMPC module for the first SHMPC phase, presentation of the nonlinear optimization problem to the GRG2 routine takes the following form:

$$\text{Min } Q1^{\text{VOID-CONTENT}}(\text{AGE, D, P, TP, DT1})$$

$$\text{P, TP, DT1}$$

subject to

$$Q1^{\text{THICKNESS}}(\text{AGE, D, P, TP, DT1}) = 0.203 \text{ in. (5.16 mm)}$$

$$[\text{or } 0.213 \text{ in. (5.41 mm)}]$$

$$P \geq 40 \text{ psi (276 kPa)}, P \leq 100 \text{ psi (689 kPa)}$$

$$DT1 \geq 45 \text{ min}, DT1 \leq 90 \text{ min}$$

$$TP \geq 0, TP \leq 1.$$

Void content, through its Q1 model component (the five-input ANN trained to estimate void content) appears in the objective function to be minimized, while thickness, through its Q1 model component, appears in the constraints. This optimization is performed at the outset of the curing run. When DR22 becomes known, the following optimization is performed:

$$\text{Min } Q2^{\text{VOID-CONTENT}}(\text{AGE, D, DR22, P, TP, DT1})$$

$$\text{P, TP, DT1}$$

subject to

$$Q2^{\text{THICKNESS}}(\text{AGE, D, DR22, P, TP, DT1})$$

$$= 0.203 \text{ in. (5.16 mm)} [\text{or } 0.213 \text{ in. (5.41 mm)}]$$

$$P \geq 40 \text{ psi (276 kPa)}, P \leq 100 \text{ psi (689 kPa)}$$

$$DT1 \geq 45 \text{ min}, DT1 \leq 90 \text{ min}$$

$$TP \geq 0, TP \leq 1.$$

When DR31 becomes known, the third SHMPC phase begins, and this optimization is performed:

$$\text{Min } Q3^{\text{VOID-CONTENT}}(\text{AGE, D, DR22, DR31, P, TP, DT1})$$

$$\text{P, TP, DT1}$$

subject to

$$Q3^{\text{THICKNESS}}(\text{AGE, D, DR22, DR31, P, TP, DT1})$$

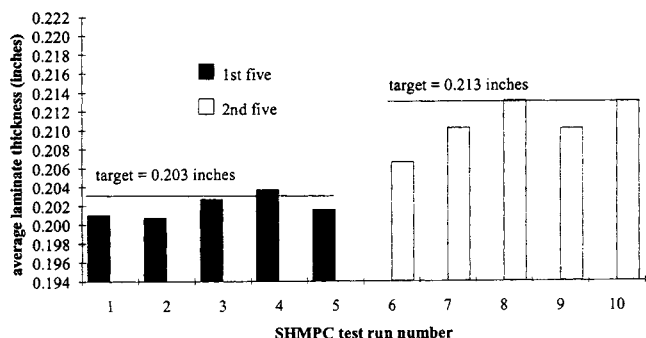
$$= 0.203 \text{ in. (5.16 mm)} [\text{or } 0.213 \text{ in. (5.41 mm)}]$$

$$P \geq 40 \text{ psi (276 kPa)}, P \leq 100 \text{ psi (689 kPa)}$$

$$DT1 \geq 45 \text{ min}, DT1 \leq 90 \text{ min}$$

$$TP \geq 0, TP \leq 1.$$

For the ten SHMPC test runs, the measured disturbances AGE and D and the secondary measurements DR22 and DR31 available for each SHMPC phase, as well as the optimal manipulated input settings P, TP, and DT1 for each SHMPC phase, are listed in detail elsewhere (Thomas, 1995, Table 7.2.2).

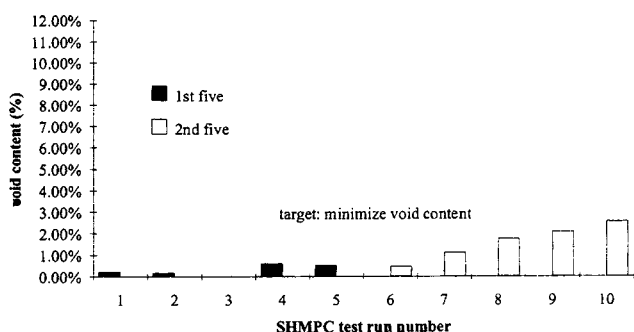


**Figure 7. Resulting thickness from ten SHMPC test runs.**

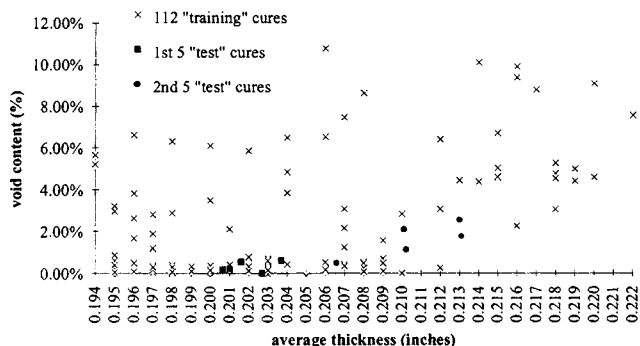
### Assessment of SHMPC test-run results

Figure 7 shows the average laminate thickness values that resulted from the ten SHMPC test runs. As with the 112 original miniclave runs, laminate thickness for each of the ten test runs was determined through an average of 25 discrete micrometer measurements, on a 0.5-in.-by-0.5-in. (12.7 mm-by-12.7-mm) grid, across the cured 3-in.-by-3-in. (76.2-mm-by-76.2-mm) laminate. The first five runs were done with a target thickness of 0.203 in. (5.16 mm). The mean of the first five test laminate thickness values was 0.2019 in. (5.128 mm), with a standard deviation of 0.0012 in. (0.030 mm). The second five runs were done with a target thickness of 0.213 in. (5.41 mm). The mean of the second five test laminate thickness values was 0.2106 in. (5.349 mm), with a standard deviation of 0.0027 in. (0.069 mm). Both mean thickness values were within a standard deviation of their respective targets.

Figure 8 illustrates the laminate void content values that resulted from the ten SHMPC test runs. As with the 112 original miniclave runs, void contents for the test runs were determined through a standard ignition loss test (ASTM D2584-68, 1990). For all ten test runs, minimized void content was the aim. The mean of the first five test void content values was 0.30%, with a standard deviation of 0.25%; the mean of the second five test void content values was 1.59%, with a standard deviation of 0.81%. For the first five test runs, maximum void content was 0.60%; for the second five, maximum was 2.54%. It is coincidental that the second five test void content values in Figure 8 show an upward trend:



**Figure 8. Resulting void contents from ten SHMPC test runs.**



**Figure 9. Comparing 112 original runs and 10 SHMPC test runs.**

the trend is not statistically significant; processing conditions fail to account physically for it; no similar appearance of a trend exists in Figure 7.

From Figure 9, it is evident that the five test runs with a 0.203-in. (5.16-mm) target thickness produced very low void content laminates. Also in this figure, there is a rectangular region bounded by thickness 0.210 in. (5.33 mm), void content 0%, thickness 0.222 in. (5.64 mm), and void content 2.55%. Four of the five SHMPC test runs with a 0.213-in. (5.41-mm) target thickness fall within this region. Of the 112 original curing runs, only two (Run 87: 0.212 in. (5.38 mm), 0.26% voids; Run 109: 0.216 in. (5.49 mm), 2.27% voids) fall within this region, while one (Run 88: 0.210 in., 0.00% voids) is on a corner of that region. Twenty-three original laminates meet 0.210 in. < thickness ≤ 0.222 in., but have void contents of greater than 2.55%; 66 original laminates meet void content ≤ 2.55%, but have thickness values of less than 0.210 in. The four SHMPC test runs within this region are in a select group; the laminates they produced show large thickness but small void content.

Attempts to replicate runs that produced low void content and large thickness proved unsuccessful when a fixed recipe was used in lieu of SHMPC. These results confirm that keeping the batch cure recipe fixed does not yield reproducible results, due to the adverse effects of disturbances. The use of secondary measurements in SHMPC allows for detecting disturbances and correcting the recipe when deviations from the norm are detected.

A study based on a one-dimensional physical model of autoclave curing (Davé et al., 1987) provides a basis for assessing SHMPC performance in controlling laminate thickness. Given the unidimensionality of this physical model, the model eliminates geometric effects that can lead to greater thickness variations. Using this model, the study predicted a cured laminate thickness per ply of 0.0057 in. (0.145 mm) for 64-, 128- and 256-ply laminates. Corresponding experimentally cured laminate thickness values per ply were 0.0054 in. on average; the laminates were produced using cure settings from the physical model predictions. The deviation for 64-ply laminates: (0.0057–0.0054) in./ply × 64 plies = 0.0192 in. (0.488 mm); the largest deviation of an SHMPC-governed laminate from its target was, by contrast, only one-third that value [0.0064 in. (0.163 mm); run 6 in Figure 7 for a similar 64-ply laminate. A competing on-line thickness control strategy is not now in industrial use for composite laminates, but this



comparison does show how the on-line SHMPC approach can produce more accurate laminate thickness values than an off-line model-optimized approach.

As an additional gauge in assessing SHMPC performance in controlling laminate thickness, consider the accepted thickness tolerances for industrially cured composites. At a prominent U.S. aerospace corporation, a process specification calls for a 7% thickness tolerance for woven cloth laminates. The Hercules E-glass/8551-7A prepreg used in this work is a woven cloth. Accordingly, the 7% tolerance would apply. For the first five SHMPC test laminates, target thickness was 0.203 in. (5.16 mm), so the 7% tolerance would allow for a laminate thickness between 0.189 and 0.217 in. (4.80 and 5.51 mm). For the second five test laminates, target thickness was 0.213 in., so the 7% tolerance would allow for a laminate thickness between 0.198 and 0.228 in. (5.03 and 5.79 mm). These industrial thickness tolerances are generally meant for complex shapes whose geometries could cause greater thickness variations. Nonetheless, each set of five test laminate thickness values fits comfortably within its respective industrially accepted tolerance limits.

### Experimental Testing of the PCM Strategy

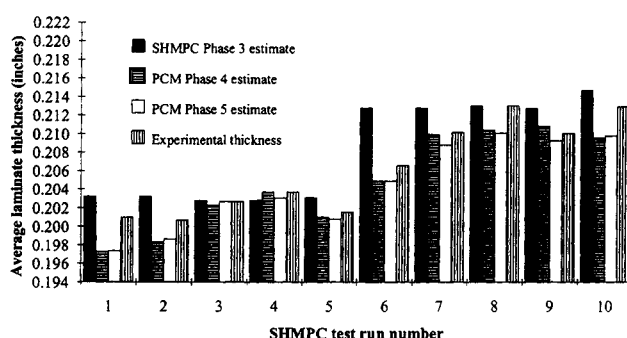
Table 3 summarizes the four successful PCM input candidates. As with the SHMPC input candidates, 17 PCM input candidates (12 secondary measurements, 3 manipulated inputs, 2 disturbances) were assessed using the same feature selection process as for SHMPC. Of these 17, four had F-test confidence levels sufficiently greater than the other 13 to warrant inclusion in the PCM strategy (Thomas and Joseph, 1997). These four inputs are thus used in the PCM strategy. All of the measured disturbances, secondary measurements, and fixed manipulated input settings from the SHMPC strategy are also available for the PCM strategy. The feature selection process, however, eliminates all but P as inputs to the PCM strategy. Secondary measurements DR61 and RAMP2 become available when  $T_{avg}$  reaches 350°F, marking the start of phase 4 (the first PCM phase). Secondary measurement TIMELG2 becomes available when  $T_{mini2}$  reaches 350°F (177°C), marking the start of phase 5 (the final PCM phase). Model components Q4 and Q5 are 3-input and 4-input, single-output ANNs (respectively). Thomas (1995) and Thomas and Joseph (1997) provide details on the development and use of these ANNs.

At the start of (post-SHMPC) phases 4 and 5, final laminate thickness and void content are estimated by the PCM

**Table 3. Four Inputs to the PCM Strategy**

PCM Candidate	Candidate Description
DR61	6th laminate surface temperature ramp rate; left thermocouple $T_{mini1}$ , measured over autoclave temperature range $T_{avg} = 310^\circ\text{F}$ to $T_{avg} = 350^\circ\text{F}$ [secondary measurement]
P	Pressure applied within miniclave to prepreg layup [manipulated input]
RAMP2	Ramp rate in $T_{avg}$ (2nd $T_{avg}$ temperature ramp, from $T_1$ to 350°F) [secondary measurement]
TIMELG2	Time lag between $T_{avg}$ reaching 350°F and $T_{mini2}$ reaching 350°F [secondary measurement]

SI conversion:  $^\circ\text{C} = (^\circ\text{F} - 32)/1.8$



**Figure 10. Comparison of SHMPC and PCM thickness estimates.**

strategy; if those estimated values have strayed too far from their targets, the curing run can be aborted, thereby saving valuable run time. Final laminate thickness and void content are measured at the end of phase 5.

### Assessing the PCM strategy

Figure 6 qualitatively illustrates the temperature-vs.-time aspects of the miniclave curing cycle. Note in this figure that phase 4 begins upon the completion of the second miniclave temperature ramp. At that point, secondary measurements DR61 and RAMP2 become known. Manipulated input P had been known since the end of the first miniclave temperature ramp, when it was fixed by the SHMPC strategy in phase 3. After the 3-h miniclave temperature hold at 350°F (177°C) begins, the laminate surface temperature as measured by  $T_{mini2}$  reaches 350°F (177°C), at which point secondary measurement TIMELG2 becomes known. At that time, phase 5 begins. The end of this latter phase coincides with the end of the curing run; upon miniclave disassembly, average laminate thickness and laminate void content can be measured.

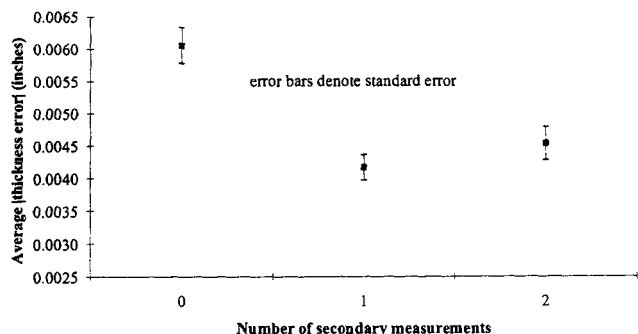
Figure 10 illustrates the average thickness of each laminate resulting from the ten SHMPC test runs. Also shown in Figure 10 are the estimated thickness values from model components  $Q3^{THICKNESS}(P, TP, DT1, AGE, D, DR22, DR31)$ ,  $Q4^{THICKNESS}(P, DR61, RAMP2)$ , and  $Q5^{THICKNESS}(P, DR61, RAMP2, TIMELG2)$ .

Table 4 summarizes the errors in thickness prediction. No mean  $|thickness\ error|$  among the PCM model components is as large as 0.0021 in. (0.053 mm). More importantly, the maximum estimation error among the PCM components is far below the maximum phase 3 (SHMPC) estimation error of 0.0062 in. (0.157 mm). Similar comparisons among phase 3 (SHMPC) and PCM void content estimators likewise revealed that the latter were competitive with the former. The

**Table 4. PCM Strategy Results: Thickness Prediction Errors**

Phase	Model Inputs	Mean Error (in.)	Max. Error (in.)
3 (SHMPC)	P, TP, DT1, AGE, D, DR22, DR31	0.0021	0.0062
4 (PCM)	P, DR61, RAMP2	0.0016	0.0037
5 (PCM)	P, DR61, RAMP2, TIMELG2	0.0017	0.0036

SI conversion: mm = in.  $\times$  25.4



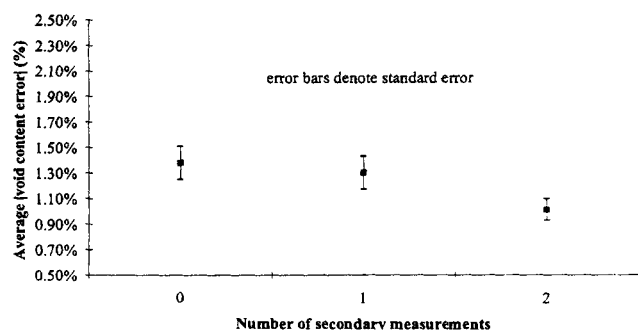
**Figure 11. Effect of number of secondary measurements on |thickness error|.**

superior performance of the PCM model components warrant use of the PCM strategy during batch-chemical process control.

### **Effects of secondary measurements on SHMPC model components**

Figure 11 plots the means of the thickness estimation-error absolute values for each of three numbers of secondary measurements (0, 1, or 2) used as SHMPC model inputs. Each point represents a mean of 210 values, generated from three different SHMPC model components prepared from seven differing subsets of regression data from the 112 original curing runs—21 model components in all—applied to each of the ten SHMPC test runs (see Thomas, 1995, sec. 7.3.2, for more details). Note that the error bars of the models with one and two secondary measurements overlap, and that the mean errors of the models with one and two secondary measurements are significantly lower than that of the model with no secondary measurement inputs. The SHMPC model components with access to on-line secondary measurements outperform those without such access, as regards laminate thickness estimation. For such estimation, all necessary information arrives with the first secondary measurement; the following secondary measurement adds little information.

Figure 12 is to laminate void content what Figure 11 is to average laminate thickness. Each point in Figure 12 also represents the mean of 210 absolute values of void content errors associated with SHMPC models. As with thickness, the void content estimation errors from the models with two secondary measurement inputs are significantly lower than those



**Figure 12. Effect of number of secondary measurements on |void content error|.**

from the models with no such inputs. The SHMPC model with the most available on-line information (by way of secondary measurements) provides superior estimation, compared to that with no available on-line information. For void content estimation, the second secondary measurement is needed for an adequate estimate; for thickness estimation, only the first secondary measurement is needed. This result is consistent with what is understood about the physical phenomena of void growth and thickness reduction during the autoclave cure process: void growth is more complex; its inherent nature is revealed later in the batch run than that of thickness reduction.

### **Summary**

For the first five SHMPC test runs, whose target was 0.203 in., the mean of the five laminate thickness values was 0.2019 in. (5.128 mm)—well under 2% deviation from the target. For the second five SHMPC test runs, whose target was 0.213 in. (5.410 mm), the mean of those five laminate thickness values was 0.2106 in. (5.349 mm)—again, well under 2% deviation from the target. Void content was to be minimized in all ten SHMPC test runs. Very few of the original 112 laminates showed an average thickness of over 0.210 in. (5.334 mm) and a void content below 2.6%: four of the second five SHMPC test laminates met both criteria. For none of the ten SHMPC test runs would the PCM strategy have called for aborting a run, and the PCM model components predicted final laminate thickness and void content at least as accurately as (and often more accurately than) their SHMPC counterparts.

Through verifying the SHMPC and PCM strategies, this work also establishes the viability of this quantitative approach, as an extension of both inferential and model-based control. As a supervisory-level control approach, the SHMPC and PCM strategies direct the process output toward target quality variables—variables not addressed by traditional regulatory control of batch chemical processes.

Neither SHMPC nor PCM strategies are limited to use of ANNs, or linear regression models, or quadratic regression models as their model components. Linear thickness models with ANN void content models could easily have been used within these strategies. The strategies can use any quantitative input/output (I/O) models that provide sufficiently accurate quality variable estimates. Feedforward ANNs now serve as minimally structured nonlinear models, whose properties have yet to be fully understood. In time, however, when a new and better quantitative I/O model is developed, that new model type can be easily used within the SHMPC and PCM strategies.

Three primary recommendations for future work in this area can be made.

The first entails building strategy model component training data out of simulated *and* experimental process data. While the former alone is often too inaccurate and assumption-riddled for practical use, the latter alone tends to be time-consuming and expensive to collect. A hybrid training set, with a large ratio of simulated-to-experimental runs, would counteract the problems using but one type of data: the preponderance of simulated data would lower the expense of data collection, while the presence of experimental data would offset errors from the simulation approach. Pre-

liminary results along these lines have been reported by Tsen et al. (1996).

The second recommendation is for the SHMPC and PCM strategies to be implemented on batch-chemical processes other than autoclave curing, that is, on processes for which measurements (related to end-product quality) are at least occasionally available during the process. That implementation would increase the breadth of SHMPC and PCM strategy applicability. It would be most informative to apply the SHMPC and PCM strategies to large-scale autoclave curing—curing for which laminate surface area and curvature must be considered. This application would introduce additional disturbance sources to the problem addressed within this work.

Finally, the stability and convergence properties of nonlinear model predictive control strategies need to be addressed. In the case study presented herein, stability and convergence were not an issue, due to the severe constraints placed on the manipulated inputs. Nonetheless, in a larger context, one should be aware of the feedback present in the system and the possibility of instabilities arising from plant-model mismatches.

## Acknowledgments

Financial support provided by NSF Grant DMI-91-23861 is gratefully acknowledged. Financial support by the McDonnell Douglas Corporation for M. M. Thomas is likewise gratefully acknowledged. The authors thank the three anonymous reviewers and appreciate their insightful feedback.

## Literature Cited

ASTM D2584-68, "Standard Test Method for Ignition Loss of Cured Reinforced Plastics," *1990 Annual Book of ASTM Standards*, Vol. 08.02: Plastics(II), ASTM, Philadelphia, p. 340 (1990).

Box, G. E. P., "Use and Abuse of Regression," *Technometrics*, **8**(4), 625 (1966).

Box, G. E. P., W. G. Hunter, and J. S. Hunter, *Statistics for Experimenters*, Wiley, New York (1978).

Cuthrell, J. E., and L. T. Biegler, "Simultaneous Optimization and Solution Methods for Batch Reactor Control Profiles," *Comput. Chem. Eng.*, **13**(1/2), 49 (1989).

Davé, R. S., "Autoclave Process Modeling," *International Encyclopedia of Composites*, S. M. Lee, ed., VCH Publishers, New York, p. 74 (1990).

Davé, R., J. L. Kardos, and M. P. Duduković, "A Model for Resin Flow During Composite Processing: Part 2—Numerical Analysis for Unidirectional Graphite/Epoxy Laminates," *Polym. Compos.*, **8**(2), 123 (1987).

Draper, N. R., and H. Smith, *Applied Regression Analysis*, 2nd ed., Wiley, New York (1981).

Eliçabe, G. E., and G. R. Meira, "Estimation and Control in Polymerization Reactors: A Review," *Polym. Eng. Sci.*, **28**(3), 121 (1988).

Garcia, C. E., and D. M. Pretz, "Advances in Industrial Model-predictive Control," *Proc. Int. Conf. on Chemical Process Control (CPC III)*, M. Morari and T. J. McAvoy, eds., Elsevier, New York, p. 245 (1986).

Garcia, C. E., D. M. Pretz, and M. Morari, "Model Predictive Control: Theory and Practice—A Survey," *Automatica*, **25**(3), 335 (1989).

Garcia, C. E., B. L. Ramaker, and J. F. Pollard, "Total Process Control—Beyond the Design of Model Predictive Controllers," *Proc. of the Int. Conf. on Chemical Process Control (CPC-IV)*, Y. Arkun and W. H. Ray, eds., CACHE, Austin, TX, p. 335 (1991).

Jang, S.-S., B. Joseph, and H. Mukai, "On-line Optimization of Constrained Multivariable Chemical Processes," *AIChE J.*, **33**(1), 26 (1987).

Joseph, B., and F. Wang Hanratty, "Predictive Control of Quality in a Batch Manufacturing Process Using Artificial Neural Network Models," *Ind. Eng. Chem. Res.*, **32**(9), 1951 (1993).

Joseph, B., F. Wang Hanratty, and J. L. Kardos, "Model Based Control of Voids and Product Thickness during Autoclave Curing of Carbon/Epoxy Composite Laminates," *J. Compos. Mater.*, **29**(8), 1000 (1995).

Larimore, W. E., and R. K. Mehra, "The Problem of Overfitting Data," *Byte*, **10**(10), 167 (1985).

Lasdon, L. S., A. D. Waren, A. Jain, and M. Ratner, "Design and Testing of a Generalized Reduced Gradient Code for Nonlinear Programming," *ACM Trans. Math. Softw.*, **4**(1), 34 (1978).

Lee, J. H., M. Morari, and C. E. García, "State-Space Interpretation of Model Predictive Control," *Automatica*, **30**(4), 707 (1994).

Morari, M., and J. H. Lee, "Model Predictive Control: the Good, the Bad, and the Ugly," *Proc. of the Int. Conf. on Chemical Process Control (CPC-IV)*, Y. Arkun and W. H. Ray, eds., CACHE, Austin, TX, p. 419 (1991).

Morari, M., "Model Predictive Control: Multivariable Control Technique of Choice in the 1990s?," Tech. Rep. CIT/CDS 93-024, Control and Dynamic Systems, California Institute of Technology, Pasadena, CA (1993).

Nomikos, P., and J. F. MacGregor, "Monitoring Batch Processes Using Multiway Principal Component Analysis," *AIChE J.*, **40**(8), 1361 (1994).

Palanki, S., C. Kravaris, and H. Y. Wang, "Synthesis of State Feedback Laws for End-point Optimization in Batch Processes," *Chem. Eng. Sci.*, **48**(1), 135 (1993).

Richalet, J., "Industrial Applications of Model Based Predictive Control," *Automatica*, **29**(5), 1251 (1993).

Ricker, N. L., "Model Predictive Control with State Estimation," *Ind. Eng. Chem. Res.*, **29**(3), 374 (1990).

Ricker, N. L., "Model-predictive Control: State of the Art," *Proc. of the Int. Conf. on Chemical Process Control (CPC-IV)*, Y. Arkun and W. H. Ray, eds., CACHE, Austin, TX, p. 271 (1991).

Sorosh, M., and C. Kravaris, "Nonlinear Control of a Batch Polymerization Reactor: An Experimental Study," *AIChE J.*, **38**(9), 1429 (1992).

Sorosh, M., and S. Valluri, "An Approach to Optimization and Control of Batch Processes," *Proc. of the American Control Conf.*, Baltimore, p. 490 (1994).

Taguchi, G., *System of Experimental Design*, UNIPUB-Kraus International Publications, White Plains, NY, and American Supplier Institute, Dearborn, MI (1987).

Terwiesch, P., and M. Agarwal, "Online Correction of Pre-optimized Input Profiles for Batch Reactors," *Comput. Chem. Eng.*, **18**(Suppl.), S433 (1994).

Thayer, A. M., "Advanced Polymer Composites Tailored for Aerospace Use," *Chem. Eng. News*, **68**(30), 37 (1990).

Thomas, M. M., "Quality Control of Batch Chemical Processes with Application to Autoclave Curing of Composite Laminate Materials," DSc diss., Washington Univ., St. Louis (1995).

Thomas, M. M., and B. Joseph, "Feedforward Network Usage in Batch Chemical Process Quality Control," *Neural Networks*, submitted (1997).

Thomas, M. M., B. Joseph, and J. L. Kardos, "Experimental Characterization of Autoclave-cured Glass-epoxy Composite Laminates: Cure Cycle Effects upon Thickness, Void Content, and Related Phenomena," *Polym. Compos.*, **18**(3), 283 (1997).

Thomas, M. M., J. L. Kardos, and B. Joseph, "Shrinking Horizon Model Predictive Control Applied to Autoclave Curing of Composite Laminate Materials," *Proc. of the American Control Conference*, Baltimore, p. 505 (1994).

Tsen, A. Y. D., S.-S. Jang, D. S. H. Wong, and B. Joseph, "Predictive Control of Quality in Batch Polymerization Using a Hybrid Artificial Neural Network Model," *AIChE J.*, **42**(2), 455 (1996).

Manuscript received Jan. 21, 1997, and revision received June 19, 1997.