NASF SURFACE TECHNOLOGY WHITE PAPERS
82 (6), 26-41 (March 2018)

The 53rd William Blum Lecture
Presented at NASF SUR/FIN 2016
in Las Vegas, Nevada
June 6, 2016

Additive Manufacturing and Surface Finishing

by
Dr. Melissa Klingenberg
Recipient of the 2015 William Blum
NASF Scientific Achievement Award

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Editor's Note: The following is the Powerpoint presentation by Dr. Klingenberg in delivering her William Blum Memorial Lecture at SUR/FIN 2016, in Las Vegas, Nevada on June 6, 2016.

Overview

- General Additive Manufacturing (AM)
- Conventional AM Technologies and Challenges
- Monitoring, Control, and Material Improvements
- Surface Finishing Opportunities
- Newer AM Technologies
- Gaseous AM/Carbonyl Processing
- Summary
- References
- Acknowledgements

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Additive Manufacturing (AM)

Definition: Any manufacturing process capable of making 3D objects from a digital model, or building-up controlled 3D features onto an existing object, typically layer by layer.

- Creating a part from the bottom up
- Significant dimensional restoration of parts with detailed features

Complementary Techniques:

- **Rapid Prototyping (RP):** a group of techniques used to quickly fabricate a scale model of a physical part or assembly using three-dimensional computer aided design
- **Rapid Tooling (RT):** production of tooling directly from an RP process

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AM Attraction

- Ability to design freely – less constraints in complexity
- Lightweight part designs
- Less parts for a system
- Faster route to production
- Highly personalized (as in medical implants)
- No need to retain expensive molds
- Materials conservation (use less materials due to design flexibility)
  - Leads to reduced costs for expensive alloys
- Ease of modifying prototype design
  - Modify CAD drawing and reprint

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AM Industry Information

- Basic development
  - Plastic → Biological materials → Metals → Ceramics → Electronics materials
  - Single material → Multi-materials → Functionally graded components
- Desirable part attributes
  - Low volume
  - Geometrically complex
- Tremendous growth potential
  - 25% annual growth rate over its 25-year history
  - Available build volume, speed and capacity increasing annually
- Production parts available today
  - Aerospace
  - Medical
- Media hype is overselling AM

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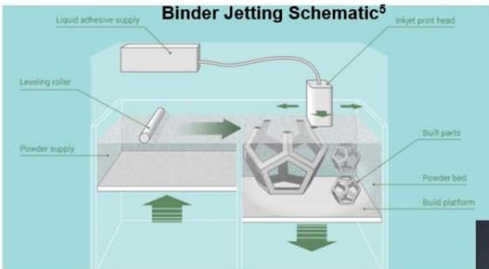
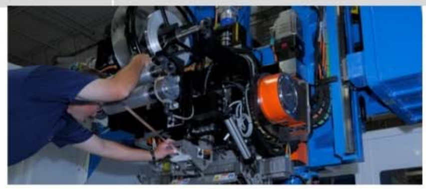
Conventional Metal AM: Unrelated to Coatings

Category	Description	Manufacturers	Materials	Envelope (cm)
Binder Jetting (3DP)	Selective liquid resin deposition – powder bonding; sinter for strength (metals, ceramics, sand or plastic)	Ex-One, Voxeljet, 3D Systems, Höganäs	Polymers, silica/sand, alumina, zircon, ceramsite, cerabeads, glass, carbon, 17-4 PH SS, 420 SS, 316 SS, WC, SiC, FeCrAl, Ti, Ag, Cu, Mg, Al, bronze, Cr-Co, Stellite, IN625, IN718, Hastelloy, Ti6Al4V, Cp Ti, M2, H13, maraging steel,M	400 x 200 x 100
Sheet Lamination (Ultrasonic Welding of Foils)	Sheets of material bonded together (paper/glue, polymer/glue or heat, and metal via welding/bolts)	Fabrisonic (UAM), Automated Dynamics (new)	Ti, steel, Cu, and Al alloys (proven metal pairs shown below)	183 x 183 x 91

Binder Jetting Schematic[5]



Binder Jetting Produced Parts[2]



Fabrisonic Unit and material pairs[3]

SiC fibers in metal laminate[3]

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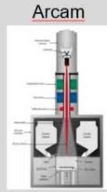
Conventional AM: Related to Coatings

Category	Description	Manufacturers	Materials	Envelope (cm)
Powder Bed Fusion – thermal energy fusion of powders	Metal Laser Sintering ~ DMLS, DMLM, SLM Spread metal powder on platform, raster laser across surface only where part features are desired for that layer under N2 or Ar; subsequent layer spread for the second layer build 1) Laser (0.1-1 kW Yb fiber – metals; CO_2 - polymers)	EOS, SLM Solutions, 3D Systems (Phenix), Concept Laser, Renishaw, ReaLizer  SLM part created by CTC	AlSi12; AlSi10Mg; AlSi7M; AlSi9Cu3; AlMg4,5MnO4; Cast Al alloys (400 and 300 series); CoCr and CoCrMo superalloys; maraging, tool, and high grade steels; IN 625 and 718, Hastelloy X; 316L, 17-4PH, and 15-5 PH SS; CP Ti, Ti6Al4V, TI6AL4V ELI, Ti6Al7Nb; bronze alloys; precious metals	80 x 40 x 50
	Spread metal powder on platform, raster electron beam across surface only where part features are desired for that layer; subsequent layer spread for the second layer build 2) Electron Beam Melting (50-3000W; several melt pools maintained)	Arcam  Ebeam arrangement[19]	Ti6Al4V, Ti6Al4V ELI, Ti Grade 2, CoCr (ASTM F75)	35 x 35 x 38

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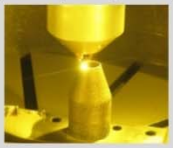
Conventional AM: Related to Coatings

Category	Description	Manufacturers	Materials	Envelope (cm)
Directed Energy Deposition (DED) - focused thermal energy melts materials as deposited	Use high power laser to melt powder into a molten pool to build or repair parts in layers 1) Laser (metal powder)  **LENS Process** [29]	Optomec, DM3D, DMG Mori, BeAM, Efesto LLC, Insstek, Accufusion, Trumpf-Sisma	CP Ti, Ti 6Al4V, Ti 6-2-4-2 Ti 6-2-4-6, Ti 48-2-2, Ti 22Al--23Nb; IN 625, IN 718, C-276, Waspalloy, Nistelle C; Stellite 6, 21, 31; Tool steel (H13, S7, A-2); Mold steel (P20, P21); P/M steel (CPM 1V, 9V, 10V, VIMCRu 20); 13-8, 17-4 PH, 15-5 PH, 304, 316, 410, 420, AM355, 309, 416; 4047 Al; GRCop-84, Cu-Ni; W, Mo, Nb; TiC, WC, CrC; 4140+WC, IN625+WC, Stellite 21+WC	90 x 150 x 90
	Feed wire, layer by layer, onto a form and electron beam heats the metal under vacuum to create near-net shape parts 2) Electron beam free form fabrication = electron beam direct melting Electron beam (metal wire) (up to 42 kW)	Sciaky, NASA (EBF[3])  **Sciacky Unit** [22]	Ti (Ti6Al4V, CP Ti); CP Ta; IN 718, IN625, Hastelloy X; L605 (Co alloy), 2319 Al, 2219 Al, CP Al; 17-4 PH, 304, 316, 418 SS; Cu-Ni (70/30, 30/70), Nb (C-103 and CP Nb), W, Mo; Cu and Ni gradient material	580 x 122 x 122  **Ebeam direct melted part** [22]

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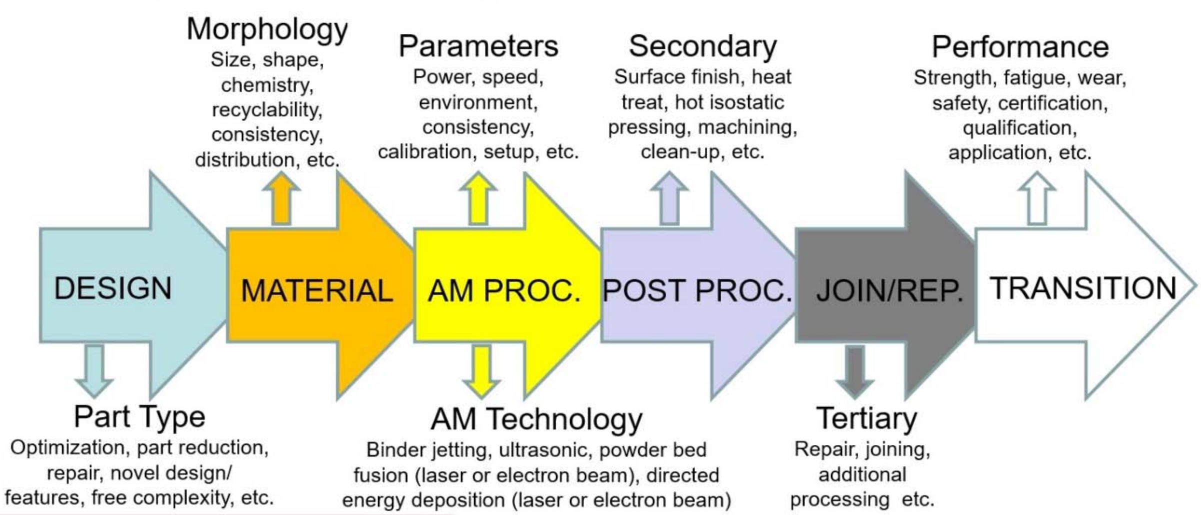
AM Cycle Challenges



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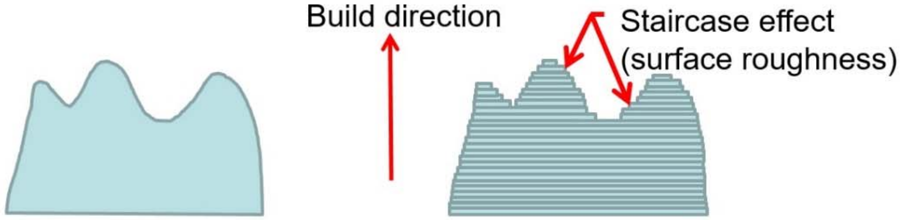
Typical Conventional AM Build Contours

- Staircase effect along edge
  - Step size depends upon AM process used and layer thickness
  - Minimal along any given horizontal layer



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Monitoring, Control, and Material Improvement

- In Process Monitoring
  - Conventional and Thermal Imaging: examine cooling and adjust heat
  - Optical Emission Spectroscopy (LENS) – compositional and lack of fusion
  - Ultrasonic Porosity Sensor – velocity decreases with increasing porosity
  - Photonic Doppler Velocimeter (sheet lamination) - resonance determines average interfacial bonded area
- Non-Destructive Inspection
  - Optical Emission Spectroscopy
  - Computed 3D Tomography (CT)/microtomography – radiography shows defects through part
  - ANSI Recognized Methods: visual, dye, acoustic, magnetic, thermal, ultrasonic, vibration, laser, neutron based methods
- Post Processing
  - Mechanical: sandblasting, cutting, grinding, peening, polishing, MMP
  - Thermal: sintering, standard or vacuum heat treatment, HIP
  - Chemical: electropolish, plating, infiltration

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Surface Finishing Opportunities

- Enhanced coating leveling ability
  - Improved leveling of functional coatings would enable reduced machining/polishing of parts
  - Enhanced adhesion on porous substrates
- Process combinations
  - Combining post-treatment operations into single batch processing
- Improved sensor technology
  - Use of sensors in coating operations could be extrapolated to AM processing for enhanced control and improved repeatability
- Expanded use of process modeling for prediction of material properties
  - Aid a user in selecting the best AM process for application needs
- Increase material choices

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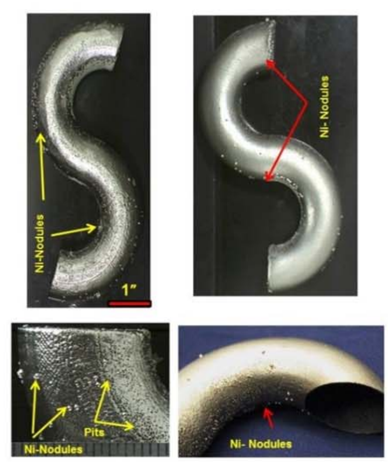
Newer AM Processing Methods

- Cold Spray
  - Extensive dimensional restoration, typically non-structural focus
- Thermal Spray
- Electroforming
  - Old process, but gaining more applications
- Structural Plating
- Deposition onto AM produced forms
  - Physical vapor deposition
  - Electrochemical plating
  - Gaseous Deposition (carbonyl)
    - Thin to thick film process
    - Use gases, evaporating liquids, or chemically gasified solids as source materials



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Deposition Onto Removable Forms

- Additively create polymer forms that maintain thermal profile
- Deposit coatings onto forms
  - Electrodeposition (electrolytic or electrolessly, depending on part complexity)
    - Rough surfaces are replicated - more surface finishing is required
    - Need multiple activation cycles to reduce coating stresses
  - Physical vapor deposition
    - Variety determined by material, part complexity, and necessary rate
    - Coating stress increases with increased thickness
  - Gaseous AM: Chemical vapor deposition
    - Variety depends on desired material and crystalline structure, compositional purity required, and part thickness necessary
- Thermally or chemically decompose form or retain form for certain applications
- Conduct post processing finishing, if necessary

PEKK part produced by OPM
Tg + 160 °C

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Gaseous AM

- Substrate surface, material reactivity, and energy input govern deposition
  - Substrate surface – roughness, surface contamination, degree of chemical bonding with arriving material
  - Reactivity - sticking coefficient = probability of incorporation into growing film
  - Energy input – substrate temperature and chemical energy in vaporized species
- Higher pressure regimes enable faster deposition rates
  - > p_{atm} more difficult to engineer gas transport
- Chemical and safety hazards exist due to toxic, corrosive, flammable, and explosive precursors
  - Overcome by engineering considerations, technology variants, precursor modification

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Gaseous AM Prior Art

- Rapid prototyping of near net shape parts using CVD
  - Continuous wave visible laser with trimethylamine alane and O_2 to make freestanding 3D alumina tweezers, robots – *Lehmann, Stuke, 1995*
  - Laser jet CVD to make complex metal, ceramic, and metal-ceramic composite parts – *Duly, 1999*
  - LCVD (100 W CO2 laser) with methane and hydrogen for C to create 3D (fibrous and helical springs) and laminate structure – Georgia Tech
- Bulk materials production and refinement of ores
  - Carbonyl precursors that can utilize inexpensive scrap materials and low purity ores
  - CVMR, Vale, INCO, Weber conduct extensive operations to produce plate, fibers, and foams

Carbonyl processing at Weber[44]

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Carbonyl Processing: Continuous Production

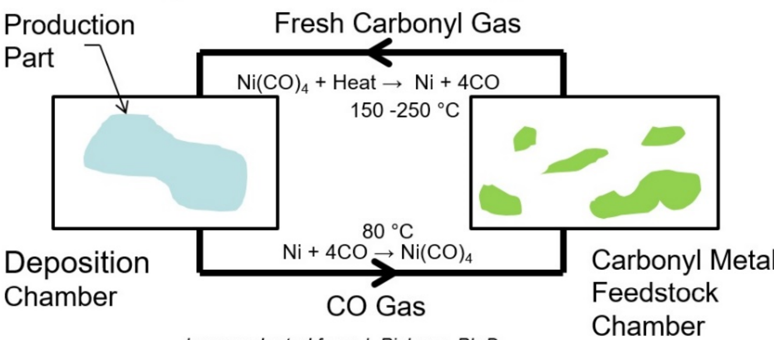


Image adapted from J. Pickens, Ph.D.

Net Shape Manufactured, Carbonyl Nickel End Cap and Tube used for Sudbury Neutrino Observatory

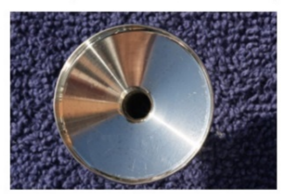
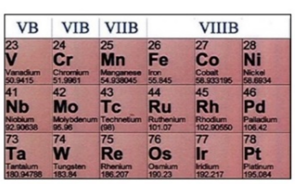


Image courtesy CVMR



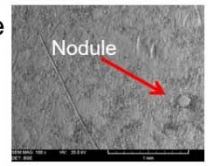
Eighteen transition elements undergo carbonyl reactions at temperatures far below their melting points

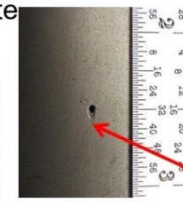
- Low operating, maintenance, and energy costs
- 50-200x deposition rate of other methods
- High builds with low stress possible
- Non-line-of-sight process

Carbonyl Nickel after multiple 90° bends – excellent ductility

Carbonyl Processing: Research

- Objective: Use conventional AM to produce forms and deposit transition metals to create light-weight, high-strength composite or hollow parts with superior structure and surface finish
- Metallurgy: smooth surface, no cracks, some pinholes and nodules, Ni with some C and O precipitates, epitaxial growth
- Hardness: Fairly consistent
- Tensile Properties: Relatively uniform UTS, YS, elongation, and modulus





Compare to annealed Ni sheet: 122 VHN

Orientation	Minimum Thickness (in)	Elastic Modulus (Msi)	UTS (ksi)	0.2% Offset YS (ksi)	Elongation (%)
0°	0.0082	24.0	88.7	64.6	32
90°	0.0062	28.2	87.8	63.1	32
180°	0.0057	--	84.3	--	36
270°	0.0076	--	87.2	--	32
Pure Nickel Sheet Annealed		**29.7**	**55–75**	**15–30**	**55–40**

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Carbonyl Processing: Challenges & Solutions

- Large reactors used extensively
  - AM is low volume work
- $(Ni(CO)_4$ toxicity concerns
  - 0.007 mg/m^3 OSHA PEL (Cr^{+6} = 0.005 mg/m^3)
- Higher deposition temperatures needed for other metals (>650 °C)
  - Exceed AM plastics T_g (<230 °C)
  - C and O contamination possible with lower dissociation temperatures

- Address applications drivers and partners for technology demonstration
- Evaluate design in CVD systems
- Consider CVD variants (atmospheric, plasma-assisted, vacuum)
  - Control crystalline structure, morphology, and orientation and process parameter modification
- Select appropriate reaction
  - Consider reversability, binary materials, reduced substrate attack
- Review different precursors
  - Halides and metallorganics - many materials at different temps.
  - Metal halides and halohydrides - more stable than hydrides
  - Metalorganics - less toxic and pyrophoric than hydrides and halides and use lower reaction temperatures
- Pursue different AM material forms

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Gaseous AM Summary

- Different reactors required for different applications
  - Partner choice selected based on needs
- Can avoid toxicity through precursor choice
  - Extreme toxicity concerns confined to $(Ni(CO)_4$
  - Halides and MOs are preferred precursors in industry
    - Wider range of materials possible, including intermetallics
    - Low deposition temperatures
- CVD temperatures often exceed T_g of AM plastics
  - Investigation of other material forms continues
- High quality components possible
  - Adjust build thickness, composition, and morphology through temperature control and distribution through form
  - Can achieve hardness, yield strength, and ultimate tensile strength greater than annealed sheet (Ni)

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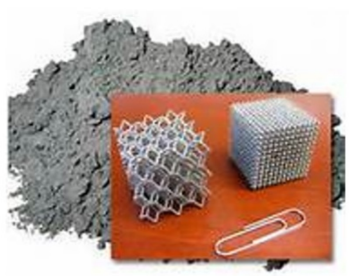
General AM Summary

- Need no tooling, setup, etc. – freedom of design
- Enables fast production of first-time, custom, or difficult to manufacture parts
- Excellent for prototypes, research and conceptualization of ideas
- Can offer better strength than wrought or cast materials, but lower ductility
- Need post processing and good in-process control/monitoring
- Repeatability across machines and runs is not consistent – requires excellent, and often expensive inspection techniques
- Material, equipment, and part qualification is significant
- Extensive opportunities for innovation
  - New AM processes
  - Surface finishing for AM parts
  - Sensor development/integration and real-time adjustments
  - Quality control/quality assurance means/methods



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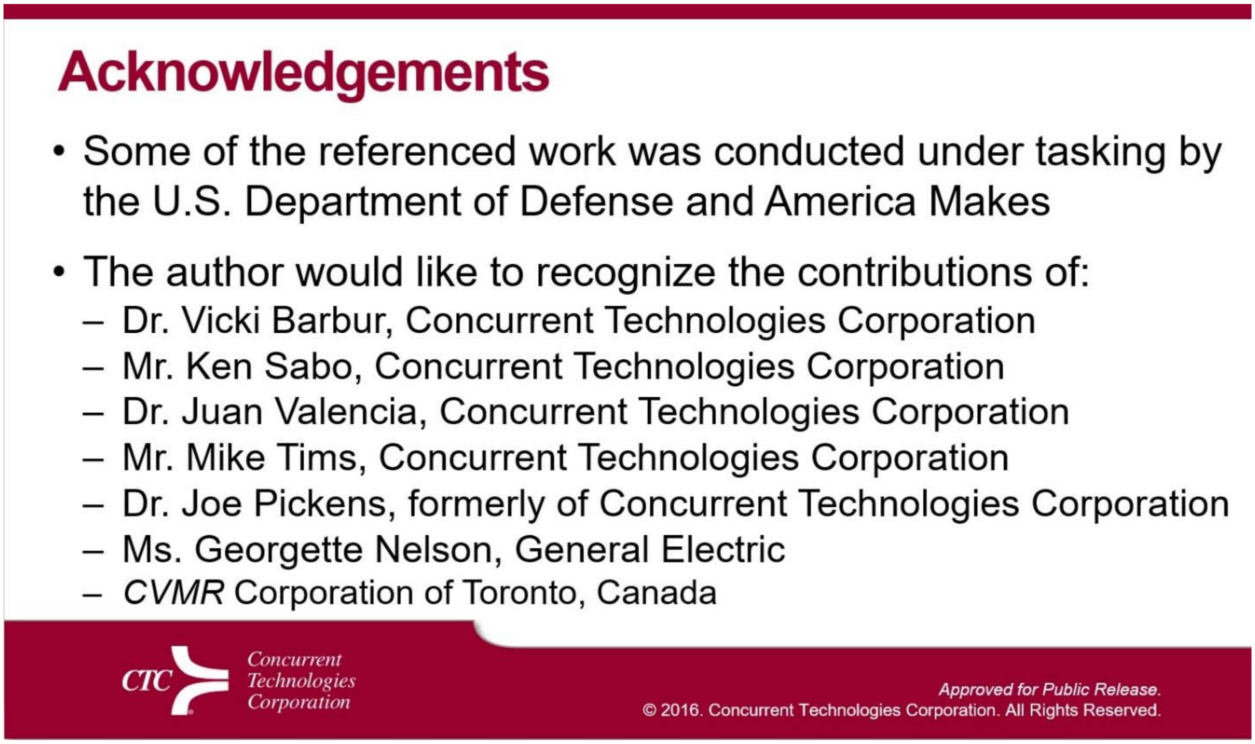

About the author

Editor's Note: The following excerpts were published by Concurrent Technologies Corporation, Johnstown, Pennsylvania, and the interview that follows was published in *Products Finishing* at the time the Dr. Klingenberg was announced as the winner of the 2015 NASF William Blum Scientific Achievement Award at SUR/FIN 2015 in Rosemont, Illinois.

Melissa Klingenberg, Ph.D., a Principal Advisor Engineer at Concurrent Technologies Corporation (CTC), has been awarded the 2015 National Association for Surface Finishing's (NASF) Scientific Achievement Award. She is the first woman to win the top honor in the 57-year history of the Scientific Achievement Award. The NASF presents the award annually to "an individual who greatly contributes to the advancement of the theory and practice of electroplating, metal finishing and the allied arts; raises the quality of processes and products; and has enhanced the dignity and status of the profession."

"It's the greatest honor of my career," Klingenberg said. "I've always thought it was the most prestigious award for science within the metal finishing industry, and I am very excited and honored to have won. I always thought this honor was beyond my reach and have admired the men who won it in the past."

Rob Mason, CTC's Principal Materials and Process Scientist and NACE Certified Corrosion Technologist, noted, "The Scientific Achievement Award is very prestigious, and Dr. Klingenberg really deserves it. She is probably the first and foremost thought leader in the industry with respect to metal finishing wear-resistant coatings. She is a working scientist, clients love her, and she is respected as the go-to authority by many people across the country. I can't emphasize enough that she is incredibly worthy of this industry-leading award." Mason nominated Dr. Klingenberg for the honor, which was voted upon by an NASF committee.

Products Finishing Editor Tim Pennington writes, "Dr. Klingenberg has done it all for the industry: serving as AESF Foundation president, serving on the AESF Council, the NASF Research Board, the Emerging Technologies Committee, the Sur/Fin

Technical Committee and as organizer and conference chair for the Surface Engineering for Defense and Aerospace Applications Conference.

"She has been an active member of NASF since 1994, and in 2008 organized and co-chaired the first ASM International/NASF Surface Engineering for Defense and Aerospace Applications conference. Her award has been especially deserving because it has been through those activities with the NASF that many feel that Dr. Klingenberg was critical in 'reinventing' the NASF to attract additional experts to conferences in the areas of innovative coating and new surface finishing technologies."

Klingenberg has more than 20 years of experience in inorganic finishing operations, specializing in research and development, technology evaluation, and implementation of innovative coatings and surface finishing processes designed to improve engineering properties and address environmental issues. She identifies, designs, integrates/installs, debugs, and implements systems including advanced deposition and plating processes, innovative coating and surface treatment technologies, and high energy and laser systems. Klingenberg is particularly known for her research in wear-resistant coatings and cadmium and chromium replacement technologies for defense applications, and she has co-authored numerous papers, articles, and presentations on these subjects. Her efforts have involved coatings deposited by physical vapor deposition (PVD) and brush plating, as well as advanced technologies and concepts, such as the use of ionic liquids for cleaning and plating.

In addition to her career as a scientist, Klingenberg is married to Scott Klingenberg, and they have one daughter, Gabrielle. They live in Windber, PA.

Klingenberg received a bachelor's of science degree in chemistry and engaged in post-baccalaureate studies in biology at the University of Pittsburgh at Johnstown. She received a master's degree in manufacturing systems engineering at the University of Pittsburgh and a Ph.D. in materials engineering at the Pennsylvania State University.

In honor of the first recipient of the Scientific Achievement Award, Dr. William Blum Sr., the newest winner is invited to present the Blum Memorial Lecture at the opening session of the NASF Annual Technical Conference. Klingenberg looks forward to delivering that presentation next year and to having the lecture published in *Products Finishing*.

Products Finishing interview

Melissa Klingenberg Ph.D., is a principal advisor engineer at Concurrent Technologies Corp., and the recipient of the 2015 NASF Scientific Achievement Award, the first woman to win the honor in the 57 years the award has been given.

PF: What does being the first woman to receive the 2015 NASF Scientific Achievement Award mean for the industry?

MK: I think that the award is something that anyone in the industry, regardless of gender, is inspired by to help innovate and broaden the industry. However, I think that having a woman honoree emphasizes that the industry supports and continues to provide excellent opportunities for women. It is my hope that this emphasis will help to continue to attract talented young women to support the industry.

PF: What does your role as principal advisor engineer entail?

MK: I collaborate with engineering, science and business staff to identify and deliver the best possible solutions for our clients. We leverage research, development, test and evaluation work to provide customized, transformative, full life-cycle solutions to support our clients' core mission objectives. I also support technical and business strategic planning, generate and explore new research ideas, and mentor technical staff.

PF: What led you to the metal finishing industry?

MK: When I started at CTC, I had no preconceived notions as to what particular industry I'd be supporting. I was assigned as a junior engineer/scientist into the inorganic finishing area, and more specifically, advanced vacuum systems processing. I was fascinated by high energy surface modification and the physics and materials science behind the processes I was using. From there, I expanded into other dry processing methods as well as wet deposition, including electrodeposition, conversion coatings, etc. I really enjoyed modifying coating compositions, crystalline structures, and surface morphologies to manipulate properties, regardless of the process being used. This fascination drove me to continue my education to better understand how to engineer the properties of coatings.

PF: Tell us about your research in wear-resistant coatings.

MK: Our initial work focused on identifying and developing coatings that were capable of maintaining the engineering properties of coated parts when replacing cadmium or chromium, but doing so using a more environmentally acceptable process or material. However, as we searched for alternatives, we found that many processes, wet or dry methods, were capable of producing coatings that possessed superior properties through the introduction of high energy, alloying compositions or use of particle co-deposition. In some instances, we were incorporating ancillary equipment into conventional deposition means or using subsequent processing to produce better microstructures and even effect crystalline changes that inherently had better wear and corrosion properties. From there, we began narrowing our investigations to examining how different crystalline structures and morphologies of a single compositional range affected adhesive and abrasive wear properties and how we could slightly modify those properties to optimize the wear protection in a wear system pair.

PF: What is the secret to your success?

MK: Teamwork. I have been very fortunate throughout my career at CTC to work with so many talented technical, business, and support staff both inside and outside of our organization. I have learned something valuable from each and every individual with whom I have worked.

PF: What advice would you give to yourself 10 years ago?

MK: I would tell myself not to be overly stressed about things that are outside of my direct control.

PF: What was your first job and what did you learn from it?

MK: Part-time at the concession stand at our community pool. Although it has been quite some time since I worked there, I think that I learned that fast and friendly customer service is foremost in any business.

In 2017, Dr. Klingenberg left Concurrent Technologies to pursue a new opportunity at the Pennsylvania State University, University Park, Pennsylvania. She is now Technical Director, Institute for Manufacturing and Sustainment Technologies.