On Identification and Control of Reactive Extrusion Processes

Swapnil C. Garge, Mark D. Wetzel and Babatunde A. Ogunnaike,

Department of Chemical Engineering

University of Delaware

Newark, DE 19716-3110

ogunnaike@che.udel.edu

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1. Introduction

Reactive extrusion is an important commercial process used for the production and modification of a wide variety of polymers and blends (e.g. Ethylene co-polymers, Polyamides, etc.). Its primary distinguishing characteristic is that chemical reactions are deliberately carried out during continuous melt extrusion to achieve desired product properties. The ever-tightening customer demands on product specs have necessitated comprehensive dynamics and control studies of these processes, which, until now, have mostly focused on the control of a single variable such as viscosity (Broadhead *et al.*, 1996).

The dynamics of the process are determined primarily by the strong interactions between the fluid mechanics, reaction kinetics, heat transfer and the extruder geometry, making effective control of product quality and end-use properties very difficult [1]. It is important to note that the usual challenges associated with the control of conventional polymer reactors (e.g. unavailability or low frequency of physical property measurements, and frequent grade transitions) are also encountered in the control of reactive extrusion.

To meet the customer's demands efficiently, it has become important to develop a method for effective control of process variables 'y' (melt pressure, melt temperature, motor power etc.), product properties 'q' (melt index, viscosity, density, etc), but more importantly, end-use physical characteristics 'w' (toughness, UV/chemical resistance, etc.), to guarantee acceptable end-use product performance. Our ultimate objective is to develop such a framework for controlling key product properties and assuring acceptable end-use performance.

Our approach to this challenging problem is to begin with an adequate mathematical representation of the relationships between variables across the entire processing chain. Such a representation will serve two crucial tasks: (i) provide estimates of the infrequently measured product properties at a much faster rate, and (ii) facilitate the development of a control system to meet the above mentioned objective.

Fig. 1 shows a schematic representation of the proposed modeling scheme, which consists of the following models: i) M_{uy} – a model that relates the manipulated variables, u, to process output variables, y, (ii) M_{uq} – a model relating the manipulated variables, u, to the internal product quality variables, $\hat{q}$, (iii) M_{qq} – a model relating internal quality variables, $\hat{q}$, to product quality variables, q, (iv) M_{qw} – relating internal quality variables, $\hat{q}$, to end use characteristics, and (v) M_{wz} – relating end-use physical characteristics, w, to product performance in end-use, z_w, a binary variable that represents acceptable performance as 1, and unacceptable performance as 0.

A modeling scheme that relates the different classes of variables sequentially, although more intuitive, is impractical in this case. This is because it is usually difficult to model the relationships between the process outputs and the measured product quality variables, since these variables are based on the measurements, which are selected on practical grounds such as the availability of sensor locations, and developing a mathematical relationship between these two classes of variables is not straightforward. To overcome this problem, an additional class of variables, called internal product quality variables '$\hat{q}$' (composition, weight average molecular weight) that constitutes an indirect way to link 'y' with 'q', is introduced in the modeling scheme.

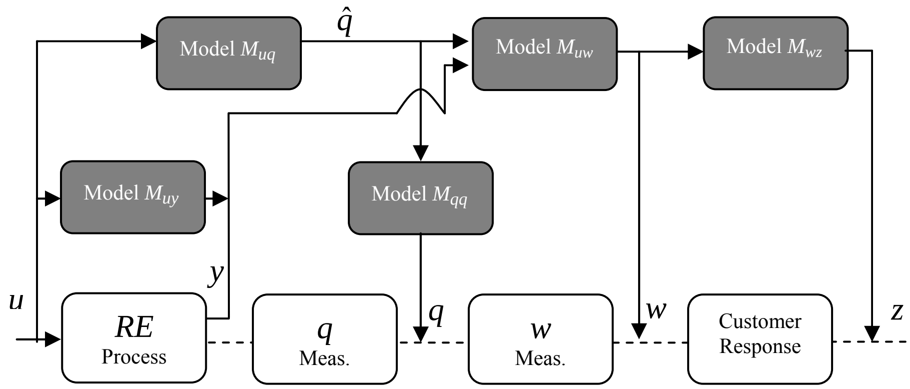

Fig. 1. Proposed modeling scheme

Our control paradigm is predicated upon using the above network of models for two important tasks: (i) to translate the customer requirements on end-use performance to set points for process variables, and (ii) to make appropriate modifications (that is, to take control action) wherever appropriate along the manufacturing chain based on all available information. For this purpose a multivariable cascade control scheme (Fig. 2) is proposed, consisting of a fast model-based controller *C1* for the inner loop between the manipulated variables and the output variables, and a slower (also model-based) controller *C2*, which will translate the end use performance objectives to set points for the output variables. In addition to these loops, there exists innermost basic regulatory control loops, which ensures that set point changes in the manipulated variables are efficiently tracked.

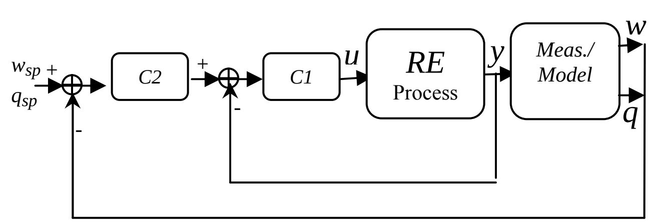

Fig. 2. Proposed control scheme. *C1* and *C2* are the inner loop and outer loop controllers respectively.

This paper is concerned with the modeling of the relationships between process manipulated inputs '*u*' and process output variables '*y*', specifically for an example process involving the reaction of a functionalized ethylene co-polymer, "Elvaloy" (Ethylene/n-Butyl Acrylate/Glycidal Methacrylate Terpolymer (E/BA/GMA)) with an acid co-polymer "Nucrel" (Ethylene/Methacrylic Acid Copolymer (E/MAA)) in a Coperion W&P ZSK-30mm co-rotating, intermeshing twin screw extruder. Due to the complexity of the interacting process mechanisms, first-principles modeling is impractical for control at this level. System identification from carefully obtained input/output data is a more practical alternative.

In this paper, we present a systematic procedure for carrying out the three major identification tests for this class of processes: (i) experimental test design and collection of input/output data, (ii) model structure and order selection and (iii) model validation. Section 2 describes the system inputs/outputs, the data collection and the data pretreatment procedure. Section 3 describes the preliminary tests and their use in obtaining the information needed for the design of the final test. Section 4 describes the design and implementation of the final test. Finally, Section 5 describes the model structure and order selection and presents the model validation results.

2. System Description

2.1. Process Inputs and Outputs

The base feed material for the example reactive extrusion system is E/MAA. The manipulated inputs for the system are screw speed, E/MAA feed-rate, E/BA/GMA feed-rate and barrel temperatures for the seven extruder zones. Changes in all the inputs are implemented manually. The barrel temperature regulatory controller loop dynamics are much slower than the process dynamics excluding the inner regulatory loops. Therefore, with the exception of a step change, any other dynamic change in the barrel temperature is impractical. The inner loop dynamics for other manipulated inputs are fast compared to the process; therefore, more rapid changes in these variables can be implemented.

The process outputs are die pressure (y_1), exit melt temperature (y_2), and motor power (y_3), and E/MAA weight fraction in the melting zone (y_4). With the exception of the E/MAA weight fraction, which can be easily obtained from the feed-rates of the two polymers, all other outputs are measured.

2.2 Data Acquisition

A specialized data acquisition system (DAQ) [3] was used to collect the input/output data. The DAQ has several key components: 1) a specialized, high-speed data acquisition hardware front end for dynamic signal capture, 2) a PC-based experiment monitoring and data processing package, and 3) a program to analyze the pulse response and steady-state data. The signals are wired to the DAQ front end including motor amperage (or torque), motor voltage, screw speed, die pressure and exit melt temperature.

In the experiments, data scanned at 60Hz were decimated to 20Hz and filtered with a 3-tap median filter to remove large spikes from the signals. In spite of the filtering, significant noise levels remain; therefore, the data were re-sampled at a frequency of 1 Hz using a moving time-average procedure to improve the Signal-to-Noise Ratio (SNR).

2.3. Data Pre-Treatment

In numerous instances, spikes were observed in the data due to severe unmeasured disturbances, process upsets and/or sensor failure. These spikes were manually removed before performing the estimation.

3. Preliminary Tests

The preliminary tests were aimed at obtaining *à priori* knowledge needed for the design of the final identification test. The tests consisted of a series of step changes as well as simultaneous staircase changes in the manipulated inputs at two operating points: (A) a low melting-zone composition of E/BA/GMA (~ 1%) inducing a low extent of reaction and a relatively small change in the product viscosity compared to the viscosity of the pure E/MAA feed, and (B) a high melting-zone composition of E/BA/GMA (~ 4 %) inducing a high extent of reaction and a significant change in the product viscosity. This selection of the operating points enabled a comprehensive study of the effect of the reaction on the process dynamic behavior.

Table 1a shows the process gains. The gains differ significantly and in some cases have a different sign at the two operating conditions, indicating that the reaction has a strong nonlinear effect on the process dynamic behavior. The results suggest that it may be necessary to build non-linear models of the process to capture the "reaction effect".

Table 1a: Scaled Gain Matrix and RGA

Scaled Gain Matrix								
	Operating Point A				Operating Point B			
O/I	u_1	u_2	u_3	u_4	u_1	u_2	u_3	u_4
y_1	-0.0617	0.0711	1.5	0.178	0.16	-0.3	1	0.533
y_2	0.0723	0.04	0.6	0.667	0.125	-0.035	0.35	0.8
y_3	0.75	0.333	0.625	-0.4667	0.381	0.3	0.188	-0.333
y_4	0	0.0205	0.4886	0	0	0.0208	0.4579	0
Relative Gain Array								
	Operating Point A				Operating Point B			
O/I	u_1	u_2	u_3	u_4	u_1	u_2	u_3	u_4
y_1	0.673	1.957	-1.732	0.102	0.279	0.8349	0.126	-0.239
y_2	0.218	-0.2156	0.136	0.862	-0.0405	-0.07211	-0.0328	1.145
y_3	0.109	0.928	-0.073	0.0363	0.763	0.148	-0.0042	0.0938
y_4	0	-1.669	2.669	0	0	0.0895	0.911	0

Table 1b: Singular Value Decomposition

Singular Value Matrix							
Operating Point A				Operating Point B			
1.84	0	0	0	1.44	0	0	0
0	1.01	0	0	0	0.714	0	0
0	0	0.47	0	0	0	0.410	0
0	0	0	0.0109	0	0	0	0.135
Condition Number(κ) = 169.3				Condition Number(κ) = 10.66			

Table 1c: *Matrix of Left Singular Vectors*

	Operating Point A				Operating Point B			
y_1	0.807	-0.201	-0.453	-0.322	-0.807	0.236	0.337	-0.425
y_2	0.368	-0.452	0.810	0.066	-0.539	-0.443	-0.675	0.241
y_3	0.381	0.869	0.314	-0.028	0.048	0.786	-0.602	-0.133
y_4	0.261	-0.011	-0.202	0.944	-0.239	0.361	0.262	0.863

Table 1d: *Matrix of Right Singular Vectors*

	Operating Point A				Operating Point B			
u_1	0.143	0.626	0.685	0.344	-0.124	0.395	-0.633	-0.654
u_2	0.111	0.255	0.214	-0.936	0.188	0.263	-0.615	0.719
u_3	0.977	-0.034	-0.203	0.060	-0.761	0.551	0.263	0.222
u_4	0.115	-0.736	0.666	-0.034	-0.609	-0.687	-0.389	0.077

The relative gain array (RGA) of the 4×4 system indicates the potential for strong loop interactions when the y's are controlled using multiple single loop controllers. To study the conditioning of the process, singular value decomposition (SVD) of the scaled gain matrix was performed. The inputs and outputs were scaled with the maximum change introduced and observed respectively for a particular variable. The condition number (κ), the ratio of the highest to the lowest singular value, was much higher at the operating point A than at the operating point B. The κ values suggest that the process is ill-conditioned at the operating point A and that the reaction improves the conditioning of the process.

A characteristic of an ill-conditioned system is that it amplifies input vectors differently based on the directions of these vectors. The process directionality is evident from Tables 1b, 1c and 1d, at the operating point A, where it is observed that the outputs y_1 and y_2 are weakly dependent on the inputs u_1 and u_2 and strongly dependent on the input u_3. Such directionality makes it difficult to identify the low gain direction from experimental data. At the same time, the process directionality is important from a control perspective since integral controllability criteria require that the identified control-relevant model, obtained using the final test, should represent the process behavior in the high as well as low gain directions.

In addition, the information about extent of the process nonlinearity is helpful in designing the final tests. For this purpose, staircase tests for the inputs u_1 and u_2, with varying stair-widths, were designed, based on the process time constants. A staircase test probes the process dynamic behavior for positive as well as negative changes in the inputs and therefore is able to provide information about the process non-linearity [4]. These tests revealed that the process is approximately linear around each operating point, as illustrated in Fig. 2.

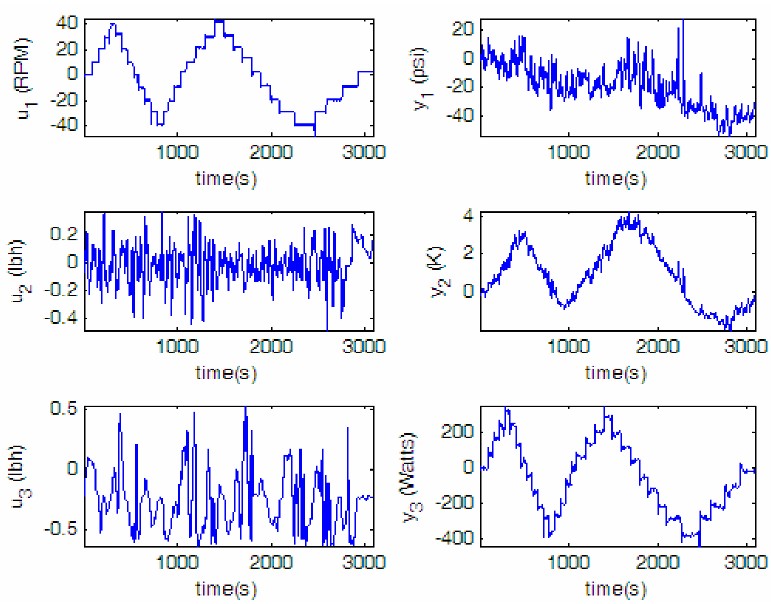

Fig.2a: Screw speed staircase test data at operating point A: Inputs, left panels; outputs right panels

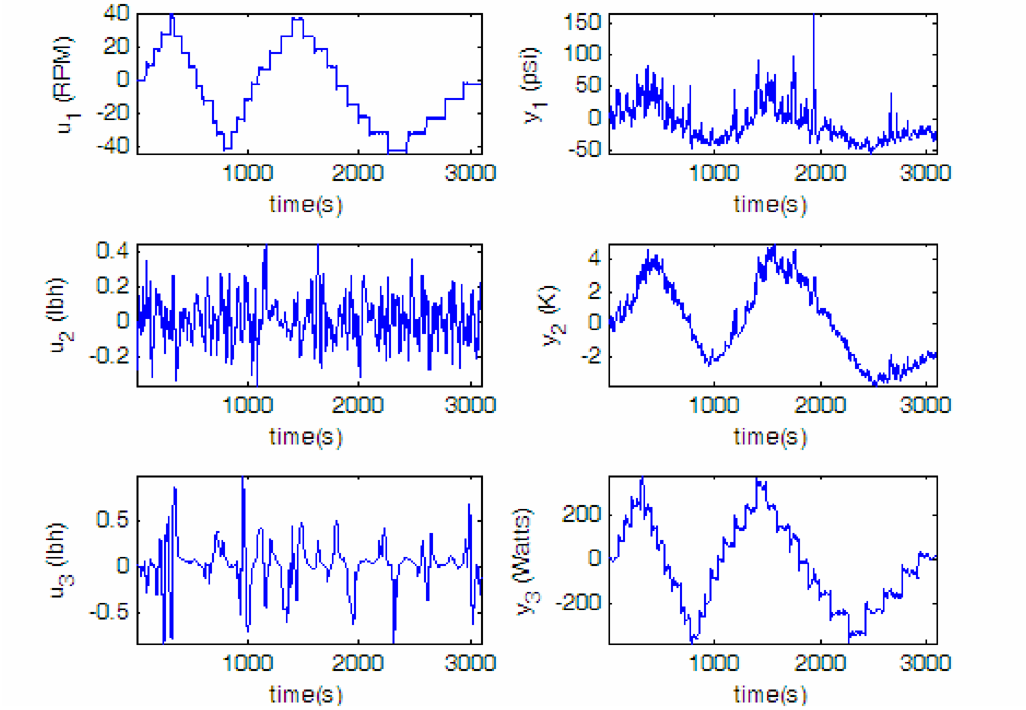

Fig. 2b: Screw speed staircase test data at operating point B: Inputs, left panels; outputs right panels

4. Final Identification Test

The design of the final identification test inherently depends on the selected model configuration, which is usually based on known process information. For the example process, the preliminary tests indicate that the process exhibits significant non-linearity and is ill-conditioned at the operating point A. The non-linear behavior can be captured with a non-linear model, such as the NARMAX, Hammerstien, or the Wiener model. It is often difficult, however, to represent the process behavior over the full range of operation with these structures [5]. Alternatively, the non-linear behavior of a process can be represented using a multi-model approach consisting of a global model obtained by an interpolation of a series of local models. This approach is particularly suitable for the process under consideration because the process behavior at each of the two operating points is approximately linear, which facilitates the use of a simple linear model at each operating point.

The final test to identify linear models consists of designing and administering suitable input excitation signals. The choice of the excitation signals used to obtain the input/output data is critical for identification. One of the desired characteristics is that the signals should be *persistently exciting* in order to guarantee that a parameter estimation algorithm will give a unique solution (see, for e.g., [4]). Generalized binary noise (GBN) signals, proposed by Tullenken [6], were selected as the excitation signals for our application because they are persistently exciting and are easy to administer. A GBN signal switches between the values of $-a$ and a (a being the magnitude of the desired change) using a switching probability (p_{sw}) based on the following rule

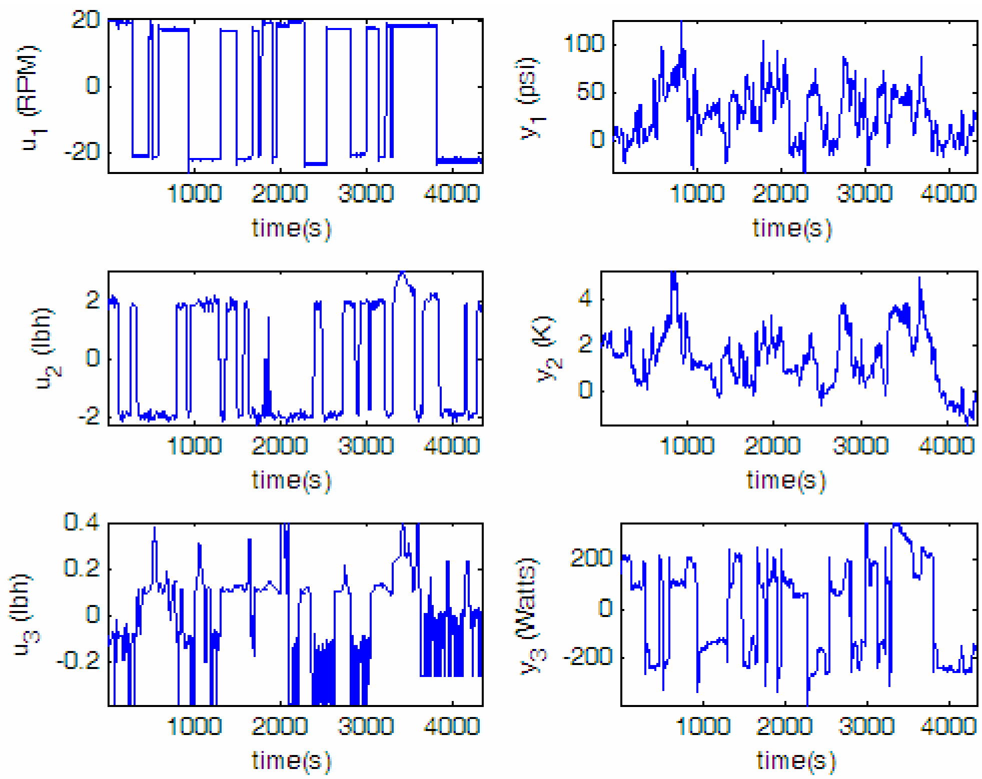

Fig .3a: GBN test data at operating point A: Inputs, left panels; outputs right panels

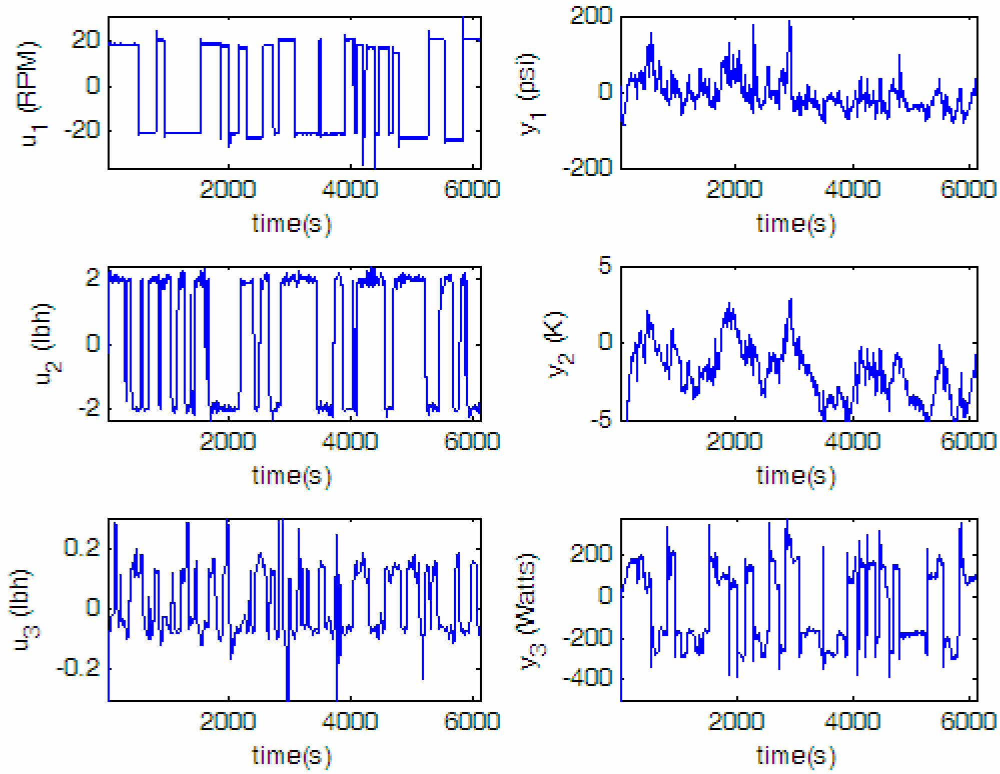

Fig. 3b: GBN test data at operating point B: Inputs, left panels; outputs right panels

$$\begin{aligned} P[u(t) = -u(t-1)] &= p_{sw} \\ P[u(t) = u(t-1)] &= 1 - p_{sw} \end{aligned} \tag{1}$$

As proposed by Zhu [4], GBN signals with a mean switching time equal to 1/3rd of the dominant process time constant (~360 s) were used in the final test. In recognition of unmeasured disturbances and high measurement noise levels (low signal to noise ratio), the duration of the test was approximately 15 times the dominant time constant.

A significant reduction in the test-duration is obtained by using specially designed signals such as GBN instead of conventional step tests. It is, in fact, possible and recommended to administer the signals simultaneously in more than one input. Three uncorrelated GBN signals were administered simultaneously in the screw speed (u_1), E/MAA feed-rate (u_2), and E/BA/GMA feed-rate (u_3) at each operating point (*Fig, 3*).

The uncorrelated signals are suitable for identifying a well-conditioned process as well as for identifying the high gain direction of an ill-conditioned process, but not the low gain direction. Many authors have, instead, suggested the use of highly correlated signals for identifying the low gain direction (at the operating point A).

An additional test, based on the open loop design suggested by Zhu [4], was used to identify the low gain direction of the process at the operating point A. The GBN signals (*Fig. 4*), which have correlated high amplitude periods combined with uncorrelated low amplitude periods, were administered only to the inputs u_1 and u_2, while the input u_3 was unchanged. The low amplitude was set at one-third the value of high amplitude.

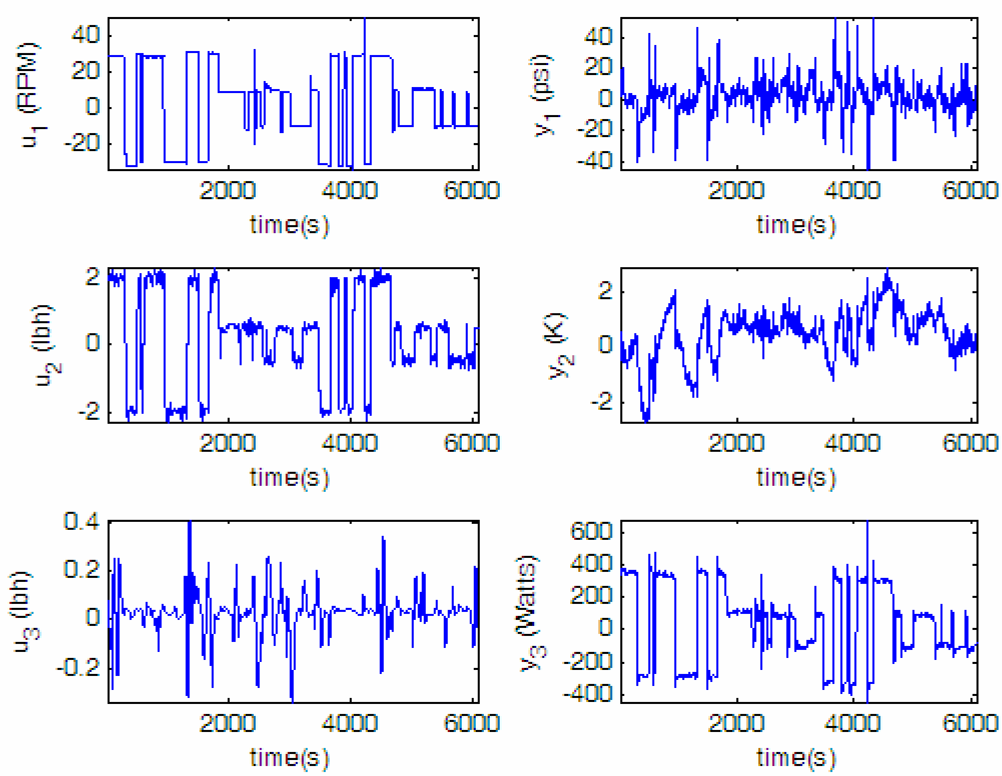

Fig. 4: Experimental data from the test for identifying the low gain direction at the operating point A: Inputs, left panel; outputs, right panel

5. Model Structure

The multi-model approach entails the identification of a separate model for distinct regions, known as premise regions [5], in the input/output variable space. The regions are defined by premise variables, which are quantitatively related to the input variables (and, sometimes, the output variables). The premise variable used for the process under consideration is the E/BA/GMA weight fraction (y_4) in the melting zone. Three premise regions are defined as follows: (I) 0 % < y_4 < 1.5%, (II) 1.5 % < y_4 < 3.5%, and (III) 3.5 % < y_4 < 5%. The global model is obtained by interpolating between local models with sigmoidal membership functions that assign weights to the outputs of the local models to generate the global model output as:

$$y_{global} = \frac{w_I y_I + w_{II} y_{II}}{w_I + w_{II}} \tag{2}$$

where,

$$\begin{aligned} w_I &= \frac{1}{1+e^{a(y_4-c)}} \\ w_{II} &= \frac{1}{1+e^{-a(y_4-c)}} \end{aligned} \tag{3}$$

In these equations, a and c are the constants that determine the shape of the sigmoid. Since the process exhibits approximately linear behavior in the premise regions I and III, a linear model was identified for each of these regions. In these regions, the selected membership function, therefore, assigns a weight '1' to the corresponding model output of a particular premise region and a weight '0' to the model output of the other region. The membership function for region II is combination of two sigmoids, each having an inflexion point at the y_3 value of 2.5% (Fig. 5).

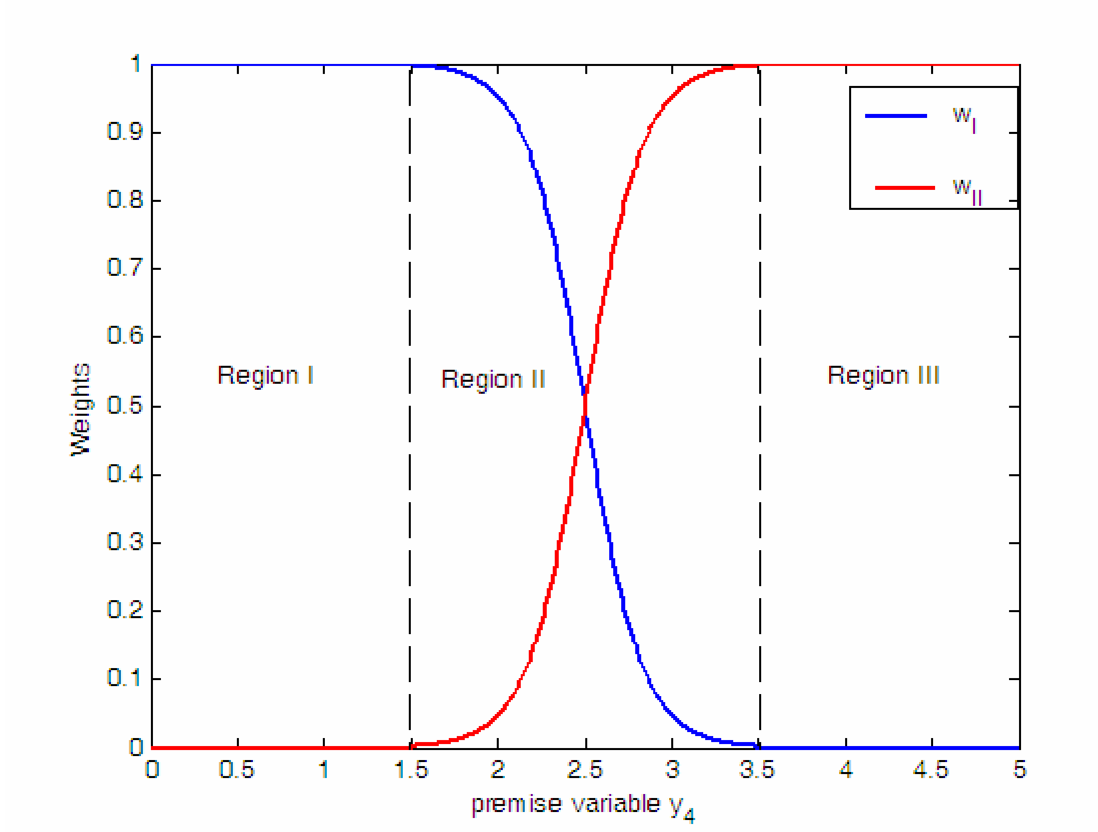

Fig. 5 Sigmoidal Membership Functions

The candidate linear models for regions I and III consists of the following models:

(1) Autoregressive with exogenous inputs (ARX)

(2) Autoregressive moving average with exogenous inputs (ARMAX): an extension of the ARX model to include a "moving average" disturbance model.

(3) Box-Jenkins (BJ): provides a complete model with disturbance modeled separately from the system dynamics.

The general (SISO) structure of these parametric models is as follows

$$F(q)y(t) = \frac{B(q)}{A(q)}u(t - n_{d1}) + \frac{C(q)}{D(q)}e(t - n_{d2}) \tag{4}$$

where,

$$
\begin{aligned}
A(q) &= 1 + a_1 q^{-1} + \ldots + a_n q^{-n_a} \\
B(q) &= \quad b_1 q^{-1} + \ldots + b_n q^{-n_b} \\
C(q) &= 1 + c_1 q^{-1} + \ldots + c_n q^{-n_c} \\
D(q) &= 1 + d_1 q^{-1} + \ldots + d_n q^{-n_d} \\
and, &\quad q^{-k} v(k) = v(t-k)
\end{aligned}
$$

For the ARX model, both multi-input multi-output (MIMO) as well as multi input single output (MISO) formulations were selected as the candidate models, whereas for ARMAX and BJ models only MISO formulations were selected. The parameter estimation was performed using the System Identification Toolbox of Matlab®. The criteria used for comparing the model structures and selecting model orders were: (i) percentage of output variation that is reproduced by the model, (ii) Akaike's final prediction error, and (iii) pole-zero diagrams to check for over parameterization.

We encountered estimation problems for MIMO formulations of ARX and MISO formulations of ARMAX structures. The MISO BJ structure provided the best fit and the smallest final prediction error compared to other MISO formulations. Table 2 shows the best fit MISO BJ model orders.

Table 2: Identified Box Jenkins Structures

Operating Point A							
	MISO Model Orders				Delay (n_{d1}, n_{d2})		
O/I	u_1 (n_a,n_b)	u_2 (n_a,n_b)	u_3 (n_a,n_b)	*Noise* (n_c,n_d)	u_1	u_2	u_3
y_1	5,5	5,5	5,5	2,2	0,0	0,0	0,0
y_2	5,5	5,5	5,5	1,1	0,0	0,0	0,0
y_3	3,3	3,3	3,3	0,0	0,0	0,0	0,0
y_4	0,0	2,2	2,2	1,1	0,0	0,0	0,0

Operating Point B							
	MISO Model Orders				Delay (n_{d1}, n_{d2})		
O/I	u_1 (n_a,n_b)	u_2 (n_a,n_b)	u_3 (n_a,n_b)	*Noise* (n_c,n_d)	u_1	U_2	u_3
y_1	5,5	5,5	5,5	3,3	1,1	1,1	1,1
y_2	4,4	4,4	4,4	1,1	0,0	0,0	0,0
y_3	3,3	3,3	3,3	1,1	0,0	0,0	0,0
y_4	0,0	2,2	2,2	1,1	0,0	0,0	0,0

5.1. Model Validation

The validation data consists of a fraction of the final test data that was not used for estimation, in addition to the preliminary test data. Some of the results are presented below. Fig. 6 shows a comparison between the model response and the experimental data for the experiments conducted at the operating point A; Fig. 7 shows the same comparison for the experiments conducted at the operation point B. The linear models are successfully able to represent the important process dynamics at the corresponding operating point.

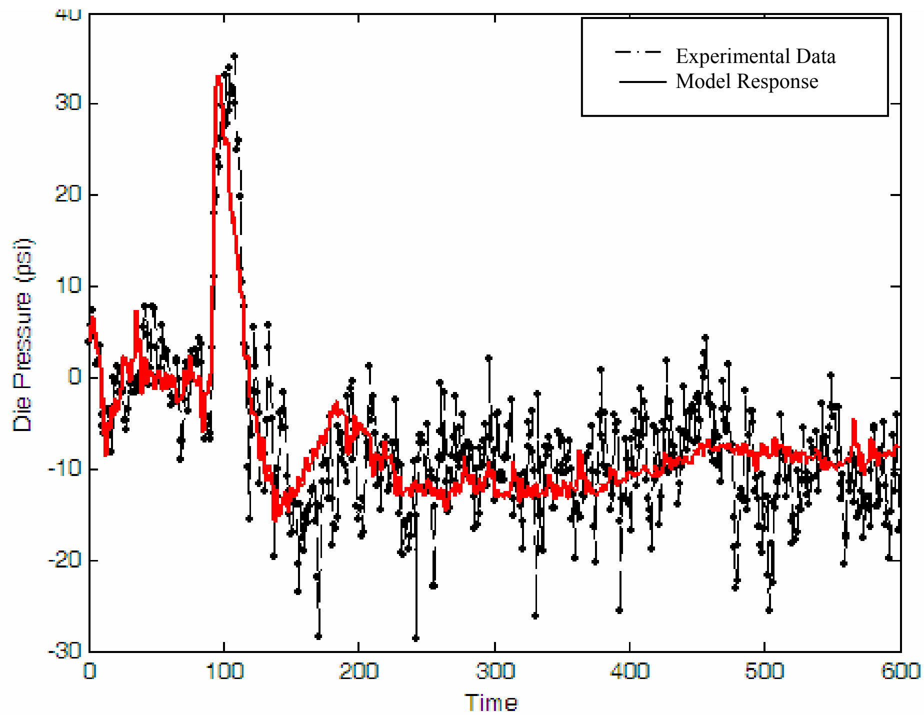

Fig. 6a: Die Pressure: Response to Step change in screw speed from 180 to 220 RPM

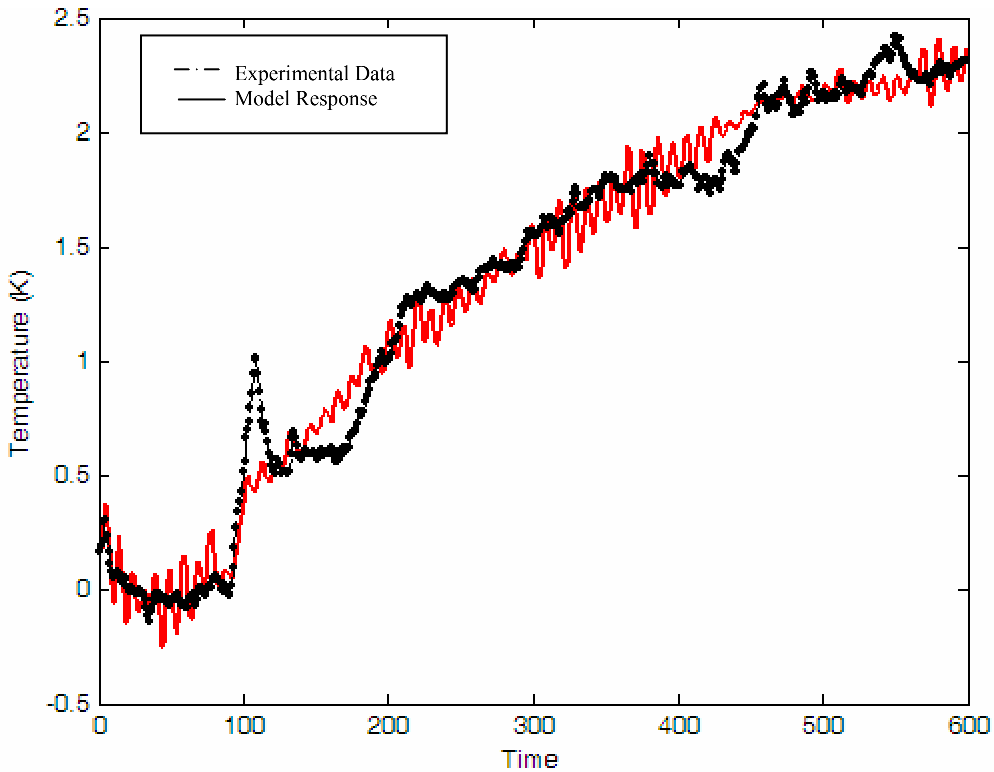

Fig. 6b: Exit Melt Temperature: Response to step change in screw speed from 180 to 220 RPM

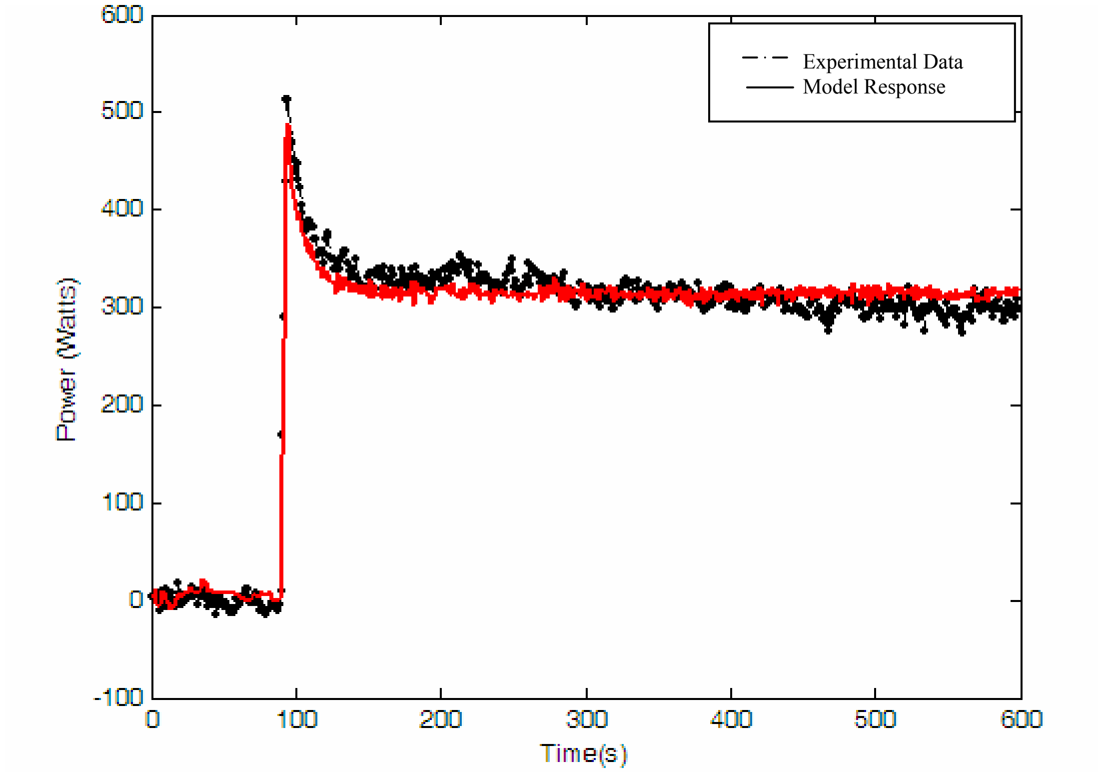

Fig. 6c: Motor Power: Response to step change in screw speed from 180 to 220 RPM

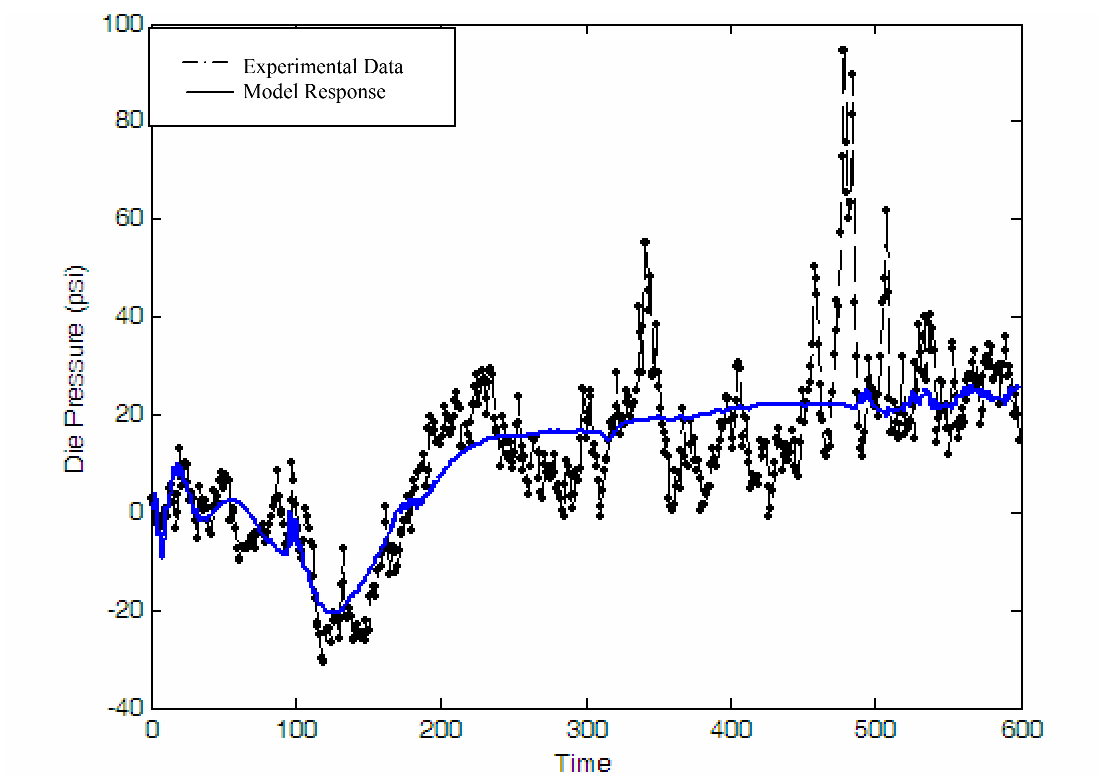

Fig. 7a: Die Pressure: Response to E/MAA feed-rate step change from 22 to 18 lbh

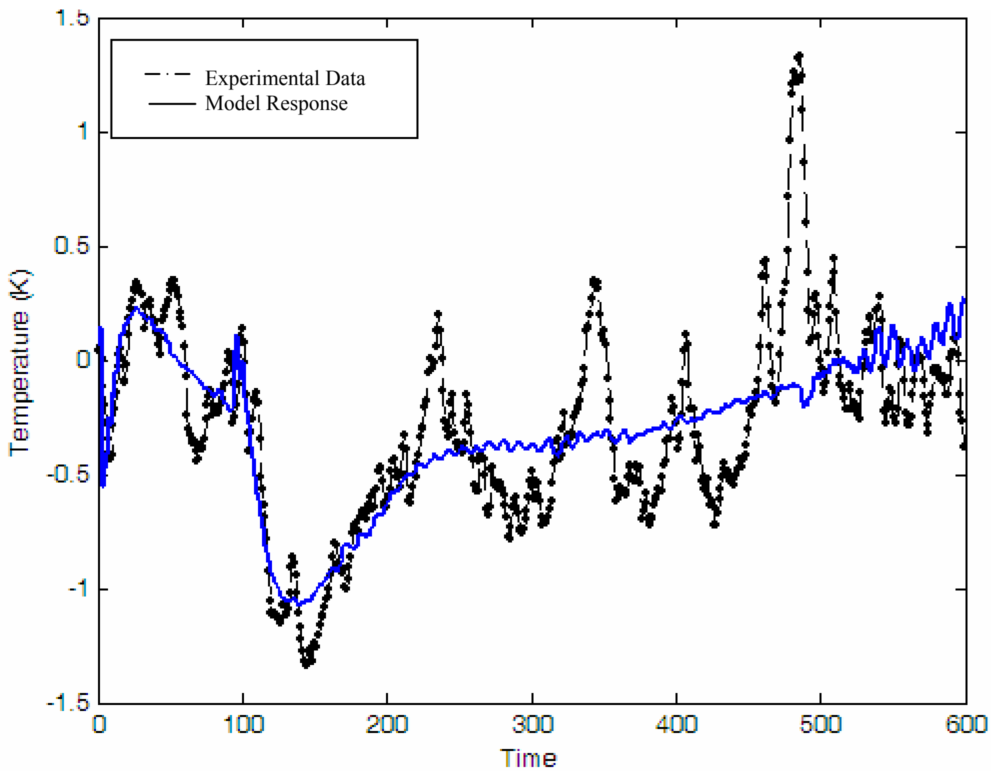

Fig. 7b: Exit Melt Temperature: Response to E/MAA feed-rate step change from 22 to 18 lbh

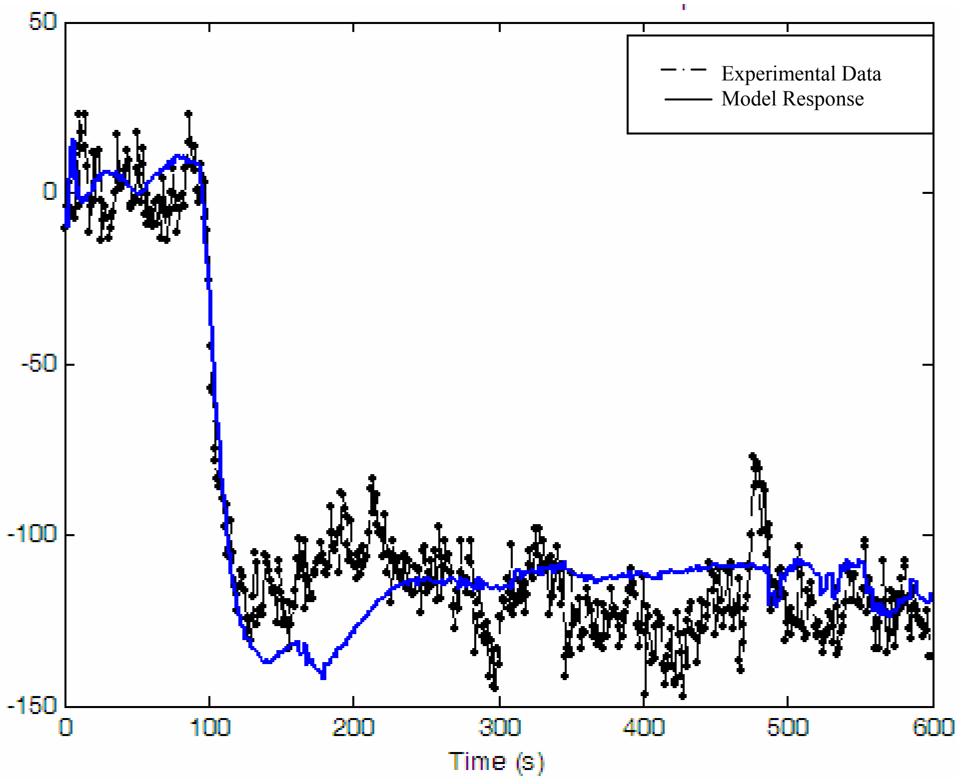

Fig. 7c: Motor Power: Response to E/MAA feed-rate step change from 22 to 18 lbh

6. Summary and Conclusions

A modeling framework has been proposed for effective control of the product end-use properties of a reactive extrusion process. The modeling framework consists of a network of models that mathematically represent the relationships between the different classes of variables across the manufacturing chain. For practical reasons, it was essential for this scheme to introduce an additional class of unmeasured variables called as internal quality variables. The proposed multivariable cascade control scheme will then use this network of models, in addition to all the available measurements, to guarantee acceptable end-use performance.

An empirical model that relates the manipulated inputs, u, and the process outputs, y, is an important component of the modeling scheme. A systematic two-stage procedure is implemented to obtain the input/output data used to develop this model; in the first stage, preliminary tests, such as step tests, are used to obtain *à priori* information, which is then used to design and implement the final identification tests in the second stage.

A multi-model structure consisting of the local linear models was a natural outcome of the preliminary test results. The Box-Jenkins parametric model structure was found suitable for local linear models. It should be noted, finally, that a non-linear model structure could have been used instead of the multi-model structure; however, this requires a costly and complicated test design. The multi-model structure, on the other hand, was obtained from relatively simple tests.

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References

1] Janssen L. P. B. M., “On the Stability of Reactive Extrusion”, *Polym. Eng. Sci.*, 38 (12), 2010 – 2019.

2] Pabedinskas, A.; Cluett, W. R., “Controller Design and Performance Analysis for a Reactive Extrusion Process”, *Polym. Eng. Sci.*, 34 (7), 585-597 (1994).

3] Wetzel M. D., Denelsbeck D. A., Latimer S. L.,Shih C-K, “A Perturbation Method to Characterize Reactive Extrusion”, *SPE ANTEC Tech. Papers*, T18, 3791, 2003.

4] Zhu Y. C., “Multivariable System Identification for Process Control”, Elsevier Science Ltd., Oxford, UK (2001).

5] Boukhris A., Mourot G., Âragot J, “Non-Linear Dynamic System Identification: A Multi-model Approach, *Int. J. Control*, 72 (7/8), 591-604 (1999).

6] Tullenken, H.J.A.F., “Generalized Binary Noise Test-signal Concept for Improved Identification Experiment Design”, *Automatica*, 26 (1), 37-49 (1990).