DMAIC in Lithium-Ion-Battery Production

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Abstract DMAIC refers to a data-driven cycle of analysis steps used for improving, optimizing and stabilizing business processes with the steps *Define*, *Measure*, *Analyze*, *Improve*, and *Control* (DMAIC). In this paper, we will demonstrate the *Define*, *Measure*, and *Analyze* steps for the calendering step in Lithium-Ion-Battery Production. It appears that the measurement system is at least questionable. Moreover, we identified influential factors for thickness and porosity of the foil.

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Archives of Data Science, Series A
(Online First)
KIT Scientific Publishing
Vol. 4, No. 1, 2018

DOI 10.5445/KSP/1000085951/09
ISSN 2363-9881

1 Introduction and Overview

DMAIC refers to a data-driven cycle of analysis steps used for improving, optimizing and stabilizing business processes with the steps *Define*, *Measure*, *Analyze*, *Improve*, and *Control* (see Figure 1). *Define* identifies the problem, *Measure* produces measurements, *Analyze* looks for relationships between quality characteristics and influential factors, *Improve* utilizes methods like design of experiments for finding the optimal factor levels for improving the process, and *Control* implements tools for controlling whether the improved process works in a stable manner.

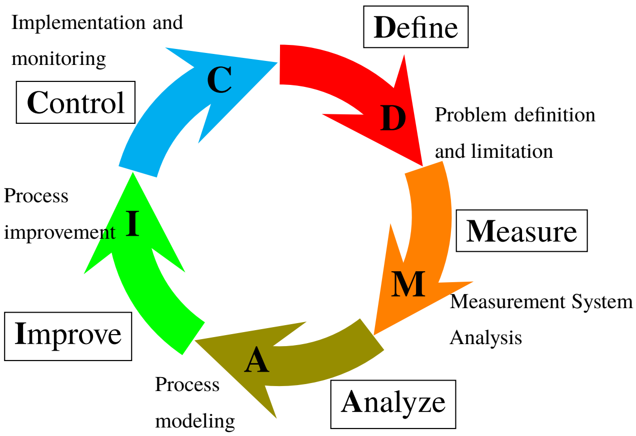


Figure 1: DMAIC cycle..

DMAIC is a combination of management methods and statistical tools. The following list gives examples for such methods (statistical methods *emphasized*):

- **Define**: VoC (Voice of the Costumer), VoB (Voice of the Business), SIPOC (Supplier, Inputs, Process, Outputs, Costumer), project charter, process map, *Pareto diagram*, Kano diagram, CTQ (Critical To Quality), RACI (Responsible, Accountable, Consulted, Informed) diagram, . . .
- **Measure**: *sampling, data exploration, process capability, control charts, Measurement System Analysis (MSA, Gauge R&R), . . .*

- **Analyze**: Ishikawa diagram, *uni-/multivariate data analysis, DoE (Design of Experiments for factor reduction), regression, statistical testing*, . . .
- **Improve**: NGT (Nominal Group Technique), FMEA (Failure Mode and Effects Analysis), Poka Yoke (error prevention), *DoE (for optimization), regression*, pilot study, . . .
- **Control**: *control charts, process capability*, process documentation, . . .

For more information on the approach and the methods see, e.g., Pyzdek and Keller (2014). In this paper, we will demonstrate such methods for one step in Lithium-Ion-Battery production (cp., e.g., Siemens (2017)).

2 DMAIC: *Define*

In the *Define* step we aim to understand the problem and define the exact task.

2.1 Project QS-Zell in the ProZell-Cluster

Figure 2: Coating Line (Source: ZSW)..

The *ProZell* Research Cluster is financed by the *Federal Ministry of Education and Research* (BMBF), founded to pool the know-how of the leading battery research facilities in Germany. The goal is to build a basis for the establishment of a competitive state-of-the-art Lithium-Ion-Battery production. In ProZell, *QS-Zell* is the project for Quality Assurance. The core of QS-Zell is the research production line (Forschungs-Plattform, FPL) at the Zentrum für Sonnenenergie- und Wasserstoff-Forschung Baden-Württemberg (Center for Solar Energy and Hydrogen Research, ZSW) (see Figure 2 for the coating part of the FPL). Finally, the optimized process steps implemented on the FPL should be used as a blue print for industrial production lines.

2.2 The Battery Production Process

The production of Lithium-Ion-Batteries can be divided into 4 major production stages which again consist of several minor sub-processes:

1. The production of coated foils that serve as electrodes (anode and cathode).
2. The casing of the coiled foils in a metal box.
3. The filling of the battery case with an electrolyte.
4. The formation (first charge and discharge) of the battery cell.

2.3 Statistical Quality Management in ProZell

In the Research Cluster, statistical tasks are the analysis and modeling of single process steps of the production chain, the modeling of the whole process chain including the development of quality uncertainty along the chain, and the development and implementation of statistical quality control. The main goal is to better understand and improve the production process.

2.4 The Calendering Process

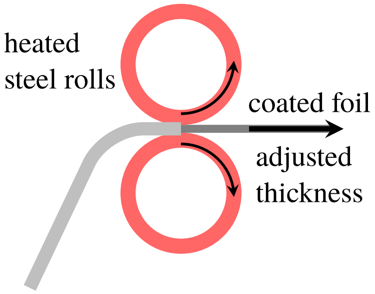


Figure 3: Calendering scheme.

Let us look more closely into the calendering process of the already coated foils in order to *define* the task: Calendering starts after the slurry is applied to the foil. The slurry needs to be compressed in order to adjust its porosity (cp. Figure 3).

Porosity influences the ions' ability to move through the slurry and the anode's ability to store these ions after charging. Thus, porosity of the coat influences the electrochemical characteristics of the final battery, like loading capacity and possible loading rate (C-rate). The following *Project Schedule* was defined:

1. *Measuring the quality before optimization* of the coated foil, in particular validation of relationship between coat thickness (non-destructive measurement) and porosity (destructive). However, thickness before calendering was not tracked, yet.
2. *DoE* for identifying important process factors for thickness/porosity and finding optimal factor levels to reduce waste.
3. *Measuring quality after optimization* of the coated foil.
4. Defining a baseline for industrial production.
5. *Process control* after optimization to ensure constant quality levels.

3 DMAIC: *Measure*

In the *Measure* step we determine how to measure quality characteristics and analyze the quality of the measurement system.

3.1 Data

Porosity data is obtained by destructive measurement after calendering. Thickness is measured on-line at three points of the foil (left/center/right). Since measurement of porosity is destructive and time consuming, we analyze thickness as a substitute since it is regarded as highly correlated with porosity. One thickness measurement per second is obtained for each position. The foil is cut in the middle after calendering.

3.2 Measurement System Analysis

For Measurement System Analysis (MSA), open questions are how to get an idea of thickness *before* calendering and how to do MSA? MSA characterizes reproducibility and repeatability (*R & R*). For *reproducibility*, note that there is only one machine and one user for the calendering process, but three measurement positions. For *repeatability*, note that variation in time should be small. For the proposed MSA procedure the coated foil is sent through the calender with only the thickness gauge activated, but no compression(!). This step is repeated 3 times and results are compared.

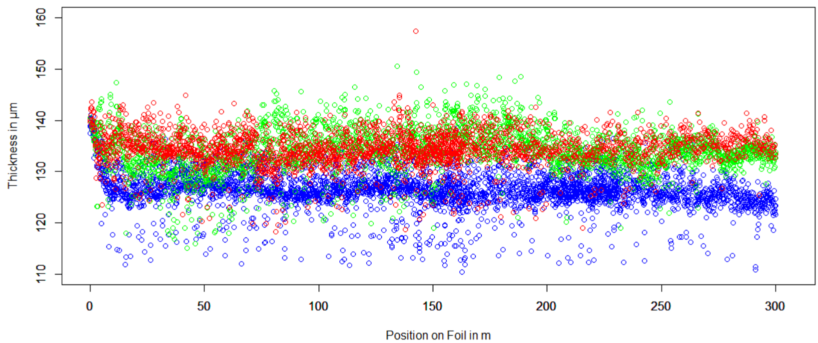


Figure 4: Thickness measurement results of coated foil from 3 runs (Data: ZSW).

Results of the 3 runs are pretty different (especially results marked in blue, see Figure 4). One problem is the matching of single measurements since the measurement position is not tracked and starting/ending points are unknown. However, speed of foil is measured every second. Thus, we identify the starting point and use the speed to match data.

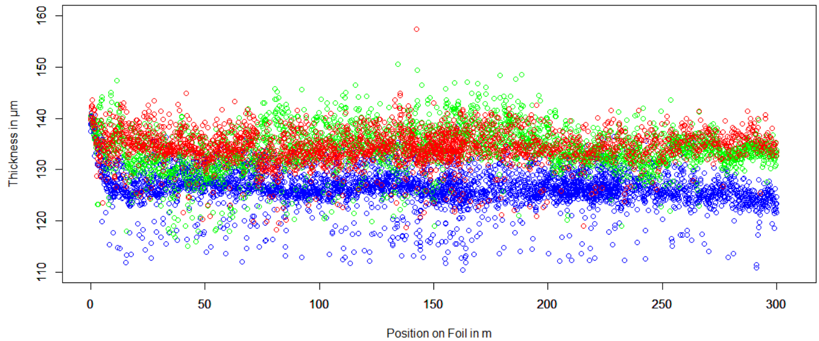
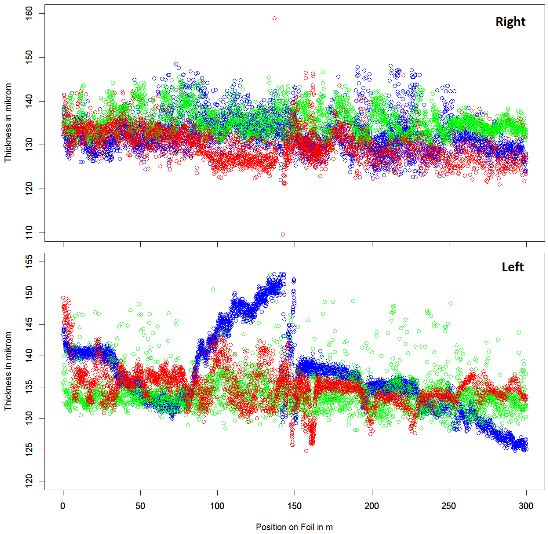


Figure 5: Transformed thickness measurements (**center**) of coated foil from 3 runs.

However, even after transformation, results are pretty different for the center position (see Figure 5). Thickness measurements of the first run (blue) are systematically lower. Also, there are differences in the variation of the runs. Since the foil is cut in the middle after calendering the left and right position should be compared in greater detail. As we know from an older result, the right position is traditionally subject to greater uncertainty. This also seems to be true in our case (bigger standard deviation (stdev), see Figure 6). Moreover, the differences between the runs are quite large for both positions. This again is a bigger problem on the right side with the standard deviation of the 3rd run (red) being more then 4 times larger than the others.



Mean	Left	Right
blue	137.20	132.40
green	134.20	135.40
red	135.10	127.70

Std.Dev.	Left	Right
blue	6.04	4.03
green	3.16	3.19
red	3.31	17.20

Figure 6: Transformed thickness measurements (**Right, Left**) and corresponding means, standard deviations (stdev).

Let us finish MSA by a formal analysis of variance (*anova*). For this, we determine the influence of the factors *position* with levels *left, center, right* and *parts* with levels corresponding to 60 consecutive measurement regions on the foil. As can be seen in Table 1 an *anova* on the mean measurements in these regions for the 3 above replicates shows that *position* has a (highly significant) effect which causes, together with the replicates, $> 99\%$ of the standard deviation of thickness. So, the difference between the measurements in the 3 positions is much too high to be acceptable, i.e. either the measurement system has to be improved or calendering systematically does not lead to the same thickness in the 3 positions which does not appear to be acceptable either.

Table 1: *Anova* for Parameters *Position* and *Parts*.

	position	*parts*	Residuals
Sum of Squares	74208.7	6684.1	1087987.6
Deg. of Freedom	2	1	7748

4 DMAIC: *Analyze*

Table 2: Factor Levels.

Abbr.	Parameter	Levels		
RT	Roller Temperature in °C	30	40	50
IR	IR-pre-heating Temp. in °C	24	70	110
RG	Roller Gap in mm	100		120
FS	Foil Speed in m/min	4		6
FT	Foil Tension in Newton	40		60

In the *Analyze* step, we try to find relationships between quality characteristics and influential factors. Before creating a DoE, possibly important process factors and their settings were identified in cooperation with ZSW researchers (see Table 2).

For *DoE* we identified 3 factors with 2 levels (linear effect over *Region of Interest (RoI)*) and 2 factors with 3 levels (possible quadratic effect over RoI). Because of limited amount of material, no interactions between factors were considered. For the construction and analysis of the DoE, the levels of the parameters were coded to -1 ("low"), 0 ("mean") and 1 ("high").

To construct the DoE, we split each 3-level factor X into 2 *surrogate factors* X_1 and X_2 with 2 levels each (Montgomery, 2017, Chapter 9.4.1, pp. 422), where $X = 1$ implies $X_1 = X_2 = 1$, $X = 0$ implies $X_1 \neq X_2$, and $X = -1$ implies $X_1 = X_2 = -1$. Then, the sum of the effects of X_1 and X_2 equals the effect of X and the product (interaction) of the effects of X_1 and X_2 the effect of X^2. A full factorial design with only the surrogate factors is created and the unused columns are utilized for the 2-level factors. If there are not enough columns left, the full factorial is expanded. The advantage of a plan designed in this manner against others like D-optimal plans is that all (including linear and quadratic) effects are uncorrelated. This was an issue of importance for our project partner, because they felt that being able to identify the impact of the process factors would be of value in further research and optimization projects.

Table 3: Full Factorial Design with Surrogates.

RT_1	RT_2	IR_1	IR_2	RT_1RT_2 RT^2	IR_1IR_2 IR^2	RT_1IR_1	RT_1IR_2	RT_2IR_1	RT_2IR_2	$RT_1RT_2IR_1$	$RT_1IR_1IR_2$ RG	$RT_2IR_1IR_2$ FS	$RT_1RT_2IR_1IR_2$ FT
-1	-1	-1	-1	+1	+1	+1	+1	+1	+1	-1	-1	-1	+1
-1	-1	-1	+1	+1	-1	+1	-1	+1	-1	-1	+1	+1	-1
-1	-1	+1	-1	+1	-1	-1	+1	-1	+1	+1	+1	+1	-1
-1	-1	+1	+1	+1	+1	-1	-1	-1	-1	+1	-1	-1	+1
-1	+1	-1	-1	-1	+1	+1	+1	-1	-1	+1	-1	+1	-1
-1	+1	-1	+1	-1	-1	+1	-1	-1	+1	+1	+1	-1	+1
-1	+1	+1	-1	-1	-1	-1	+1	+1	-1	-1	+1	-1	+1
-1	+1	+1	+1	-1	+1	-1	-1	+1	+1	-1	-1	+1	-1
+1	-1	-1	-1	-1	+1	-1	-1	+1	+1	+1	+1	-1	-1
+1	-1	-1	+1	-1	-1	-1	+1	+1	-1	+1	-1	+1	+1
+1	-1	+1	-1	-1	-1	+1	-1	-1	+1	-1	-1	+1	+1
+1	-1	+1	+1	-1	+1	+1	+1	-1	-1	-1	+1	-1	-1
+1	+1	-1	-1	+1	+1	-1	-1	-1	-1	-1	+1	+1	+1
+1	+1	-1	+1	+1	-1	-1	+1	-1	+1	-1	-1	-1	-1
+1	+1	+1	-1	+1	-1	+1	-1	+1	-1	+1	-1	-1	-1
+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1	+1

However, since the amount of material available for our experiments was highly limited, we needed to find a way to reduce the number of experiments if possible. Considering the full factorial 2^4 design for the surrogates including the remaining 2-level factors (see Table 3), 4 level combinations exist twice (only the signs of the surrogates IR1 and IR2 are interchanged, e.g., in experiments 2 and 3). Therefore, the number of experiments was reduced from 16 to 12 by leaving out experiments 3, 6, 11, and 15. This left us with a still nearly D-optimal design and also gave us the opportunity to repeat the 4 experiments in question at the end if there was still enough material left.

Table 4: First Design.

No.	**RT**	**IR**	**RG**	**FS**	**FT**
1	-1	-1	-1	-1	+1
2	-1	0	+1	+1	-1
3	-1	+1	-1	-1	+1
4	0	-1	-1	+1	-1
5	0	-1	+1	-1	-1
6	0	0	-1	+1	+1
7	0	+1	+1	-1	-1
8	0	+1	-1	+1	-1
9	0	0	+1	-1	+1
10	+1	0	-1	-1	-1
11	+1	-1	+1	+1	+1
12	+1	+1	+1	+1	+1

For practical reasons, the Roller Temperature was not randomized. Therefore, the surrogate design leads to the experiments in Table 4.

For *Data Collection*, choose the correct level combination, start the machine and wait until the previous foil has passed thickness measurement (depending on speed). Wait 30 more seconds for the process to adjust (just to be sure). Mark the foil right before the thickness measurement unit. And collect thickness data for 60 seconds (around 50 measurements for each level combination).

The first 5 experiments went through without major problems. However, the foil started to rupture in experiment number 6. Such a problem never occurred before with other types of foils. After several attempts, in order not to waste more material, the experiments were stopped. The maximum foil tension (FT) appeared to be too high. A new DoE had to be constructed.

Table 5: Second Design.

No.	**RT**	**IR**	**RG**	**FS**
1	-1	0	+1	+1
2	0	-1	-1	+1
3	0	-1	+1	-1
4	0	0	-1	+1
5	0	0	+1	-1
6	+1	0	-1	-1
7	+1	-1	+1	+1
8	-1	-1	-1	-1

By the first DoE, a lot of material was already used/lost. Running 12 new experiments appeared to be impossible. Therefore, the idea was to reduce the number of experiments needed and incorporate old experiments in the new design. This is realized by excluding foil tension as an influential factor fixing its level to -1 and by reducing the number of levels for IR-temperature to 2. The new DoE was constructed in the same manner as the first one, but needed only 8 experiments (see Table 5), 3 of which match experiments already performed (marked in red). This means that all considered factors, including the quadratic effect of the roller temperature (RT^2), can be estimated without any correlations. With the remaining material we were barely able to run the 5 new experiments needed.

Table 6: Linear Model for the Left Part of the Foil.

	Estimate	Std. Err.	t-value	p-value
Intercept	101.89	0.25	403.08	< 0.001
IR	1.45	0.25	5.72	0.029
RG	7.73	0.25	30.56	0.001
FS	0.86	0.25	3.38	0.077
RT^2	−1.15	0.25	−4.56	0.045
RT	−3.41	0.25	−9.55	0.011

$RMSE$	0.7149 on 2 degrees of freedom
R^2	0.9982

Regression is applied on the means of the observations for each level combination. In the model for the left part of the foil (see Table 6), all factors are significant. For all three models, i.e. for the left/center/right parts of the foil, the fit is > 0.99. Note that such a high goodness of fit is facilitated by using observation means. Nevertheless, linear dependence on the selected features is impressively shown. Moreover, coefficient signs are the same for all factors in all the thickness models and all signs make sense. However, only factors RG and RT are significant for the center and the right part of the foil, mean thickness is significantly higher in the center, and variation is lower on the left.

4.1 Models for Thickness and Porosity

Comparing the results for thickness and porosity (see Table 7) in the center of the foil, the same factors are identified as significant and thickness appears to be a good surrogate for porosity, which is even harder to predict (lower R^2).

5 DMAIC: *Improve* - What to do next

Up to now, we considered the *Define*, *Measure*, and *Analyze* DMAIC steps. In the next steps, in order to improve the process, first the measurement system has to be adapted to avoid different results in different positions (left/center/right). Moreover, optimal factor levels are to be found for future production. Also,

Table 7: Linear Model of Porosity (Left) and Thickness (Right) for the Center.

	Estimate	Std. Err.	t-value	p-value	Estimate	Std. Err.	t-value	p-value
Intercept	41.50	0.37	111.09	< 0.001	104.71	0.51	207.28	< 0.001
IR	0.61	0.37	1.63	0.245	1.00	0.51	1.97	0.187
RG	3.59	0.37	9.61	0.011	7.53	0.51	14.91	0.004
FS	−0.05	0.37	−0.12	0.915	0.78	0.51	1.55	0.261
RT^2	0.12	0.37	0.33	0.774	−1.11	0.51	−2.19	0.160
RT	−2.17	0.53	−4.11	0.054	−3.16	0.71	−4.42	0.047
RSE	1.056 on 2 degrees of freedom				1.429 on 2 degrees of freedom			
R^2	0.9824				0.9922			

optimized process results have to be compared with the present ones and specification limits for thickness and porosity have to fixed.

Acknowledgements The authors thank the German Federal Ministry of Education and Research (BMBF) under the Grant 03XP0076B (project QS-Zell) for financial support. The authors also thank Stefan Rößler, Stefan Mähr, Hai Yen Tran and Wolfgang Braunwarth (ZSW) for realizing the experiments and for fruitful discussions in analyzing the data.

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