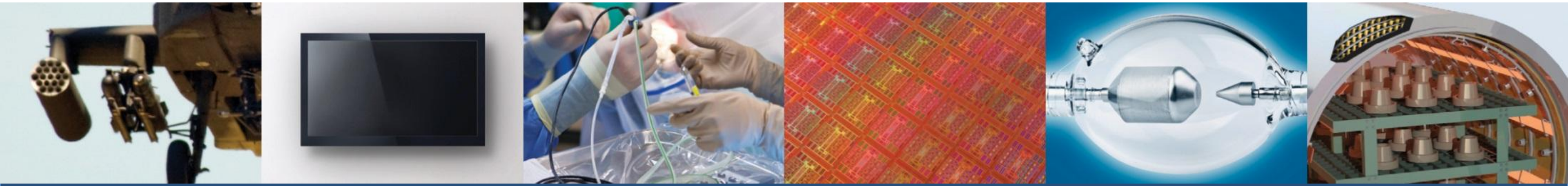
ELMET TECHNOLOGIES

Your Edge in Performance Metals

Invited Talk presented at the Predictive Analytics World for Manufacturing June 2016 Chicago IL, USA

Dr. Peter Frankwicz
Senior Process Engineer



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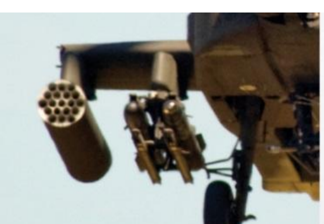
Your Edge in Performance Metals

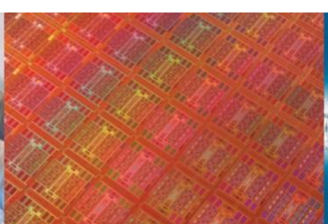
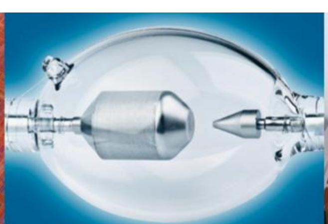
Improved Statistical Process Control of Mature Manufacturing Processes Using Multiple Available Data Streams

Dr. Pete Frankwicz

Senior Metallurgical Process Engineer

June 2016




Rev. 5

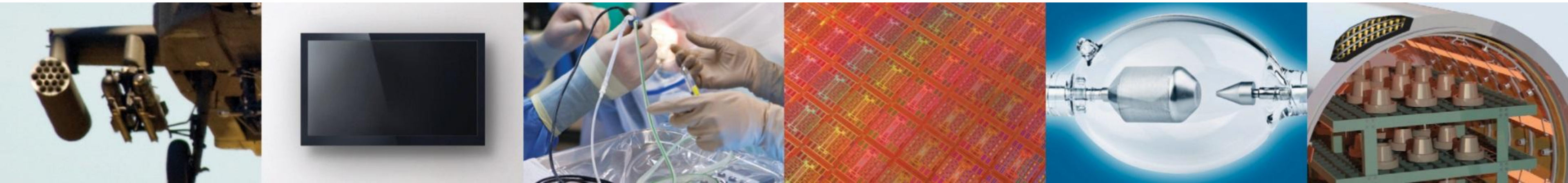
Outline

ELMET TECHNOLOGIES

- Introduction to Elmet Technologies LLC, Lewiston Maine USA
- Two industrial case studies
  - Targeting of doped tungsten products
  - Optimization of Mo powder production – sintering unit process operations
- Tools for predictive analytics in small business manufacturing
- Conclusions

Elmet Technologies

- Tungsten and Molybdenum refractory metal products
- Fully integrated manufacturing
- Unique and diversified product lines: electronic materials sputter targets to 22µm Mo-W aerospace defrosting wire



87 Year Manufacturing Lineage

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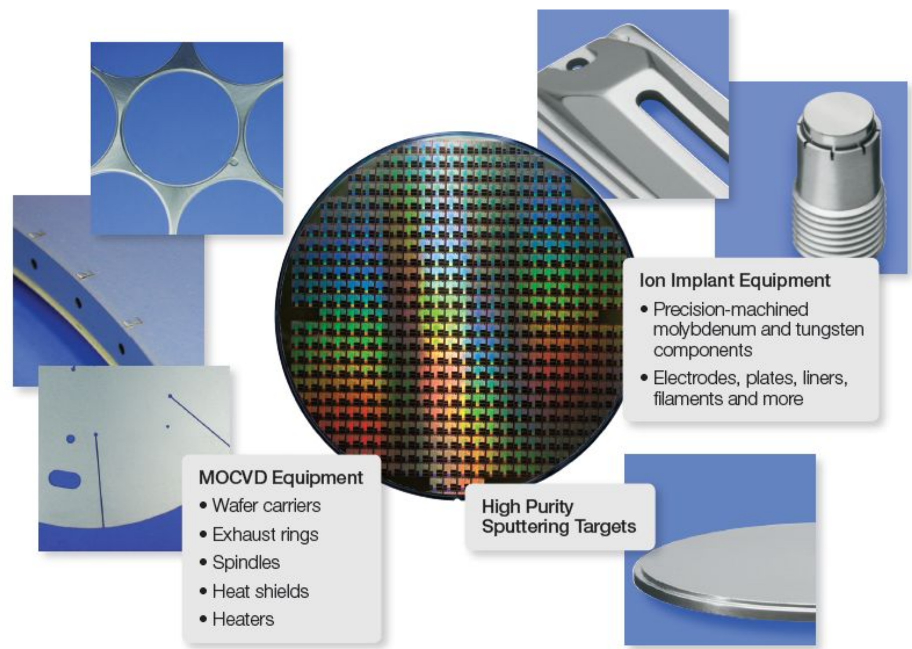
- Founded 1929 in Lewiston Maine USA under name **American Electometals Inc.**
- Acquired by Royal Philips Electronics of the Netherlands.
  - Majority of the world share of tungsten incandescent filaments for residential & industrial applications
- Re-organized in 2015 as Limited Liability Corporation with Anania & Associates Investment Co. of Windham, Maine.



Industrial Case Study Overview:

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- ***Common process engineer in uncommon situations***
- This is not a talk on advanced statistical or mathematical theory
- Case studies are about:
  - Agile data mining
  - Statistical and predictive process control
  - Unit process integration
- “Real manufacturing” data
- Physical units generalized to avoid divulging proprietary material specifications or manufacturing processing

JMP: “John’s* Macintosh Program”

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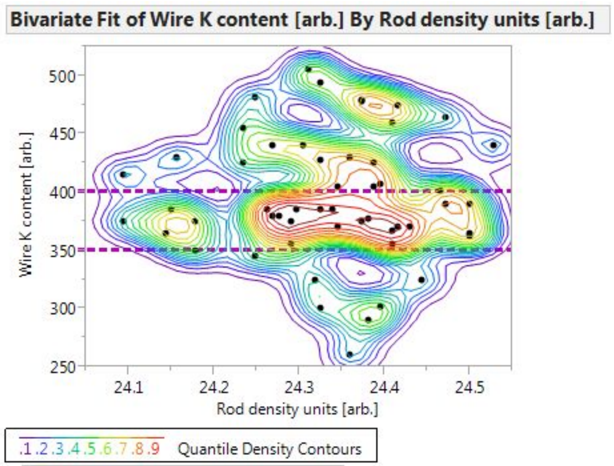
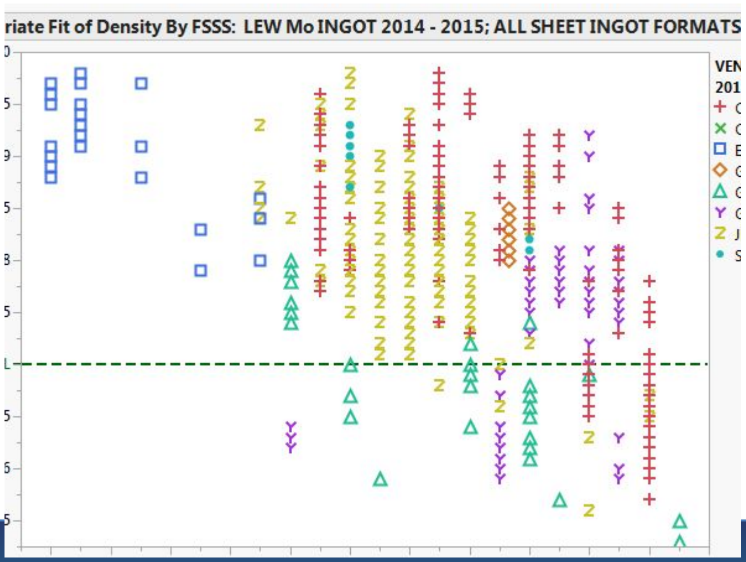
- Front end platform for mathematical analysis and visualization
  - Semiconductor, financial and pharmaceuticals sectors
  - Statistical process control of industrial manufacturing
- High powered computational environment using multi-core 64-bit computing horsepower
- No code or programing required
- Java-like scripting options available
- *Creator & Chief Architect John Sall of SAS Corporation



Nonparametric Bivariate Density

Variable	Kernel Std
Rod density units [arb.]	0.02715
Wire K content [arb.]	13.75459



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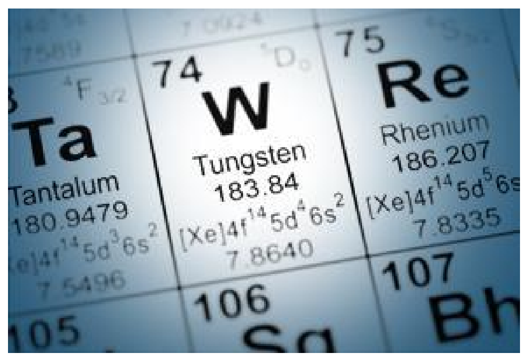
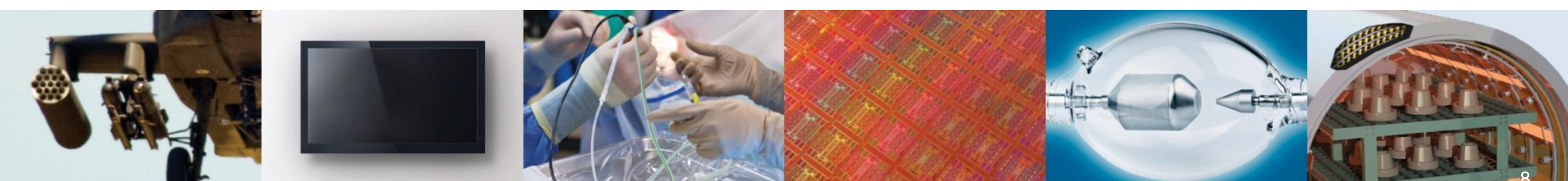
Industrial Case Study One:

Targeting of doped tungsten products





Heirloom Elmet Products

- “Non-sag” tungsten
- Creep resistance wire at high temperatures (T>2200ºC)
- Dispersion strengthen alloy system
- Microstructure with potassium (K) filled bubbles or dispersoid
- K-dispersoid act as barriers for grain boundary migration

- Applications
  - Historical: incandescent filaments
  - Elmet manufacturing over **17,000 kilometers** fine tungsten wire per month
  - Growing market in micro-electromechanical applications

Alpha Filament Technology

- Alpha Filament Technology manufactures high end industrial tungsten filaments and support members
- AFC requires doped tungsten wire to be within a 50 part-per-million potassium (K) content window
- The 50 ppm retained K level has been problematic for the Elmet manufacturing for at least ten years
- Identification of passing production material was a mixture of blind luck and folklore. “*en-masse*” sampling of work-in-progress
- Six financial quarter historical passing rate mean: **15.3%**
- **Lack of Predictive Testing Protocol**

Elmet Tungsten Production Flow

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- First Reduction
- Second Reduction
- Blending
- Ingot production
- Sintering
- Flat product: heavy plate to foils
- Round product: thick rod to fine wire

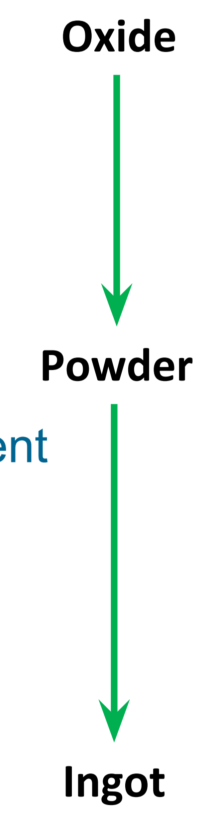
Tungsten Facts:

- Symbol: W
- Category: Transition Metal, Group VI
- Atomic Number: 74
- Density: 19.25 g / cm^3
- Melting Point: 3420 °C, highest metal, 2nd highest of elements
- Discovered: 1781

Available Data Streams

Available Data Streams for exploration and predictive model development

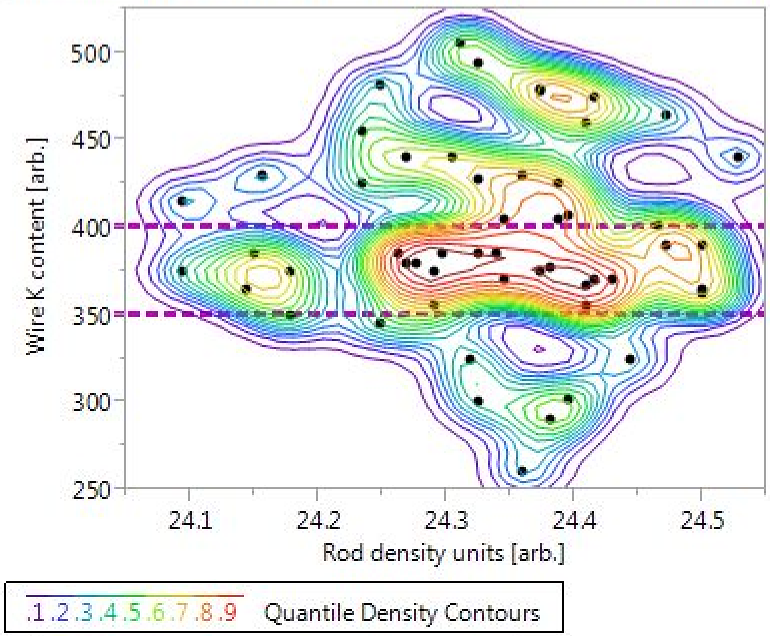
- Doped tungsten reduction:
  - Feed stock APT lot
  - Reduction conditions
  - Doped potassium content
- Blended tungsten batches:
  - Particle size distribution
  - Blend tap and bulk density
  - Predicted blend weight averaged density and particle size of component
  - Blend Doped potassium content
- Sintered tungsten rods: **{Physical entity + data attributes}**
  - Sintered density
  - Sintering furnace
- Sintered tungsten rod batch
  - Density mean; range



Preliminary Data Mining

- Bivariate density estimations used for exploration of unstructured data
- Density contours identify process “sweet spots”.

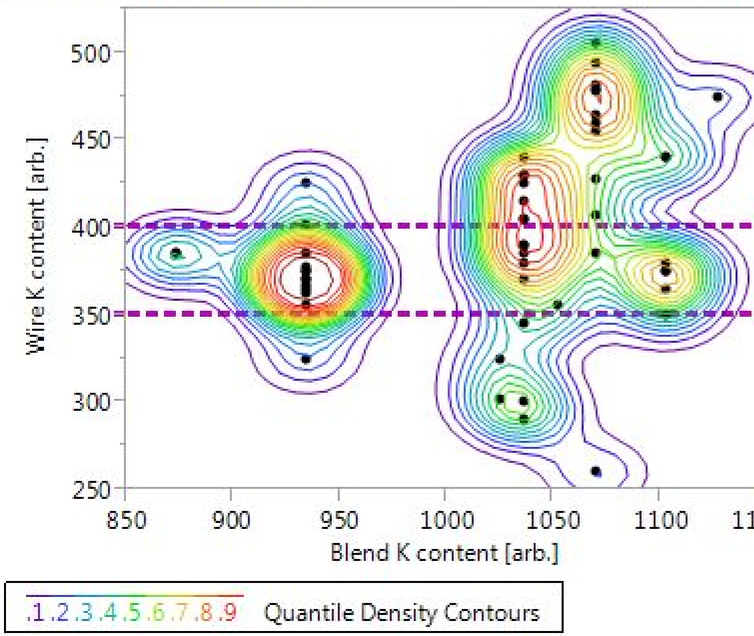
Bivariate Fit of Wire K content [arb.] By Rod density units [arb.]



Nonparametric Bivariate Density

Variable	Kernel Std
Rod density units [arb.]	0.02715
Wire K content [arb.]	13.75459

Bivariate Fit of Wire K content [arb.] By Blend K content [arb.]



Nonparametric Bivariate Density

Variable	Kernel Std
Blend K content [arb.]	17.27932
Wire K content [arb.]	13.75459

Data Mining with Stepwise Regression

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- What parameters are important?
- Stepwise regression of available data parameters can be used to identify statistically significant process knobs.
- Used to screen large sets of X factors for a select set of Y responses.
- Example: Identifies only one factor particle size as statistically significant with Adjusted RSqaure = 0.269!
- Counter initiative to metallurgical knowledge base.

Stepwise Fit for K content [AT 235]

Stepwise Regression Control

Stopping Rule: Minimum BIC

Direction: Forward

rows not used due to excluded rows or missing values.

SSE	DFE	RMSE	RSquare	RSquare Adj	Cp	p	AICc	BIC
3450.3189	35	9.9287733	0.2579	0.2367	5.8802753	2	279.535	283.6405

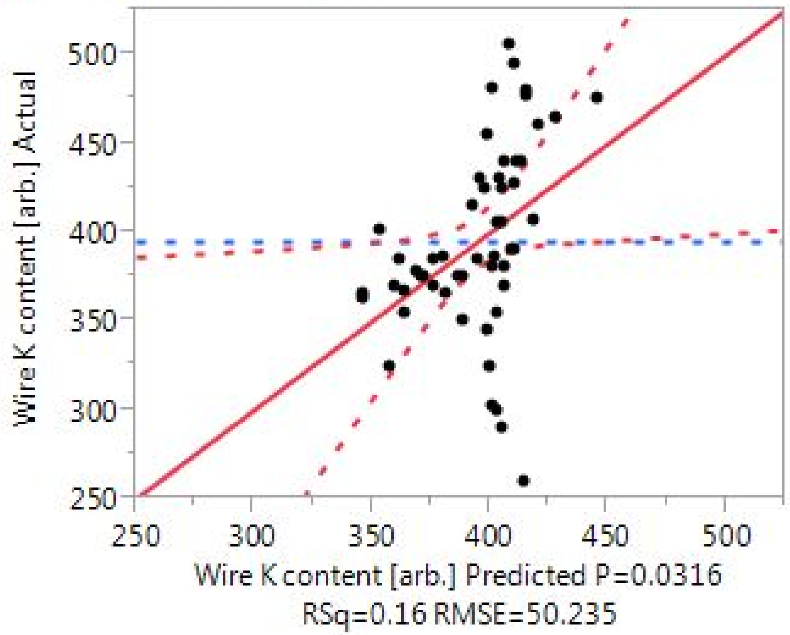
Current Estimates

Lock	Entered	Parameter	Estimate	nDF	SS	"F Ratio"	"Prob>F"
☑	☑	Intercept	153.16294	1	0	0.000	1
☐	☐	Blend wt% FSSS	0	1	11.08347	0.110	0.74267
☐	☐	Blend wt% K	0	1	90.4955	0.916	0.34534
☐	☑	Fsss	-20.382904	1	1198.947	12.162	0.00133
☐	☐	Bulk Density	0	1	314.0958	3.405	0.07372
☐	☐	Tap Density	0	1	0.117584	0.001	0.97304
☐	☐	K, pow avg	0	1	202.8466	2.124	0.15421
☐	☐	BUNDLE MEAN	0	1	111.6859	1.137	0.29372

General Linear Modeling

- Can I predict potassium content?
- Least squared modeling can be used to examine major effects of selected physical parameters but suffer from low fitting metrics [0.5 > RSquare].
- Example: Least square model of retained K versus particle size, chemical content and ingot sintered density. Adjusted RSquare = 0.11
- Predictive models for a very small potassium content range have limited utility in a production environment.

Actual by Predicted Plot



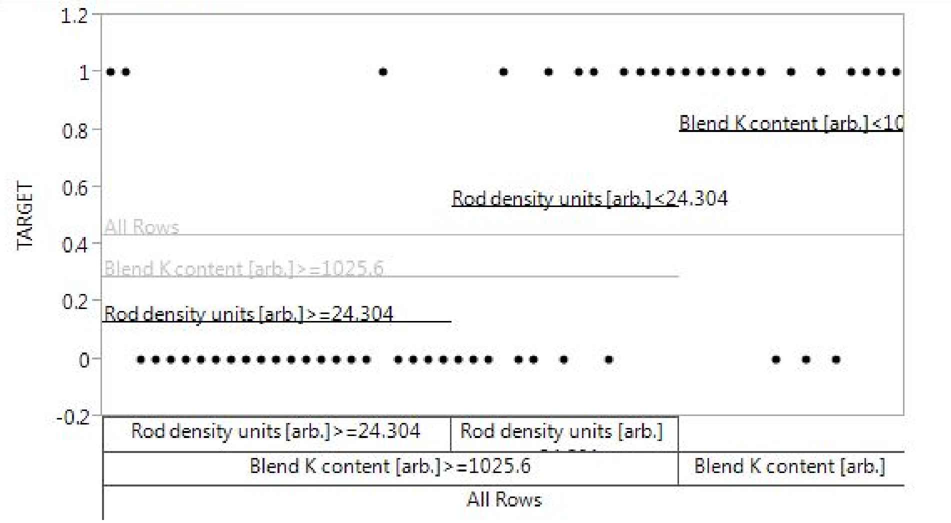
Summary of Fit

RSquare	0.163466
RSquare Adj	0.11225
Root Mean Square Error	50.23452
Mean of Response	394.3943
Observations (or Sum Wgts)	53

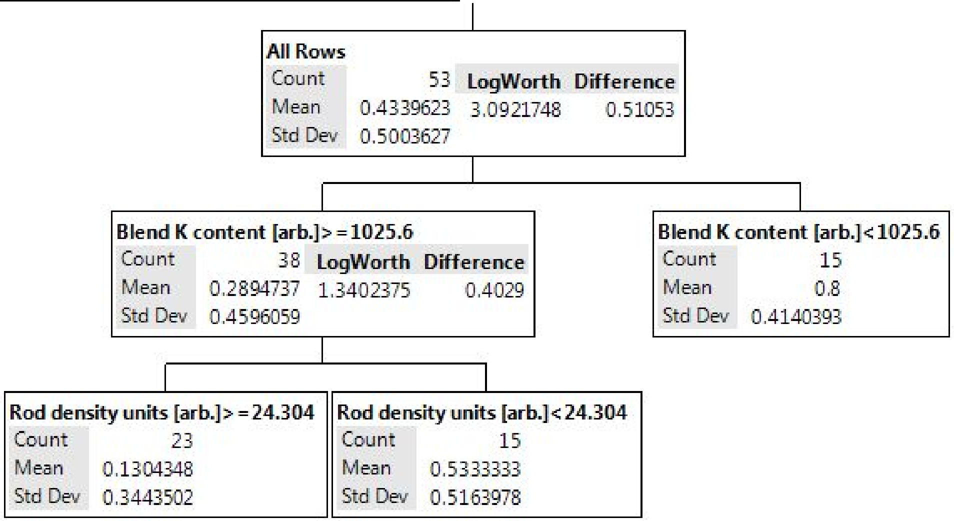
Recursive Partitioning

- Agile Idea: Do not need to model actual potassium content but rather passing product in the 50 ppm window.
- Assign "target" factor: 1=pass, 0 fail. Comparable to a Gini Index.
- Recursive Partitioning with Target as Y response and X factors from the available parameters.
- Defines physical parameter based testing criteria:
  - Blend K < 1025 K content
  - If K> 1025 K content;
  - Then < density less than 24 units

Partition for TARGET



RSquare	RMSE	N	Number of Splits	AICc
0.329	0.4061329	53	2	63.7269



Predictive Analytics Positive Impacts

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1. Simple Predictive Model Criteria: Defined testing criteria based on available process data rather than *en-masse* product testing.
2. Increase testing success rate from mean 15.3% to **66.7%** in last financial quarter
3. Smoothing out of product inventory and manufacturing flow in fine wire.
4. Significant reduction of wire potassium chemistry testing.

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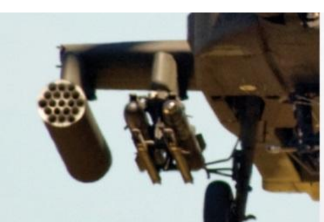
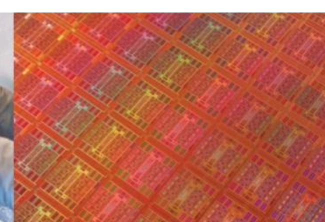
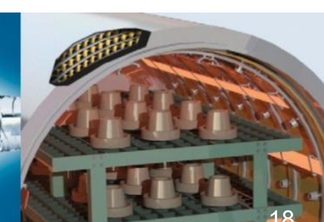
Industrial Case Study Two:

Optimization of Mo powder production – sintering unit process operations





Process Change Management

- 2014-2015: Transition to multiple Mo powder sources
  - Elmet in full production converting MoO_3 to Mo
  - Introduced blend to blend variations in powder metallurgical parameters such as particle size distributions and powder densities.
- Variation in sintered density
  - Multiple feedstock sources resulted in unanticipated variation in sintered density of ingots after process of record sintering.
- Downstream manufacturing instability
  - Hot rolling department experienced heightened levels of rolling defects and processing difficulties.

– Requires re-optimization of unit process integration

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Available Data Streams

- Mo-oxide first and second reduction :
  - Feed stock MoO_3
  - MoO_2 Reduction conditions
- Molybdenum Blend:
  - MoO_3 feedstock lot
  - Particle size distribution
  - Blend tap and bulk density
- Sintered Mo ingots: **{Physical entity + data attributes}**
  - Blend lot
  - Ingot type
  - Sintered density
  - Sintering furnace

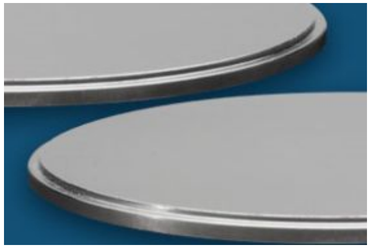
Molybdenum Facts:

- Symbol: Mo
- Category: Transition Metal, Group VI
- Atomic Number: 42
- Density: 10.28 g / cm^3
- Discovered: 1778
- High melting temperature, 2620 °C

Oxide

Powder

Ingot



Mo Sputter Targets

Preliminary Data Mining

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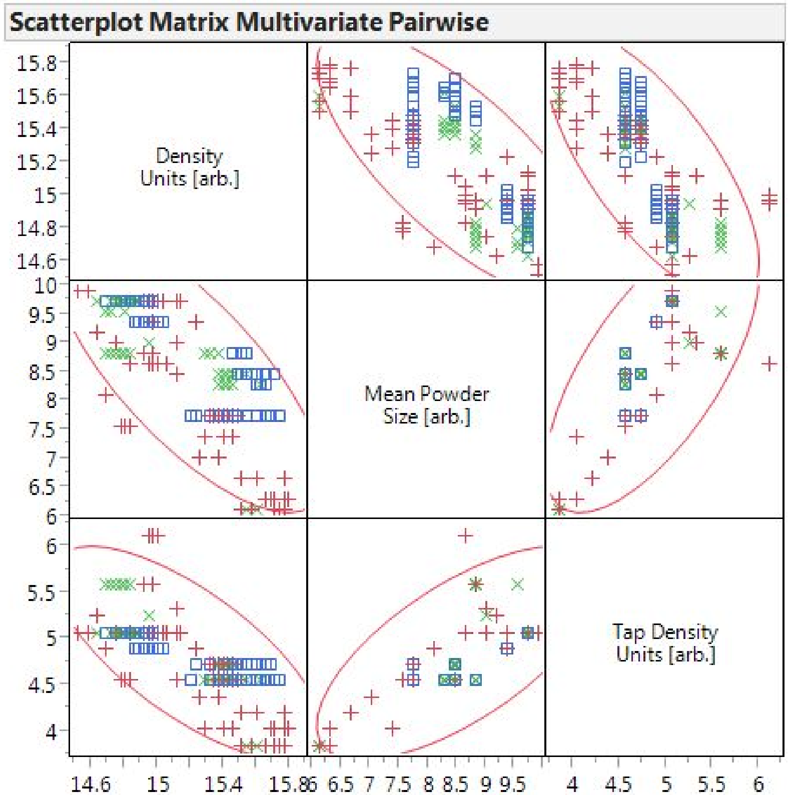
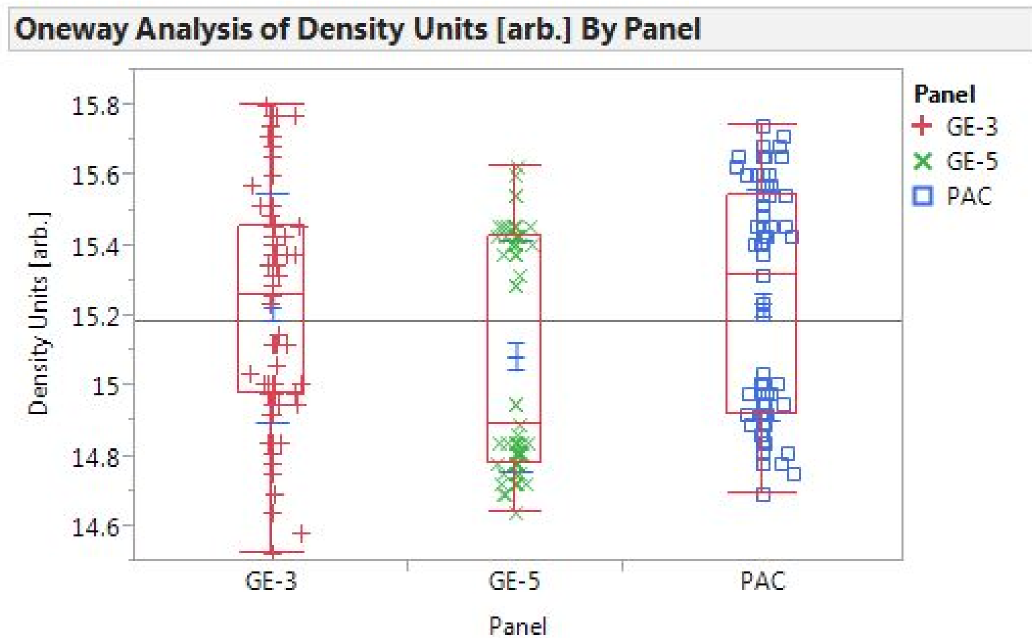
- Sintered density is inversely related to mean particle size. The correlation value equal -0.75 and is statistically significant.
- Interesting categorical factor: sintering furnace





Pairwise Correlations

Variable	by Variable	Correlation	Count	Lower 95%	Upper 95%	Signif Prob	-.8 -.6 -.4 -.2 0 .2 .4 .6 .8
Mean Powder Size [arb.]	Density Units [arb.]	-0.7473	249	-0.7975	-0.6868	<.0001*	
Tap Density Units [arb.]	Density Units [arb.]	-0.7200	249	-0.7750	-0.6542	<.0001*	
Tap Density Units [arb.]	Mean Powder Size [arb.]	0.6787	249	0.6055	0.7406	<.0001*	

Bivariate Density Estimation

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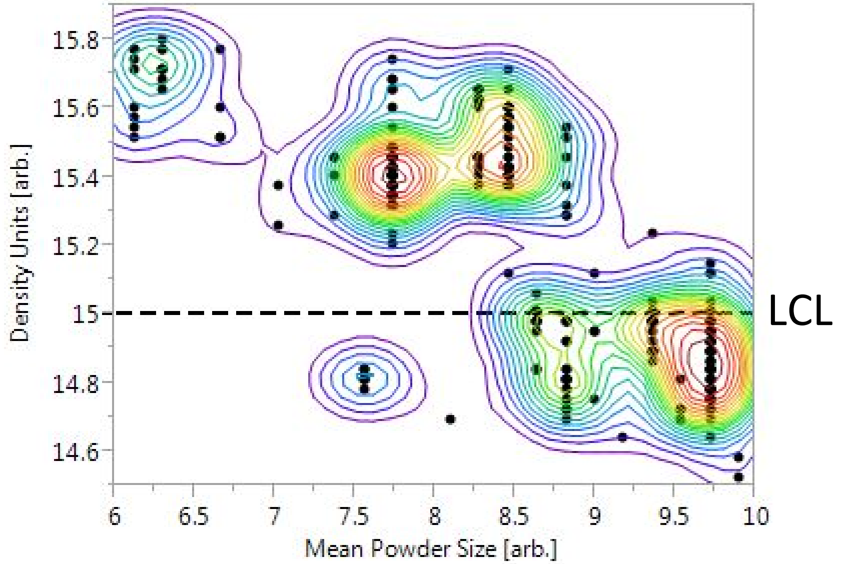
- Strong bivariate relationships
- Map display interdependent process space involving multiple factors

Fit Group

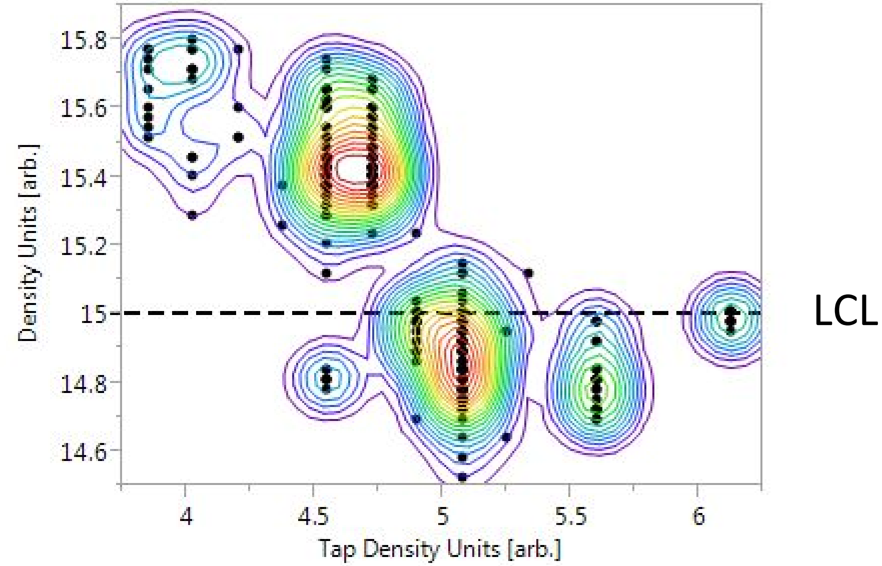
Bivariate Fit of Density Units [arb.] By Mean Powder Size [arb.]



Nonparametric Bivariate Density

Variable	Kernel Std
Mean Powder Size [arb.]	0.199924
Density Units [arb.]	0.066427

Bivariate Fit of Density Units [arb.] By Tap Density Units [arb.]



Nonparametric Bivariate Density

Variable	Kernel Std
Tap Density Units [arb.]	0.09411
Density Units [arb.]	0.066427

Data Mining to Predictive Process Control

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- High priority to develop predictive modeling to relate metal powder properties to sintered ingot density
- Modeling data set:
  - Production data of 2014 and 2015
  - Statistically significant data set: N=249 ingots
  - 15,100 kilograms of Mo powder
  - Three independent sintering furnaces
- Simple least square linear regression with main & cross effects only.
- Units are generalized to “arb. unit” that completely preserve relevant process trends and relationships.

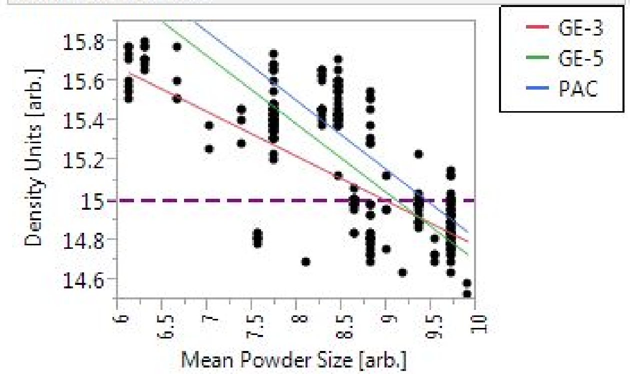
Model 01: Powder size – Furnace

- Model 01:
- Model response: sintered ingot density
- Model factors: mean powder size and sintering furnace entity
- Continuous and categorical factors
- N=249 production ingots
- RSquare = 0.66
- Predictive model capability to density lower control limit (LCL) of 15.0 density units.

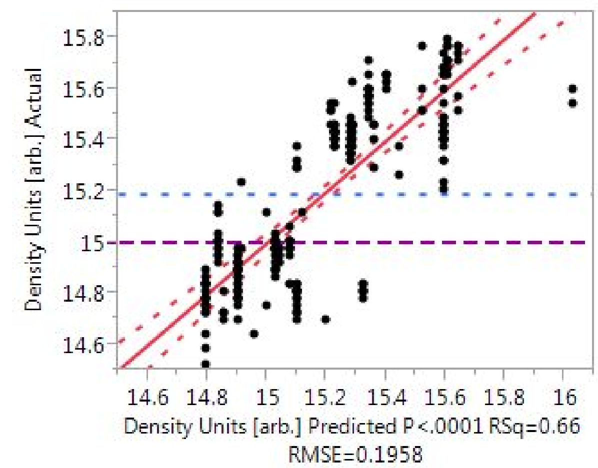
- Major predictive model findings:

Reveals opportunities for continuous process improvement (CPI) of sintering unit process

Regression Plot



Actual by Predicted Plot



Summary of Fit

RSquare	0.661801
RSquare Adj	0.654843
Root Mean Square Error	0.195771
Mean of Response	15.18835
Observations (or Sum Wgts)	249

Sintering Furnace Performance

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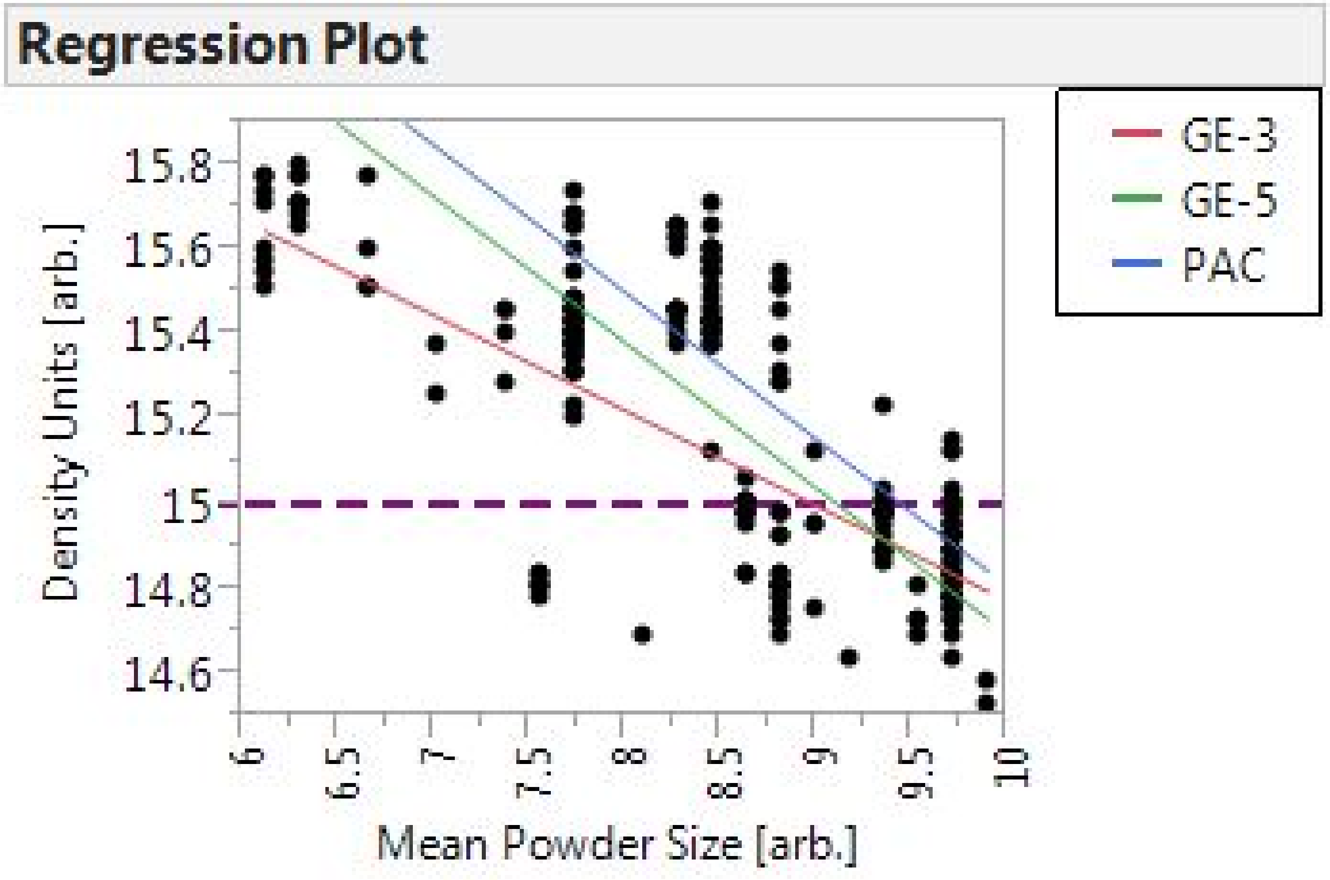
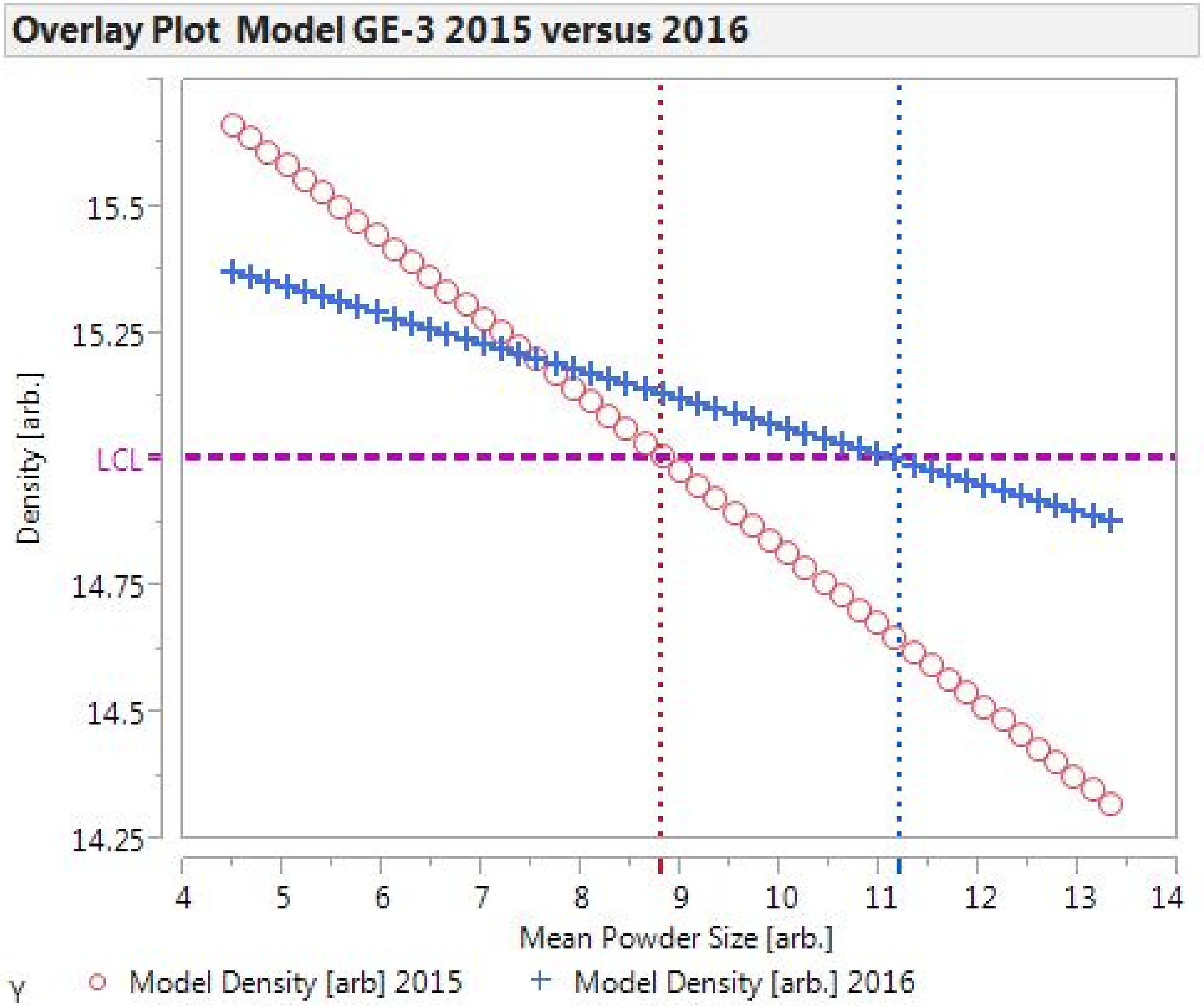
- Model 01 exhibits different response slope for furnace GE-3
- Opportunity for furnace optimization to reduce process variation



Modeling based on predictive expressions of Model 01

- Modeling of density versus powder size for furnace GE-3 2015 and 2016
- Improved density performance for larger size powder blends in 2016 after process improvements

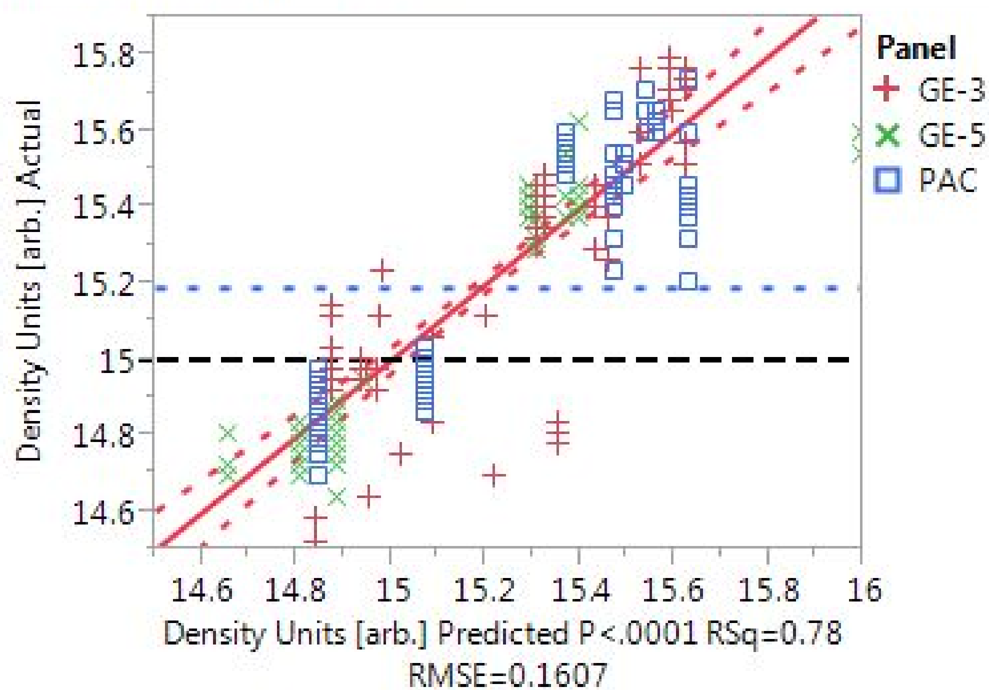


2016
N=347 ingot

Model 02 Three Factors

- Model 02:
- Model response: sintered ingot density
- Model factors: mean powder size, tap density and furnace entity
- RSquare = 0.77
- Added factor improved predictive
- Basis for simple predictive process control (PPC)

Actual by Predicted Plot

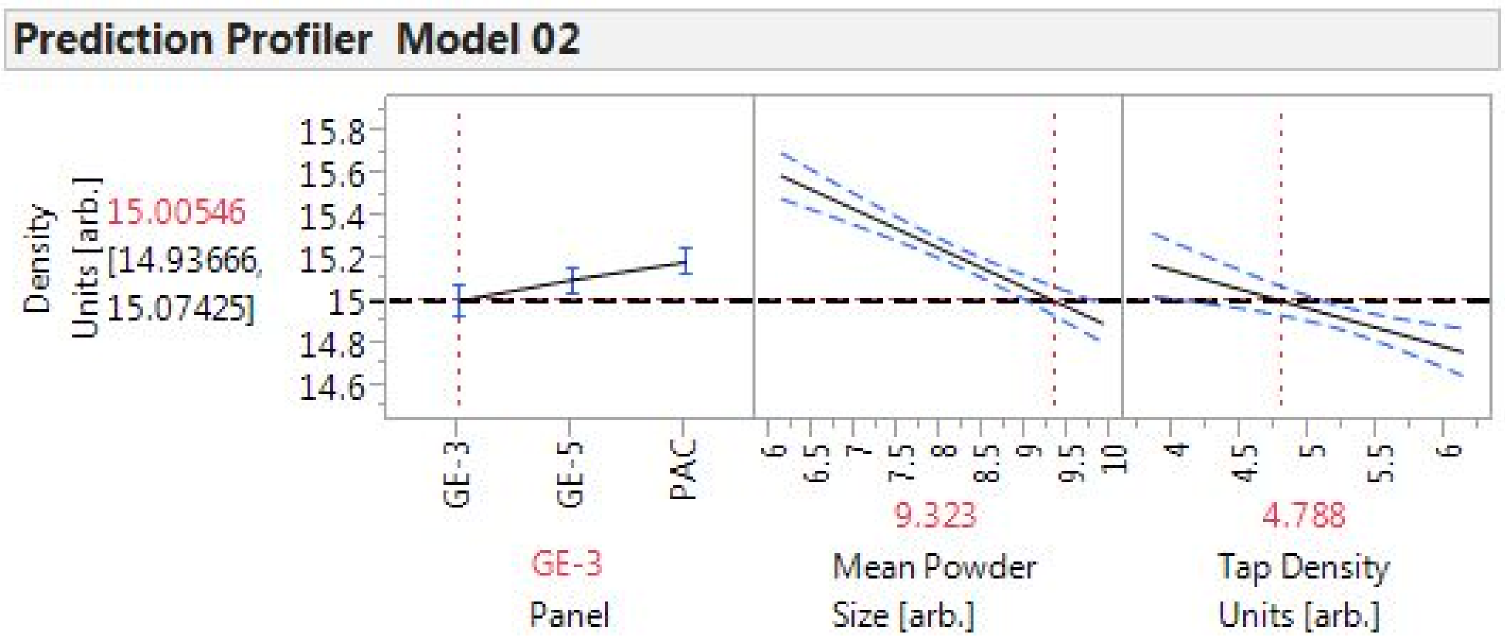


Summary of Fit

RSquare	0.775997
RSquare Adj	0.767562
Root Mean Square Error	0.160655
Mean of Response	15.18835
Observations (or Sum Wgts)	249

Predictive Process Control Regime

- Simple predictive process control: Use JMP model profiler to “dial-in” mean powder, tap density and sintering furnace to achieve optimized density
- Creates process capability to direct Mo powder blends to a specific sintering furnace for optimal downstream manufacturing



Predictive Analytics Positive Impacts

- Decrease of rolling mill scrap
  - **Savings of $25K in first month**
  - Stabilized manufacturing in the downstream Rolling Mill Department
- Predictive process control (PPC) has optimized Elmet sintering furnace unit process operations
- Predictive modeling results have been used in technical discussions with an external suppliers to Elmet
  - More quantitatively rigorous and statistically supported discussions to drive external suppliers to provide higher quality metal powder.

Predictive Analytics & Small Businesses

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- Small business manufacturing seascape
- How do small businesses navigate to predictive analytics?

Ram Island Ledge Lighthouse at entrance to Portland Harbor

Robert F. Bukaty/AP File/2009

Small Business Manufacturing Seascape

- Lack of integration in manufacturing systems
  - Multiple generation process tools in manufacturing line
  - Fleet mentality: no discrete tool process control but the ensemble is treated as a fleet or herd
  - No global control of unit process tool set [i.e. recipe download; "golden recipe comparison, or data upload etc.]
- Statistical process control (SPC) is "rear facing"
  - Most cases: SPC primarily used for past scrap product or manufacturing crashes (i.e. historical)
  - Worst case: process data is collected in separate data silos and never mined or examined by process engineering.
  - No basis for proactive decisions on unit process control

- Risky reliance on ***"…it has always worked this way. What happen?"***

Tools for Predictive Analytics

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Providing small business manufacturing the tools of predictive analytics:

- Integrate the factory floor
- Establish “value added” statistical process control
- Cultivate predictive process control to reduce scrap
- Bottom line: Predictive analytics must be simple, address the most significant factory manufacturing issues and be simple.

Conclusions

- Small businesses can transition to smart manufacturing by implementing simple predictive process control (PPC)
- The agile mining and exploration of available data streams will generate new and significant manufacturing learnings:
  - Sintering furnace optimization
  - Identification and reduction of product variation

Acknowledgments

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- Alec Brown — Material Scientist
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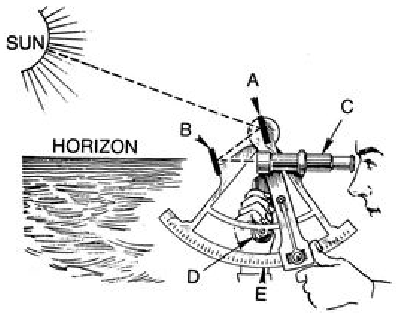
- Sara Dunne — Analytic Sciences Laboratory Director
- Dave Littlefield — Operations Manufacturing Engineer
- Robert Marcus — Manufacturing Engineer
- Thomas Sauberlich — Manufacturing Engineer
- Bruce Tremblay — Operations Equipment Engineer



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Professor Perepezko — University of Wisconsin

Elmet D1 Manufacturing Group

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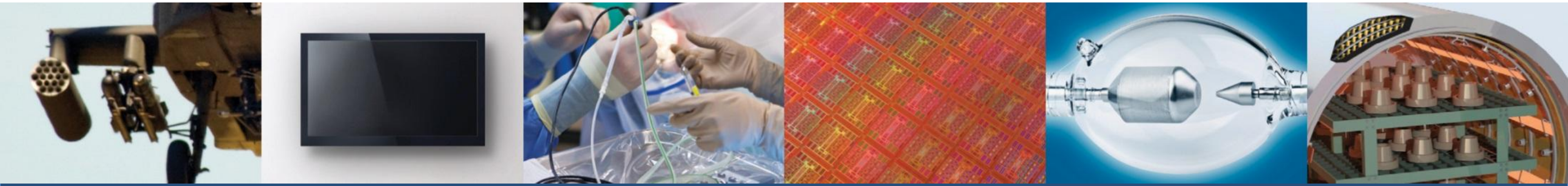
Thanks for your attention. Questions and some answers

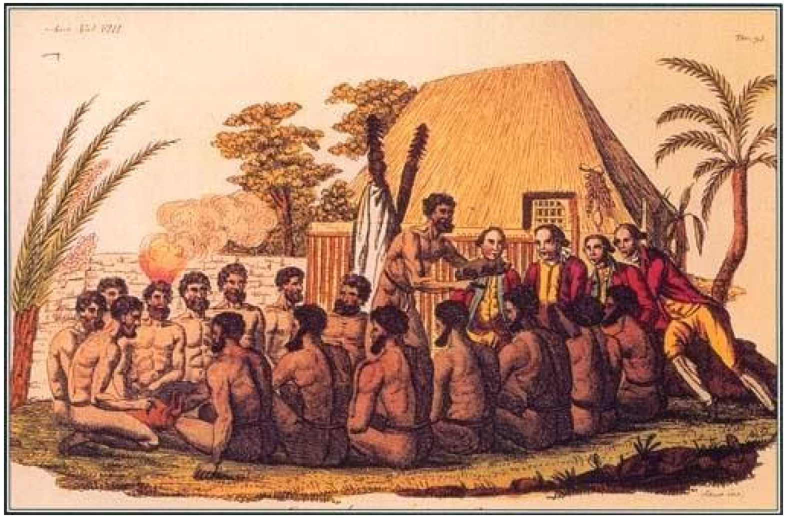
www.elmettechnologies.com



Select Smart Manufacturing Papers:

- **"Reduction Of Greenhouse Gas Emissions By Metal Interconnect Etch Process Optimization"**
- P. S. Frankwicz, L. Gardner and T. Moutinho, Proceedings of the 220th Electrochemical Society Meeting, Boston, MA October 2011; ECS Transactions, 41 (34) 1-7 (2012)
- **"Process Monitoring And Control Of Semiconductor Production Tools Using JMP"**
- P.S. Frankwicz, D. Scipione, S. Coleman and J. Devlin, Proceedings of New England SAS Users Group Meeting NESUG 2010, Baltimore, MD October 2010.
- **"Process Excursion Detection Using Statistical Analysis Methodologies In High Volume Semiconductor Production"**
- P.S. Frankwicz, S.E. Romano and T. Moutinho, Proceedings of New England SAS Users Group Meeting NESUG 2009, Burlington, VT September 2009.
- **"Comprehensive Interconnect Etch Tool Qualification Methodology For High Volume Mixed Technology Node Production"**
- P. S. Frankwicz, R. Clark, K. Hayes, M. Johnson, L. Kennedy, D. Scipione, C. Viera and T. Moutinho; Proceedings of International Symposium on Semiconductor Manufacturing; Santa Clara, California, USA; ISSM 2007, pgs.350-353.

Pacific Indigenous Marine Navigation



Using the tools we have.

Polynesian Voyaging Society