

AMMT Program Material Development

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AMMT Program Material Development Technical Area Lead PNNL Senior Technical Advisor Advanced Material Systems

AMMT Industry Workshop, Manufacturing Demonstration Facility at Oak Ridge National Laboratory, May 23-24, 2023

Acknowledgements

- Multi National Laboratory and -Stakeholders effort to accelerate development and implementation of existing and new materials in nuclear reactors
- ANL: Srinivas Mantri, Xuan Zhang
- INL: Mike McMurtrey, M. Mulholland
- LANL: Miles Beaux, Bryant Kanies, Robin Adriana Montoya,
- ORNL: Sebastien Dryepondt, Soumya Nag, TS Byun, Peeyush Nandwana, H. Hyer, F. List, S. Taller,
- PNNL: Mageshwari Komarasamy, Ram Devanathan, Ken Ross, Mohan, Nartu, Ankit Roy
- EPRI: Marc Albert, Dave Gandy
- NEI: Hilary Lane



Material Identification

Decision making through Diverse Surveying

Develop materials as an integrated part of advanced manufacturing (AM).





We need Understanding & *Partnerships*



Examples of Specific Reactor Type Score Card

Material Score Cards 2020/2021

		Guidance	MSR						VHTR									
ID	Criteria		316	316H	304	Alloy 800H	Alloy N	Graphite	SiC	НТ9	IN617	316	304	Alloy 800H	Graphite	SiC	IN617	IN718
1	Code Availability	Codes are available for all areas = 5 For two of the three areas = 3 For one area = 1	3 (3)	0 (1)	3 (3)	1 (2)	0	1 (2)	2 (2)	2	1 (2)	1 (3)	2 (3)	1 (2)	1 (2)	2 (2)	1 (2)	1 (1)
2	Minimal Gaps in Data Availability for Performance Values and Measurement s	No or few gaps in data availability = 5 Moderate gaps in data availability = 3 Large gap in available data = 1	3 (3)	1 (1)	3 (3)	1 (2)	0 (1	Pacific	N	lumi efere	<u>per of</u> enced	f <u>rea</u> I; co	<u>ctors</u> mbii	<u>s</u> the ned fo	materia or all re	l typ acto	e wa r type	s es
3	Technical Maturity for End Use/Developm ent Stage	TRL 8-9 and/or MRL 8-10 = 5 TRL/MRL 7-8 = 4; TRL/MRL 5- 6 = 3; TRL/MRL 3-4 = 2; TRL/MRL1-2 = 1	3 (3)	1 (1)	3 (3)	2 (3)	0 (1	ettionar reposition	SINCE					1915 - 11 - 11 - 11 - 11 - 11 - 11 - 11	8778 800H	80.80		Austenitic Steel Ferritic/Martensit Carbon Alloy St Nickel Alloy Other
4	Deployment readiness requirements	Ready for industry deployment within 2 years = 5; In 3-5 years = 4; In 6-7 years = 3; In 8-9 years = 2; In \ge 10 years = 1	3 (5)	1 (1)	3 (5)	2 (5)	0 (1	er of Reacto					I.	Incom	AUD	 		
5	Supply Chain Availability	No anticipated supply chain risks or impacts = 5; Moderate impacts = 3; Major impacts = 1	3 (3)	1 (1)	3 (3)	3 (3)	1 (3	oquin N 0		Ш	ш		$\ $	Ш				1
6	Programmatic Factors	Applications across all reactor types and/or multiple industry entities interested in a reactor type = 5	5 (5)	1 (1)	5 (5)	4 (4)	4 (1	Preliminary Data Se	191555 pt 2021	D9 Steel Alloy 709 15-15 Ti	Alf Steel A187 SA213 HT9 Steel	ODS Steels Grade 92 SA533 Steel cA608 Steel	2.25Cr-1.0Mo Steel	A690 SA540 Steel Pure N	Alloy 800H Alloy N Alloy 230 Hastelloy X HEAs	SICISIC CIC Carbon Mo Allov	Nib Alloy Zr3Si2 Aluminum Nätride YHx	Concrete

Decision making through Diverse Surveying

Stakeholder Input

EPRI - DOE AMMT materials integrations technical meeting

- Identify and prioritize material gaps and data needs for AR deployment across various advanced non-LWR reactor (coolant) types
- Discuss potential strategies for executing necessary work
 - What funding is needed, when, currently funded scope, options to executed unfunded scope, and overall impact.
- Being to develop a draft integrated priority material development list with execution plan

Stakeholder's input, needs, requirements



March 13-14, 2023, EPRI Charlotte Offices

Diverse Surveying

Direct funded projects

Stakeholder's input, needs, requirements



AMMT workshops

Decision Criteria Matrix: Multi-National Laboratory Effort

Category	Criteria						
Manufacturing: Powder/Precursors	Powder Availability	Powder Properties	Powder Chemistry	Cost	Recyclability	Production Method TRL	
Manufacturing: Components	Printability / Scalability	Defects	Post Treatment	Processing window	Weldability	Surface Roughness Surface Finish	
History & Applications	NE Experience	Other Industry Experience	Data availability	Code & Standards Availability	Experience with non-LPBF AM	Component Versatility	
Mechanical Properties	Creep	Fatigue	Creep-fatigue	High Temp tensile strength	Room temp tensile strength	Fracture Toughness	
Environmental Effects	Radiation Resistance	Oxidation Resistance	Stress Corrosion Cracking	Molten Salt	Liquid Metal	Transmutation / Neutronics	
Physical Properties	Thermal Conductivity	Solidification-relevant properties	Other modeling- relevant properties	Digital Manu. relevant-properties	Thermal Ca tempe	apacity: melting rature, etc.	
Microstructure	Material Homogeneity	Microstructure Stability	LPBF Microstructure Specificity	Scope for Microstructural Enhancement	Microstruct	ural Dependency	

The idea behind this matrix is to choose what criteria to consider for material selection. Scoring can be done based on the selected reactor technology.



Decision Criteria Matrix : Examples

Criteria	What is being evaluated?	Ferritic ODS	PBAM W	Cast (Ni ₂ Co ₂ FeCr) ₉₂ Al ₄ Nb ₄	IN-718 (bulk)	Grade-91 Steel (bulk)
Code & Standards Availability	The availability of codes and standards which govern the production, material quality/standards, and implementation of a material.	1	3	1	3	4
Environmental Effects	Radiation resistance, oxidation resistance, SCC, high temperature effects	3	2	No Data Available	4	3
Time Dependent Properties	Risk of losing dimension stability in long-term service (fatigue, creep, creep fatigue)	4	5	No Data Available	4	4
Reproducibility, Consistency, & Scalability	Degree of reproducibility and consistency in product quality for various manufacturing routes/methods of the same material (e.g. For the same material, 3D printing is not consistent, but casting is)	2	4	5	4	3
Cost	Overall cost for production of components (considering the same concern as Reproducibility/Consistency)	2	3	3	3	1
Microstructure	Material homogeneity, microstructure stability, tailorable	4	4	4	4	4

- Are these criteria the most relevant?
- Did we fail to consider any important criteria?
- How would you assign weight to the criteria?

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Prioritization of Current Reactor Materials for Advanced Manufacturing



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ANI



Michael D. McMurtrey INL

AMMT Industry Workshop, Manufacturing Demonstration Facility at Oak Ridge National Laboratory, May 23-24, 2023

Prioritization of current reactor materials for advanced manufacturing: Fe-based alloys ANL & PNNL

Alloys of interest

Austenitic	Ferritic/Martensitic
A-709	HT-9
AFA alloys	Grade-91
Ti-modified 316SS (D9)	Grade -92
ODS	



- 9Cr-1Mo, Grade-91 & Grade-92 are code qualified
- HT9: Has a wider cross-industry appeal
- A-709: Ongoing code qualification. Higher temperature capability compared to 316H
- D-9: Higher strength compared to 316H
- AFA Steels. Cross-industry appeal. High strength & better corrosion resistance in various environments due alumina formation

Significant work is needed for all these alloys since very limited AM research has been published to date



- Commercial powders not available
- Ongoing single-track experiments to determine optimum process parameters
- Custom-made powders have been ordered for initial printing trials

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Advanced Methodologies to Assess Alloy Printability

- Advanced computational tools to assess impact of chemistry on defect formation
 - accelerate alloy selection
 - determine optimum printing parameters
- Example: neural network to predict balling probability





Balling probability vs Carbon wt%

Database

Neural network

• Collaboration with other labs to generate tailored relevant database



Refined AM Microstructure Can Lead to Superior Properties For NE-relevant Austenitic Alloys

- Exciting opportunity to Develop Unique Refined Microstructure due to Rapid Cooling
- Fine Distribution of Nano Precipitates
- Example: AFA (Fe-22Ni-17Cr-4.5Al) developed for ICE applications (EERE/VTO/PMCP)





Courtesy Yuki Yamamoto







Refined AM Microstructure Can Lead to Superior Properties For NE-relevant Austenitic Alloys

- High density of nano precipitates results in superior creep strength
- Example: High Temp. Cast HK30Nb (Fe-25Cr-21Ni-1.3Nb-0.22C)





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 Work needed on chemistry optimization and heat treatment selection to further increase properties and reduce anisotropic behavior of relevant steels

Prioritization of current reactor materials for advanced manufacturing: Ni-based alloys INL & ORNL

High temp high strength & corrosion resistant

Three categories based on potential applications

Low Cobalt	High temperature strength	Molten salt (Low Cr)		
718 (20Cr-5Nb-3Mo)	230 (22Cr-14W-<5Co) , 233	Hastelloy N (7Cr-16Mo)		
625 (22Cr-9Mo-3.5Nb)	740H, 282 (20Cr-10Co-8Mo)	244 (8Cr-22.5Mo-6W)		
800H (32Ni-21Cr-40Fe)	617 (22Cr-12.5Co-9Mo)			

718: used in various reactors, well-known & available AM alloy

- Irradiation campaign initiated under TCR
- Creep data are needed & getting generated
- 625: Well-known & available AM alloy
 - Creep testing planned

800H: Code qualified but difficulty in procuring powder and limited AM work



- Data consistent with wrought 718
- Similar results between as printed and heat treated (2h@1174°C+6h@1204°C+1h@945°C+8h@718°C, 8h@621°C)
- Similar results along and perpendicular to BD



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Prioritization of current reactor materials for advanced manufacturing: Ni-based alloys INL & ORNL

282 and 230: Not currently used by NE industry but high strength, **powders are available** & several studies on AM 282

- Ongoing LPBF printing of 282 at ORNL
- Microstructure characterization at INL

617: Code qualified alloy but limited AM work & powder not commercially available

Hastelloy N. Superior performance in molten salt compared to 316H but no AM work & powder not commercially available

Haynes 244. Much higher strength compared to Hastelloy N.

 Ongoing effort to purchase powder to print and characterize the alloy



Zeiss build for parameter optimization and sensors calibration

Optimizing LPBF 282 heat treatment









Advanced Materials and Manufacturing Technologies (AMMT) Program

Preliminary Feasibility Studies of New Materials for Advanced Manufacturing

PIs: TS Byun (ORNL), Miles Beaux (LANL), Isabella van Rooyen (PNNL)







AMMT Industry Workshop, Manufacturing Demonstration Facility at Oak Ridge National Laboratory, May 23-24, 2023

Preliminary Feasibility Studies of New Materials for Advanced Manufacturing - Needs of New Materials

- All advanced reactors (SFR, MSR, and Fusion Reactor) require high temperature (>500 °C) and high irradiation dose operation for high thermal and economic efficiencies.
- No practical materials with qualification are available for high temperature (>550 °C) reactor structures.
- We have very little understanding of high dose (>200 dpa) performance of structural materials.
- Embrittlement (due to hardening, phase changes, and transmutation gases), dimensional instability (thermal & irradiation creep, and void/gas-bubble swelling), and environmental damages during high temperature, high dose irradiation are the main concerns.
- Capabilities of existing structural materials are limited within a <550 °C/<150 dpa envelope.



Preliminary feasibility studies of new materials for advanced manufacturing – ORNL







Characterization and Testing

- 700 °C and 800 °C for post-build TMP.
- Testing Temperatures: Room Temperature 600 °C

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Preliminary feasibility studies of new materials for advanced manufacturing – ORNL

ODS Alloy: Determination of Post-Build TMP Condition



Material ID.	Base Materials
HR-1	14YWT alloy (with Y & O)
HR-2 HR-3	14YWT base+Fe ₂ O ₃ 14YWT base+Y ₂ O ₂
HR-4	10YWT base+Y ₂ O ₃

- HR-2 and HR-3 materials processed by 900 °C consolidation and 600 °C continuous TMP demonstrate high enough strength.
- HR-4 material also showed similar strengths after final TMP at a lower temperature .
- Consider consolidation temperature was low (800 850 °C extrusion for mechanically alloyed 14YWT NFAs).
- 700 °C and 800 °C were decided for the temperature of post-build TMP.



Material(Consolidation/TMP Temperature)

Preliminary Feasibility Studies of New Materials for Advanced Manufacturing - LANL

- Feasibility studies of fabrication techniques for materials typically difficult to manufacture
 - Additive manufacturing of bulk refractory alloys
 - Refractory coatings on composite materials

A weld pool in a titaniumzirconium-molybdenum (TZM) alloy.



- Refractory Alloys:
 - High melting temperatures (>1850 °C)
 - High temperature stability
 - Superior strength at high-temperatures
 - Good thermal creep resistance
 - High wear resistance
 - Compatibility with liquid metal coolants
 - Good thermal conductivity
 - High void swelling resistance relative to FCC materials
- Additive manufacturing of refractory alloys overcomes the difficulty in conventional fabrication & machining

LBPF TZM Alloy

- Composites (C/C & SiC/SiC) are candidates for cladding, heat pipes, and structural components
- Advantages of composites
 - Lightweight (microreactors)
 - Neutronic compatibility
 - High temperature stability
 - Good mechanical & thermal properties
- Refractory coatings increase compatibility of composites with reactor environments
 - Protects against corrosion of fine-grained fibers and fiber-matrix interphase
 - Reduces high-temperature oxidation of C/C
- Refractory coatings allows for combination of advantages of composite and coating materials



Preliminary Feasibility Studies of New Materials for AM: HEAs PNNL

Addressing challenges and needs for upscaling the current manufacturing processes.

Why High Entropy Alloys (HEAs)?

- exhibit unusual lattice distortion and sluggish diffusion, immobilize the radiationinduced defects decreased swelling and segregation
- Tuned microstructure by exploiting their varied phase stability in different temperature regimes for enhanced sink strength.

Bulk manufacturing processing

Solid-State Processing Methods:

• Cold/Hot Pressing, Spark Plasma Sintering, Friction Stir Additive, .etc.

Liquid-State Casting Methods:

- Arc Melting, Vacuum Induction/levitation Melting, Directional Solidification, .etc.
- Liquid-State Additive Manufacturing:

Properties

>

 $V_{35}Ti_{35}Fe_{15}Cr_{10}Zr_5$

Al₂₀Be₂₀Fe₁₀Si₁₅Ti₃₅

• Directed Energy Deposition (wire and powder fed), Powder Bed Fusion, etc.

FeCrAl and Zr-4.

- USNRC report on "Use of High Entropy Alloys (HEAs) in Future Nuclear Applications (Jan 2023)
 - identifies AlCuCrFeNi and AlCrFeMnNi HEAs for nuclear applications.
- AlBeFeSiTi system: provide lightweight alternatives to the current nuclear alloys.



- multiple interfaces either by secondary phase precipitation or by oxide dispersion enhances the sink strength of the alloys leading to better radiation resistance
- Transformation induced plasticity (TRIP) HEAs possessing self-healing ability
 - transformation that occurs during the irradiation reverses during heating phase allowing the alloy to self-heal from the damage caused by radiation.
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Preliminary Work on composite Al_{0.3}Ti_{0.2}Co_{0.7}CrFeNi_{1.7} HEA (Precipitation strengthened)



Al_{0.3}Ti_{0.2}Co_{0.7}CrFeNi_{1.7} processed via *Directed Energy Deposition* and *Selective Laser Melting*.

- Hierarchically heterogeneous microstructures (secondary L1₂-phase precipitates) obtained via simple one-step annealing:
 - significant better performance than the nearly homogeneous microstructures in the as-deposited state
 - Smaller than 6.6% reduction in hardness values at 500°C compared to RT, while the as-deposited conditions showed a greater than 18% reduction in the hardness
 - No microstructural instability is observed for heterogenous microstructures during nano-indentation deformation at 500°C

AM method	Condition	RT	250°C	500°C	% Change
	AD	4.157±0.287	3.763±0.212	3.197±0.233	23.13 ↓
DED	AD+800°C (HT)	4.710±0.123	4.663±0.183	4.387±0.153	6.5↓
<u>a</u>	AD	4.116±0.145	3.569±0.171	3.560±0.121	13.3↓
SLM	AD+800°C (HT)	4.654±0.171	4.494±0.132	4.427±0.104	4.9↓

[Mohan Sai Kiran Kumar Yadav Nartu, Shristy Jha, Advika Chesetti, Sundeep Mukherjee, Isabella van Rooyen, Rajarshi Banerjee, *Microstructure and Temperature Dependent Indentation Response of Additively Manufactured Precipitation-strengthened* Al_{0.3}Ti_{0.2}Co_{0.7}CrFeNi_{1.7} High Entropy Alloy, Submitted to JOM, May 2, 2023]



Develop 316H and ODS Steels by Solid State Manufacturing Processes – PNNL



Extrusion-based fused filament fabrication (FFF) Process and Team

ODS Powder 1-14YWT 3

successfully printed 3 bricks and 1 honeycomb structure



Shear Assisted Processing and Extrusion (ShAPE)

A picture of a first-of-its kind ShAPE machine



ickness: 1.1±0.05 m Zircaloy-4 puck with Ni on inside and Defect-free tube with fine-grained microstructure was obtained outside Ni cladding is successfully produced on inner diameter and partially on outer diamete Interior is pure Zircaloy without any oxidized region. ENERGY

Developed at PNNL, ShAPE[™] is a unique method for consolidating/extruding materials, in which linear and rotational shear are combined to plasticize.

Benefits include:

- Grain refinement
- Texture alignment
- Breakdown of 2nd phases ٠
- Mixing and homogenization
- Single step process
- Lower force and energy





ShAPE Experiments to Date



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- Modify tooling for next experiment on horizontal indirect extrusion
- Perform ShAPE (indirect) experiments on SPS ODS with improved parameters from lessons learned: • decrease the processing temperature below 1000°C which might reduce the waviness in the tubes and also preserve the number density of the oxide dispersion
- Direct Extrusion FY2024: improved parameters on SPS and HIPed ODS samples

Indirect extrusion of PM2000 alloy

First experiment was carried out using a vertical extruder

Input: The ShAPE extrusion was carried out at the die rotational speed of 400 RPM and die advance rate of 5 mm/min.

Output: The die temperature was approximately 1250°C during the steady-state region.

Forces and torque were lower than expected, hence the processing temperature can be reduced further to below 1000°C to preserve the density of oxide dispersions.



Picture of the PM2000 tube extruded via the ShAPE process



Cross-section showing that the extrudate is a tube for the most part.





Advanced Materials and Manufacturing Technologies (AMMT) Program

Large Scale Additive Manufacturing

Pls: Ken Ross (PNNL), Soumya Nag(ORNL)





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Large Scale AM for Nuclear Applications: Why AM?

Large scale DED AM to fabricate components for pressure boundary applications relevant to nuclear community

demonstration of a Hot
Isostatic Press (HIP) can



Semi-Open Impeller (Simple)

From 'HIP of AM parts' to 'AM of HIP cans'

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Closed Impeller (Complex)



How much of AM vs PM is optimal for a component?

Dam Turbine Impeller (Large Scale)

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ORNL team: Luke Meyer, Andrzej Nycz, Fred List, James Haley, Mithulan Paramanathan, Tom Feldhausen, Soumya Nag

Large scale AM Cans – Machines and Monitoring

Simple Concentric Cylinder



Complex T-valve



Successful build to assess build guality and feasibility. Weld mode tuned after bottom section.

Challenge: Tool path strategy with minimal defects

Processing : <u>Signal</u> : **Structure : Property**



IR - Temperature





Monitor and control stress evolution via in-situ DIC+IR

DIC tracks surface features in 3D over time

Multispectral IR measures temperature and emissivity

Target detectable information:

Cracking and delamination, Volumetric phase changes, CTE, thermal conductivity

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ORNL team: Luke Meyer, Andrzej Nycz, Fred List, James Haley, Mithulan Paramanathan, Tom Feldhausen, Soumya Nag

Additive Friction Surfacing (AM) Process Description

Nascent large scale manufacturing process with the potential for improved properties, cost and lead time



316-H bar stock deposited directly onto 316-L plate at PNNL

PNNL team: David Garcia, Mayur Pole, Ken Ross

- Refined grain structure produces improved properties demonstrated in aluminum
- Overmatched properties possible in austenitic stainless steels
- Potentially order of magnitude cost staving on material alone compared to fusion based methods
- High deposition rate
- Application space
 - Near-net shape additive manufacturing
 - Component life extension
 - Cladding
 - Functionally graded and dissimilar materials









Additive Friction Surfacing (AM) SEM. EBSD Results

- Grains are significantly refined
 - Average grain size between 4 and
 6.8 μm
 - Approximately an order of magnitude reduction compared to forging specifications (assuming 6-4 max ASTM#)
 - Grains size is approximately ¼ that of the extruded plate
 - Grains are further refined at layer boundaries (b, d).
- Use of argon cover gas is anticipated to eliminate oxide layers in next round of experiments
- No visible interface between horizontal layers



Etched optical micrograph of *first attempt* of additive friction surfacing with 316H





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Material Development Technical Area

Advanced Adv

The vision of the AMMT program is to develop materials as an integrated part of advanced manufacturing (AM).

Optimize & Manufacture Existing Reactor Material

Improve and optimize LPBF 316SS to improve AM material heterogeneity (Peeyush Nandwana)

Prioritization of current Reactor Materials for Advanced Manufacturing Sebastien Dryepondt (ORNL) Mantri Srinivas (ANL) Mike McMurtrey (INL) Isabella van Rooyen (PNNL)

Develop 316H and ODS steels by solid state manufacturing processes (Isabella van Rooyen)

Develop & Manufacture New Reactor Materials

Preliminary Feasibility Study of New Materials for Advanced Manufacturing TS Byun (ORNL) Miles Beaux (LANL) Isabella van Rooyen (PNNL)

Large Scale Manufacturing

Large Scale Manufacturing for Nuclear Applications Soumya Nag (ORNL)

Critical Minerals

Critical Minerals Isabella van Rooyen (PNNL))

- To develop advanced materials & manufacturing technologies that have cross-reactor impacts.
- To establish a comprehensive framework for rapid qualification.
- Technology demonstration and deployment.

