Engineering Laboratory

Materials Standards for Additive Manufacturing

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National Institute of Standards and Technology
U.S. Department of Commerce

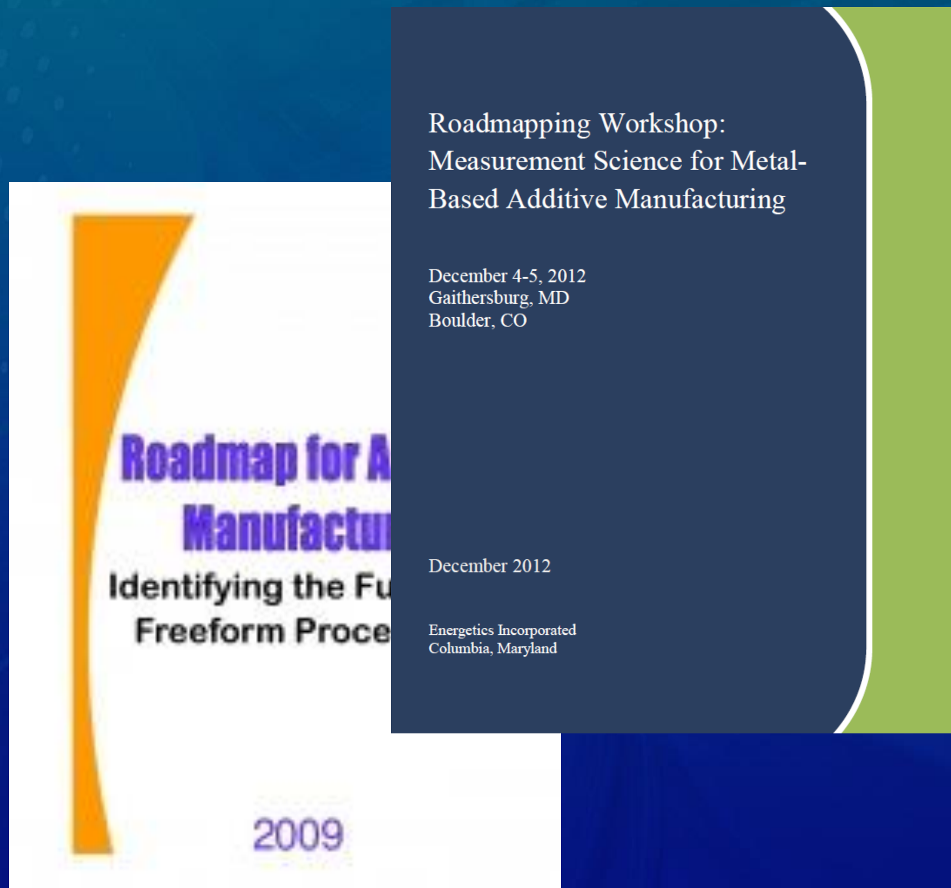
Material Standards for Additive Manufacturing

Powder

Process

Part

AM Materials is a top priority…



AM#2: Project Overview

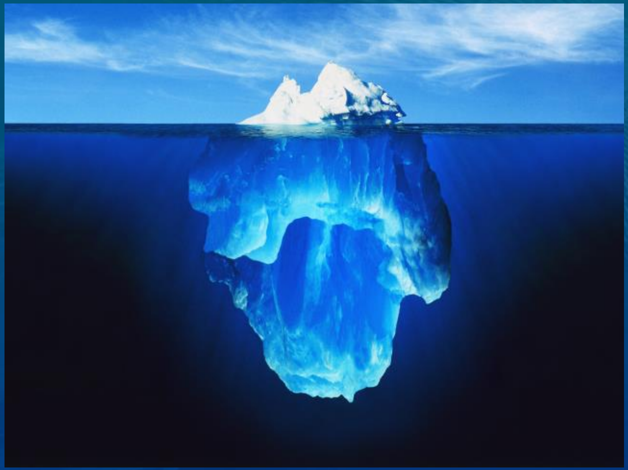
- Materials Properties and Qualification remains a significant barrier to more widespread adoption of AM technologies
- Currently, the additive manufacturing (AM) industry does not have the confidence, and is unable to rigorously verify, that nominally identical AM powders are in fact identical, resulting in unconfirmed powder properties.
- This lack of confidence in material properties is also true for parts produced by AM.
- Need publically available, published property data for both powders and AM materials.
- Project's Technical Focus:
  - Standard test methods for metal powder characterization
  - Standard test methods to obtain material properties of AM parts
  - Test protocols, procedures, and analysis methods for industry round robin testing of AM materials for consensus material property data

Collaborators

- Internal:
  - Engineering Laboratory - Materials and Structural Systems Division
  - Material Measurement Laboratory - Materials Science and Engineering Division
  - Information Technology Laboratory - Statistical Engineering Division
  - Center for Neutron Research
- External:
  - Oak Ridge National Laboratory, Morris Technologies, U. of Louisville, Picatinny Arsenal, Aberdeen Proving Ground, UTEP, GE Global Research, Carpenter Powder Products, Oxford Performance Materials, ASTM

Results to Date

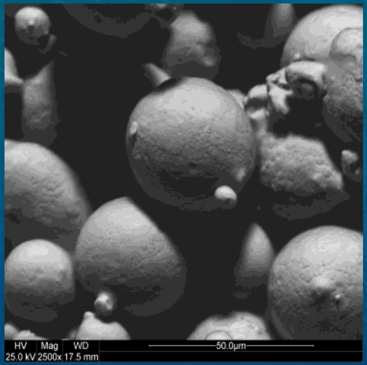
- Background Studies
- Powder Characterization
- Material Properties
- Stress
- Porosity for Process Monitoring



Background Studies

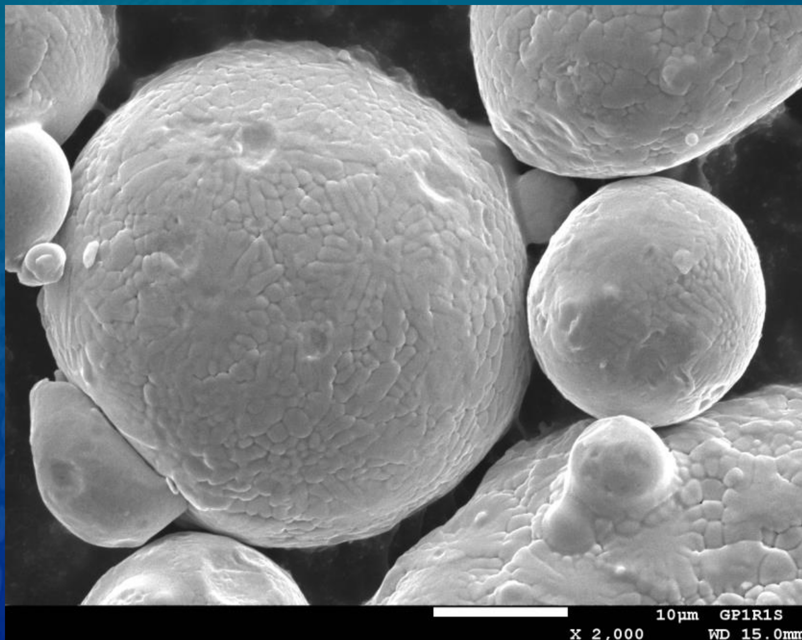
- NISTIR 7847 assessed current state-of-the-art for material property testing of bulk metal material properties. (Slotwinski, Cooke, Moylan)
- NISTIR 7873 assessed current state-of-the-art methods for characterizing metal powder. (Cooke, Slotwinski)
- Determined the applicability of current state-of-the-art methods for AM parts and AM powder and documented conclusions in internal report, results to be published in 2013. (Slotwinski, Moylan, Cooke)

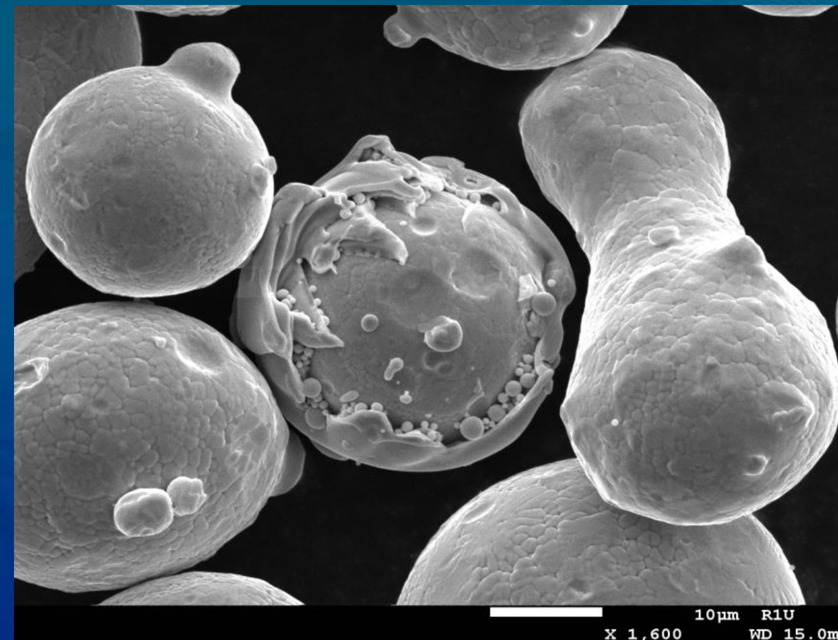
Powder Characterization

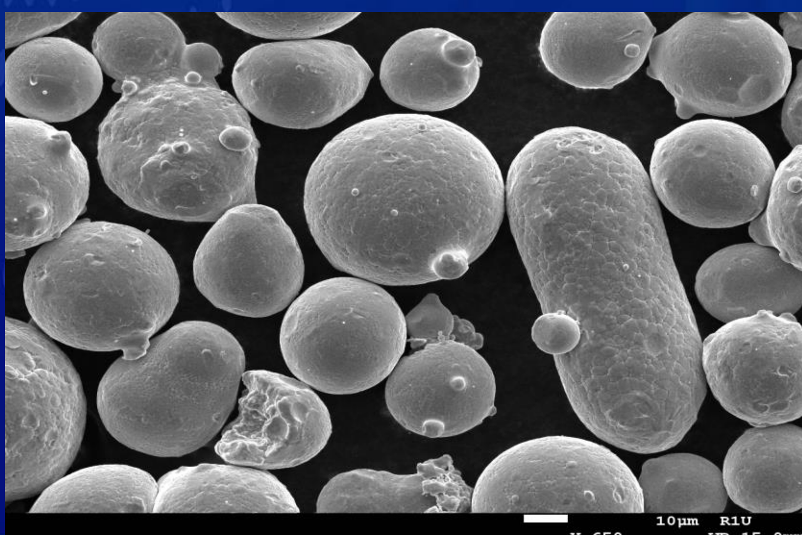


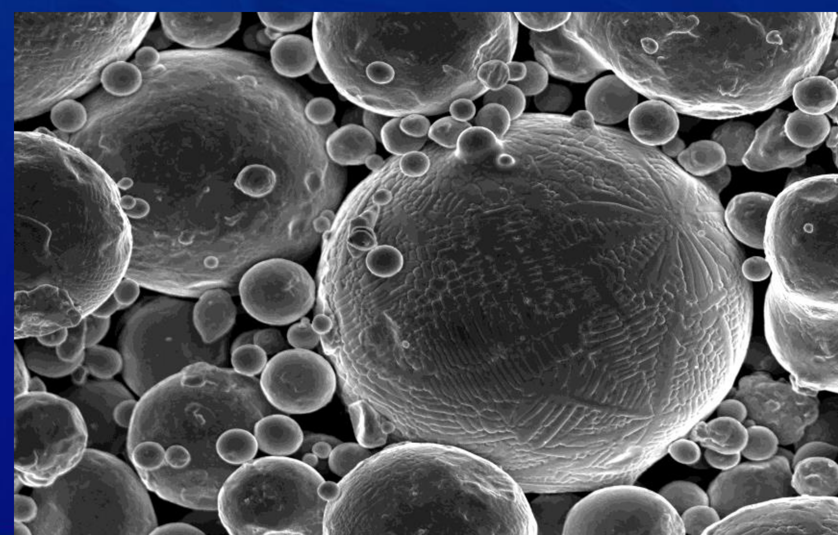
- Current Foci:
  - Variability of nominally identical powder, effects of recycling (e.g., exposure of powder to multiple builds), documented properties of round robin powder (for potential future correlation with mechanical properties)
- Characteristics of Interest:
  - Size (and size distribution), morphology, chemical composition, flow, thermal properties…
- Measurement Methods:
  - SEM (size, morphology), Quantitative X-Ray Diffraction (chemical composition), Laser Diffraction (size distribution), X-Ray Computed Tomography (morphology), X-Ray Photoelectron Spectroscopy (gives photoelectron energy characteristic of elemental chemical states)
- Results:
  - Recycling reduces austenite, increases ferrite content in Stainless Steel (QXRD), but does not change surface chemistry/atomic concentration (XRPS)
  - Nominally identical Stain Steel and Round Robin CoCr powder lots have same base chemical composition (QXRD)
  - CoCr and Stainless Steel powder morphology is “quasi-spherical”
  - Laser diffraction measurements and analysis are currently underway.

“Quasi-Spherical” Powder









Powder Characterization Laboratory

- Setting up powder characterization lab to:
  - Develop appropriate measurement techniques and standards
  - Compare industrially common benchtop techniques to more advanced measurement methods (such SEM, laser diffraction, energy-dispersive X-ray, X-ray computed tomography…)
- Drafted powder characterization standard, recently submitted to ASTM (WK40606 – *Standard Guide for Characterizing Properties of Metal Powders Used for Additive Manufacturing*) and chairing task group.

Future Work on Powder Characterization

- Finalizing WK40606 into ASTM Standard
- Determining effects of customized powder lots (e.g., skewed size distributions, controlled morphologies, etc.) on mechanical properties
- Flow, thermal properties measurements
- More specific AM powder characterization standards

Material Properties Round Robins

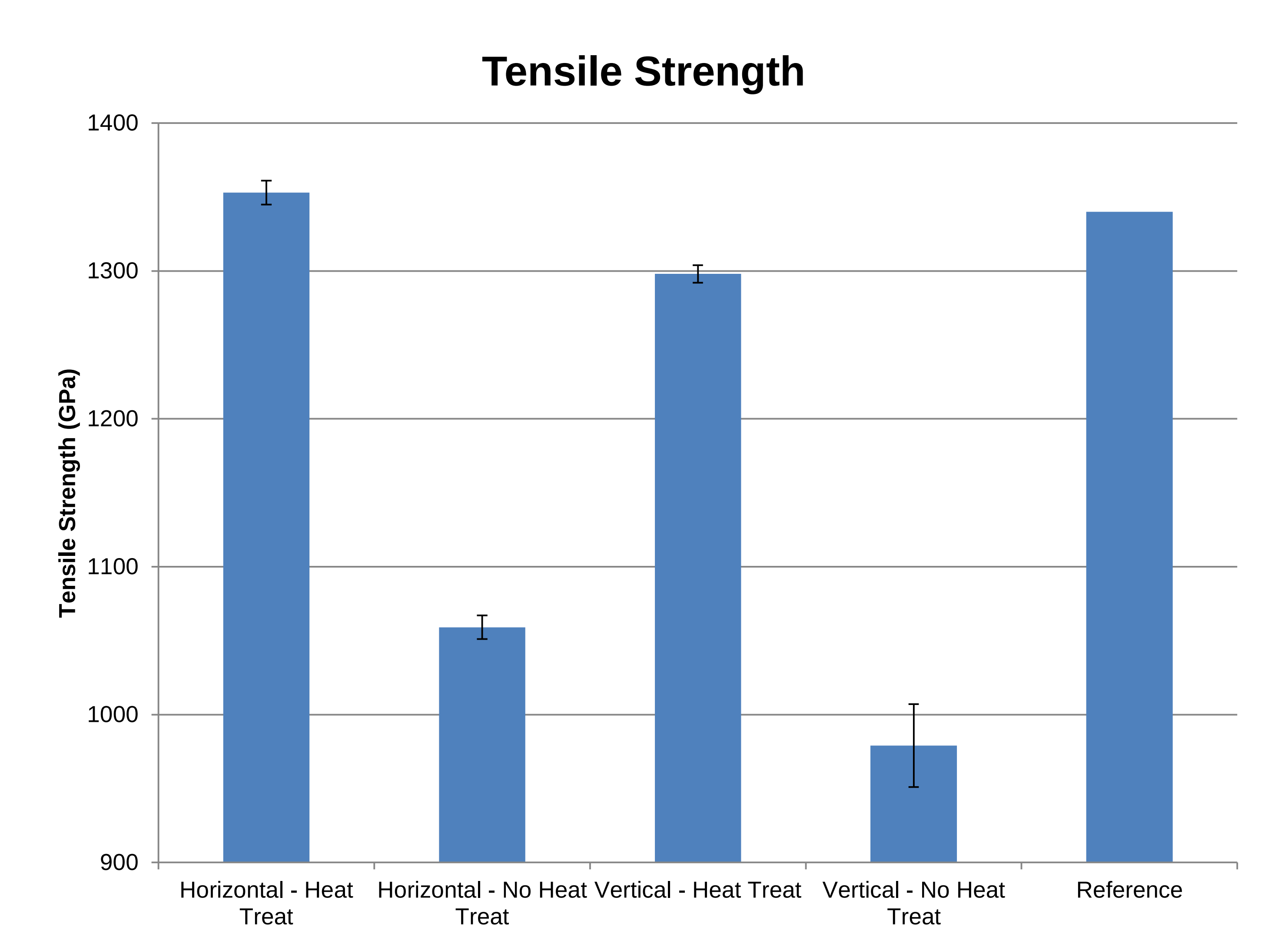
- Two NIST-funded round robin tests (one internally led, one externally led)
- Mainly focused on laser-based DMLS powder bed systems, but internal study also includes two e-beam (ARCAM) AM systems for comparison
- Preparation of test protocols, procedures, test specimens, powder specifications, and analysis methods
- NIST statistical and material science expertise for design of experiments and analysis of internally led round robin
- Both have careful controls and procedures on powder, build parameters, post processing and material property measurements
- “Tests to develop the test”

Material Properties Round Robins

	AMC	NIST
Material	Inconel 625	Cobalt Chrome (MP1)
Status	Final Planning	Underway
Process	3x DMLS (M270)	6x DMLS (M270) + 2x ARCAM
Types of Specimens	Tensile, high-cycle fatigue, low-cycle fatigue (room temperature and high temperature)	Tensile (room temperature)
Number of Specimens	120	64
Participants	5	8
Goal	Establish protocol for making and testing material coupons for additive processes; establish protocol for generating and reporting DMLS mechanical property data with the intent of possible inclusion in a design allowables database (such as MMPDS)	Same; baseline study of material property variability for nominally identical builds

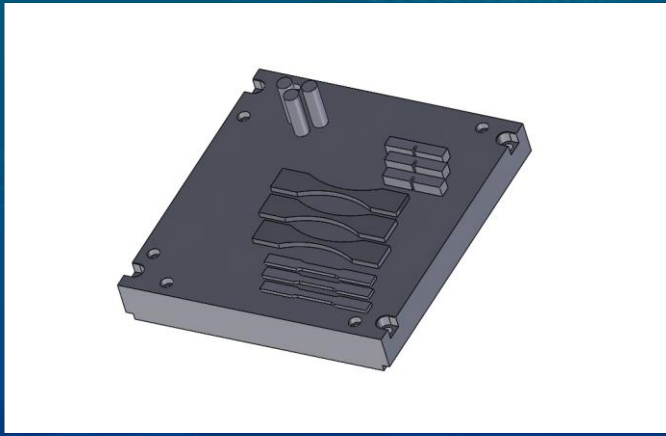
Tensile Properties: EOS GP1 Stainless Steel (17-4)

- Stress-strain behavior of NIST-made tensile bars is very repeatable
- Stress-strain behavior qualitatively is very similar to vendor data and reminiscent of cold steel
- NIST measured Tensile Strength (1000 MPa typical) is generally higher than vendor data (930 MPa ± 50 MPa) and NIST measured Elastic Modulus (160 GPa typical) is generally in consonance with vendor data (170 GPa ± 30 GPa)
- Material exhibits discontinuous yielding, and has significant work hardening (strengthening of material during plastic deformation)
- Vendor-recommend heat-treatment results in increased yield strength (not decreased!) and decreased ductility (not increased!)
- Slight directional-dependent anisotropy present - specimens are slightly weaker (7%) when build vertically, heat-treatment improves this slightly (4%).



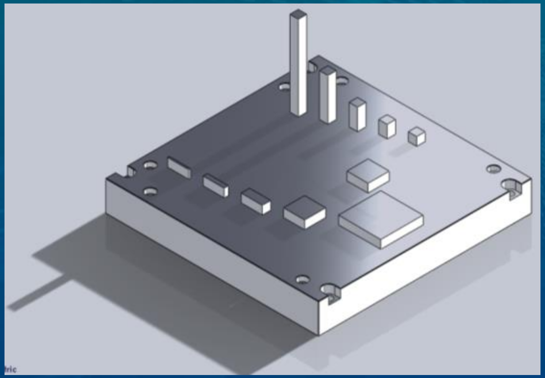
Tensile Strength
Tensile Strength (GPa)
1400
1300
1200
1100
1000
900
Horizontal - Heat Treat
Horizontal - No Heat Treat
Vertical - Heat Treat
Vertical - No Heat Treat
Reference

Additional Mechanical Properties Work



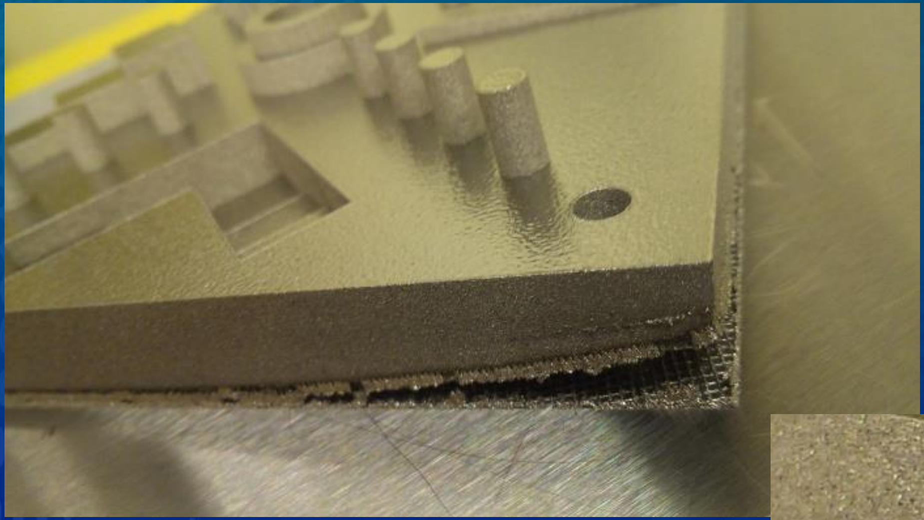
- A variety of other test specimens made in three different configurations:
  - As-built
  - Machined to final shape from similarly-sized and shaped solids
  - Machined to final shape from large blocks
- Compression, high-cycle fatigue, charpy, tension
- Awaiting heat-treatment, removal, and mechanical testing
- Results will feed into development of AM mechanical testing standards

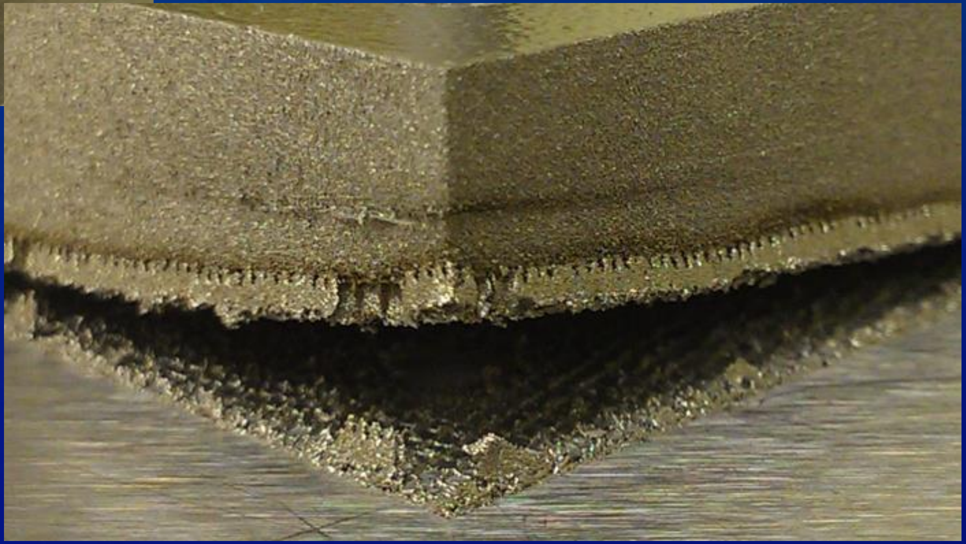
Thermal Stress



- The extremely rapid and localized melting and cooling results in residual thermal stresses
- Interest in residual thermal stresses present after a build, as well as the effects of post-processing (shot-peening, heat treatments) and part removal on stress.
- Working with both ORNL and NCNR for neutron imaging of stress (complimentary capabilities)
- Have delivered test samples to both ORNL and NCNR, awaiting results

AM Thermal Stress Can Be Significant…

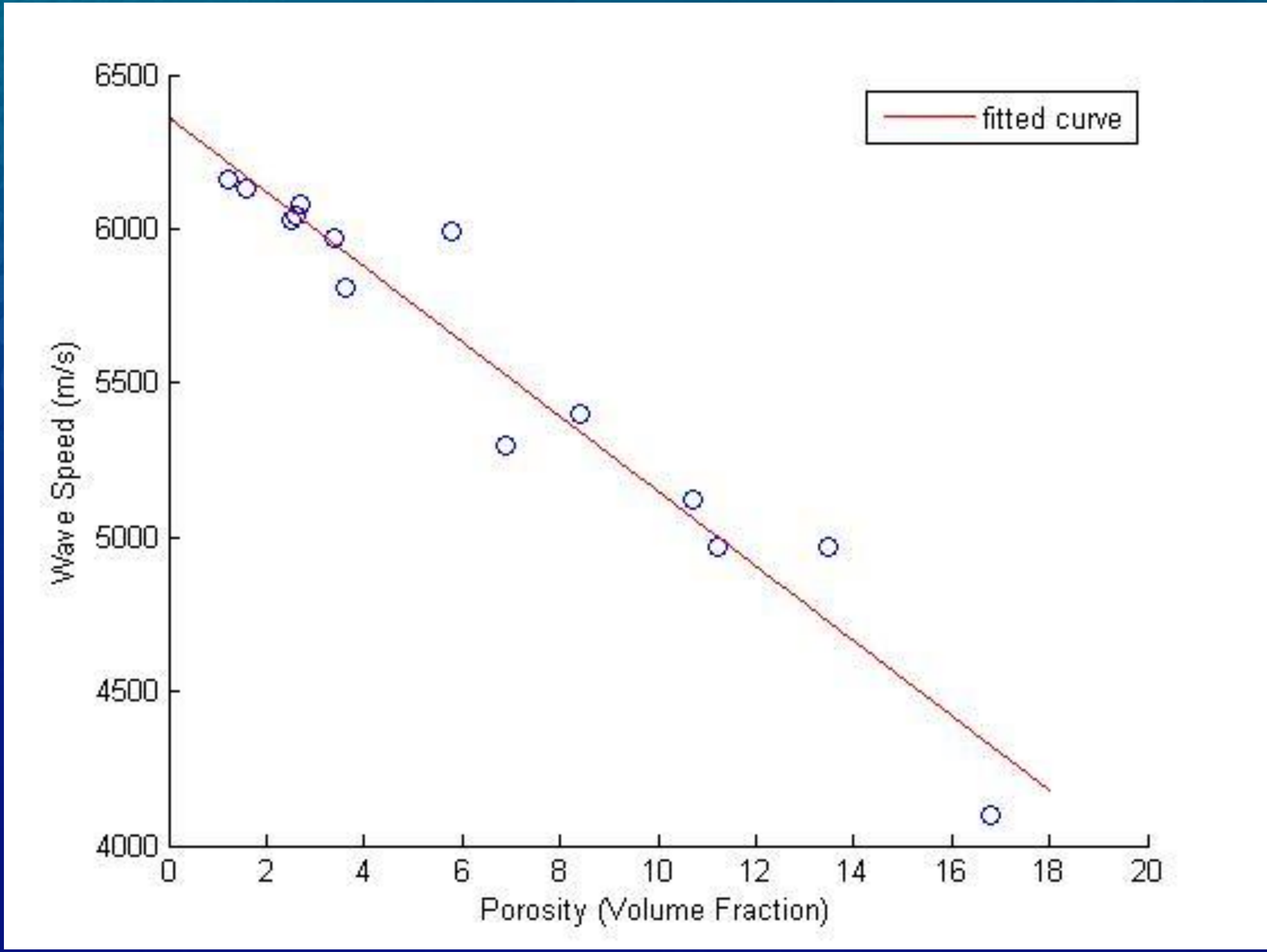




Ultrasonic Porosity Sensor: Process Monitoring

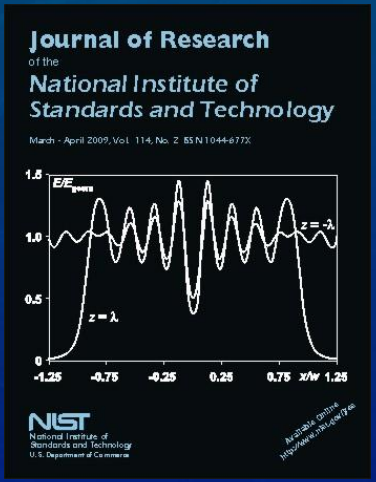
- Ultrasonic velocity in material decreases with increasing porosity (models vary, most are linear)
- Different models all predicted that a 0.2% change in porosity would be detectable ultrasonically ($\Delta \cong 0.02$ mm/μs)
- Porosity samples
  - Partnered with Morris Technologies to produce three sets of CoCr samples with varying porosity (0% - 72%)
  - Measured ultrasonic velocity with three different techniques
  - Collaborating with others to determine “final” porosity (Archimedes, X-ray computed tomography, Mass/Volume, Optical) as well as porosity morphology
- Designed sensor system for use in EOS M270 for process monitoring

Ultrasonic Porosity Sensor



NIST Journal of Research: Special Issue on Additive Manufacturing, Summer 2013 – J. Slotwinski, Guest Editor

- Overview of Additive Manufacturing
- Test Artifact
- Powder Characterization
- Mechanical Properties of AM Parts
- Sustainability of AM Processes
- Theory, laboratory experimentation and sensor design for UT porosity sensor
- Z-Axis Interferometer Measurements



Summary

- Additive manufacturing is gaining momentum in the US, but there are significant materials-related issues to overcome before gaining wider adoption
- Current methods for mechanical testing and powder characterizatoin assessed, round robins started
- Focus on powder metrology, AM materials mechanical data and standardizing AM materials measurement methods
- Highly inter-disciplinary, with multiple participants and partners.
- Significant publications this year.