The Role of Process Characterization in Process Validation

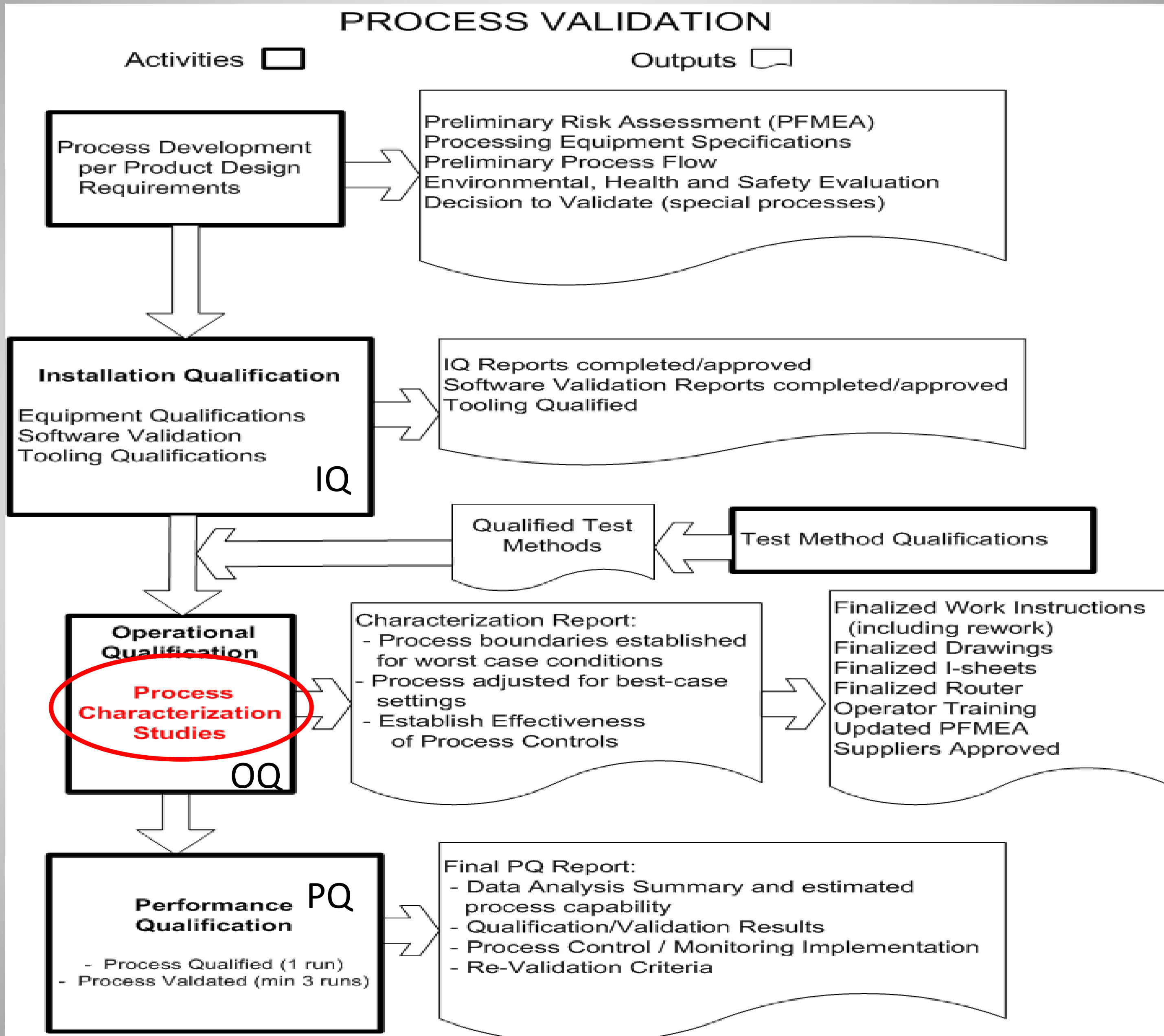
Speaker

David A. Goodrich, P.E., CQE, CQA

Presented at
Greater Fort Worth ASQ Section 1416
2016 Cowtown Quality Roundup
22 April 2016

Process Validation
Establishing by objective evidence that a process *consistently* produces a result or product meeting its predetermined specifications.

Process Characterization - Identifying and quantifying all significant sources of variation, especially characterization of variation inherent to the materials and technology as applied to the specific product design.


PROCESS VALIDATION
Activities
Outputs
Process Development per Product Design Requirements
Preliminary Risk Assessment (PFMEA)
Processing Equipment Specifications
Preliminary Process Flow
Environmental, Health and Safety Evaluation
Decision to Validate (special processes)
Installation Qualification
Equipment Qualifications
Software Validation
Tooling Qualifications
IQ
IQ Reports completed/approved
Software Validation Reports completed/approved
Tooling Qualified
Qualified Test Methods
Test Method Qualifications
Operational Qualification
Process Characterization Studies
OQ
Characterization Report:
- Process boundaries established for worst case conditions
- Process adjusted for best-case settings
- Establish Effectiveness of Process Controls
Finalized Work Instructions (including rework)
Finalized Drawings
Finalized I-sheets
Finalized Router
Operator Training
Updated PFMEA
Suppliers Approved
Performance Qualification
PQ
- Process Qualified (1 run)
- Process Valdated (min 3 runs)
Final PQ Report:
- Data Analysis Summary and estimated process capability
- Qualification/Validation Results
- Process Control / Monitoring Implementation
- Re-Validation Criteria

Characterization studies determine what happens when conditions occur which stress the process. These conditions are considered the "most challenging" or worst-case.

Must be completed prior to qualification testing, for processes chosen to validate.

Worst-Case

A set of process settings and conditions encompassing upper and lower processing limits. These settings pose the greatest chance of process or product failure when compared to ideal conditions. Such conditions do not necessarily induce product or process failure.

When Should We Begin Characterization?

Pre-Characterization Requirements:

- Product and Process Specifications
- Equipment Installation Qualifications
- Software Validations (as applicable)
- Process Risk Assessment Complete
- Characteristics of materials verified to meet requirements
- Applicable test methods qualified

Characterization may use pre-production work instructions.

When is a Characterization Study not Required?

Transferring a Mature (Robust) Process to Another Facility –

When operating limits are established at the developing or transferring facility, the decision may be made to test the operating limits, eliminating the need for a process characterization study.

Replicating a Process with Identical Equipment –

When operating limits are established, the decision may be made to test the operating limits, eliminating the need for a process characterization study (usually done when several identical machines are being validated at one time). Must demonstrate consistency between processes.

The amount of qualification and validation testing required should be determined based on risk assessment.

Look for Potential Sources of Process Variation

- Equipment
- Process
- Materials
- Measurement
- People
- Environment

Process Characterization Studies should identify and control the sources that can affect the process

What are the effects of ***materials*** variation on the process?

Some Common Examples:

- Critical Component dimensions - MMC, LMC
- Material composition / hardness range
- Component variation from multiple mold cavities
- Cleanliness / Residual Process Materials
  - Mold release agents
  - Lubricants
  - Particulate
- "Fresh" material vs. near end of shelf life
- ***May require advance planning to procure materials for characterization activities ($$$ well spent!)***

Risk Analysis

Process Failure Modes and Effects Analysis (PFMEA)

Identifies sources of process variability affecting essential design requirements, and ***scores potential critical and key process parameters***.

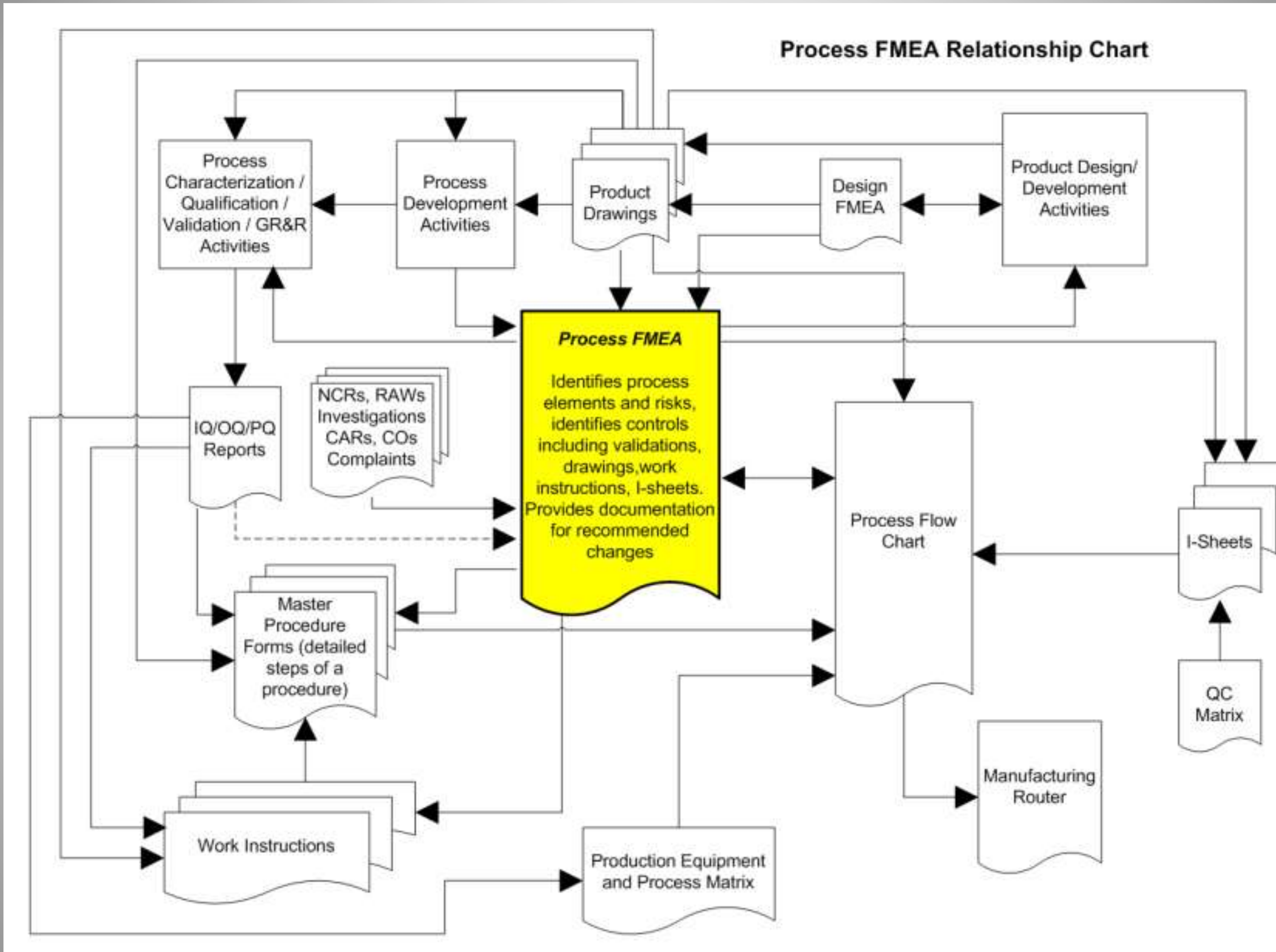
Failures are prioritized according to how serious their consequences are **(Severity),** how frequently they occur **(Occurrence)** and how easily they can be detected **(Detection).**

Calculate the **risk priority number,** or **RPN,** which equals **S × O × D**

The purpose of the FMEA is to take actions to eliminate or reduce failures, starting with the highest-priority ones.

Provides a rationale for risk-based confidence and reliability requirements (and corresponding sample size requirements)

http://asq.org/learn-about-quality/process-analysis-tools/overview/fmea.html



David A. Goodrich, P.E.

Risk-based Confidence and Reliability Requirements

- Assign a Risk Rating from the PFMEA Risk Priority Number (RPN)
  - Example of typical Risk Ratings and associated RPNs (based on a 1 to 10 scale for severity, occurrence and detection)
    - Low Risk (RPN 1- 300)
    - Medium Risk (RPN 301-600)
    - High Risk (RPN 601 – 1000)
- Confidence and Reliability "Requirements" (this may vary depending upon type of risks associated with the product, industry, etc.)

Low Risk	95% Confidence / 90 % Reliability
Medium Risk	95% Confidence / 95 % Reliability
High Risk	99% Confidence / 99% Reliability

Process Characterization
Which process parameters should we study?

- Only the parameters thought to be critical should be included in the characterization.

- Start with the essential design requirements and study the process parameters that deliver those.

- **Design of Experiments (DOE)** is a recommended tool for process characterization. The power of the DOE tool can identify significant variation factors, and their interaction(s), that impact process performance, product performance, and quality.

Design of Experiments (DOE)

DOE is an efficient and effective method for quantifying process factors and testing the limits of the process and/or technology. **Factorial DOE** provides the objective evidence of interaction factors and the **Response Surface Method (RSM)** can be used to predict both Worst-Case, as well as best case or optimum conditions or settings.

The output of DOE can include a transfer function, or mathematical model of process behavior useful in describing process performance and understanding relationships between various process factors.

Design of Experiments (DOE)

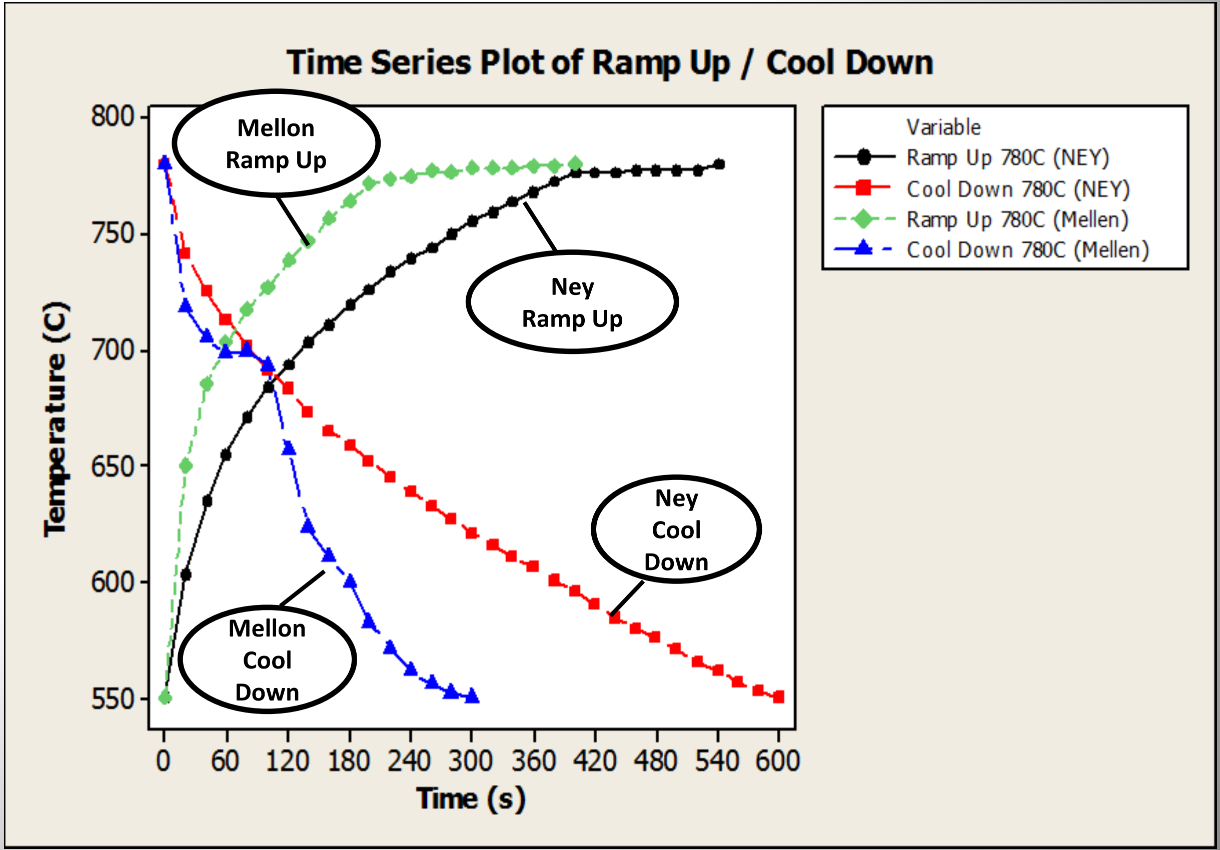
- Identify the factors that could impact the process
- Determine what levels of each factor to evaluate
- Design the experiment
- Run the experiment
- Analyze the results

Case Study 1: Kiln-Fired Ceramic Coating Process

Existing Production Process

- Poor Process Yield (coating thickness variation, pits and bubbles, poor adhesion)
- High Rework Level (re-coat, re-fire, re-inspect)
- High Reject Rate (rework largely not effective)

Initial review of process identified considerable process variability

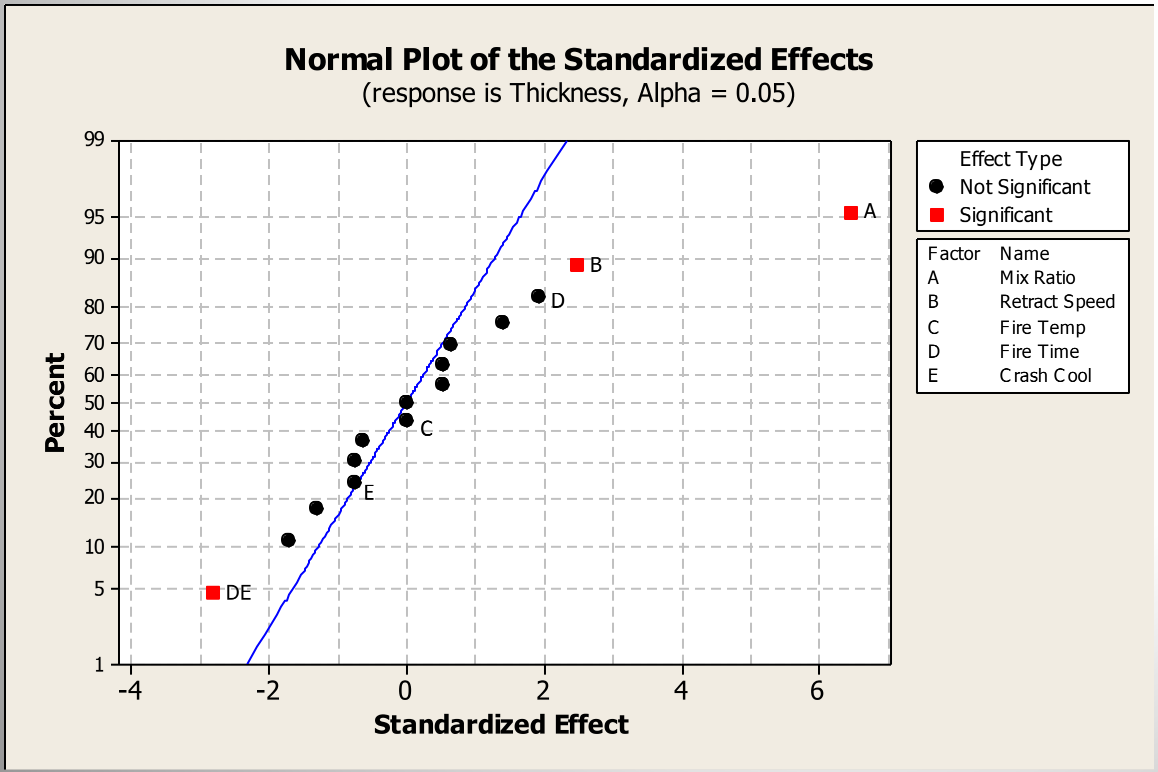


Case Study 1: Kiln-Fired Ceramic Coating Process

Design of Experiments – Coating Thickness

Factorial DOE using the Existing Process to Identify Significant and Non-significant Variables and Interactions

Design-Expert Output



Stepwise Regression:

Alpha-to-Enter: 0.15 Alpha-to-Remove: 0.15

Response is Thickness on 5 predictors, with N = 32

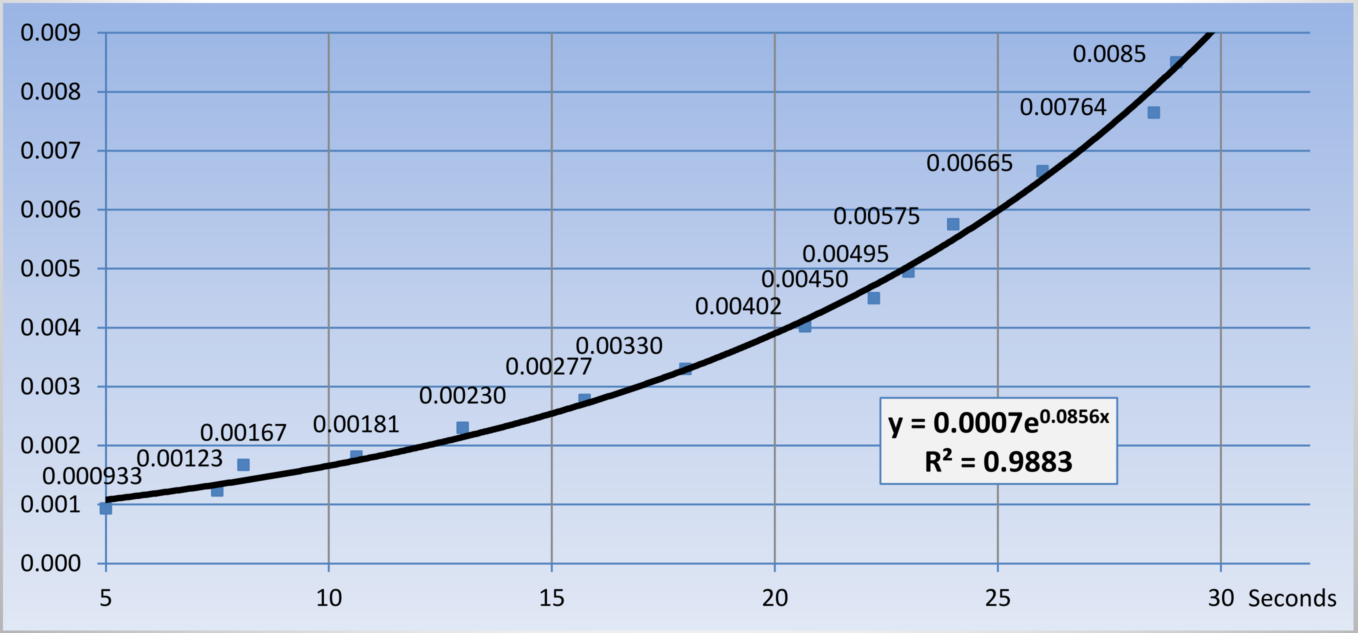
Step	1	2	3
Constant	-0.03184	-0.03472	-0.03753
Mix Ratio	0.00075	0.00075	0.00075
T-Value	5.41	5.74	5.96
P-Value	0.000	0.000	0.000
Retract Speed		0.0029	0.0029
T-Value		2.20	2.28
P-Value		0.036	0.030
Fire Time			0.00019
T-Value			1.79
P-Value			0.085
S	0.00196	0.00185	0.00178
R-Sq	49.35	56.60	61.04

Case Study 1: Kiln-Fired Ceramic Coating Process

Controls for the Most Significant Factor (Factor A – Mix Ratio)

Coating Thickness vs. Viscosity Cup Drain Time

Measuring Viscosity Directly (instead of mix ratio) Compensated for Solvent Evaporation (Mix Ratio change) and Dip Temperature Variation



Enamel Ratio Operating Range 76% - 82% (68F – 78F)

David A. Goodrich, P.E.

Case Study 1: Kiln-Fired Ceramic Coating Process

Controls for 2nd Most Significant Factor (Factor B – Retract Speed)

One-Speed Setting on Dipping Machine - Eliminated operator variation (easy fix!)

Controls for Interaction Factor (Factor DE – Fire Time/Crash Cool)

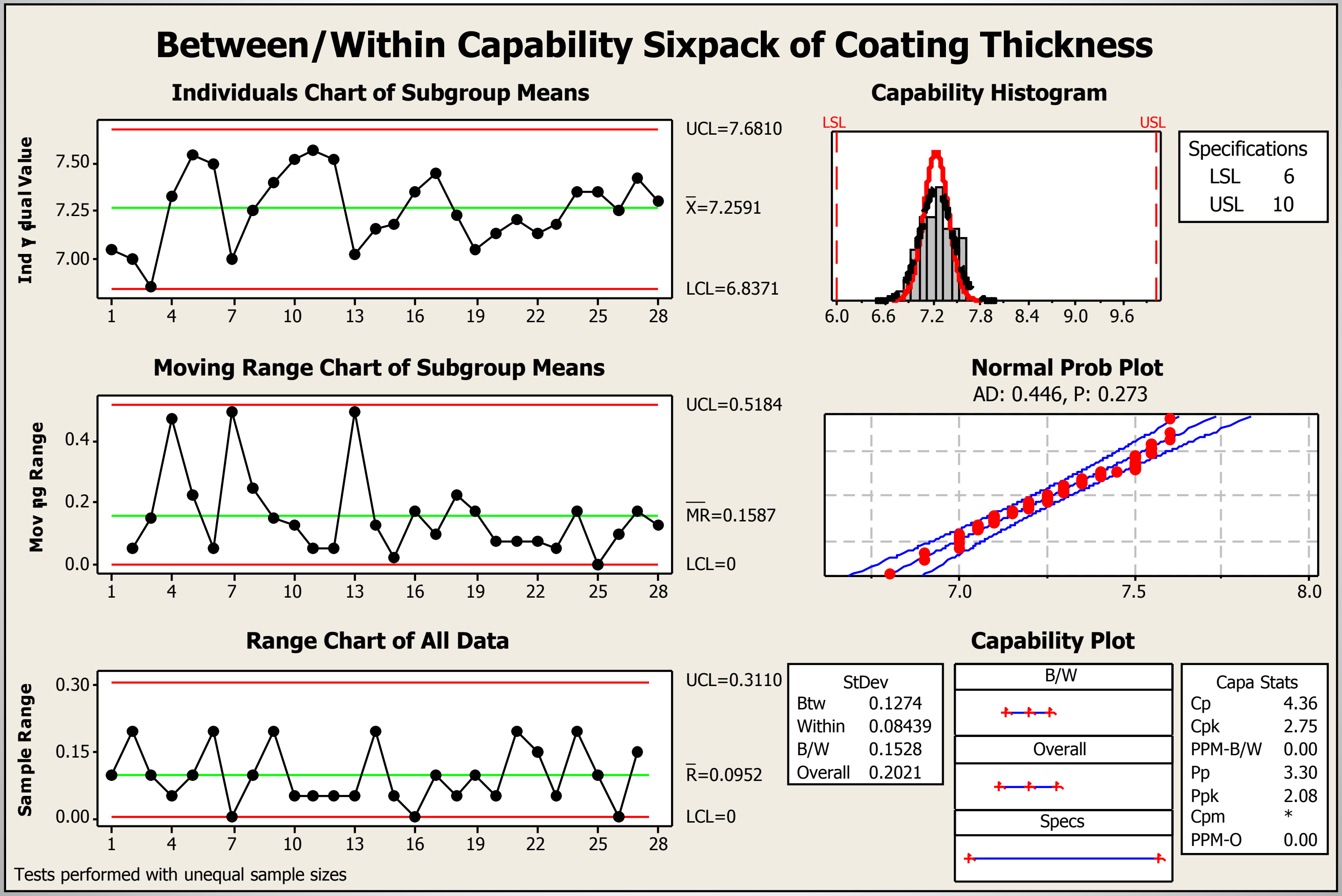
- **Installed Programmable Cycle Furnaces – Identical Units**
- **Less than 5% variation between units (Ramp up / Cool Down Cycle)**

Before

After

Case Study 1: Kiln-Fired Ceramic Coating Process

Resulting Process (Coating Thickness) – Cpk > 2.0



David A. Goodrich, P.E.

Case Study 1: Kiln-Fired Ceramic Coating Process

Source of Variation	Controls Implemented
- Manual “Brush” process for Pre-Coat Cleaning	- New Cleaning Method (Ultrasonic Cleaning Line)

- Inadequate Coating Controls
  - Solvent Ratios based on “operator experience”
  - Dipping speed unknown
  - No storage controls
	- New Coating Application Controls
	  - Solvent Ratios based on viscosity measurements
	  - Dipping speed controlled
	  - Storage controls (temperature)

- Extreme variation between firing ramp up/down	- New PLC controlled furnaces provide highly repeatable furnace ramp up/down
- Unqualified inspection methods	- Gage R&R’s (Attribute Agreement Analyses) for every inspection method
- Other general improvements	- Calibration required process equipment - Material Cert. of analysis /Shelf life - Adhesion specification and test developed - Detailed Work Instructions

Case Study 1: Kiln-Fired Ceramic Coating Process

Pre-Improvement Process Results

- High Coating Thickness reject rate required 100% inspection multiple times
- High levels of Pit and Bubbles requiring significant rework
- Coating Adhesion “events” with no known cause or controls
- First pass yield less than 50%
- Final yield approximately 60%

Post Improvement Process Results

- Coating Thickness Cpk (> 2.0)
- Statistically-based sample inspection for coating thickness
- Near Elimination of Pits and Bubbles
- 2x Improvement in Coating Adhesion strength
- Adhesion strength monitoring implemented
- Elimination of adhesion “events”
- First pass yields > 99%

Case Study 2: Ultrasonic Welding Process

Process to weld a cap onto a cartridge used to contain oil.

- Five Welding Process variables:
  - Weld Pressure
  - Weld Time
  - Weld Amplitude
  - Hold Time
  - Trigger Force

- Essential Design Requirements
  - Burst Pressure – 500 psi minimum
  - No flash at weld seam – 0.030” max weld collapse
  - No allowable leakage at weld seam – vacuum leak test (pass/fail)

- An initial 5-factor, two-level full factorial screening experiment identified three significant process factors:
  - **Weld Pressure**
  - **Weld Time**
  - **Weld Amplitude**

Case Study 2: Ultrasonic Welding Process

Response Surface DOE

Performed a Central Composite Design - Response Surface DOE to find the optimized process settings

3 Factors - Weld Pressure, Weld Time, Weld Amplitude

2 - Responses – **Burst Pressure and Weld Collapse**

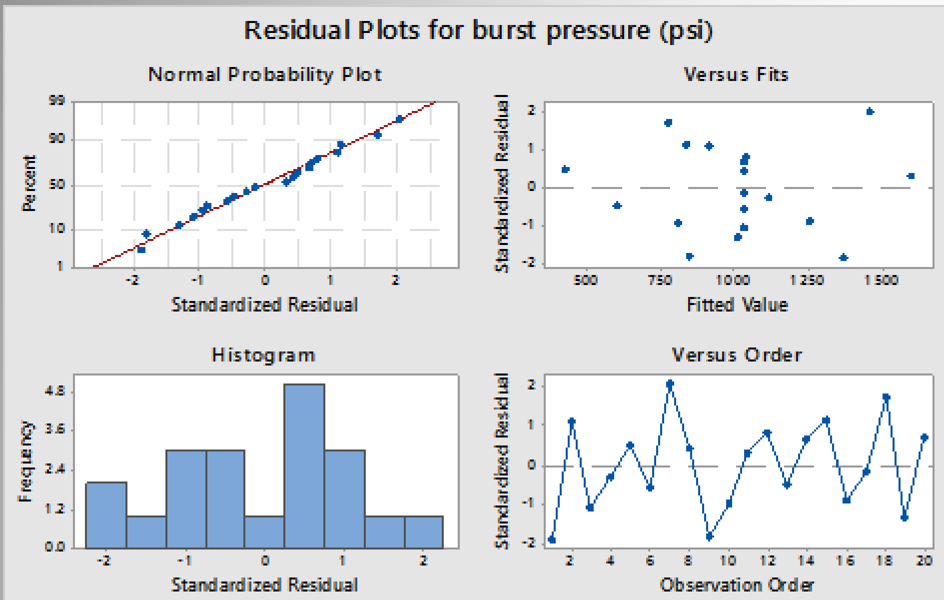
Design Summary

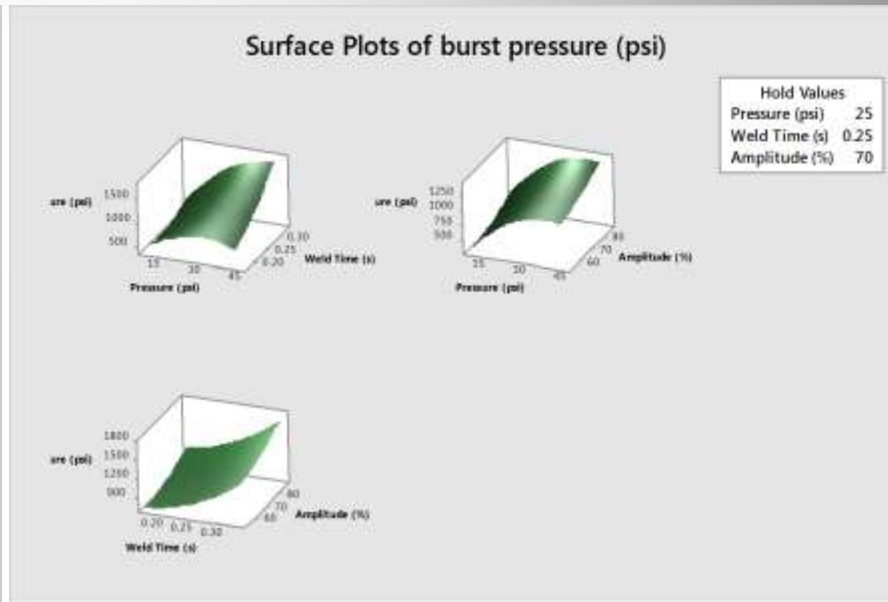
Study Type	Response Surface		**Experiments**	20			
Initial Design	Central Composite		**Blocks**	No Blocks			
Design Model	Quadratic						
Response	**Name**	**Units**	**Obs**	**Minimum**	**Maximum**	**Trans**	**Model**
Y1	Collapse	in	20	0.015	0.028	None	Quadratic
Y2	busrt pressure	psi	20	800	1632	None	Quadratic

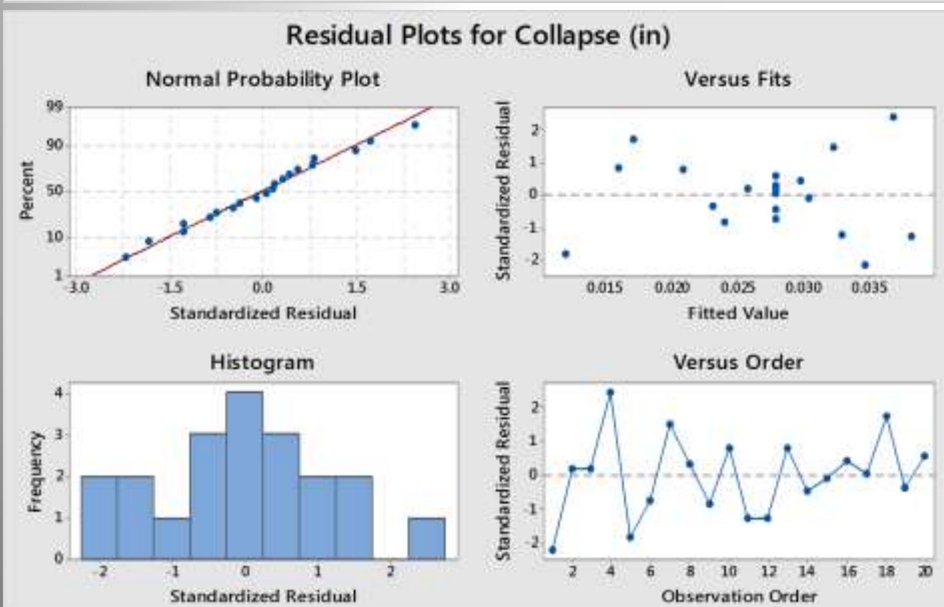
Factor	**Name**	**Units**	**Type**	**Low Actual**	**High Actual**	**Low Coded**	**High Coded**
A	Pressure	psi	Numeric	15	35	-1	1
B	Weld Time	s	Numeric	0.2	0.3	-1	1
C	Amplitude	%	Numeric	60	80	-1	1

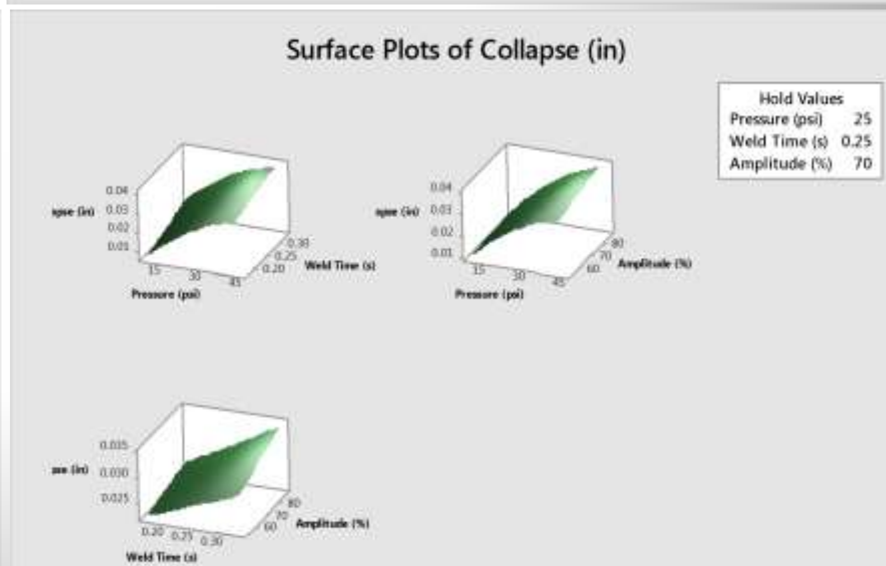
Case Study 2: Ultrasonic Welding Process

Response Surface DOE – Minitab Output









David A. Goodrich, P.E.

Case Study 2: Ultrasonic Welding Process

Response Surface DOE – Minitab Output

Response Optimization: burst pressure (psi), Collapse (in)

Parameters

Response	Goal	Lower	Target	Upper	Weight	Importance
burst pressure (psi)	Maximum	800.000	1632.00		1	1
Collapse (in)	Target	0.011	0.026	0.028	1	1

Variable Ranges

Variable	Values
Pressure (psi)	(20, 30)
Weld Time (s)	(0.22, 0.28)
Amplitude (%)	(65, 75)

Solution	Pressure (psi)	Weld Time (s)	Amplitude (%)	burst pressure (psi)	Collapse (in) Fit	Fit	Composite Desirability
1	20	0.277468	75	1059.58	0.0260000		0.558568

Multiple Response Prediction

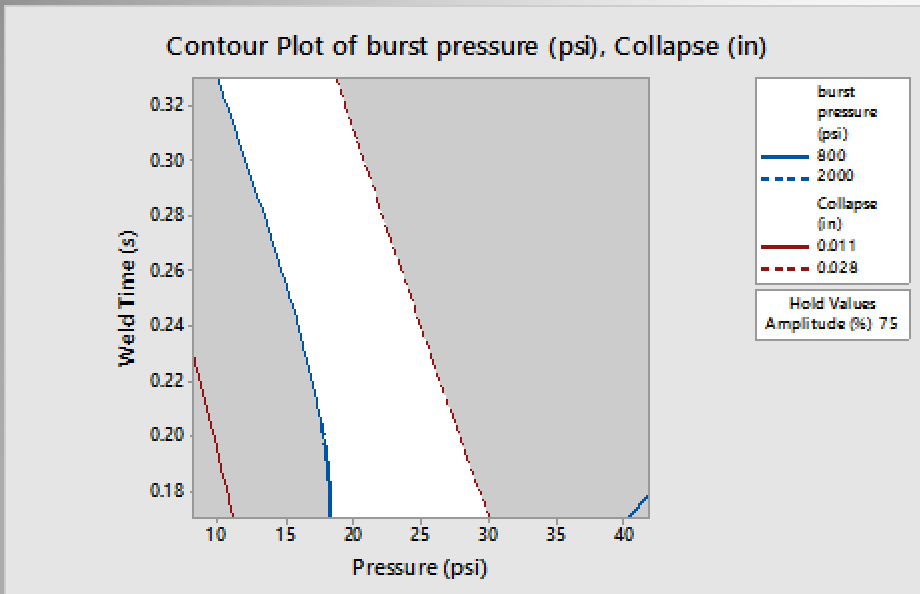
Optimized "Nominal" Process Settings

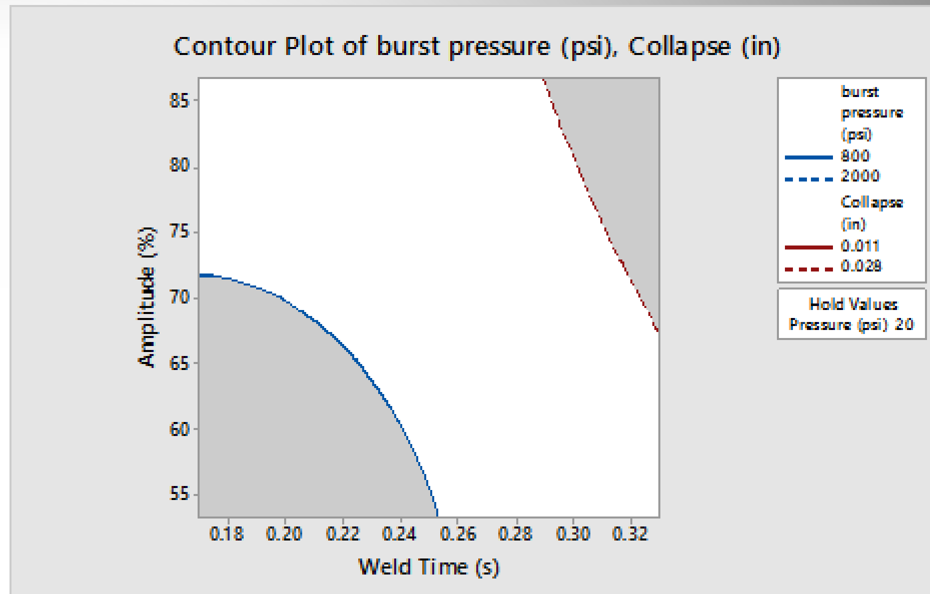
Variable	***Setting***
Pressure (psi)	***20***
Weld Time (s)	***0.277468***
Amplitude (%)	***75***

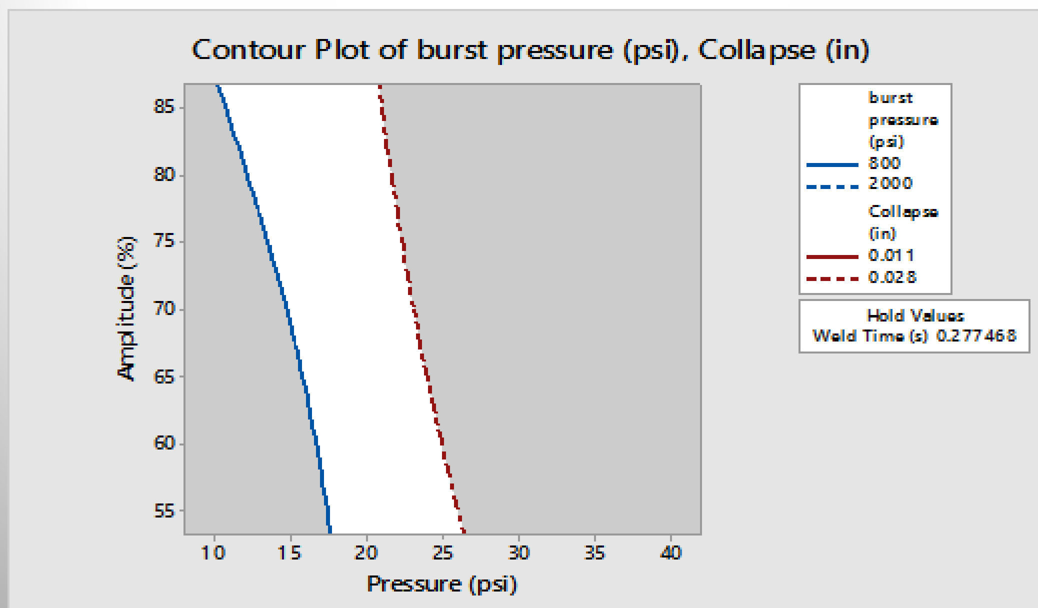
Response	Fit	SE Fit	95% CI	95% PI
burst pressure (psi)	**1059.6**	54.9	(937.2, 1182.0)	(745.0, 1374.1)
Collapse (in)	**0.026000**	0.000355	(0.025208, 0.026792)	(0.023964, 0.028036)

Case Study 2: Ultrasonic Welding Process

Response Surface DOE – Minitab Output







Case Study 2: Ultrasonic Welding Process

Response Surface DOE – Minitab Output

Prediction for Collapse (in)

Variable	Setting
Pressure (psi)	**18**
Weld Time (s)	**0.26**
Amplitude (%)	**72**

Fit	SE Fit	95% CI	95% PI
0.0228894	0.0003434	(0.0221243, 0.0236544)	(0.0208639, 0.0249148)

Variable	Setting
Pressure (psi)	**22**
Weld Time (s)	**0.29**
Amplitude (%)	**78**

Fit	SE Fit	95% CI	95% PI
0.0287903	0.0004147	(0.0278663, 0.0297142)	(0.0266996, 0.0308809)

Process "Minimum" Settings

Process "Maximum" Settings

Prediction for burst pressure (psi)

Variable	Setting
Pressure (psi)	**18**
Weld Time (s)	**0.26**
Amplitude (%)	**72**

Fit	SE Fit	95% CI	95% PI
892.425	53.0526	(774.216, 1010.63)	(579.468, 1205.38)

Variable	Setting
Pressure (psi)	**22**
Weld Time (s)	**0.29**
Amplitude (%)	**78**

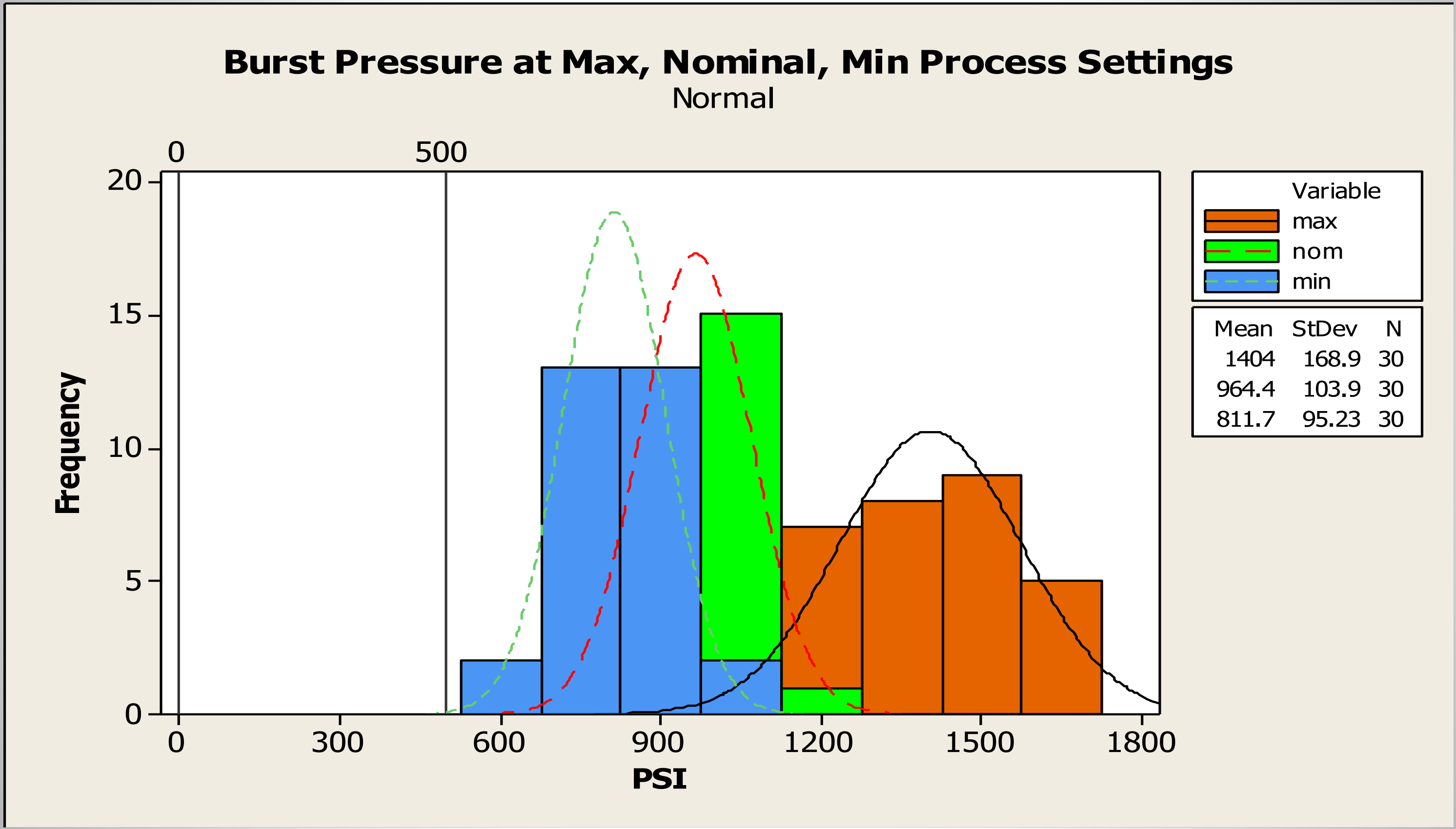
Fit	SE Fit	95% CI	95% PI
1222.80	64.0725	(1080.04, 1365.56)	(899.768, 1545.83)

Case Study 2: Ultrasonic Welding Process

Response Surface DOE

Burst Pressure Results

Minimum, Maximum, and Nominal Process Settings



Burst Pressure results became more dispersed (less capable) at the Maximum Process settings. This was caused by increasing variation in the weld process as the Weld Collapse approached the allowable maximum (0.030 inch).

Case Study 2: Ultrasonic Welding Process

Response Surface DOE

Using the Response Surface Model:
“What If” the Target Collapse is increased from 0.026” to 0.036”?
(This would require a component design change)

Response Optimization: burst pressure (psi), Collapse (in)

Parameters

Response		Goal	Lower	Target	Upper
burst pressure (psi)	Maximum	800.000	2000.00		
Collapse (in)		Target	0.011	**0.036**	0.038

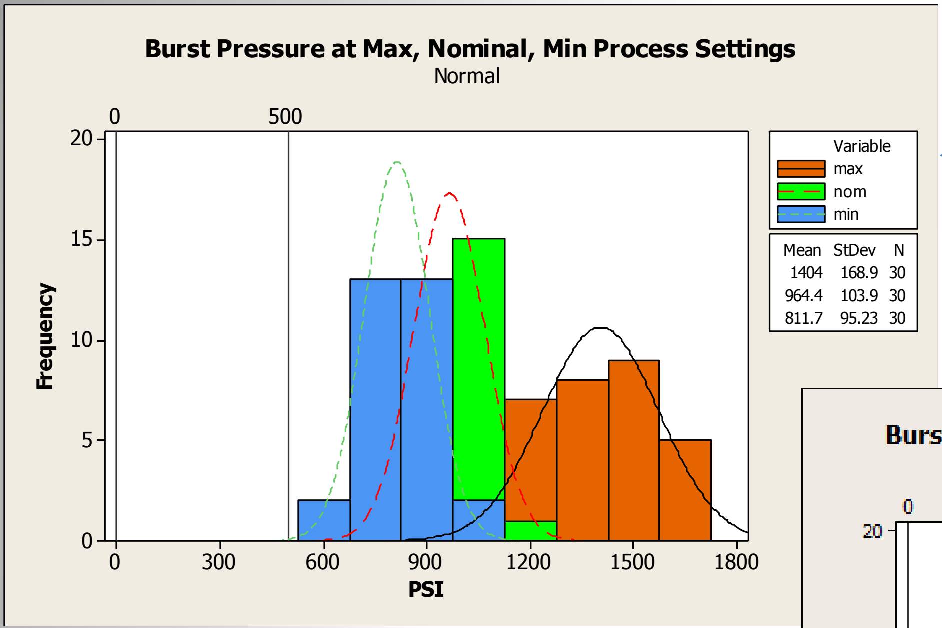
Solution

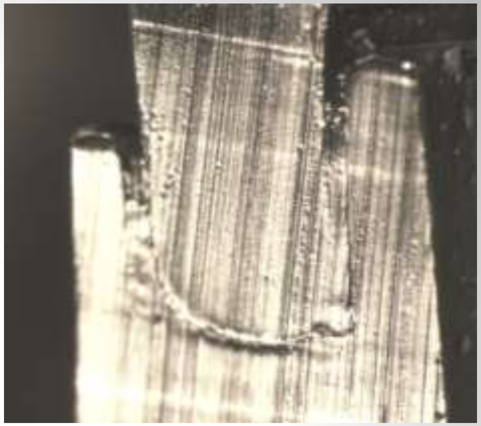
Solution	Pressure (psi)	Weld Time (s)	Amplitude (%)	burst pressure (psi)	Collapse (in)
1	26.5463	0.33	86.82	1753.97	0.0360000

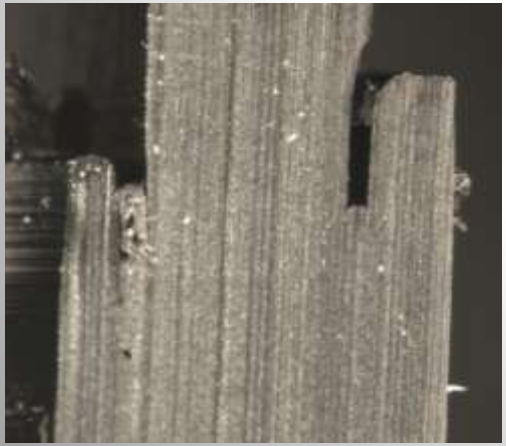
Burst Pressure prediction increases significantly, indicating a much more robust weld

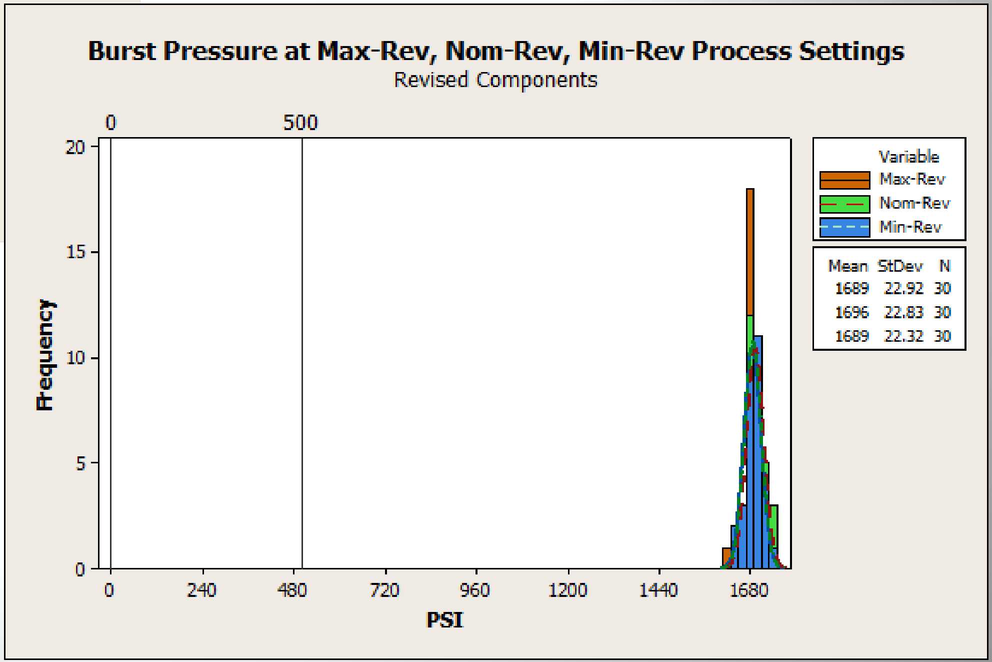
Case Study 2: Ultrasonic Welding Process

Burst Pressure Prior to Component Design Optimization





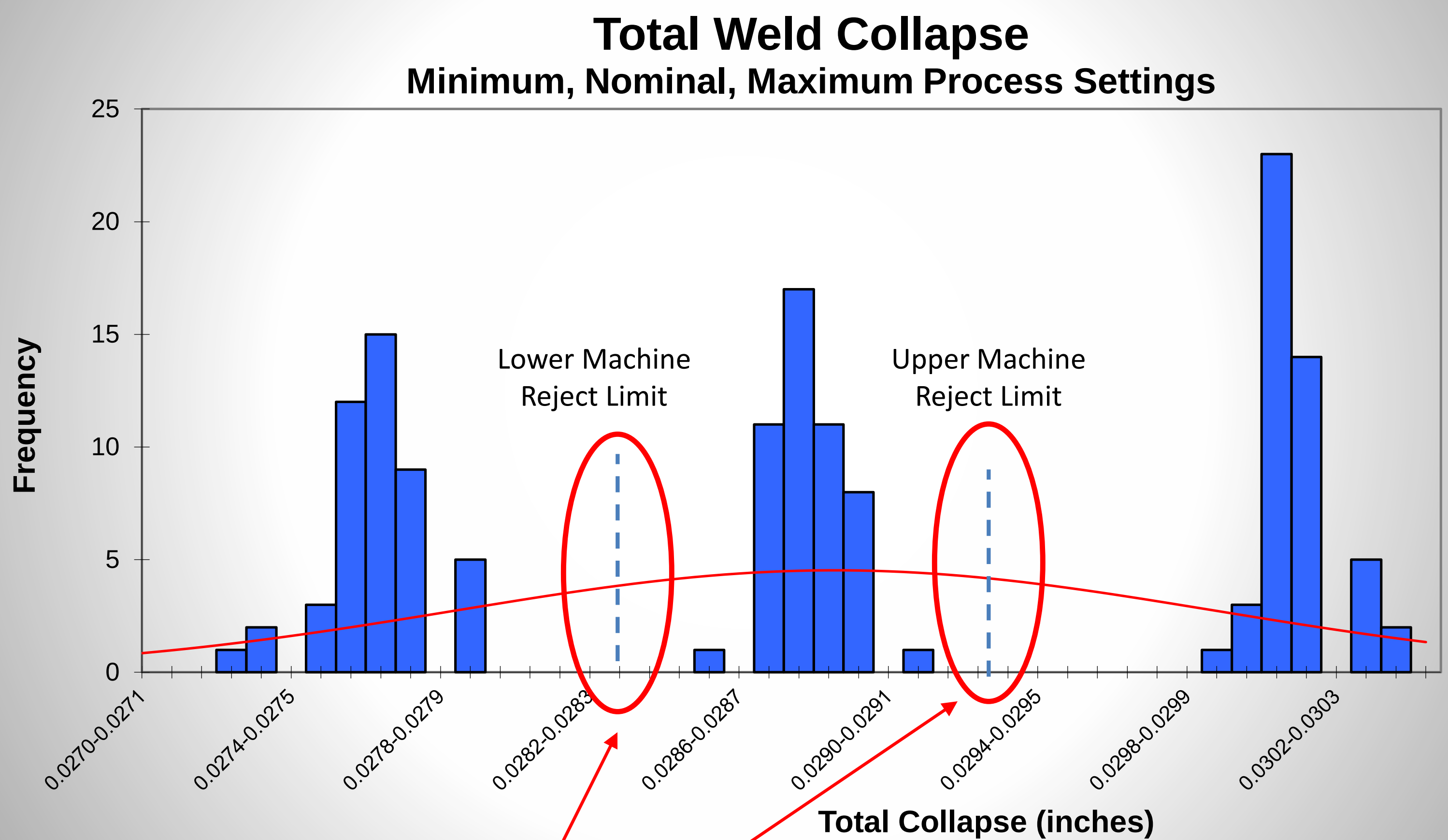




Burst Pressure After Component Design Optimization

Case Study 2: Ultrasonic Welding Process

In-Process Monitoring – Validated Welding Process



Automated Machine Shut Down
Based on these ***Process Reject Limits***

Process Characterization Study
Deliverables

- A fully defined process with appropriate process controls established.
- Products (product family) covered by the characterization.
- Material limitations (where applicable).
- Measurable process characteristics.
- Identification of Sources of anticipated significant process variation.
- Proposed process controls strategy (recommendations) such as:
  - setup parameters,
  - process instructions,
  - nominal process settings (optimized for "best case"),
  - operating limits of the process identified (anticipated operating ranges including the "worst case" (min/max) settings.
  - actions to be taken if control limits are exceeded

Characterization Study **Deliverables**

- Gauge R&R Studies Completed (where applicable)
- Attribute Agreement Studies Completed (where applicable)
- Include test data and methods used during the characterization study
- Updated Process Risk Analysis (e.g. Process FMEA, FTA)
- ***Formal (Approved) Characterization Report***

Process Qualification runs that challenge the anticipated operating ranges including the "worst case" (min/max) settings are typically done after the approved characterization report as a part of an Operational Qualification Process Challenge (risk based sample sizes apply here!)

Questions?

Comments?

Criticisms?

Design-Expert Software Version 10 (45-day free trial)

http://www.statease.com/dx10.html

Minitab Software Release 17 (30-day free trial)

http://www.minitab.com/en-us/

Data Used for Case Study 2: Ultrasonic Welding Process Surface Response DOE Example

Factor	Factor	Factor	Response	Response
Pressure	Weld Time	Amplitude	Collapse	Burst Pressure
psi	s	percent	in	psi
35	0.3	60	0.0337	1229
25	0.25	53.18	0.0259	1002
25	0.25	70	0.0281	904
41.82	0.25	70	0.0381	1096
8.18	0.25	70	0.0109	465
25	0.25	70	0.0274	963
25	0.33	70	0.0331	1632
25	0.25	70	0.0282	1083
25	0.17	70	0.0235	693
15	0.3	60	0.0213	735
35	0.3	80	0.0377	1624
35	0.2	80	0.0323	1099
15	0.2	60	0.0163	565
25	0.25	70	0.0276	1112
35	0.2	60	0.0304	923
25	0.25	86.82	0.03	1183
25	0.25	70	0.028	1013
15	0.2	80	0.0179	904
15	0.3	80	0.023	915
25	0.25	70	0.0284	1116