LINEAR GROWTH RATE BASED ADAPTIVE BATCH CRYSTALLIZATION CONTROL

Christopher D. Rhoten
Consultant, 318 Millbrae Loop, Hendersonville, NC 28791

Introduction

The ideal methodology for batch crystallization control has generally been focused towards measurement and control of supersaturation[1,2]. Such a control approach seeks to control the growth rate of the crystal via the control of supersaturation within the metastable zone for orderly crystal growth. The advent of reliable on-line mother liquor and massecuite concentration measurement has made possible, at least in theory, the realization of on-line supersaturation measurement and control.

However, in practice, this approach results in a rather complex computational and programming problem. In order to accurately measure and control supersaturation, a number of reliable and relatively accurate process inputs are required. Among the more important inputs are the purity and temperature of the mother liquor as well as the concentration of the solution provided by a refractometric measurement. While the refractometer will give reliable and relatively accurate measurement if it is properly calibrated it is, none-the-less, a "point measurement" and not necessarily representative of the entire contents of the pan. Mother liquor purity cannot easily be measured directly on line and thus, must be either estimated or input from relatively infrequent laboratory measurements that may themselves be subject to a certain degree of analytical error.

Temperature measurement is another significant problem due to the fact that in full scale production equipment, there is significant variation of temperature depending on the measurement location in the pan. Temperature is also affected by hydrostatic head as the pan increases in volume. In addition, it is relatively difficult to control absolute pressure at a precise pre-set value leading to additional variation in process temperature. Thus, it becomes a significant question as to what is the "correct" temperature to use as an input to the super-saturation based control program which, in turn affects the computed supersaturation value.

In the practical reality of batch crystallization, there exists a relatively broad supersaturation range within the boiling mass in a production pan. So, the question becomes one of speculative compromise as to the selection of the appropriate supersaturation value at which to control the process. This is not to imply that supersaturation control is not an acceptable control approach. Only that it is a relatively complex approach to such control with a number of possible sources of error.

Upon critical examination of the batch crystallization process as a whole, the ultimate goal is to manage crystal growth rate at a maximum value while assuring orderly crystal growth without the formation of secondary grain. Thus, it follows that, given the measurement and management of crystal growth rate, made possible through the use of simultaneous measurement of mother liquor and massecuite concentrations, it is possible to manage crystal growth rate directly without resorting to a relatively complex calculation of supersaturation. This is made possible through the use of the dual measurement of mother liquor (refractometric) and massecuite (microwave) concentrations. By measuring the actual crystal growth rate by differential concentration measurement and using an ideal growth rate algorithm for feed management and the control of mother liquor concentration, all other related control variables are automatically accounted for within the growth rate measurement and may essentially be ignored as process inputs to the control programming. Such an approach to batch pan control utilizing a single polynomial mass crystal growth rate algorithm results in a relatively simple and effective control solution. This paper describes the development, installation and operating results of such a control approach.

Process Modeling

Defining a proper control algorithm for crystal growth rate control depends on the construction of a suitable, linear growth rate based crystallization model incorporating linear growth rate, time, sucrose solubility and supersaturation inputs. Such a model was developed, an example of which is found in Appendix 1. The basic growth rate portion of model is shown in Figure 1.

Linear Growth Rate Model																						
Supersaturation		1.18	1.18	1.19	1.20	1.20	1.21	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.16	1.12	1.06
Elapsed Time: Minutes		5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85	90	95	100
Linear Rate/min	0.0063																				
Seed Crystal Size	0.0100	0.0413	0.0727	0.1040	0.1353	0.1666	0.1980	0.2293	0.2606	0.2919	0.3233	0.3546	0.3859	0.4172	0.4486	0.4799	0.5112	0.5425	0.5739	0.6052	0.6365
Crystal Number	5.06E+10																				
Wt Seed/Crystal, lb	0.2724	19	104	306	675	1,260	2,113	3,283	4,821	6,777	9,200	12,143	15,654	19,784	24,582	30,101	36,389	43,496	51,474	60,372	70,241
% Crystals by Weight		0.04	0.19	0.46	0.88	1.46	2.24	3.27	4.65	6.41	8.52	11.00	13.86	17.14	20.84	24.94	29.40	34.92	41.18	47.99	55.43

Figure 1

The basis of the growth rate control model is the selection of an appropriate linear crystal growth rate and crystal shape around which to develop the growth rate algorithm. In this model, a very simple cubic crystal shape is used with all three axis growing at a constant linear rate from a seed size of 10 microns to a selected final grain size. In addition, a time factor and supersaturation for the overall crystal growth is imposed within which the total increase in grain size was required to occur. To “adjust” the model to optimal growth rate, the linear growth rate / unit time is adjusted to achieve approximately 30% crystals by weight by the end of the batch feed cycle. Effectively,

these parameters define the mother liquor concentration at which the pan is controlled. While this is not a "perfect" model due to the choice of a "cubic" crystal shape, it is sufficiently accurate to define a suitable control algorithm for control of the process requiring only an adjustment in seed volume to fine tune the final grain size in practice.

The crystal content by weight calculated from the growth rate analysis is utilized to balance the overall batch control model and establish the relationship between the mother liquor and the massecuite concentrations during the feeding cycle of the pan operation. The results of this balance are shown in Figure 2. The difference between the mother liquor and massecuite concentrations relative to pan level is the basis for the growth rate based pan control algorithm. The calculated mother liquor concentration maintains a supersaturation of 1.2 for the duration of the feeding cycle.

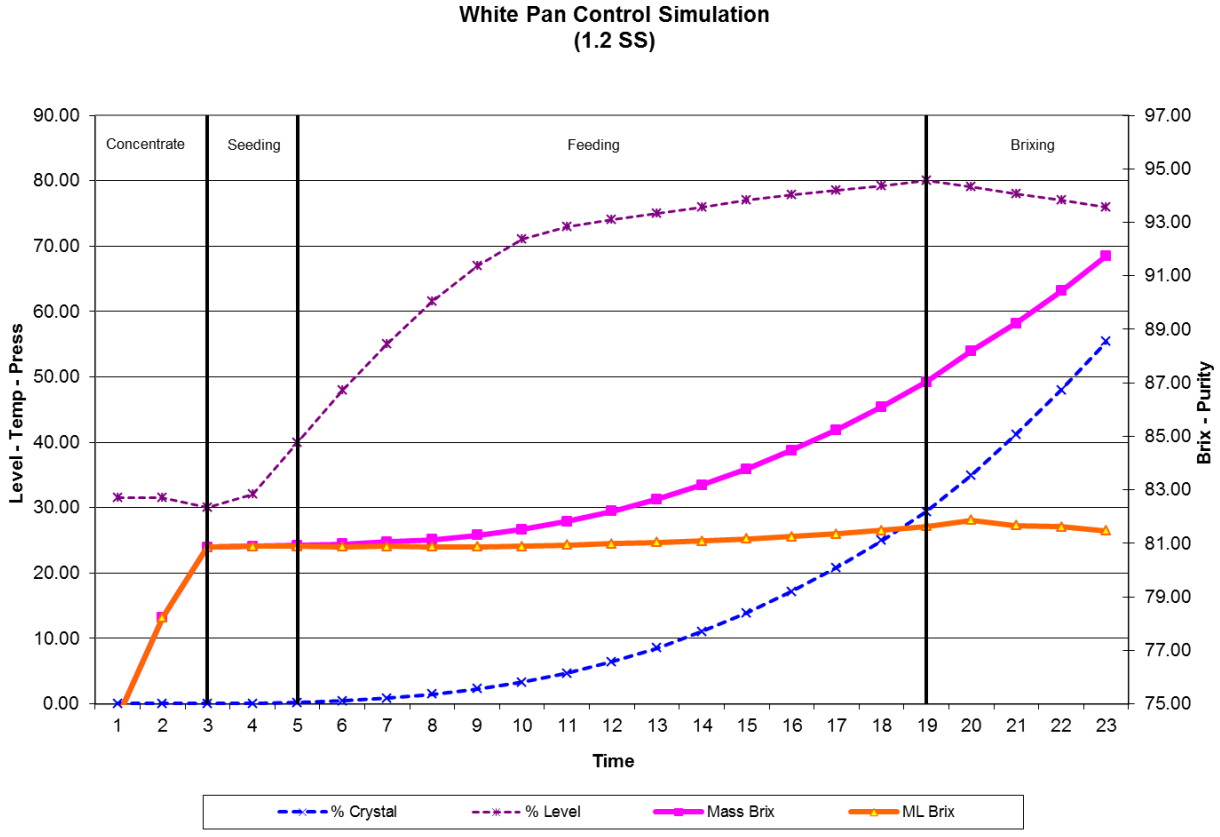

Figure 2

The plot of the difference in mother liquor and massecuite concentration vs pan level results in a 6th order polynomial distribution as shown in Figure 3 below. Regardless of the purity at which this relationship is calculated and plotted, within the normally encountered limits of white pan operation, the curves are effectively identical. The superimposed curves for 92, 93 and 94 purity standard liquor are shown in Figure 4. Thus, for any shift in the standard liquor feed purity only

an adjustment to the mother liquor concentration (refractometric set point) during the feed cycle of the batch pan cycle is required to compensate for any change in feed purity. Similarly, any change in absolute pressure, operating temperature, standard liquor feed concentration or operating steam pressure are observed as insitu effects on the crystal growth rate during the progress of the pan cycle and compensated for by the control algorithm and the continuous adjustment of the mother liquor concentration set point based on the control algorithm to maintain optimal crystal growth rate.

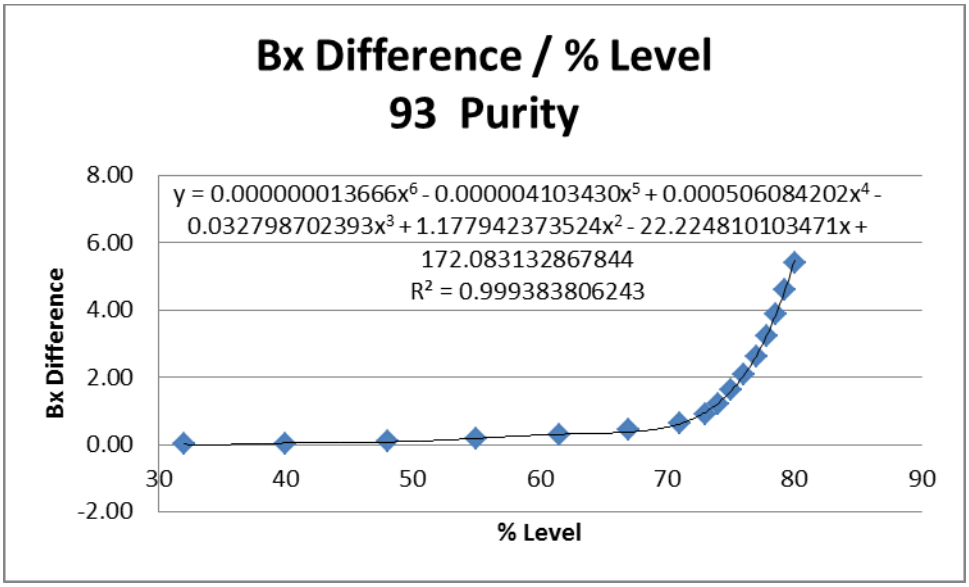

Figure 3

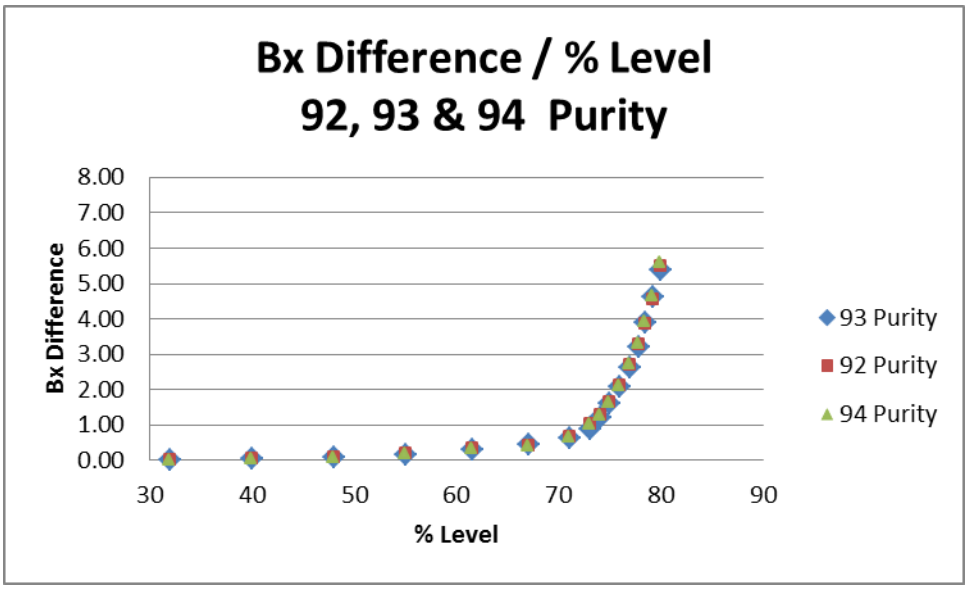

Figure 4

Pan Control Architecture

The control architecture for this control approach is quite simple. The output of the microwave signal (massecuite concentration) is monitored. The output of the control algorithm relative to pan level is subtracted from the microwave concentration output. The difference in the two values is the instantaneous set point for the refractometric (mother liquor concentration) based feed control to the pan. No other control inputs or outputs are required for the overall control of the feed cycle. Additional control loops for the control of absolute pressure and calandria steam pressure are independent control loops yet fully compensated for by virtue of the crystal growth rate based refractometric feed control.

As stated above, the basic control scheme for the boiling control consists of three independent control loops. One for absolute pressure control and a second for calandria steam pressure control. These two loops control the pan boiling temperature and rate of evaporation or speed at which the pan is operated. A third independent feed control loop is based on the growth rate algorithm and seeks to control the difference between the massecuite and the mother liquor concentration. The difference, relative to pan level, represents the crystal content in the strike at any given point and is managed by adjustment of the refractometric set point on a continuous basis during the course of the feed cycle. In turn, the feed rate to the pan is controlled via the refractometric based feed control loop to maintain the correct development of crystal content as the pan is fed to maximum volume.

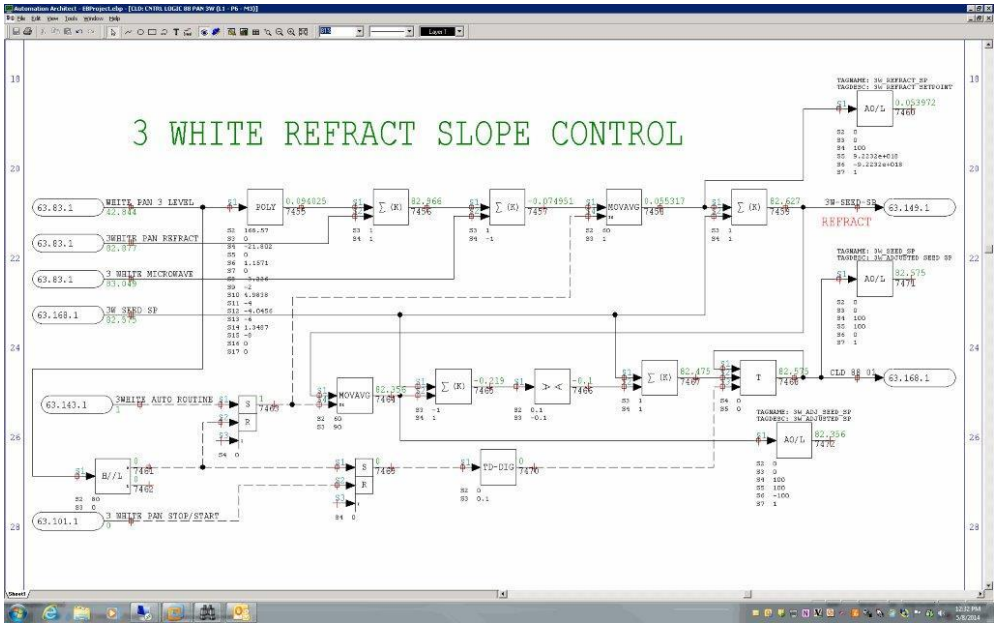

Figure 5

In addition to these three basic control loops there may also be certain process control safety features to insure that sensors are properly cleaned between each cycle, for the initiation of pan operation including establishing vacuum and charging, for seed point detection and alarming (or

automatic seeding), absolute pressure manipulation (sometimes required with high concentration and/or high purity feed syrup), for dropping and cleaning of the pan between cycles and for automatic seeding set point correction from strike to strike. Figure 5 is an example of the basic control programming for the feed control and the automatic updating of the seeding set point.

Calibration of Control Inputs

Of critical importance to the correct operation of any crystallization control system is the correct and consistent calibration of the refractometer and microwave instruments. Due to the nature of the control architecture, the refractometer and microwave instruments must give essentially identical output values at the seed point. The microwave instrument itself must also have a sufficiently accurate calibration over the course of the strike from the beginning to the end of the feed cycle.

To satisfy these requirements, a calibration procedure was developed to insure reproducible and relatively accurate calibration performance. The refractometer is bench calibrated at standard temperature using standard calibration oils for the density range to be encountered in a given batch crystallization cycle. For white, high purity, crystallization, three oils are utilized giving calibration points at 65, 75 and 85 brix. The microwave instrument is then calibrated to the output of the refractometer at approximately 75 brix (standard liquor concentration), the seed set point (approximately 82-83 brix) and at the final brix for the strike as measured by laboratory analysis of the final massecuite resulting in a three-point microwave instrument calibration. With each subsequent re-calibration of the instruments, the old calibration points previously entered into the instruments are discarded and replaced with the updated points.

Instrument calibration is generally performed on a monthly basis for all similarly equipped pans regardless of service. If it is necessary to perform interim re-calibration due to instrument failure or non-routine cleaning, the entire calibration procedure for both instruments is followed to insure proper calibration of both instruments relative to the current operating environment. With such a calibration regimen, few, if any, operational difficulties arise as a result of calibration issues or errors.

Operational Performance

The operational stability and repeatability of the control method described herein is extremely dependable and relatively immune to rather significant process variation normally affecting the batch crystallization process. In comparison to the refractometric based control system described in “An Innovative Approach to Self-Optimizing Batch Pan Control”,[3] presented at the 2013 37th Biennial ASSBT Conference, the control program described herein gives more consistent and improved control of MA and CV and is significantly more resistant to process variation and upset. Since installation and debugging of the current control program, the only ongoing

operational requirement has been the routine cleaning and calibration of the refractometer and microwave instruments.

The operational stability of the control approach is evident in the repeatability of the control outputs as shown in Figure 6. Note in the diagram the very consistent nature of the increase in massecuite concentration from strike to strike while the refractometric set point and output and

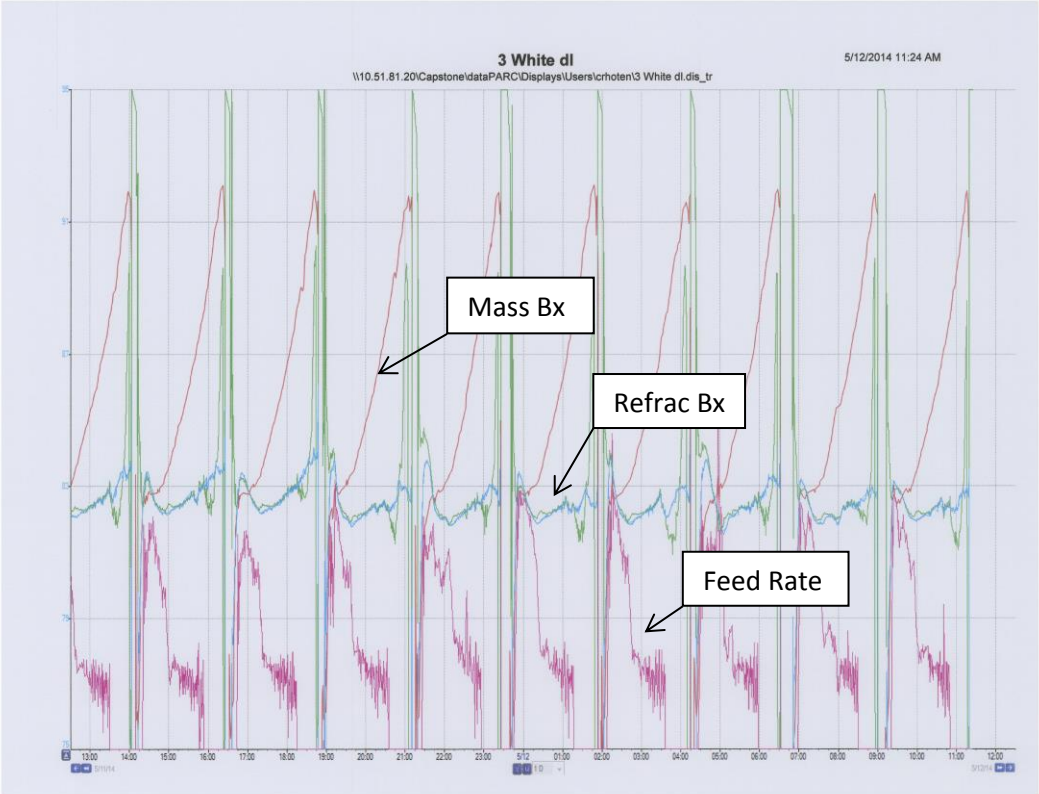

Figure 6

the feed rate (feed valve position) show greater variation. The variation in the refractometric output and feed rate are proportional to the variation in feed purity and concentration, absolute pressure (pan operating temperature) and calandria pressure, all of which are compensated for in the control algorithm by virtue of the direct measurement and control of crystal growth rate. The relatively simple continuous adjustment of the mother liquor refractometric concentration set point keeps the crystal growth rate constant for the actual operating conditions encountered during the course of the strike providing for a very consistent granulation quality from strike to strike.

An added feature in the control architecture provides for the automatic adjustment of the seed set point from strike to strike. This is accomplished by simply averaging the refractometric brix set

point variation during the course of the feed cycle and re-setting of the seed set point with this computed average on a strike to strike basis. The typical variation in the seed set point over a 30-day period is shown in Figure 7. The variation roughly tracks the minor changes in standard liquor purity but is also affected by variation in absolute pressure and minor errors in the microwave instrument calibration.

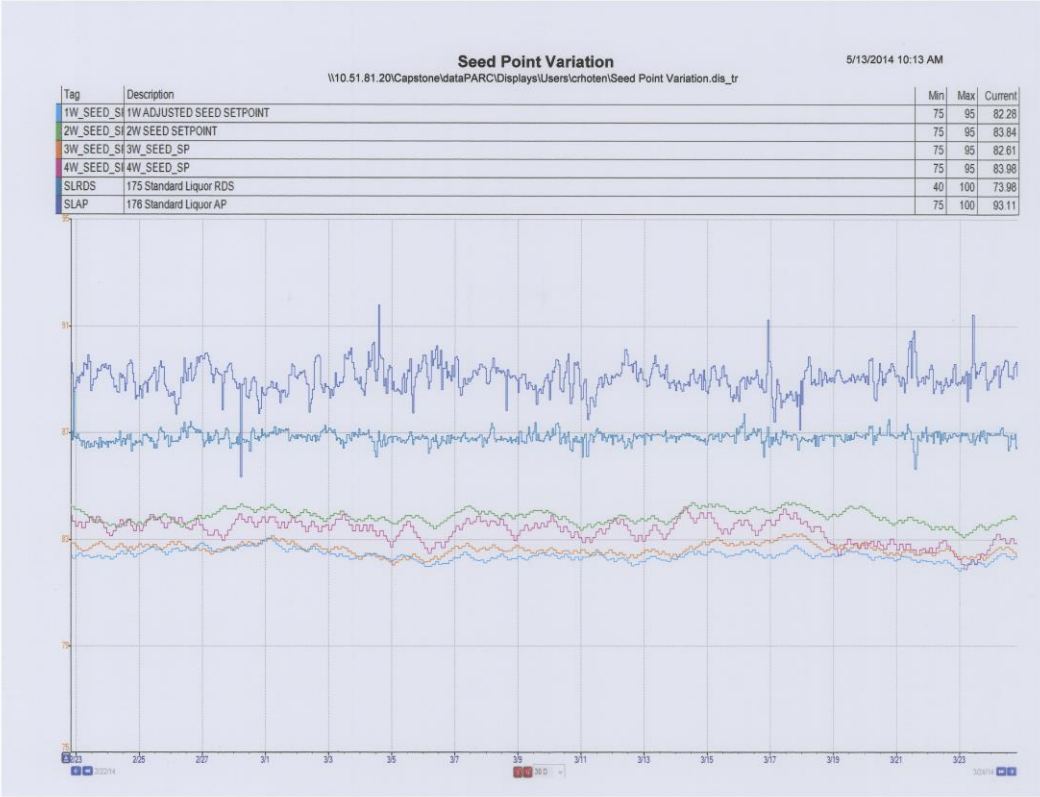

Figure 7

Effectively, this feature maintains the seed set point (starting point) for the next strike at the same conditions at which the previous strike was boiled. This adjusted seeding setpoint only serves to adjust the starting point to as close to current conditions as possible. Once the strike is seeded, the control adjusts the refractometric set point to the actual conditions of the current strike to achieve the calculated ideal crystal growth rate for the strike in progress.

As currently installed, operator interaction with the pan control is limited to initiation of the pan cycle, manual seeding of the strike at the alarmed automatically set seed point and dropping of the strike to the mixer upon reaching the final density for the prevailing massecuite purity conditions followed by preparation of the pan for the next batch cycle. Of course, all of these "manual" interactions may also be automated but what then would the pan men do besides read a good book and take credit for the excellent results while awaiting the rare but eventual equipment failure requiring his expert intervention to keep the process operating. It has been interesting to observe with this control scheme that rather than the control scheme attempting to

emulate what the pan men do, that the pan men now attempt to emulate what the control scheme does in terms of feed and concentration profile when there is a problem with a process sensor. In that respect, the control approach is a good "teacher" as well as a good "partner" to the pan men and their acceptance of it has been enthusiastic.

Comparative Results

Operational massecuite and production sugar quality are quite consistent with the LGR control strategy. The system has been in operation for more than two campaigns requiring only routine sensor calibration to maintain consistent operational output and quality. Compared to previous systems installed and tested, the current LGR system is giving more consistent and superior results as indicated in Figure 8. This data is based on individual MA and CV results from individual strikes over similar comparative time periods. Figure 9 shows the actual data for a typical 30-day period for the control scheme described herein.

Comparison of Control Performance

Average Control	MA	Std Dev	CV	Std Dev
LGR	395.7	17.8	28.9	1.0
Ref	398.1	22.1	29.3	1.0
MW	387.0	24.9	32.0	2.0

Figure 8

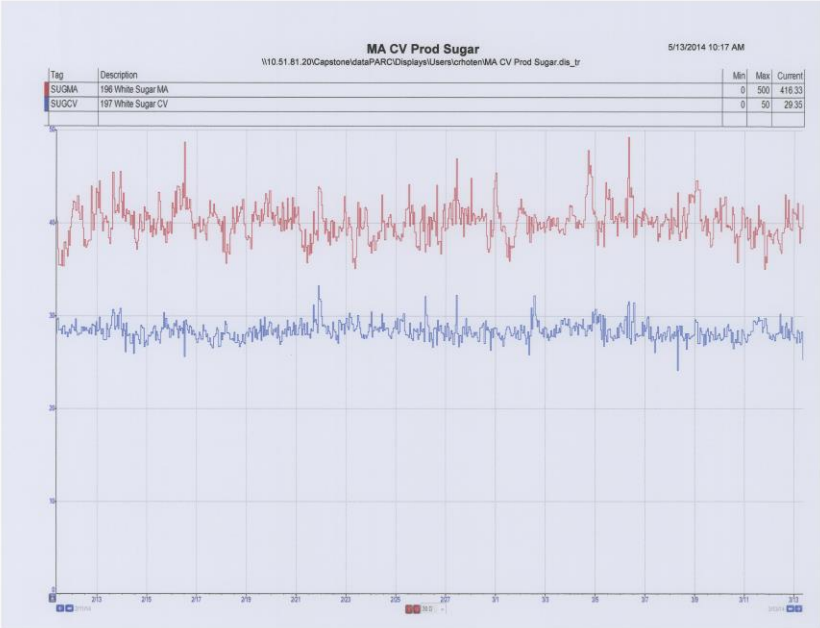

Figure 9

In Summary

- Linear Growth Rate (LGR) based batch pan control is a relatively simple control approach in comparison to relatively more complex supersaturation based control.
- LGR based control gives quite repeatable batch pan performance and is self-compensating for all variables affecting the batch crystallization process.
- For any dual concentration measurement control strategy, a suitable and repeatable methodology for routine instrument calibration is essential to the optimum operation of the control.
- Quality of massecuite and granulated sugar produced from LGR based batch pan control is excellent in terms of both product consistency and overall product quality.

Literature References

1. Lajos Rozsa, Ph.D., Giovanni M. Arriaza, Marco T. Romero: Advanced Control of Crystallization Based on the Direct Use of On-line Data on Supersaturation: Theory and Practice. SIT (May 12-15) Guangzhou, China.

2. Osama Z.El-abdein, Yassin M. Tmerek, Ibrahim D. Abdullah, Serif T. Amin: Design and Evaluation of a Full Control Program for Sucrose Crystallization Based on Soft Sensor Approach. SIT Paper #1026.

3. Christopher D. Rhoten: An Innovative Approach to Self-Optimizing Batch Pan Control. 37th Biennial ASSBT Conference, Anaheim, California.

White Pan Simulation

Seed Point

Pan Simulation: "Control"

		Concentration			Graining		Boiling Up														Final Brixing			
Process Inputs:		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23
	Temp	73.0	73.0	73.0	74.5	74.5	73.9	73.3	72.7	72.1	73.0	73.1	73.2	73.3	73.4	73.5	73.6	73.7	73.8	73.9	74.0	74.1	74.3	74.4
	Ws	77.14	77.14	77.14	77.48	77.48	77.34	77.21	77.08	76.95	77.14	77.17	77.19	77.21	77.23	77.26	77.28	77.30	77.32	77.34	77.37	77.39	77.43	77.45
	Z	3.3753	3.3753	3.3753	3.4397	3.4397	3.4138	3.3881	3.3627	3.3375	3.3753	3.3796	3.3838	3.3881	3.3923	3.3966	3.4009	3.4052	3.4095	3.4138	3.4181	3.4224	3.4310	3.4354
	SS	0.83	1.02	1.20	1.18	1.18	1.19	1.20	1.20	1.21	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.20	1.16	1.12	1.06
	Purity	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00	93.00
	Mass Brix	74.50	78.23	80.85	80.89	80.92	80.96	81.05	81.14	81.30	81.52	81.82	82.20	82.65	83.17	83.78	84.47	85.24	86.10	87.04	88.20	89.22	90.44	91.74
	Lbs/Gal	11.554																						
Calculated Values																								
	S	69.29	72.76	75.19	75.23	75.26	75.30	75.37	75.46	75.61	75.81	76.09	76.44	76.86	77.35	77.91	78.55	79.27	80.07	80.94	82.03	82.97	84.11	85.32
	N	5.22	5.48	5.66	5.66	5.66	5.67	5.67	5.68	5.69	5.71	5.73	5.75	5.79	5.82	5.86	5.91	5.97	6.03	6.09	6.17	6.25	6.33	6.42
	W	25.50	21.77	19.15	19.11	19.08	19.04	18.95	18.86	18.70	18.48	18.18	17.80	17.35	16.83	16.22	15.53	14.76	13.90	12.96	11.80	10.78	9.56	8.26
	C	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.97	0.98	0.98	0.99
	ML Purity	**93.00**	**93.00**	**93.00**	**93.00**	**92.98**	**92.96**	**92.92**	**92.87**	**92.80**	**92.71**	**92.58**	**92.41**	**92.20**	**91.93**	**91.61**	**91.22**	**90.73**	**90.15**	**89.43**	**88.41**	**87.00**	**85.09**	**82.32**
	ML Brix	74.50	78.23	80.85	80.88	80.88	80.88	80.88	80.86	80.87	80.89	80.93	80.98	81.03	81.09	81.17	81.25	81.36	81.48	81.64	81.87	81.67	81.61	81.47
	%Crystal	**0.00**	**0.00**	**0.00**	**0.04**	**0.19**	**0.46**	**0.88**	**1.46**	**2.24**	**3.27**	**4.65**	**6.41**	**8.52**	**11.00**	**13.86**	**17.14**	**20.84**	**24.94**	**29.40**	**34.92**	**41.18**	**47.99**	**55.43**
	% Crystn	0.00	0.00	0.00	0.06	0.25	0.61	1.16	1.94	2.97	4.32	6.11	8.39	11.09	14.23	17.79	21.82	26.29	31.15	36.32	42.58	49.63	57.06	64.97
Extrapolated Values																								
	%Level	31.5	31.5	30.0	32.0	40.0	48.0	55.0	61.5	67.0	71.0	73.0	74.0	75.0	76.0	77.0	77.8	78.5	79.2	80.0	79.0	78.0	77.0	76.0
	Stm Press	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0	13.0
	Lb/Ft3	85.83	87.34	88.42	88.43	88.45	88.46	88.50	88.54	88.60	88.69	88.82	88.98	89.17	89.39	89.64	89.93	90.26	90.63	91.03	91.53	91.97	92.50	93.07
	Lb DS/Ft3	64.19	68.55	71.67	71.72	71.76	71.81	71.91	72.03	72.22	72.48	72.85	73.31	73.85	74.49	75.23	76.08	77.04	78.11	79.28	80.74	82.03	83.59	85.26
	Cu. Ft.	662	662	630	672	840	1,008	1,155	1,292	1,407	1,491	1,533	1,554	1,575	1,596	1,617	1,634	1,649	1,663	1,680	1,659	1,638	1,617	1,596
	S (Total)	39,336	42,035	41,883	44,706	55,910	67,141	77,045	86,288	94,259	100,258	103,608	105,701	107,944	110,350	112,932	115,418	117,956	120,696	123,786	124,550	124,995	125,797	126,729
	S (ML)	39,335	42,035	41,882	44,687	55,806	66,835	76,369	85,027	92,145	96,975	98,788	98,923	98,743	98,208	97,278	95,634	93,374	90,595	87,397	81,054	73,521	65,426	56,488
	S (Crystal)	1	0	0	19	104	306	675	1,260	2,113	3,283	4,821	6,777	9,200	12,143	15,654	19,784	24,582	30,101	36,389	43,496	51,474	60,372	70,241
	N	2,961	3,164	3,152	3,365	4,208	5,054	5,799	6,495	7,095	7,546	7,798	7,956	8,125	8,306	8,500	8,687	8,878	9,085	9,317	9,375	9,408	9,469	9,539
	W	14,477	12,575	10,667	11,356	14,175	16,976	19,372	21,564	23,311	24,439	24,754	24,615	24,368	24,005	23,516	22,825	21,960	20,949	19,823	17,919	16,241	14,305	12,272
	Sum	56,774	57,774	55,702	59,427	74,294	89,170	102,216	114,346	124,665	132,243	136,161	138,271	140,436	142,661	144,948	146,930	148,794	150,730	152,926	151,844	150,645	149,571	148,539
	Bx Chk	74.50%	78.23%	80.85%	80.89%	80.92%	80.96%	81.05%	81.14%	81.30%	81.52%	81.82%	82.20%	82.65%	83.17%	83.78%	84.47%	85.24%	86.10%	87.04%	88.20%	89.22%	90.44%	91.74%

Linear Growth Rate Model

Linear Rate/min	0.0063																						
Seed Crystal Size	0.0100	0.0413	0.0727	0.1040	0.1353	0.1666	0.1980	0.2293	0.2606	0.2919	0.3233	0.3546	0.3859	0.4172	0.4486	0.4799	0.5112	0.5425	0.5739	0.6052	0.6365		
Crystal Number	5.06E+10																						
Wt Seed/Crystal, lb	0.2724	19	104	306	675	1,260	2,113	3,283	4,821	6,777	9,200	12,143	15,654	19,784	24,582	30,101	36,389	43,496	51,474	60,372	70,241		