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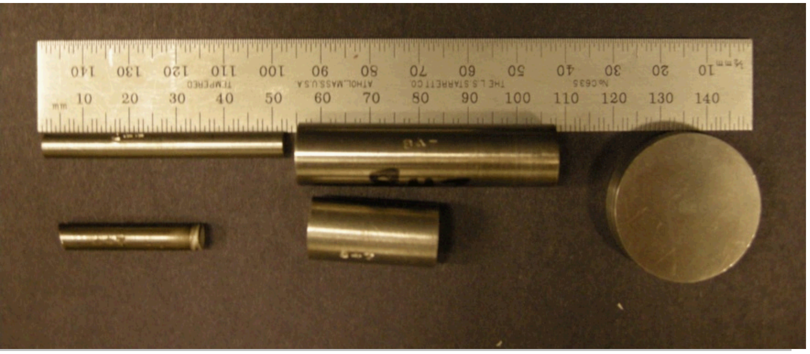

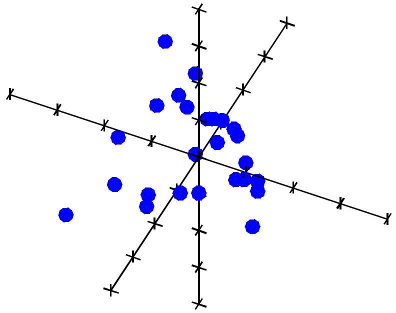

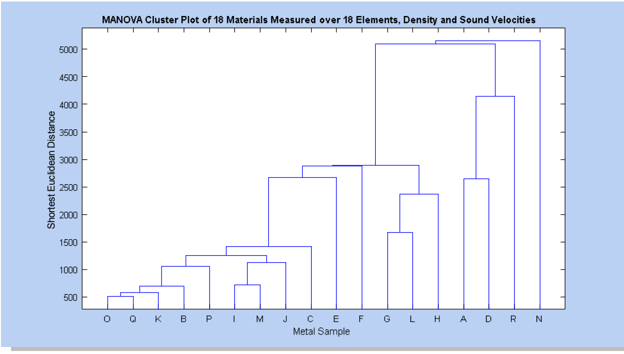

Materials Assurance Through Orthogonal Materials Measurements

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Materials Science and Engineering Center

Sandia National Laboratories

U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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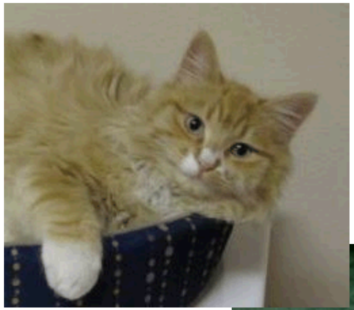
Overview

- Purpose for the research
- Orthogonal Testing
  - Develop a combinatorial materials “signature” based upon a broad set of simple materials tests
  - Develop algorithms to use very different kinds of data sets. The combined data set or “spectrum” will contain orthogonal interrogation properties
- Material Measurements
  - XRF
  - Density
  - Sound Velocity
- Multivariate Analysis of Variance (MANOVA)
  - Probabilities and dendrogram
- Conclusions
- Next Steps

Motivation

- **Problems necessitating materials assurance**
  - Counterfeit and adulterated materials put products *and people* at risk
  - In 2007 pet foods adulteration using melamine to deceive protein tests. In 2008, melamine in infant formula in China.
  - Counterfeit electronic parts are becoming a concern in the DoD acquisition process
- **Most materials specifications are not enough**
  - Simply passing the specification does not ensure correct material
  - Specs may miss alterations in material
- **Robust specifications can be cost prohibitive**



"We do not want a $12 million missile defense interceptor's reliability compromised by a $2 counterfeit part."

General Patrick O'Reilly
Director, Missile Defense Agency
November 8, 2011

Category 1 Requested authentic part numbers for obsolete and rare parts							
Analysis performed	**DAA6**	**DAA6**	**IHH1**	**MLL1**	**MLL1**	**YCC7**	**YCC7**
Visual Inspection	Fail	Fail	Fail	Fail	Fail	Fail	Fail
Resistance to Solvents (RTS) and Scrape Test	N/A	N/A	Fail	N/A	N/A	Pass	Pass
Package Configuration and Dimensions	Pass	Pass	Pass	Pass	Pass	Pass	Fail
X-Ray Florescence Elemental Analysis	Fail	Fail	Pass	Fail	Fail	Pass	Pass
Real-Time X-ray Analysis	Pass	Fail	Pass	Pass	Pass	Fail	Pass
Scanning Electron Microscopy (SEM) Analysis	Fail	Fail	Fail	Pass	Pass	Fail	Fail
Solderability Test	Pass	Pass	Pass	Pass	Pass	Pass	Pass
Dynasolve Test	N/A	N/A	Fail	N/A	N/A	N/A	Fail
Delidding and Die Microscopy	Fail	Fail	Fail	Fail	Fail	Fail	Pass
Suspect counterfeit	**Yes**	**Yes**	**Yes**	**Yes**	**Yes**	**Yes**	**Yes**

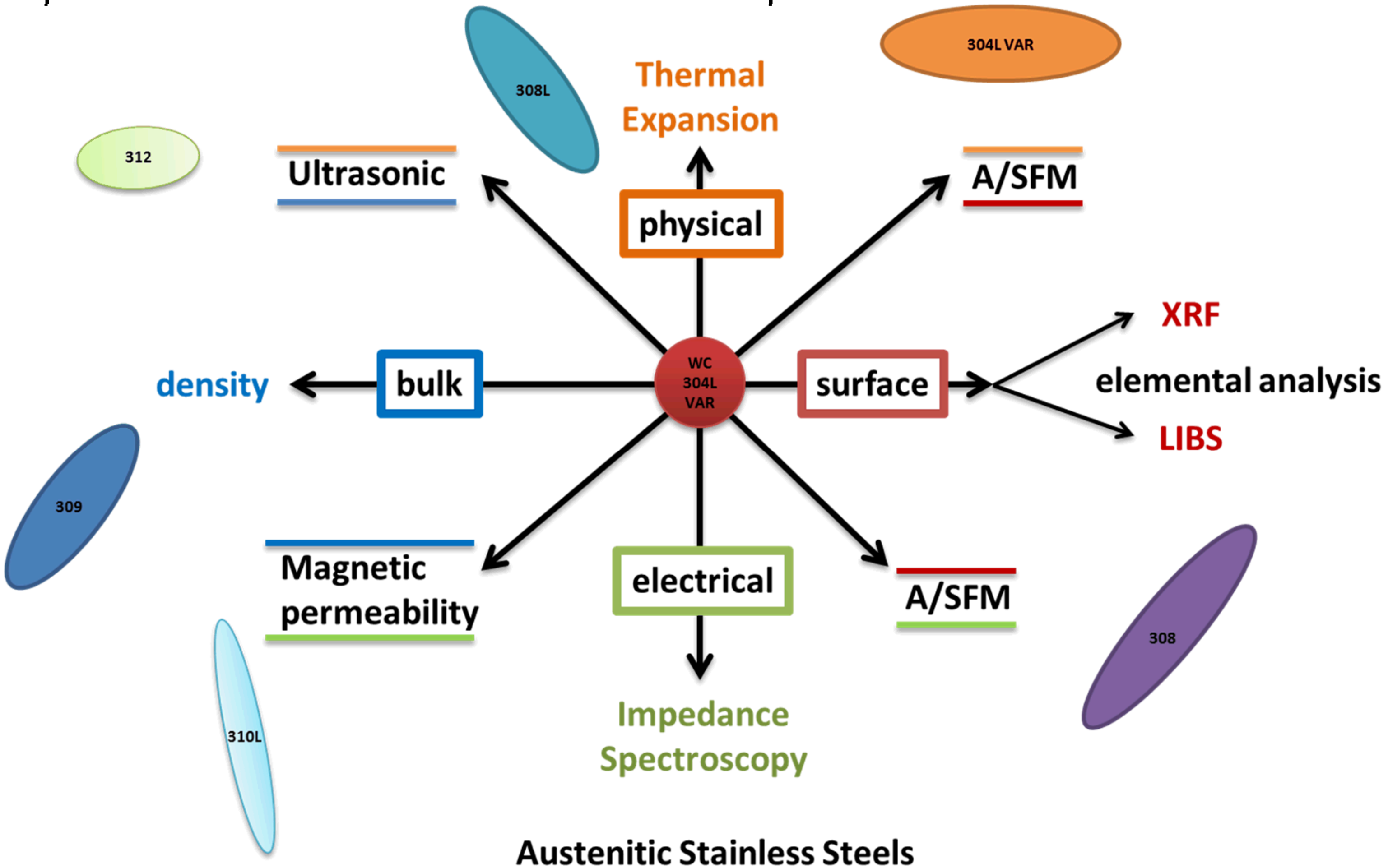
Source: GAO analysis of SMT test results.

Senate Inquiry Into Counterfeit Electronic Parts in the Department of Defense Supply Chain
May 21, 2012

Orthogonal Testing

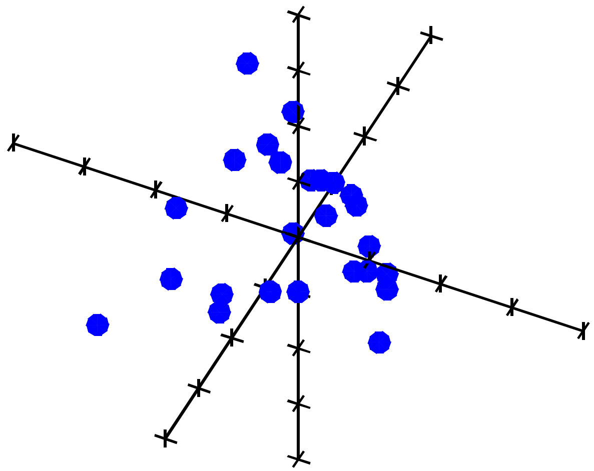
Non-destructive, non-specification test space

⇒ can be performed on raw material and/or finished parts

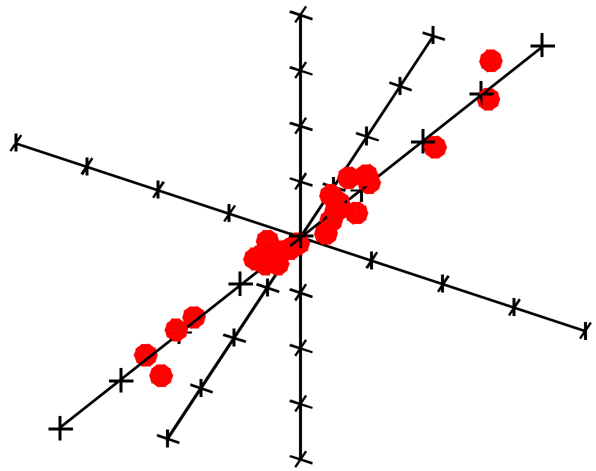


Orthogonal tests measure diverse, complementary, and ideally, uncorrelated properties.

Variable Correlations



Uncorrelated Variables



Highly Correlated Variables

Steel Samples

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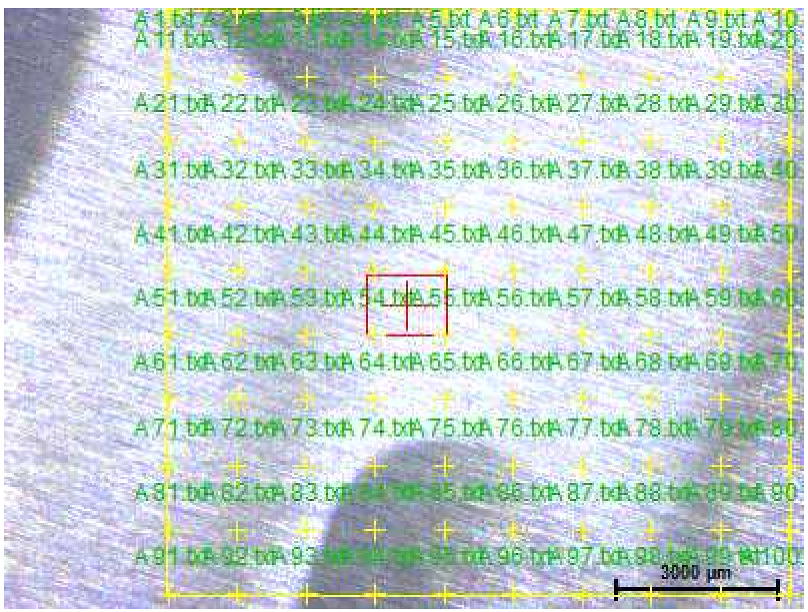
Samples chosen for diversity within available steels on-hand. Showing 18 of the 19 samples in our current regime.

Sample ID	Type	Description
A	SRM	Cr 13-Mo 0.9 modified AISI 410
B	SRM	Cr 18-Ni 9 modified AISI 321
C	SRM	Cr 24-Ni 13 modified AISI 309
D	SRM	Cr 9-Mo 0.3 modified AISI 403
E	SRM	16 Cr - 10Ni
F	SRM	18.5 Cr - 9.5 Ni
G	CarTech	316
H	CarTech	316L
I	CarTech	866 type 303-Se
J	CarTech	type 347 #538
K	BAS	austenitic stainless steel
L	BAS	austenitic stainless steel
M	BAS	austenitic stainless steel
N	BAS	austenitic stainless steel
O	BAS	austenitic stainless steel
P	BAS	austenitic stainless steel
Q	BAS	austenitic stainless steel
R	BAS	austenitic stainless steel

SRM – Standard reference material
BAS – Bureau of analyzed samples, ltd.
CarTech – Carpenter Technology Corporation

X-ray Fluorescence (XRF)

- Elemental analysis
- Surface technique
- Fast, non-destructive, in common use



- Bruker M4 Tornado m-XRF

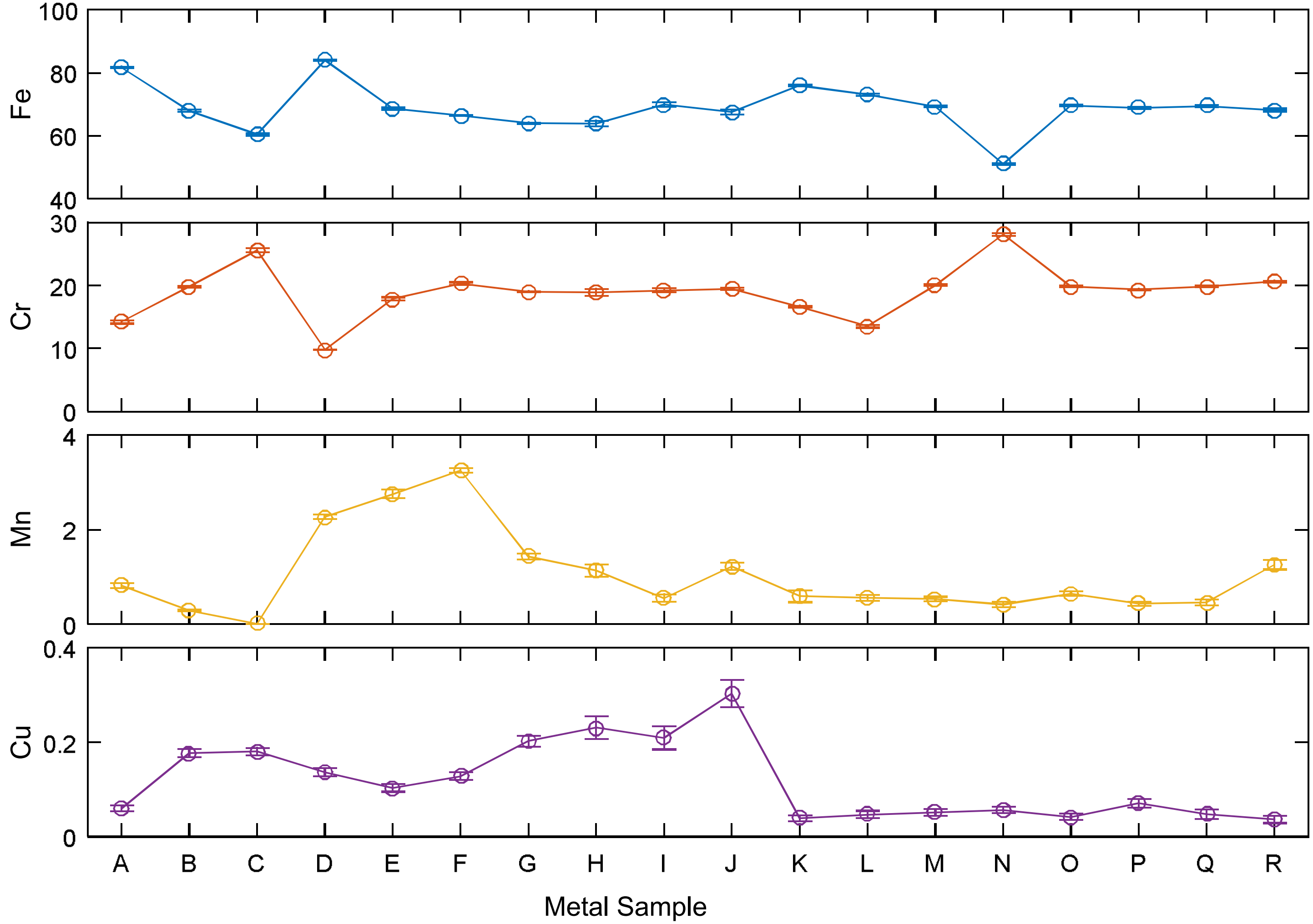
- 100 point measurements
  - Rh X-ray source focused to 25um
  - each spot was measured for 30s
  - X-ray energy 50kV and 200uA
  - Chamber under vacuum at 19 mBar

Image source: www.bruker.com

Elemental Compositions

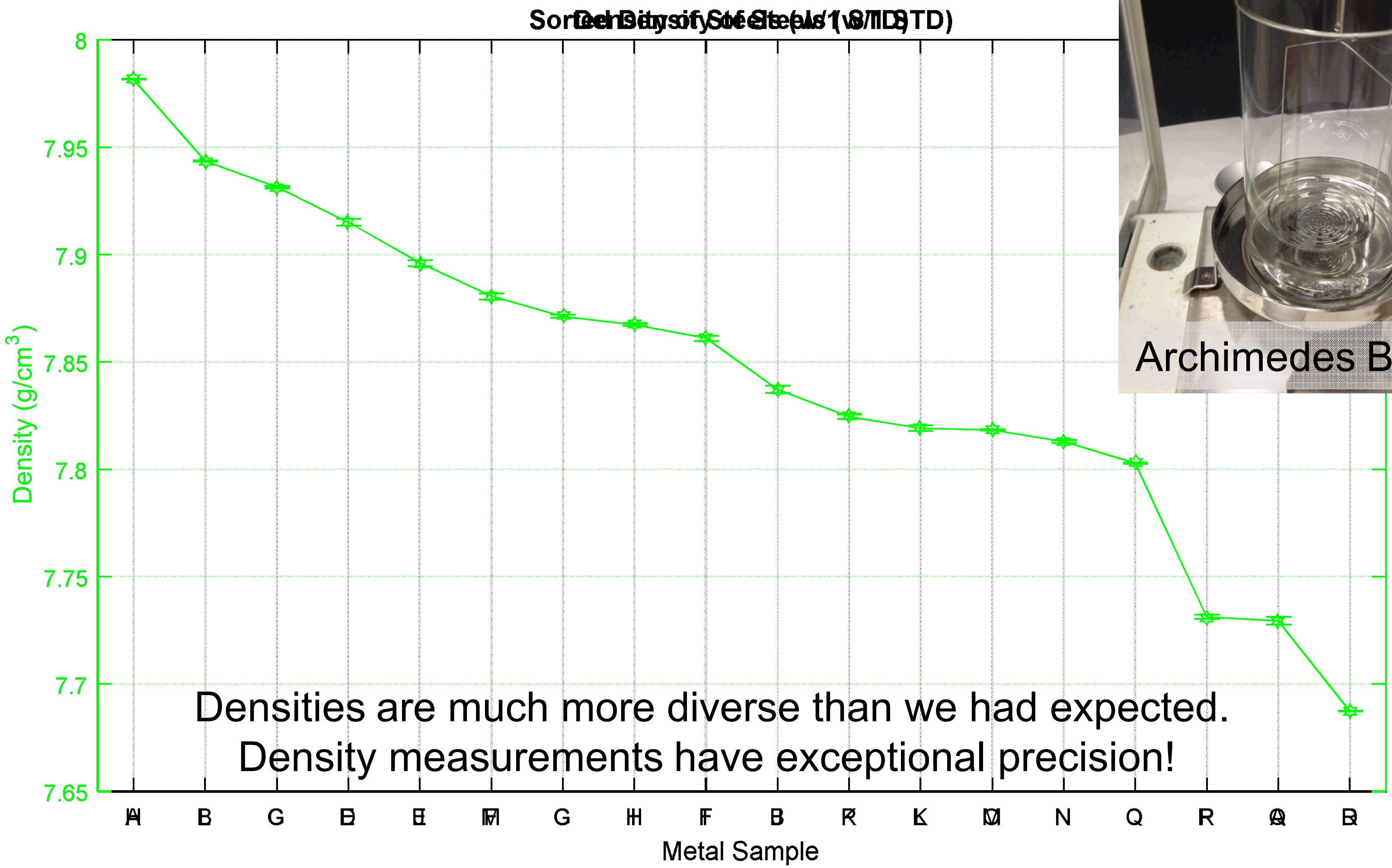
Average Composition

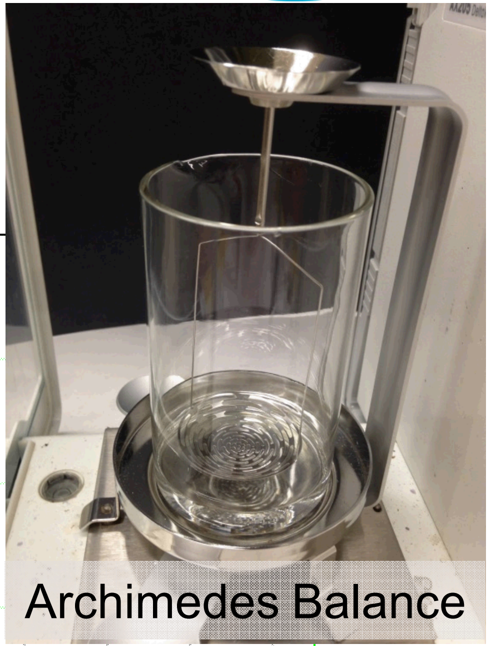
Element	Mass %
Fe	68.91
Cr	19.00
Ni	8.43
Mn	1.03
Si	0.58
Mo	0.52
W	0.26
Sn	0.24
Al	0.20
Pb	0.19
Nb	0.17
Ag	0.13
Cu	0.12
Ti	7E-02
Se	4E-02
V	4E-02
Zr	2E-02
S	2E-02
Ta	1E-02
Co	1E-02
P	7E-03
As	2E-04



Good sample variability – very small variances!

Density – A bulk property





Archimedes Balance

Densities are much more diverse than we had expected.
Density measurements have exceptional precision!

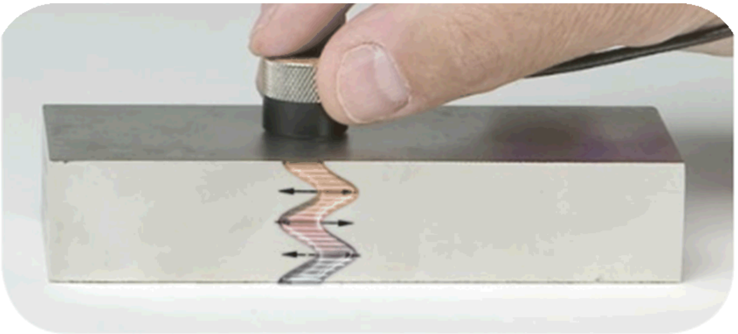
Density measured using Mettler Toledo model AX205 DeltaRange, means and standard deviations from three repeat measures.

Acoustic Properties

- Measurement system:
  - 2.25 MHz, 5 MHz Panametric -NDT single element longitudinal and shear wave transducers.
  - Olympus Panametrics –NDT model 5800 computer controlled Pulse/Receiver
  - NIPXI GPIB, NI-PXI1031 data acquisition unit
  - NI-PXI-5124 12 bit 200 MS/s Digitizer


*

Longitudinal wave transducers – A106S and V109 (Pulse-echo Technique)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Shear (transverse) wave transducers – V154 and V155

* Graphics from www.olympus-ims.com/

Sound Velocities in Steels

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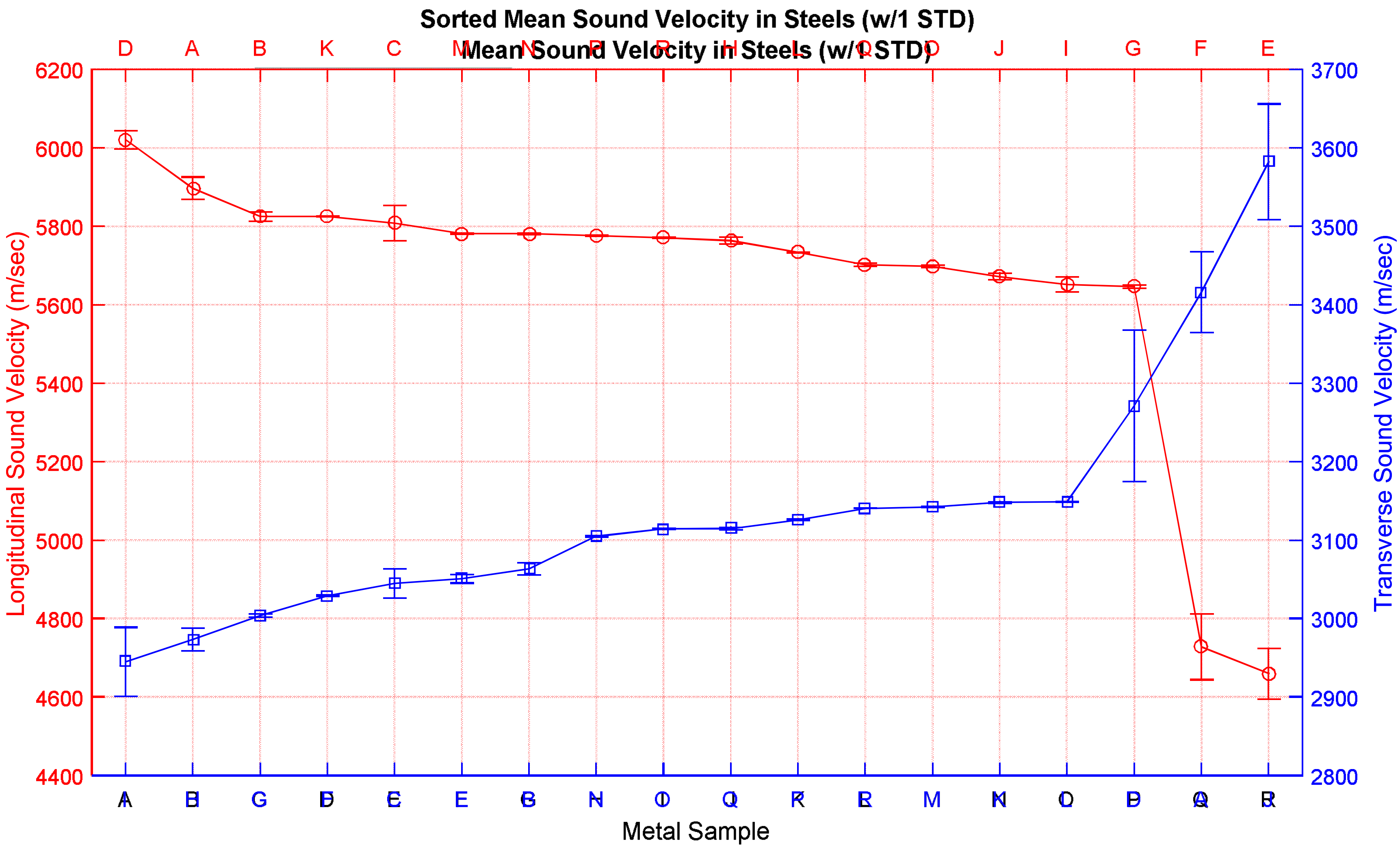
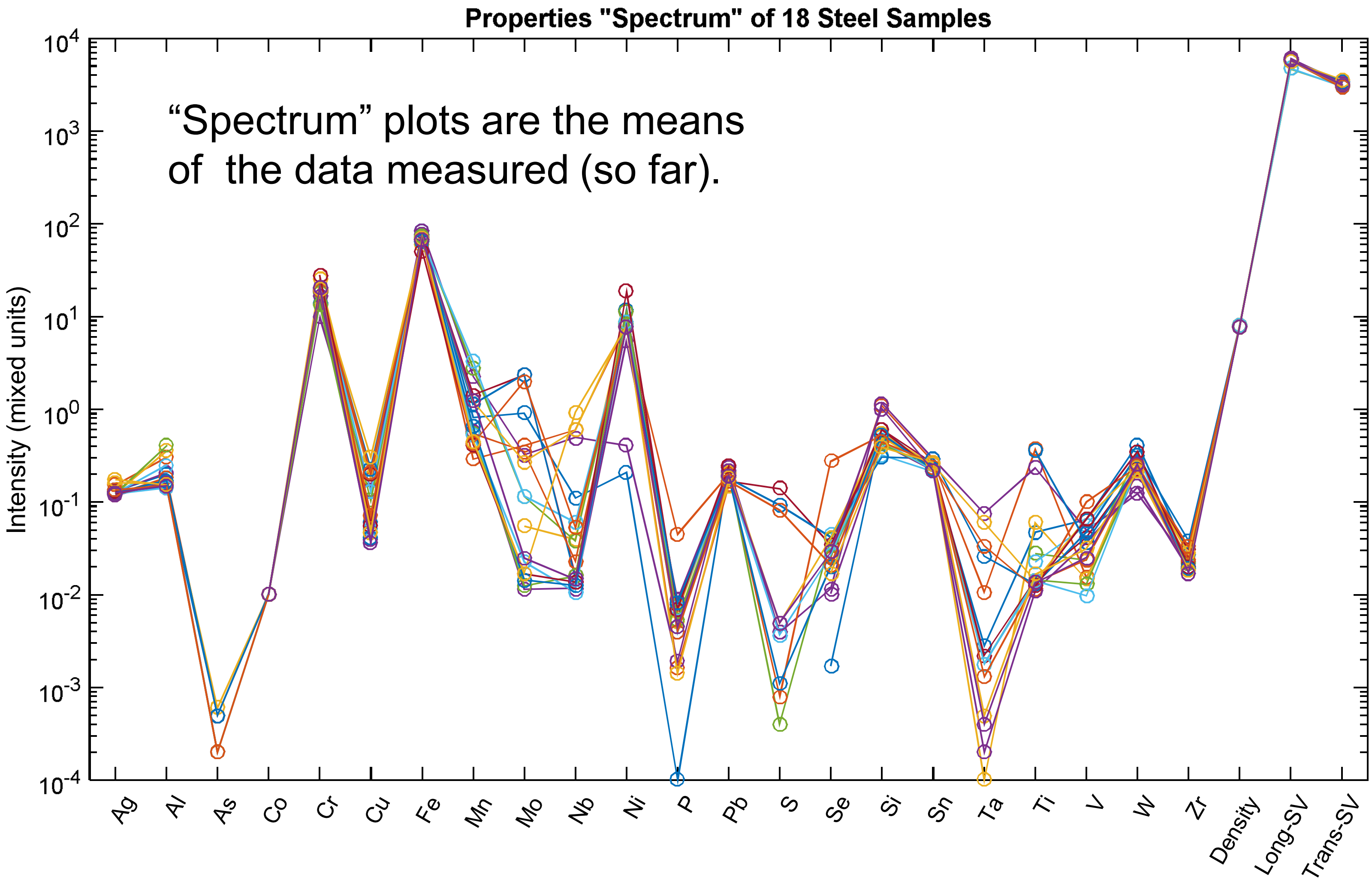


Larger variances associated with inhomogeneity in steel samples.

Means and standard deviations from several repeat measures.

Materials Spectra



Pairwise MANOVA for Steels

Compute pairwise one-way multivariate analysis of variance using the materials "spectra" as variables. H_0 is the means are the same.

Simulate values for density and sound velocities using means and standard deviations from random normal distribution.

For this analysis omitted these elements due to all-zero values or zero variance:
As, Co, S, & Ta

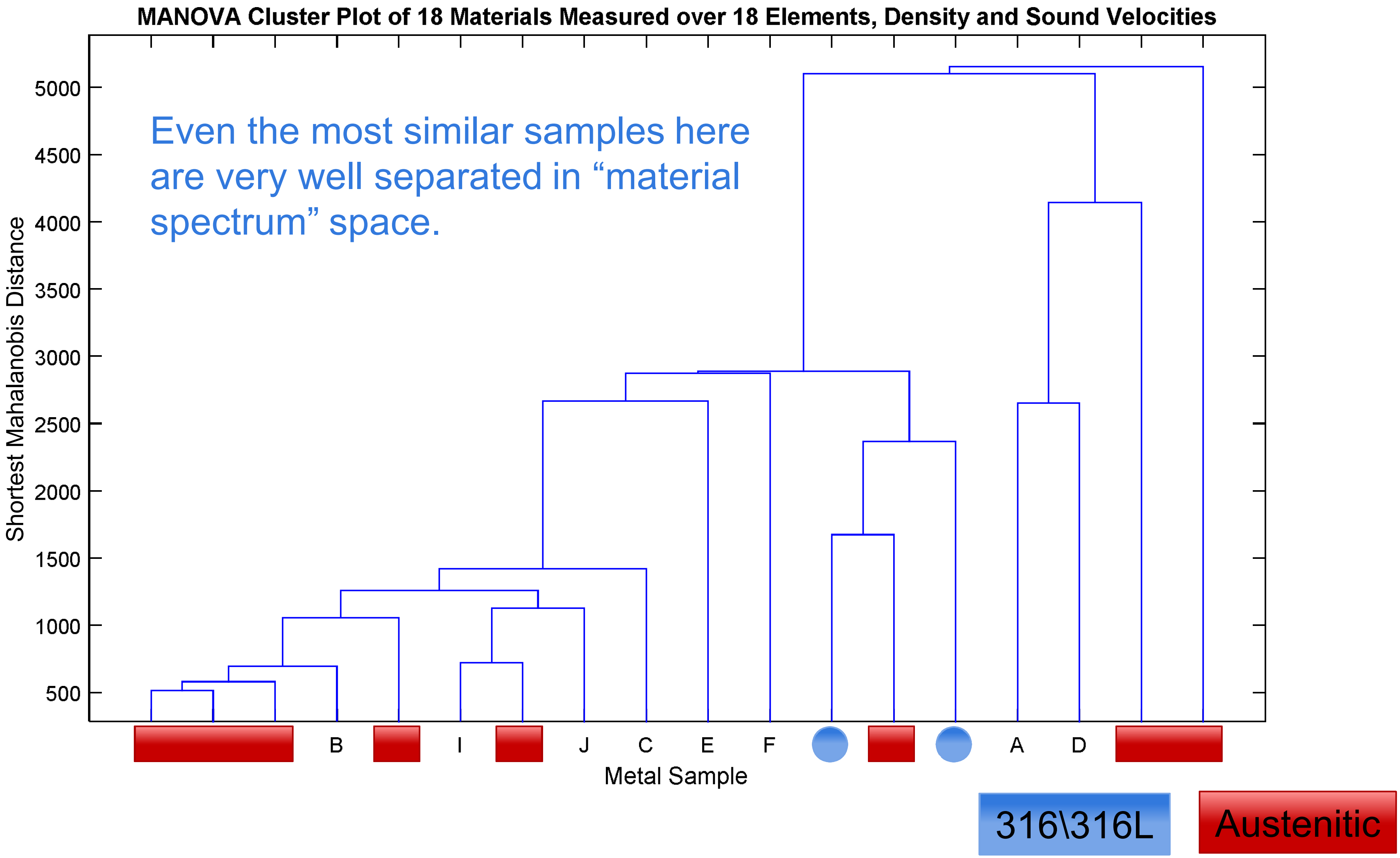
Table contains p-values for comparing population mean vectors. $p<.05$ considered significant.

Here, all pairs are significant, thus all H_0 are rejected.

	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R
A	1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
B		1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
C			1	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
D				1	0	0	0	0	0	0	0	0	0	0	0	0	0	0
E					1	0	0	0	0	0	0	0	0	0	0	0	0	0
F						1	0	0	0	0	0	0	0	0	0	0	0	0
G							1	0	0	0	0	0	0	0	0	0	0	0
H								1	0	0	0	0	0	0	0	0	0	0
I									1	0	0	0	0	0	0	0	0	0
J										1	0	0	0	0	0	0	0	0
K											1	0	0	0	0	0	0	0
L												1	0	0	0	0	0	0
M													1	0	0	0	0	0
N														1	0	0	0	0
O															1	0	0	0
P																1	0	0
Q																	1	0
R																		1

Materials "spectra" show perfect discrimination!

MANOVA Single Linkage Dendrogram

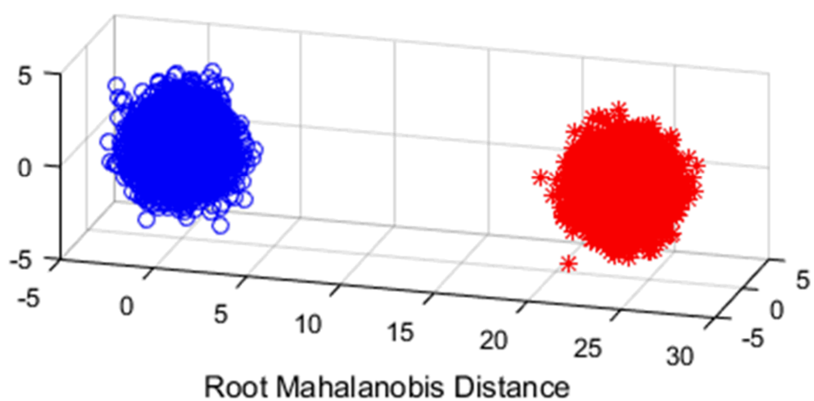


Mahalanobis Distance

$$D = [\bar{\mathbf{x}}_1 - \bar{\mathbf{x}}_2]^{\mathrm{T}} \mathbf{S}^{-1} [\bar{\mathbf{x}}_1 - \bar{\mathbf{x}}_2]$$

$\bar{\mathbf{x}}_i$ is the mean of the $\boldsymbol{i}^{\text{th}}$ group and $\boldsymbol{S}$ is the covariance matrix.
Here, Mahalanobis is a squared distance.
When $\boldsymbol{S}$ is the identity matrix, Mahalanobis distance is equal to the square of the Euclidean distance between the two groups.

$\bar{\mathbf{x}}_1 = 0$, $\boldsymbol{\sigma}_1 = 1$. $\bar{\mathbf{x}}_2 = 1$, $\boldsymbol{\sigma}_2 = 1$. $\bar{\mathbf{x}}_2 = 10$, $\bar{\mathbf{x}}_2 = 20$, $\bar{\mathbf{x}}_2 = \sqrt{516}$, $\boldsymbol{\sigma}_2 = 1$.



Two groups each having 10,000 points, with multivariate-normal distributions.

Conclusions

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- Selected a sampling of metals with limited diversity to evaluate our orthogonal measurements
  - Carefully chose 19 standard samples for their diversity within the family of steels
- Performed initial materials tests on at least 18 similar metals
  - XRF, density, sound velocity
  - Creating a materials characteristic "spectrum"
- Conducted multivariate analysis on the current set of measurements
  - MANOVA shows very nice separation of the multivariate means
- Orthogonal materials measurements shows excellent promise as a tool for materials assurance!

Next Steps

- Complete additional measurements on materials properties
  - Ferrite number
  - Electrical conductivity
  - Thermal conductivity
  - Thermal expansion
- Perform simulations with larger variance terms
  - Determine level of variance effect on materials separation
- Perform identical analyses on a “corrupted” sample

Acknowledgements

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