UCRL-TR-234888

LAWRENCE LIVERMORE NATIONAL LABORATORY

DOE-DARPA High-Performance Corrosion-Resistant Materials (HPCRM), Annual HPCRM Team Meeting & Technical Review

J. Farmer, B. Brown, B. Bayles, T. Lemieux, J-S. Choi, L. Ajdelsztajn, J. Dannenberg, E. Lavernia, J. Schoenung, D. Branagan, C. Blue, B. Peter, B. Beardsley, O. Graeve, L. Aprigliano, N. Yang, J. Perepezko, K. Hildal, L. Kaufman, J. Lewandowski, J. Perepezko, K. Hildal, L. Kaufman, J. Lewandowski, J. Boudreau

September 24, 2007

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DOE & DARPA Sponsored
High Performance Corrosion Resistant Materials (HPCRM) Program
Annual HPCRM Team Meeting & Technical Review
DoubleTree Grand Key Resort
Key West, Florida
HPCRM Team Meeting & Technical Review – January 17-19, 2006
Sponsor's Meeting – January 20, 2006

MEETING AGENDA

Tuesday, January 17, 2006

1:00 pm — HPCRM Investigator's Meeting:
- Brown Bag Lunch
- Reporting Progress at Meeting
- Review Planned FY06 Work Scope
- Collaborations with Other DOE & DOD Projects
- Planned Publications

5:00 pm — Adjourn

Wednesday, January 18, 2006

7:30 am — Continental Breakfast

***Opening*:**

8:00 am	Welcome to Key West	*Jef Walker (DOE)*
8:10 am	Logistics for Meeting	*Lesa Christman (LLNL)*
8:20 am	Opening Remarks	*Leo Christodoulou (DARPA)* *Capt. Chris Earl (USN/DARPA)*
8:30 am	Yucca Mountain Perspective	*Russ Dyer (Dep. Director OCRWM)*

Program Overview:

8:45 am — Overview & Accomplishments: — *Joe Farmer (LLNL)*
- HPCRM Overview
- FY05 Tasks & Deliverables

DOD (Navy) Applications:

9:45 am	Submarine Applications	*Bob Brown (NRL)*
10:15 am	Surface Ship & Other Applications	*Bob Bayles (NRL)*
10:45 am	Break	*All*

Wednesday, January 18, 2006 – continued

DOE (Repository) Applications:

11:00 am	Potential DOE Applications	*Jef Walker (DOE)*
11:30 am	Steps in the Path for Licensing	*Paige Russell (DOE)*
12:00 pm	Working Lunch & Discussion	*All*
1:00 pm	Possible Economic Impact of Coatings for Repository	*Jon Kirkwood (BAH)*

Criticality Control Applications

1:30 pm Basket Application — *Jor-Shan Choi (LLNL)*
- Criticality analysis modeling?
- Path forward for demonstrating coated basket?

***Synthesis*:**

2:00 pm Gas Atomization & Thermal Spray — *Leonardo Ajdelsztajn (UCD)*

2:30 pm SAM2X5 Powder Production — *Dan Branagan (TNC)*
- TNC Engineering Perspective
  - Is SAM2X5 ready for commercial use?
  - What remains to be done?

3:00 pm SAM1651 Feed Powder — *Craig Blue (ORNL)*
- ORNL Engineering Perspective
  - Is SAM1651 ready for commercial use?
  - What remains to be done?

3:30 pm Break — *All*

3:45 pm Industrial Scale HVOF Processing — *Brad Beardsley (CAT)*
- FY05 Developments & Proposed Work
  - Coating Navy Parts & Alloy C-22 Plates
  - Advanced Process Development by Progressive Technology
  - Robust Industrial-Scale Processing
- CAT Engineering Perspective
  - Are materials ready for commercial use?
  - What remains to be done?

4:30 pm Nanoparticle Additives — *Olivia Graeve (UNR)*

Recognition Ceremony & Group Photograph:

6:00 pm Hors D'oeuvres & Cash Bar — *All*

<u>Thursday, January 19, 2006</u>

Breakfast:

7:30 am	Working Continental Breakfast	*All*

Corrosion Resistance:

8:00 am	Concentrated Brine Corrosion	*Joe Farmer (LLNL)*
8:45 am	Salt Fog Corrosion	*Lou Aprigliano (NSWC)*
9:00 am	Related Activities in the Materials Performance Thrust Team	*Joe Payer (CWRU)*
9:30 pm	Break	*All*

Microstructure & Thermal Stability:

9:45 pm	Microstructure Studies	*Nancy Yang (SNL California)*
10:15 am	Thermal Stability	*Kjetil Hildal (UWM)*
10:30 am	Predictive Modeling	*Larry Kaufman (CALPHAD)*

Damage Tolerance:

11:00 pm	Mechanical Properties	*John Lewandowski (CWRU)*

Planned FY06 HPCRM Work Scope:

11:30 pm	Planned FY06 Work Scope	*Joe Farmer (LLNL)*
12:00 pm	Working Lunch	*All*

Thursday, January 19, 2006 – continued

Tunnel Boring Applications:

1:30 pm	Tunnel Boring Application • What are we trying to achieve? o Economics o Goals o Impacts	*Frank Wong (LLNL)*
2:00 pm	Cutting Disks & Bits • Spin-off applications? • Transfer of technology to industry?	*Craig Blue (ORNL)*
2:30 pm	Break	*All*
3:00 pm	Testing at Colorado School	*Levent Ozdemir (CSM)*
3:30 pm	HPCRM Summary & Discussion: • Compile List of Issues • Define Strategy for Resolution	*Jay Boudreau (BLE)*
4:00 pm	Closing Remarks	*Jef Walker (DOE)*

<u>Friday, January 20, 2006</u>

Meeting of Sponsors – Invitation Only:

7:30 am — Working Continental Breakfast

8:00 am — Discussion of FY'06 Activities:

- Review of Meeting Outcomes & Establish Path Forward — *Jay Boudreau (BLE)*
  - Comments from Reviewers DOE/DOD
  - Review of Issues on White Board
- Review of Gantt Chart, Finalize FY06 Schedule & Milestones: — *Jon Kirkwood (BAH)*
  - Milestones
  - Programmatic Logic
  - DOE/DOD Coordination
- Intellectual Property Status — *Al Thompson (LLNL Attorney)*
  - Any Novel Compositions
  - New Spray Atomization & Thermal Spray Technology
  - Hard Facing of Cutting Tools

12:00 pm — Working Lunch

- Milestones & Deliverables

1:00 pm — FY06 Fabrication Activities — *Joe Farmer & Brad Beardlsey*

- Schedule & Tasks for Coating and Testing of Large Parks
- Review Spray Atomization & Deposition Quality Control
- Schedule & Tasks for Coating and Testing of Fuel Baskets
- Schedule & Tasks for Coating and Testing Navy Parts

3:00 pm — Summary — *Jay Boudreau & Chip Smith*

4:00 pm — Closing Remarks — *Jef Walker (DOE)*
Leo Christodoulou (DARPA)

High-Performance Corrosion-Resistant Materials: Overview of Program

Joseph C. Farmer
Lawrence Livermore National Laboratory
Livermore, California

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ADC Signature

This document contains no sensitive subjects:

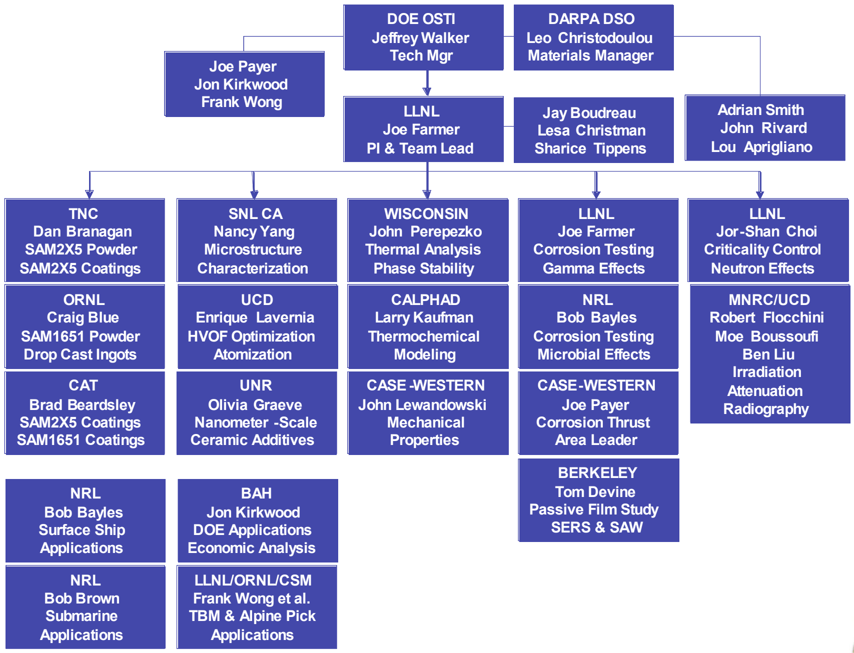
Sensitive Subject Reviewer Signature

Acknowledgements

- DARPA
  - Tony Tether, Director
  - Captain Chris Earl, Special Assistant to the Director
  - Steve Wax, Director, Defense Science Office (DSO)
  - Leo Christodoulou, DSO Materials Program Manager
  - Heather Heigele, DSO Program Analyst
  - Nichole Hoffman, DSO Budget Analyst
- DOE
  - John Wengle, Director, Office of Science & Technology International
  - Jef Walker, Manager, Technology
  - Dick Spence, Yucca Mountain Project
  - Paige Russell, Yucca Mountain Project
- Government Consultants
  - Chip Smith, Directed Technology, Consultant to DARPA
  - Joe Payer, Case Western Reserve University, Consultant to DOE
  - Jon Kirkwood, Booz-Allen, Consultant to DOE

HPCRM Team

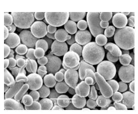
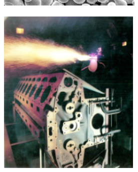
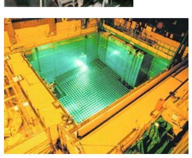
HPCRM Team Photograph – KW2004



Lawrence Livermore National Laboratory (LLNL): Corrosion & HLW/SNF Applications
Joe Farmer, Jor-Shan Choi, Jeff Haslam, Dan Day, Lesa Christman & Sharice Tippens / Frank Wong – TBM
Sandia National Laboratory California (SNL CA): Microstructure Characterization
Nancy Yang, T. Headley, G. Lucadamo, J. L Yio, J. Chames, A. Gardea & M. Clift
Oak Ridge National Laboratory (ORNL): SAM1651 Synthesis & Tunnel Boring Machine (TBM) Applications
Craig A. Blue & W. Peters
Naval Surface Warfare Center (NSWC) / Strategic Analysis (SA): Salt Fog Corrosion & Low-Carbon Variants
Lou F. Aprigliano (NSWC Retired)
Naval Research Laboratory (NRL) & Geo-Centers Corporation (GCC): Corrosion & Navy Applications
Bob Bayles, R. Brown, E. J. Lemieux, T. M. Wolejsza & F. J. Martin
The NanoSteel Company (TNC) / Idaho Natl. Lab. (INL): SAM2X5 Synthesis, Atomization & Thermal Spray
Dan J. Branagan, M. C. Marshall, B. E. Meacham & E. J. Buffa / Dave Swank et al.
Caterpillar (CAT): Industrial Scale Demonstrations & Process Scale-up
M. Brad Beardsley et al.
University of California Davis (UCD): Gas Atomization, Cryogenic Milling & Thermal Spray
Erique J. Lavernia, J. Schoenung, L. Ajdelsztajn & J. Dannenberg
McClellan Neutron Radiation Center (MNRC/UCD): Neutron Irradiation, Attenuation & Radiography
Robert G. Flocchini, Moe Boussoufi, H. Ben Liu et al.
Case Western Reserve University (CWRU): Corrosion Science Thrust Area / Mechanical Properties / Guidance
Joe Payer et al. / John Lewandowki et al. / Arthur Heur et al.
University of Wisconsin Madison (UWM): Thermal Analysis & Phase Stability
John H. Perepezko & K. Hildal
CALPHAD/MIT (CALPHAD/MIT): Thermochemical Modeling & Computational Materials Science
Larry Kaufman
University of Nevada Reno (UNR): Ceramic Nanoparticle Additives for Enhanced Damage Tolerance
Olivia Graeve et al.
Colorado School of Mines (CSM): Tunnel Boring Machine (TBM) & Alpine Pick Applications
Levent Ozdemir et al.

Introduction

HPCRM Objectives

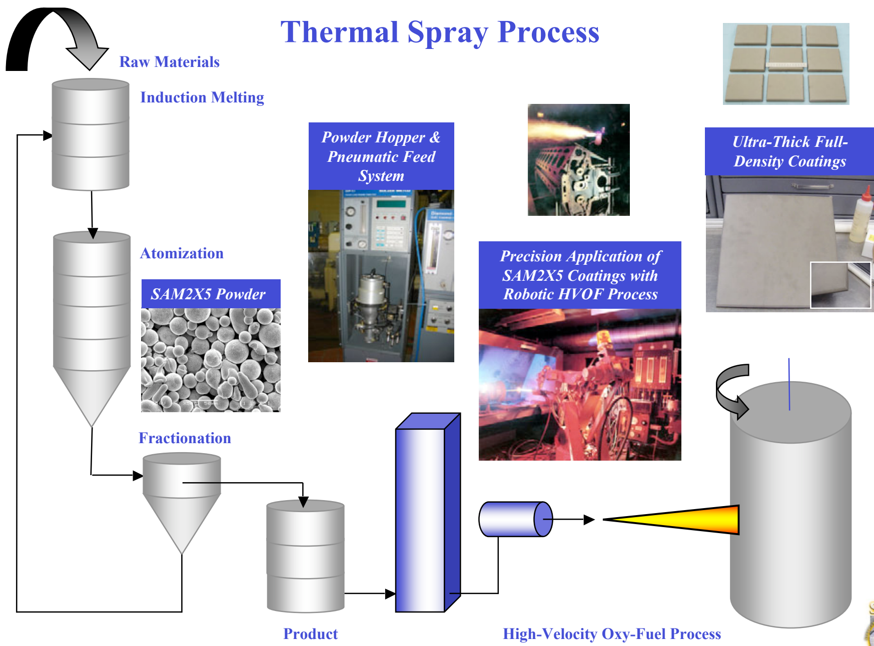

Overall Goal

Develop high-performance corrosion-resistant iron-based amorphous-metal coatings for prolonged trouble-free use in very aggressive environments: seawater & hot geothermal brines

Specific Technical Objectives

1. **Synthesize Fe-based amorphous-metal coating with corrosion resistance comparable/superior to Ni-based Alloy C-22**
2. **Establish processing parameter windows for applying and controlling coating attributes (porosity, density, bonding)**
3. **Assess possible cost savings through substitution of Fe-based material for more expensive Ni-based Alloy C-22**
4. **Demonstrate practical fabrication processes**
5. **Produce quality materials and data with complete traceability for nuclear applications**
6. **Develop, validate and calibrate computational models to enable life prediction and process design**

Thermal Spray Process



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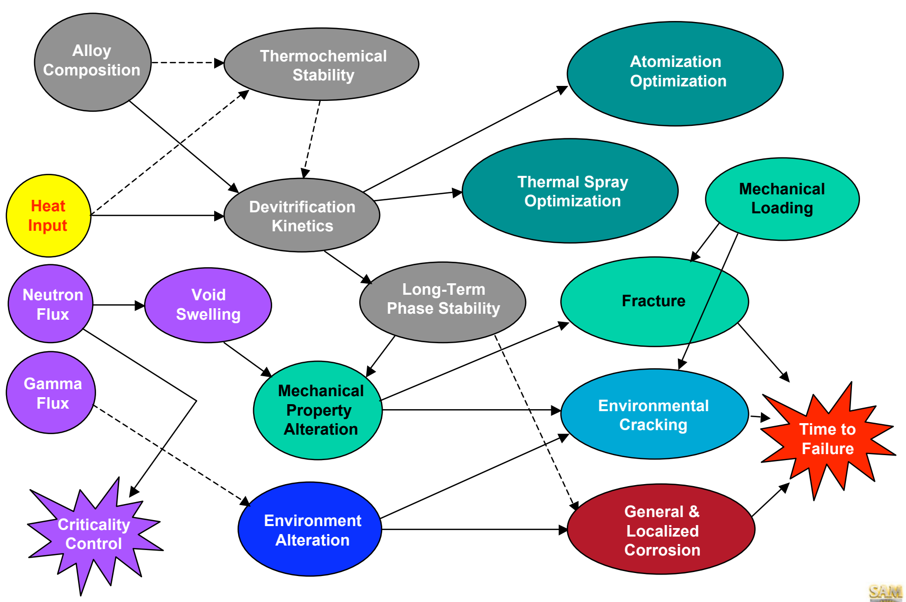
Overview of Accomplishments

- Fe-based amorphous metal formulations have been identified with corrosion resistance comparable to that of Ni-based Alloy C-22
  - Cr & Mo provide corrosion resistance
  - B enables glass formation
  - Y lowers critical cooling rate
    - SAM1651 = 80 K/s (yttrium added)
    - SAM2X7 = 610 K/s (no yttrium) … similar to SAM2X5
- These materials are also extremely hard provide enhanced resistance to abrasion and gouges (stress risers) from backfill operation
  - Type 316L Stainless Steel = 150 VHN
  - Alloy C-22 = 250 VHN
  - HVOF SAM2X5 = 1100-1300 VHN
- Optimization has been used to produce high-quality coatings in laboratory
  - Full-density pore-free completely amorphous coatings can be achieved by limiting the powder’s size distribution so that the critical cooling rate can be maintained within particles during thermal spray (PSO)

10 HPCRM Third Annual Program Meeting & Review – Key West 2006

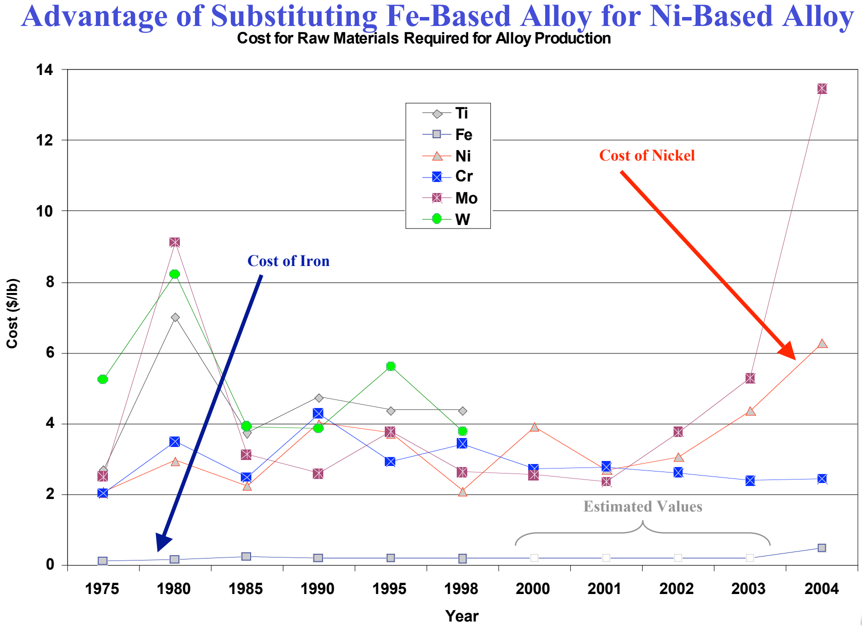
Technology Provides Several Potential Benefits

- These new materials provide a viable coating option for repository engineers
  - SAM2X5 & SAM1651 coatings can be applied with thermal spray processes without any significant loss of corrosion resistance
  - Both Alloy C-22 and Type 316L stainless lose their resistance to corrosion during thermal spraying
- SNF/HLW containers with corrosion resistant coatings are envisioned
  - Enhanced multi-purpose container (MPC) … leverage existing capability
  - Protected closure weld … eliminate need for stress mitigation
  - Integral drip shield … elimination of titanium drip shield
  - Thicken areas where greater corrosion is expected (crevices)
- Both SAM2X5 & SAM1651 have high boron content which enable them to absorb neutrons and therefore be used for criticality control in baskets
  - Alloy C-22 and 316L have no neutron absorber
  - Problems encountered with borated stainless and Gd-doped Ni-Cr-Mo
  - Variable thickness absorber

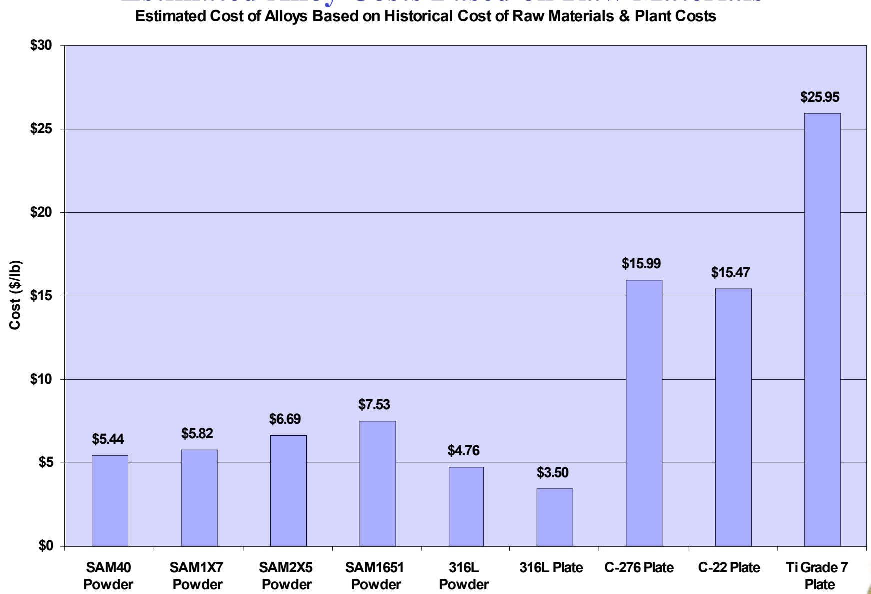


Process Economics

Advantage of Substituting Fe-Based Alloy for Ni-Based Alloy

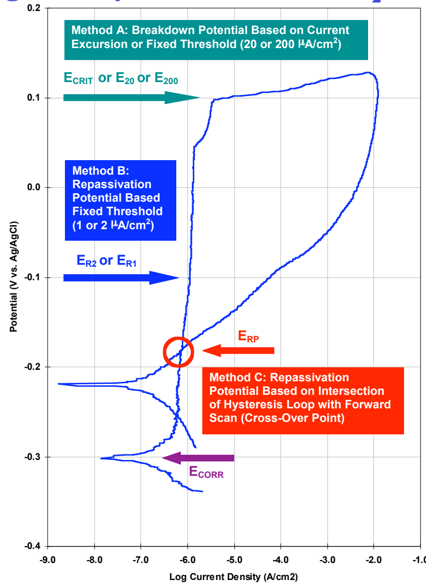


Estimated Alloy Costs Based on Raw Materials



Alloy Screening

Definition of Critical & Repassivation Potentials Wrought Alloy C-22 in 5M $CaCl_2$ at 105°C



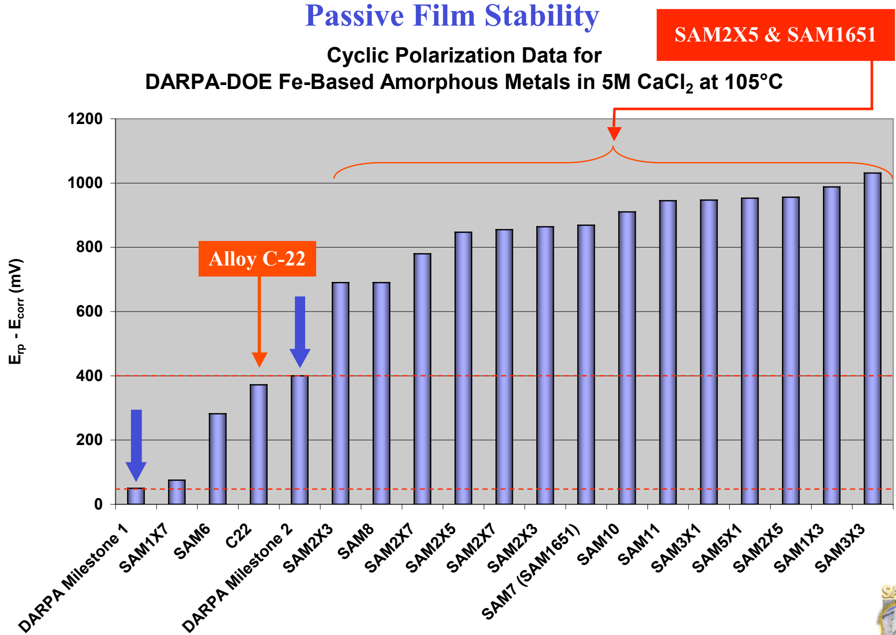
Synthesis & Screening of More Than Forty Candidates

Formulation	Formula	Fe	Cr	Mn	Mo	W	B	C	Si	Y	Zr	Ti	Co	Ni	Al	P	Other	Total
SAM27	$(Fe_{0.8}Cr_{0.2})_{73}Mo_2W_2B_{16}C_4Si_1Mn_2$	58.4	14.6	2.0	2.0	2.0	16.0	4.0	1.0									100.0
SAM35	$Fe_{54.5}Mn_2Cr_{15}Mo_2W_{1.5}B_{16}C_4Si_5$	54.2	15.0	2.0	2.0	1.5	16.0	4.0	5.0								0.3	100.0
SAM40	$Fe_{52.3}Mn_2Cr_{19}Mo_{2.5}W_{1.7}B_{16}C_4Si_{2.5}$	52.3	19.0	2.0	2.5	1.7	16.0	4.0	2.5									100.0
SAM40X3	$Fe_{50.7}Mn_{1.9}Cr_{18.4}Mo_{5.4}W_{1.6}B_{15.5}C_{3.9}Si_{2.4}$	50.7	18.4	1.9	5.4	1.6	15.5	3.9	2.4								0.2	100.0
SAM1	$(SAM40)_{100-x}+Ni_x$																	
SAM1X1	$(SAM40)_{99}+Ni_1$	51.8	18.8	2.0	2.5	1.7	15.8	4.0	2.5					1.0				100.0
SAM1X3	$(SAM40)_{97}+Ni_3$	50.7	18.4	1.9	2.4	1.6	15.5	3.9	2.4					3.0				100.0
SAM1X5	$(SAM40)_{95}+Ni_5$	49.7	18.1	1.9	2.4	1.6	15.2	3.8	2.4					5.0				100.0
SAM1X7	$(SAM40)_{93}+Ni_7$	48.6	17.7	1.9	2.3	1.6	14.9	3.7	2.3					7.0				100.0
SAM2	$(SAM40)_{100-x}+Mo_x$																	
SAM2X1	$(SAM40)_{99}+Mo_1$	51.8	18.8	2.0	3.5	1.7	15.8	4.0	2.5									100.0
SAM2X3	$(SAM40)_{97}+Mo_3$	50.7	18.4	1.9	5.4	1.6	15.5	3.9	2.4									100.0
SAM2X5	$(SAM40)_{95}+Mo_5$	49.7	18.1	1.9	7.4	1.6	15.2	3.8	2.4									100.0
SAM2X7	$(SAM40)_{93}+Mo_7$	48.6	17.7	1.9	9.3	1.6	14.9	3.7	2.3									100.0
SAM3	$(SAM40)_{100-x}+Y_x$																	
SAM4	$(SAM40)_{100-x}+Ti_x$																	
SAM5	$(SAM40)_{100-x}+Zr_x$																	
SAM6	$Fe_{43}Cr_{16}Mo_{16}B_5C_{10}P_{10}$	43.0	16.0		16.0		5.0	10.0								10.0		100.0
SAM7 (SAM1651)	$Fe_{48}Mo_{14}Cr_{15}Y_2C_{15}B_6$	48.0	15.0		14.0		6.0	15.0		2.0								100.0
SAM8	$(Fe_{48}Mo_{14}Cr_{15}Y_2C_{15}B_6)_{97}+W_3$	46.6	14.6		13.6	3.0	5.8	14.6		1.9								100.0
SAM9	$(SAM40)_{90}+Mo_7+Y_3$	47.1	17.1	1.8	9.3	1.5	14.4	3.6	2.3	3.0								100.0
SAM10	$Fe_{57.3}Cr_{21.4}Mo_{2.6}W_{1.8}B_{16.9}$	57.3	21.4		2.6	1.8	16.9											100.0

HPCRM MSR Samples by TNC

Passive Film Stability

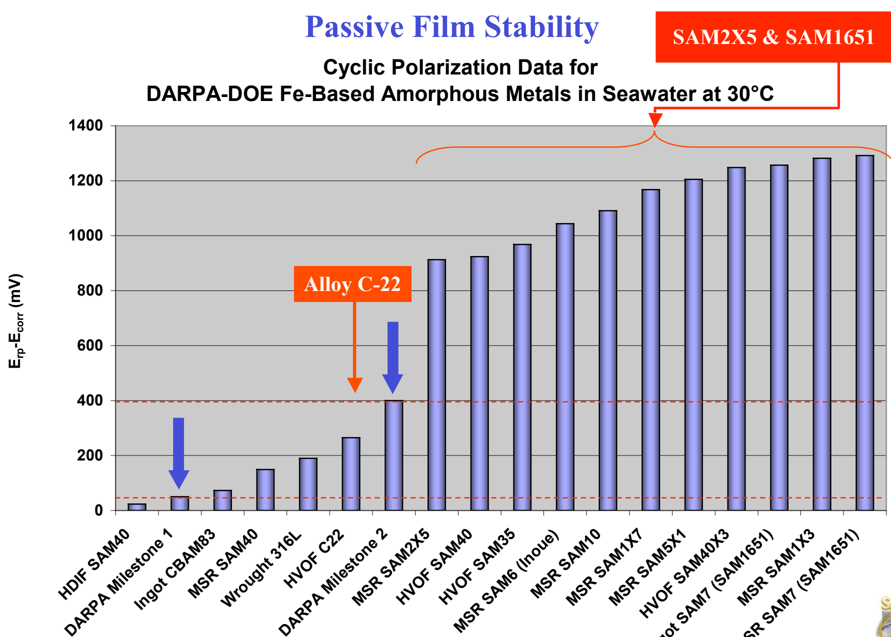
Cyclic Polarization Data for
DARPA-DOE Fe-Based Amorphous Metals in 5M $CaCl_2$ at 105°C



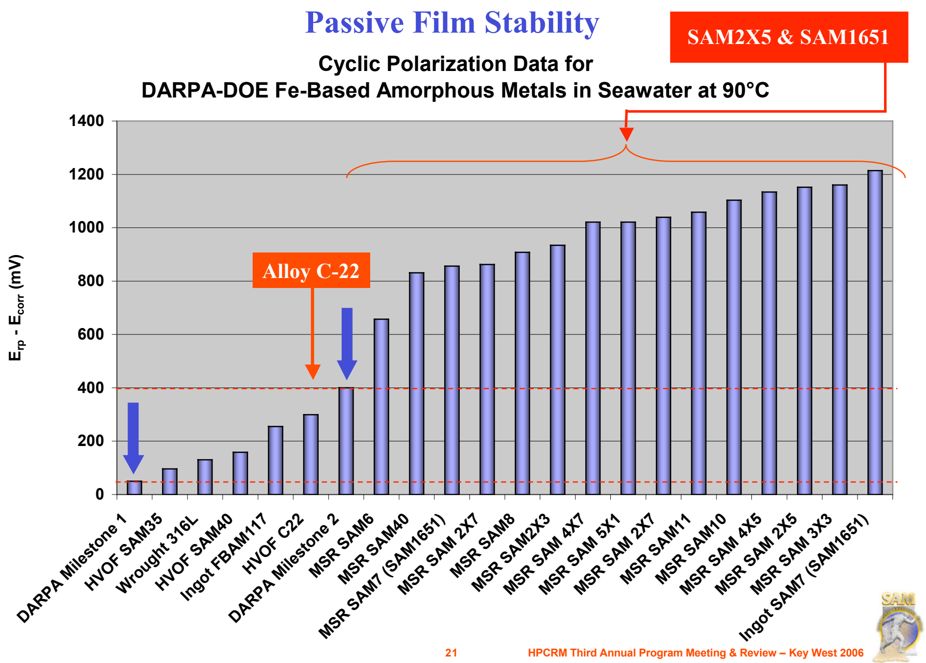
19 HPCRM Third Annual Program Meeting & Review – Key West 2006

Passive Film Stability

Cyclic Polarization Data for
DARPA-DOE Fe-Based Amorphous Metals in Seawater at 30°C



20 HPCRM Third Annual Program Meeting & Review – Key West 2006


Passive Film Stability
Cyclic Polarization Data for
DARPA-DOE Fe-Based Amorphous Metals in Seawater at 90°C
SAM2X5 & SAM1651
Alloy C-22
Erp - Ecorr (mV)
1400
1200
1000
800
600
400
200
0
DARPA Milestone 1
HVOF SAM35
Wrought 316L
HVOF SAM40
Ingot FBAM117
HVOF C22
DARPA Milestone 2
MSR SAM6
MSR SAM40
MSR SAM7 (SAM1651)
MSR SAM 2X7
MSR SAM8
MSR SAM2X3
MSR SAM 4X7
MSR SAM 5X1
MSR SAM 2X7
MSR SAM11
MSR SAM10
MSR SAM 4X5
MSR SAM 2X5
MSR SAM 3X3
Ingot SAM7 (SAM1651)
21
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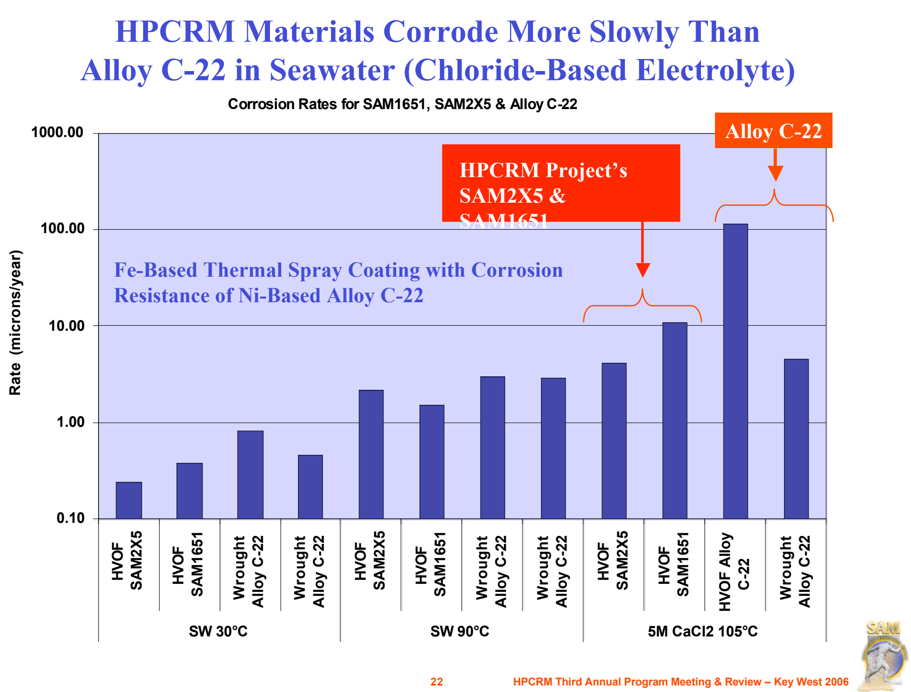
HPCRM Materials Corrode More Slowly Than
Alloy C-22 in Seawater (Chloride-Based Electrolyte)
Corrosion Rates for SAM1651, SAM2X5 & Alloy C-22
Alloy C-22
HPCRM Project's SAM2X5 & SAM1651
Fe-Based Thermal Spray Coating with Corrosion Resistance of Ni-Based Alloy C-22
Rate (microns/year)
1000.00
100.00
10.00
1.00
0.10
HVOF SAM2X5
HVOF SAM1651
Wrought Alloy C-22
Wrought Alloy C-22
HVOF SAM2X5
HVOF SAM1651
Wrought Alloy C-22
Wrought Alloy C-22
HVOF SAM2X5
HVOF SAM1651
HVOF Alloy C-22
Wrought Alloy C-22
SW 30°C
SW 90°C
5M CaCl2 105°C
22
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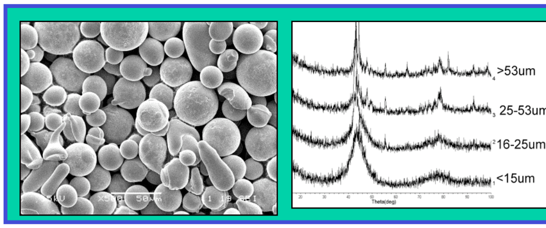
Ingots & Powder

SAM2X5 Powder Has More Consistent and Predictable Morpology Than SAM1651 Powder – SAM2X5 More Easily Sprayed

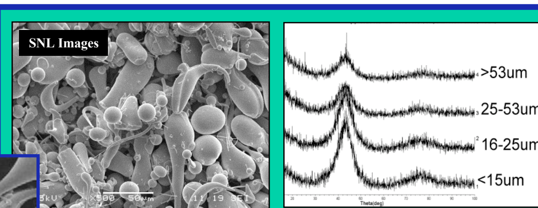
SAM2X5

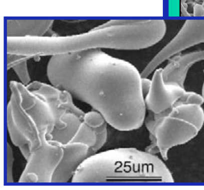
High critical cooling rate: ***small powders remain amorphous, but large powders devitrified***



SAM1651

Low critical cooling rate: ***amorphous structure for all particle sizes***

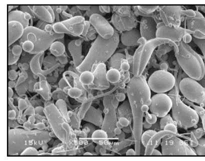




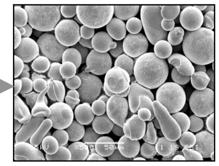
Irregular shaped SAM1651 powders flow through HVOF system with greater difficulty than SAM2X5

Wire-fed HVOF will enable deposition of SAM1651 without the need for further optimization of powder atomization process

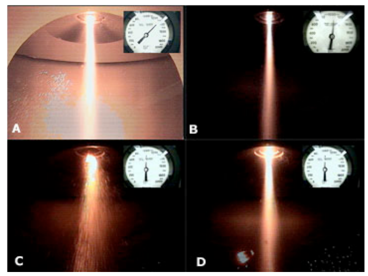
Need to Optimize of SAM1651 Gas Atomization Process



Non-Spherical SAM1651
Low Critical Cooling Rate
Completely Amorphous



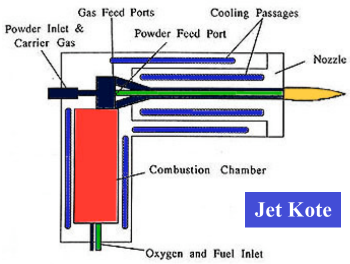
Spherical SAM2X5
High Critical Cooling Rate
Residual Crystaline Phase



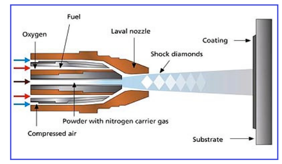
State-of-the-art gas atomization facility with laser diagnostics at UCD will be used to achieve spherical SAM1651 powder morphology for pneumatic conveyance in HVOF systems

Thermal Spray

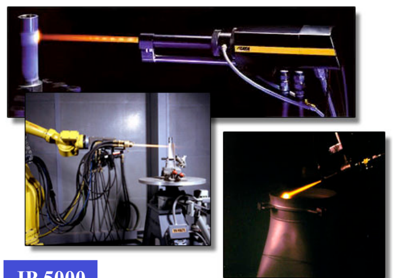
Thermal Spray Coating – UCD/INL /Caterpillar



Jet Kote



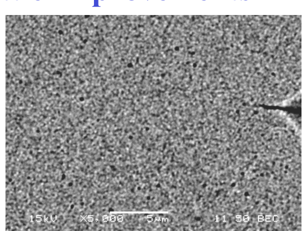
DJ2600 / Air Cooled Nozzle (UCD)



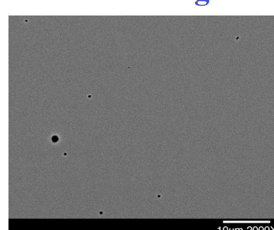
JP 5000

DJ2700 / Water Cooled Nozzle (UCD)

Dramatic Improvements in Microstructure & Coating Morphology

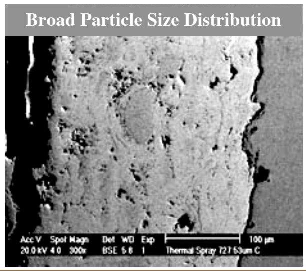


DAR40 at 800°C for 1 Hour

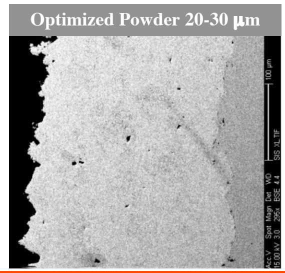


SAM1651 at 800°C for 1 Hour

Broad Particle Size Distribution



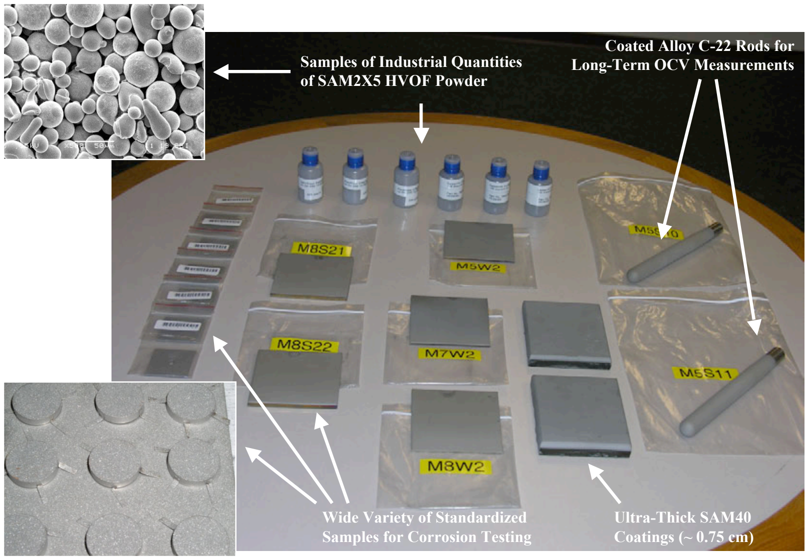
Optimized Powder 20-30 µm



Early Amorphous Metal Formula & Typical Coating Process

New SAM1651 Formula & Optimized Coating Process

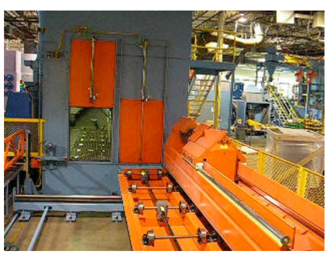
SAM2X5 Powder & HVOF Samples – TNC/INL/Caterpillar

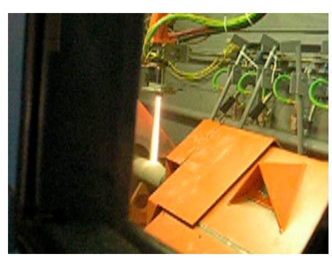


SAM2X5 on Alloy C-22 Plates – HVOF by Caterpillar

Automated Process

Caterpillar's Robotic Thermal Spray at Progressive Technology





Phase Stability

Reduced Glass Transition Temperature Indicative of Glass Forming Ability

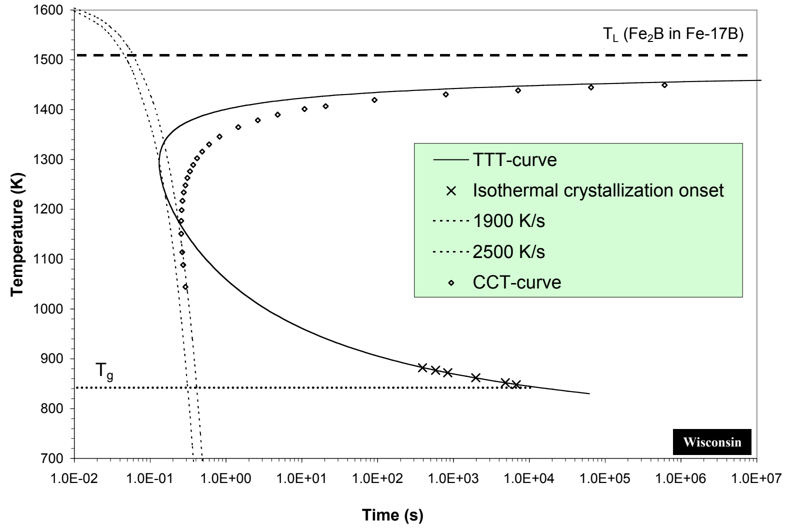
SAM2X5 → (SAM2X5)

SAM1651 → (SAM7)

Alloy	T_g (°C)	T_x (°C)	T_m (°C)	T_L (°C)	T_{rg}
SAM35	545-565	613	1074	1350	0.51
SAM40	568-574	623	1110	1338	0.53
SAM40X3	561-567	630	1130	1260	0.55
SAM1X1	not clear	612	1121	min. 1270	N.A.
SAM1X3	560	589	1119	min. 1300	0.53
SAM1X5	540	572	1115	min. 1300	0.52
SAM1X7	510	545	1112	min. 1300	0.50
SAM2X1	575	620	1124	1190-1210	0.57
SAM2X3	578	626	1131	1190-1210	0.57
SAM2X5	579	628	1133	1190-1210	0.57
SAM2X7	573	630	1137	1190-1210	0.57
SAM3X1	560	614	1108	min. 1320	0.52
SAM3X3	573	659	1138	min. 1380	0.51
SAM3X5	590	677	1143	min. 1400	0.52
SAM3X7	not clear	697	1164	min. 1420	
SAM4X1	573	621	1135	min. 1300	0.54
SAM4X3	568	623	1146	min. 1320	0.53
SAM4X5	580	623	1194	1290	0.55
SAM4X7	558	616	1198	1255	0.54
SAM5X1	570	622	1134	min. 1360	0.52
SAM5X3	575	641	1147	min. 1410	0.50
SAM5X5	596	659	1193	min. 1420	0.51
SAM6	580	623 [2)]	995	1238-1250	0.56
SAM7	584	653 [2)]	1121	1290	0.55
SAM8	565	637 [2)]	1137	1350-1370	0.52
SAM9	572	677 [2)]	1146	1223	0.56
SAM10	535	568 [1)]	1210	1350-1370	0.50
SAM11	535	572 [1)]	1202	1365-1395	0.49

Wisconsin

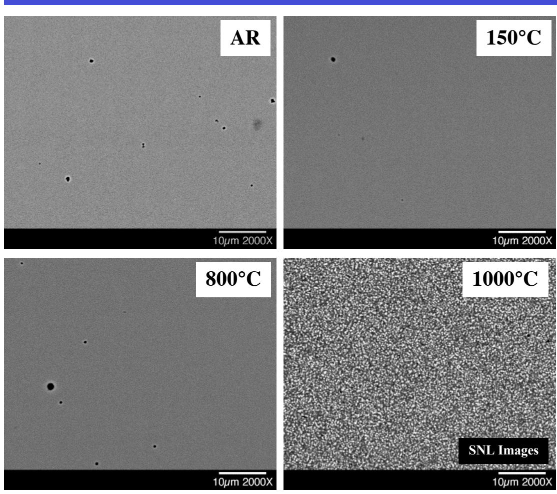
TTT/CCT Diagrams for SAM35 Based Upon Experimental Data, Kinetics Analysis and Estimated Thermodynamics



Full Thermodynamic Analysis in Progress – Application of Model to SAM2X5 & SAM1651

Thermal Stability of SAM1651 Drop-Cast Ingot

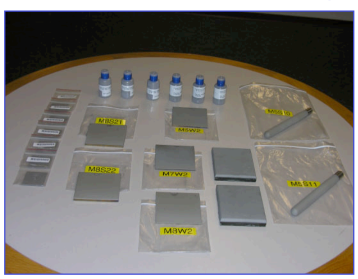
Microstructure of amorphous SAM1651 appeared to be stable up to 800°C/1hr. Beyond 1000°C, the matrix transformed into submicron crystalline phase(s).



Y-oxide particles remained unaffected in matrix of SAM1651 drop-cast ingot.

Corrosion & Impact

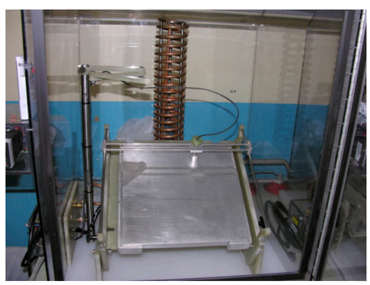
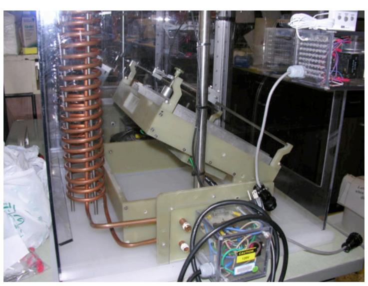
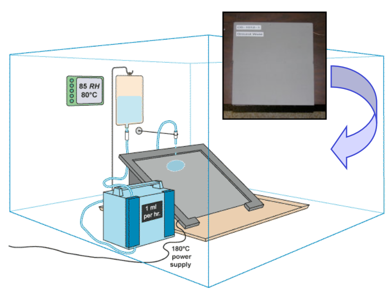
Long-Term Immersion Testing of SAM2X5 & SAM1651 Corrosion Potential, Weight Loss & Crevice Corrosion – LLNL



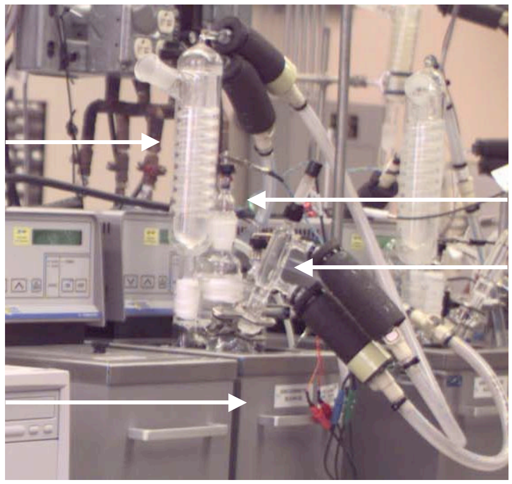
Corrosion Test Matrix at LLNL

Test Matrix at Lawrence Livermore National Laboratory						
Test Solution Type	NaCl	KNO_3	T	$CaCl_2$	$Ca(NO_3)_2$	T
	M or m	M or m	°C	M or m	M or m	°C
Half Moon Bay SW			30, 90			
Half Moon Bay SW			30, 90			
Chloride-Nitrate	1 M	None	30, 90			
Chloride-Nitrate	3.5 m	None	30, 90			
Chloride-Nitrate	3.5 m	0.175 m	30, 90			
Chloride-Nitrate	3.5 m	0.525 m	30, 90			
Chloride-Nitrate	6.0 m	None	30, 90			
Chloride-Nitrate	6.0 m	0.300 m	30, 90			
Chloride-Nitrate	6.0 m	0.900 m	30, 90			
Calcium Chloride				5 M	None	105
Calcium Chloride				12 m	None	130
Calcium Chloride				12 m	6 m	130
Published References: PVP 2005 -71173; 71174; 71175; 71176.						

Exposure of Hot Plate to Dripping Geothermal Brines – LLNL

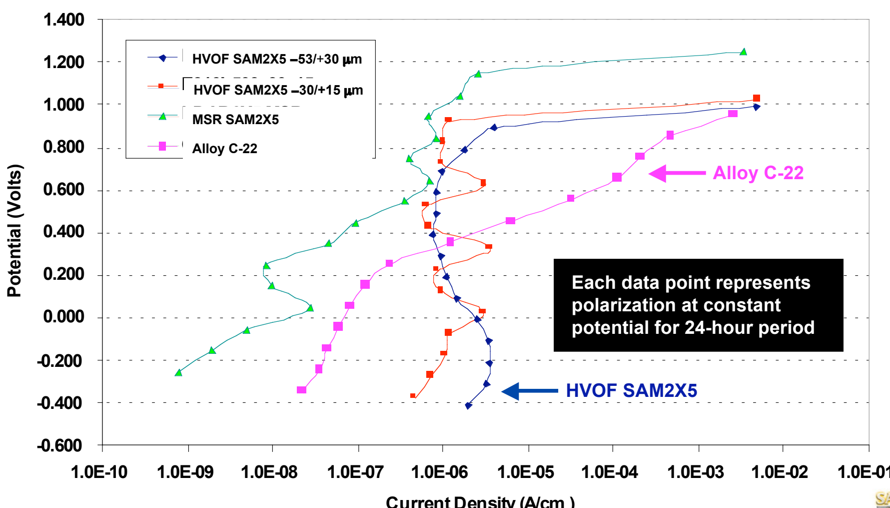





Electrochemical Measurements in Temperature Controlled Cells - LLNL

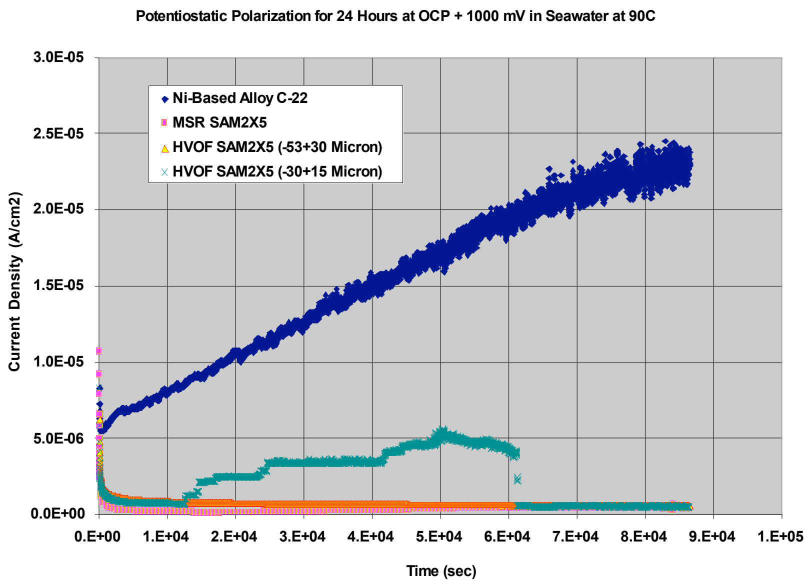


Potentiostatic Polarization of Alloys in Hot Seawater

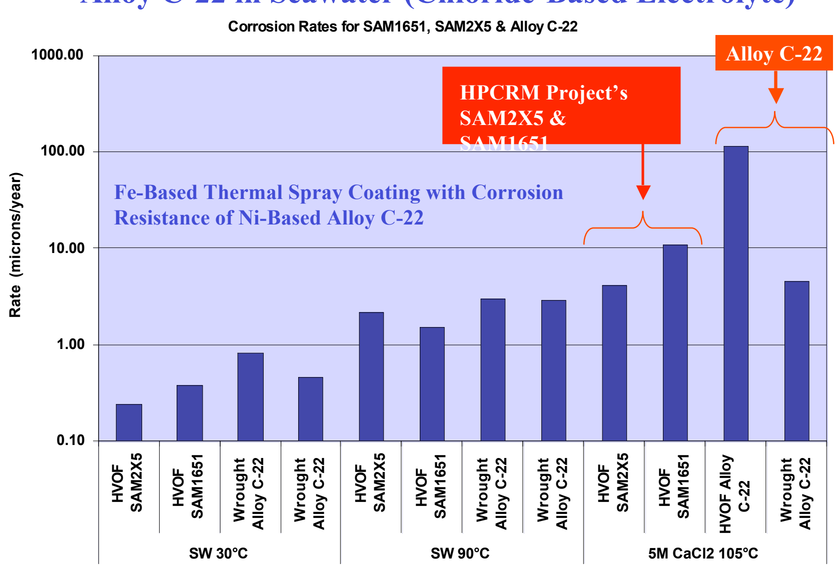
HVOF & MSR SAM2X5 Fe-Based Amorphous Metal Compared to Wrought Alloy C-22
Potentiostatic 100 mV Step Test in 90°C Seawater



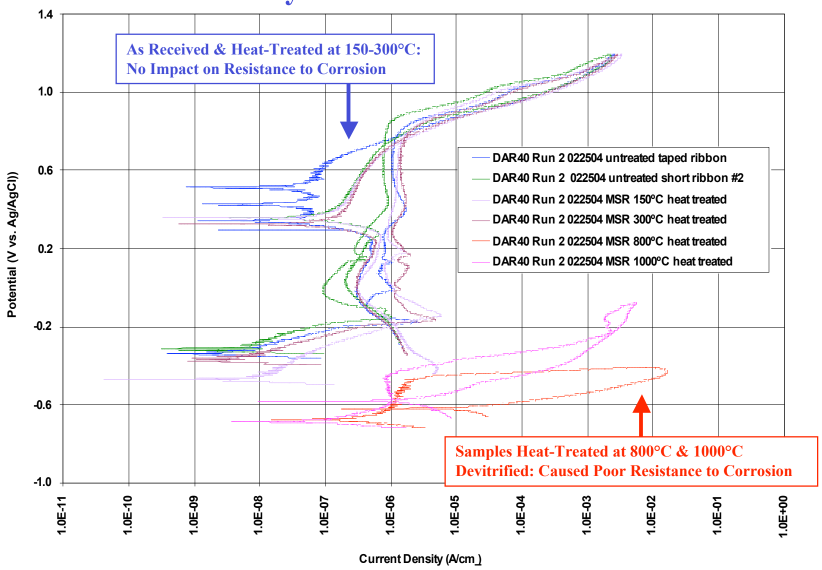
Potentiostatic Polarization of SAM2X5 in Hot Seawater



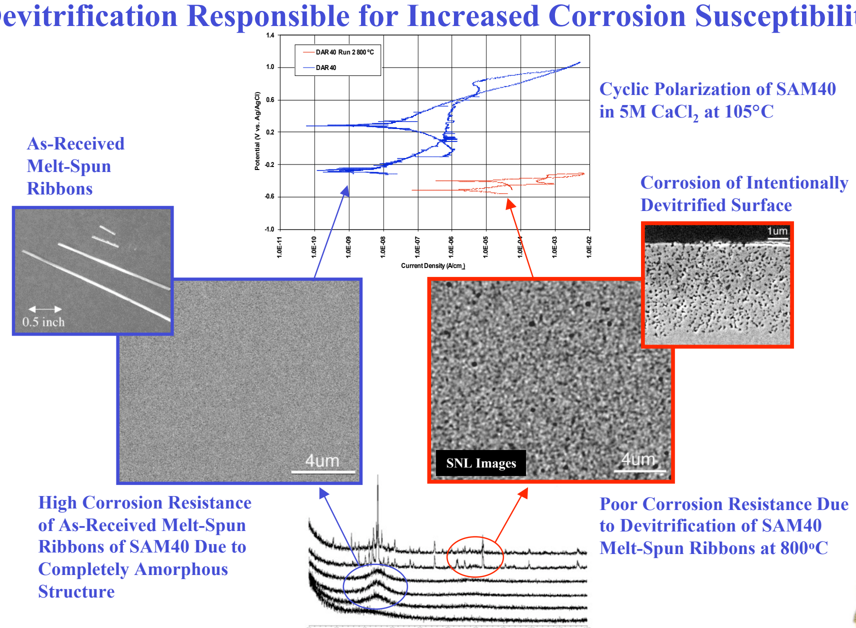
HPCRM Materials Corrode More Slowly Than Alloy C-22 in Seawater (Chloride-Based Electrolyte)



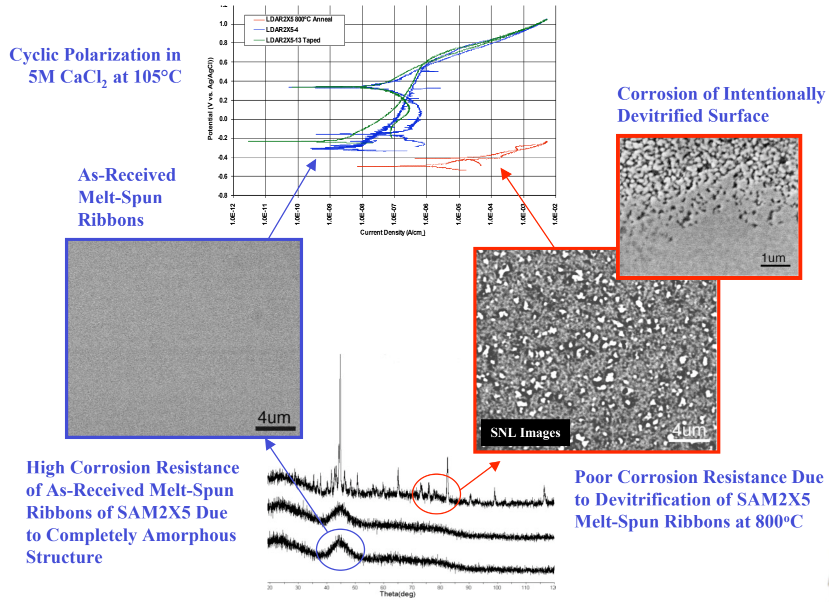
Effect of High Temperature on Corrosion Resistance of Early SAM40 Formulation



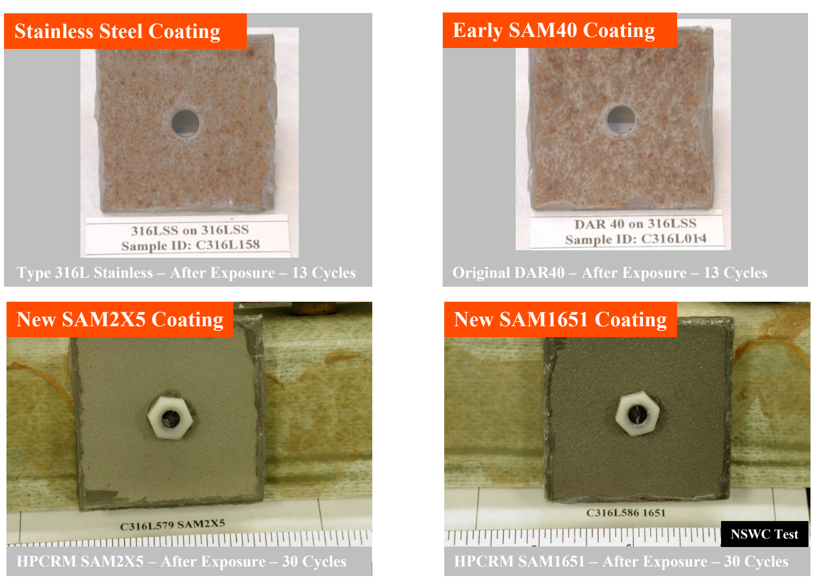
SAM40 (DAR40) Devitrifies After 1 Hour at 800°C: Devitrification Responsible for Increased Corrosion Susceptibility



SAM2X5 (LDAR2X5) Devitrifies After 1 Hour at 800°C: Devitrification Responsible for Increased Corrosion Susceptibility

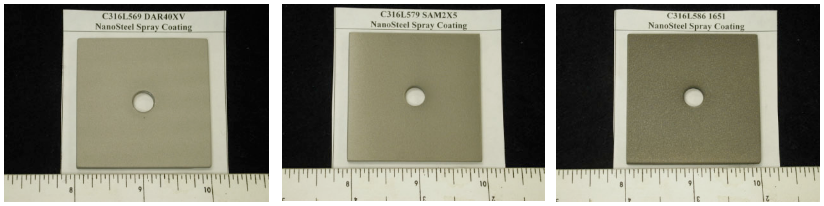


No Corrosion Observed During Salt Fog Testing

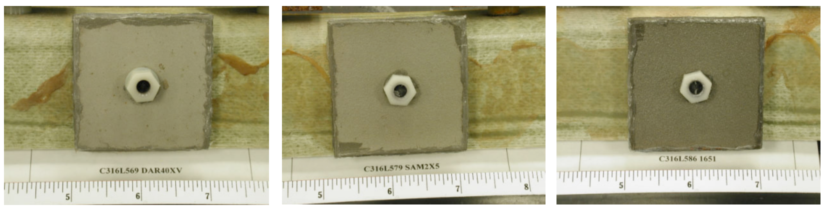


Standard Salt Fog Testing – NSWC

As-Received HVOF SAM40XV, SAM2X5 & SAM1651 on 316L Substrates



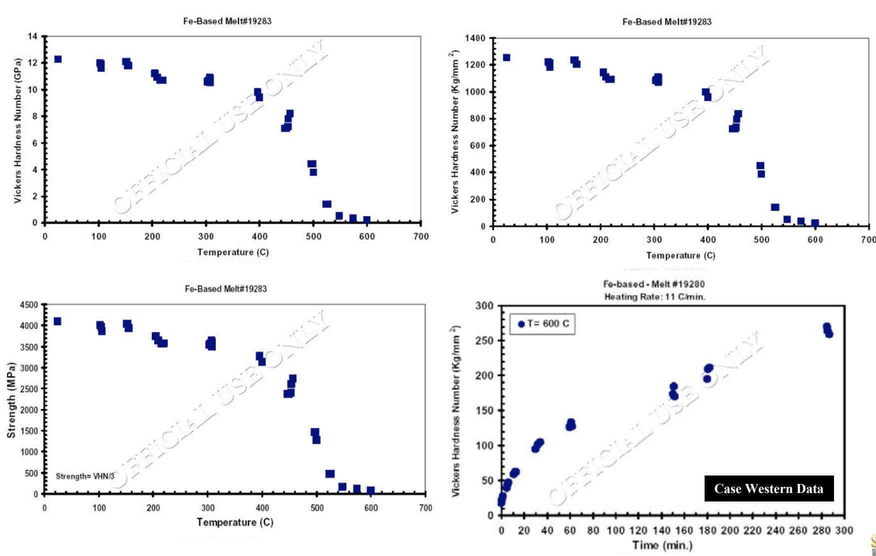
30-60 Cycles in Salt Fog:
Slight Attack of SAM40XV / No Attack of SAM2X5 or SAM1651



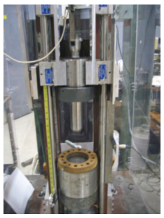
Electrochemical Corrosion Testing in Gamma Pit at LLNL

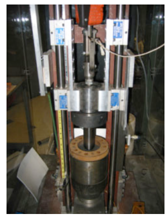
Mechanical Properties

Hot Hardness Measurements of SAM1651 Ingots (Lewandowski)

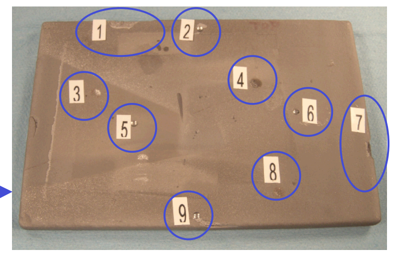


Impact Testing with Fully Instrumented Drop Towers – LLNL



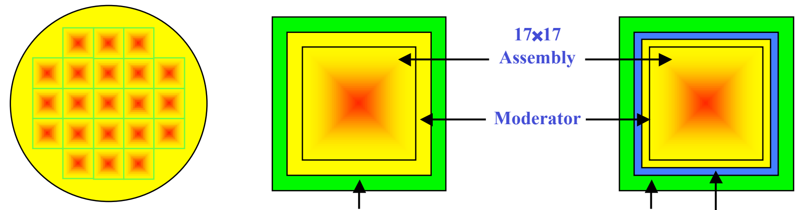


Preliminary Damage Testing of HY80 Plate:
- Impact from Side: 1
- Impact Directly on Top: 2, 5, 6, 9
- Ball Peen Hammer Top: 3, 4, 8
- Ball Peen Hammer on Edge: 7



Applications

Criticality Safety Analysis Results



	¼-inch (6.4 mm) Stainless Steel Basket								
	No Boron	0.12 wt. % Boron	1 wt. % Boron	2 wt. % Boron	No Boron 1-mm SAM2X5	0.12 wt. % Boron 1-mm SAM2X5	No Boron 1-mm SAM1651	0.12 wt. % Boron 1-mm SAM1651	¼-inch Ni-Gd Basket Material
k_{eff}	0.96	0.91	0.85	0.83	0.87	0.86	0.90	0.88	0.86
Δk_{eff}	0.0	0.05	0.11	0.13	0.09	0.10	0.06	0.08	0.10

Neutron attenuation effectiveness of SAM will be tested at radiation facilities

Irradiation, Attenuation & Radiography at TRIGA Reactor

Unperturbed Neutron Fluxes & Heating at 1.5 MW Operating Power in MNRC's TRIGA Reactor

Facility	Thermal < 0.1 eV	Fast > 1 MeV	$\varphi_{fast} / \varphi_{thermal}$	Heating in Aluminum	Diameter	Length
	n cm^{-2} sec^{-1}	n cm^{-2} sec^{-1}	%	W g^{-1}	cm	cm
Central Irradiation Facility (CIF)	1.5×10^{13}	7.6×10^{12}	50	0.16	4.4	38
Pneumatic Transfer System (PTS)	7.6×10^{12}	3.7×10^{12}	50	0.084	1.5	10
Neutron Transmutation Doping (NTD)	4.4×10^{11}	1.1×10^{13}	2.5	0.0027	8.8	22

Ex-Portsmouth 688 Class Submarine Sail

Sail of Ex-Portsmouth 688 Class Submarine with Black Epoxy Coating on Exterior Surface

Sail Cover Plates Ready for Coating at Caterpillar

Exterior Forward Sail Cover Plates Removed

Internal Aft Sail Corrosion

Internal Forward Sail Corrosion

Summary
Benfits & Risks

Benefits of Iron-Based Amorphous Metals

- Fe-based amorphous metal formulations have been identified with corrosion resistance comparable to that of Ni-based Alloy C-22
  - Cr & Mo provide corrosion resistance
  - B enables glass formation
  - Y lowers critical cooling rate
    - SAM1651 = 80 K/s (yttrium added)
    - SAM2X7 = 610 K/s (no yttrium) … similar to SAM2X5
- These materials provide a viable coating option for repository engineers
  - SAM2X5 & SAM1651 coatings can be applied with thermal spray processes without any significant loss of corrosion resistance
  - C-22 and 316L lose their resistance to corrosion during thermal spraying
- SNF/HLW containers with corrosion resistant coatings are envisioned
  - Enhanced multi-purpose container (MPC) … leverage existing capability
  - Protected closure weld … eliminate need for stress mitigation
  - Integral drip shield … elimination of titanium drip shield
  - Thicken areas where greater corrosion is expected (crevices)

Benefits of Iron-Based Amorphous Metals

- These materials are extremely hard and provide enhanced resistance to abrasion and gouges (stress risers) from backfill operations
  - Type 316L SS = 150 VHN
  - Alloy C-22 = 250 VHN
  - HVOF SAM2X5 = 1100-1300 VHN
- Both SAM2X5 & SAM1651 have high boron content which enable them to absorb neutrons and therefore be used for criticality control in baskets
  - Alloy C-22 and 316L have no neutron absorber
  - Problems encountered with borated stainless and Gd-doped Ni-Cr-Mo
  - Variable thickness absorber
- Cost savings through substitution of Fe-based alloy for Ni-based material
  - Fe = $0.50 (USGS 2004) / $0.19 (USGS 1998)
  - Ni = $6.28 (USGS 2004) / $2.10 (USGS 1998)
- Further improvements in such thermal spray coatings are possible
  - Amorphous metal transitioning into ceramic outer layer (proprietary)
  - Advanced graded and multilayer coatings

Technical Risks Associated with Fe-Based Amorphous Metals

- Both amorphous metal formulations have strengths and weaknesses
  - SAM1651 (yttrium added)
    - Low critical cooling rate (CCR) = amorphous in 'as sprayed' condition
    - Irregular powder = difficult to atomize and spray
    - Possible need for cryogenic milling of powder
  - SAM2X5 (no yttrium)
    - High critical cooling rate = potential problem with devitrification
    - Spherical powder = more easily atomized and thermally sprayed
- While SAM1651 powders can be rendered as fully amorphous coatings in the 'as sprayed condition' … irregular particle shapes cause coating porosity
  - Additional process optimization is required to identify atomization conditions where spherical particles can be produced at high yield
- In the case of 'high CCR alloys' such as SAM2X5 … variability in cooling rate may cause devitrification
  - Some devitrification will enhance damage tolerance … and is beneficial
  - However … too much devitrification has been observed to compromise corrosion resistance in both electrochemical and salt fog testing

61 HPCRM Third Annual Program Meeting & Review – Key West 2006

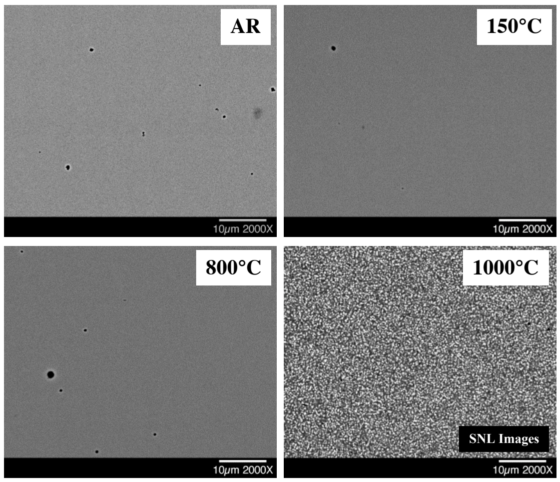
Technical Risks Associated with Fe-Based Amorphous Metals

- Coatings are being produced by different manufacturers
  - Thermal spray parameters believed to be optimal by one manufacturer may be different than those parameters preferred by another
  - Better standardization and quality control is needed … which will inevitably occur during the process of qualification (achieving QSL status)
- Broad composition specifications have been suggested by powder suppliers
  - These specifications were developed without accounting for the sensitivity of corrosion resistance to variability/uncertainty in elemental composition
  - Sensitivity studies are needed (consider heat-to-heat variability issue)
- Alloy C-22 is an outstanding corrosion-resistant engineering material
  - Even so … crevice corrosion has been observed with C-22 in hot sodium chloride environments without buffer or inhibitor
  - Comparable metallic alloys such as SAM2X5 and SAM1651 may also experience crevice corrosion under sufficiently harsh conditions
  - Accelerated crevice corrosion tests are now being conducted to intentionally induce crevice corrosion … and to determine those environmental conditions where such localized attack occurs

62 HPCRM Third Annual Program Meeting & Review – Key West 2006

Thermal Stability of SAM1651 Drop-Cast Ingot

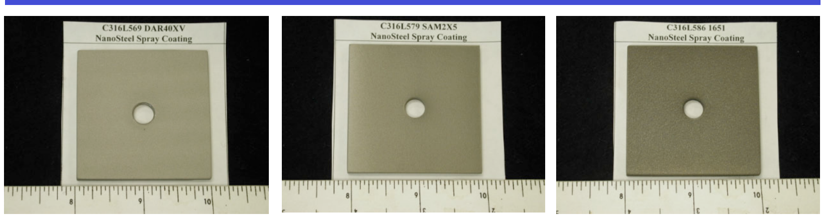
Microstructure of amorphous SAM1651 appeared to be stable up to 800°C/1hr. Beyond 1000°C, the matrix transformed into submicron crystalline phase(s).



Y-oxide particles remained unaffected in matrix of SAM1651 drop-cast ingot.

Standard Salt Fog Testing – NSWC

As-Received HVOF SAM40XV, SAM2X5 & SAM1651 on 316L Substrates



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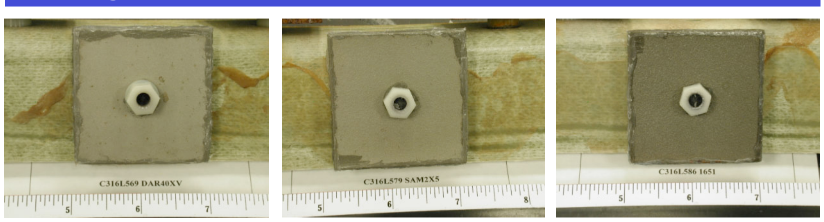
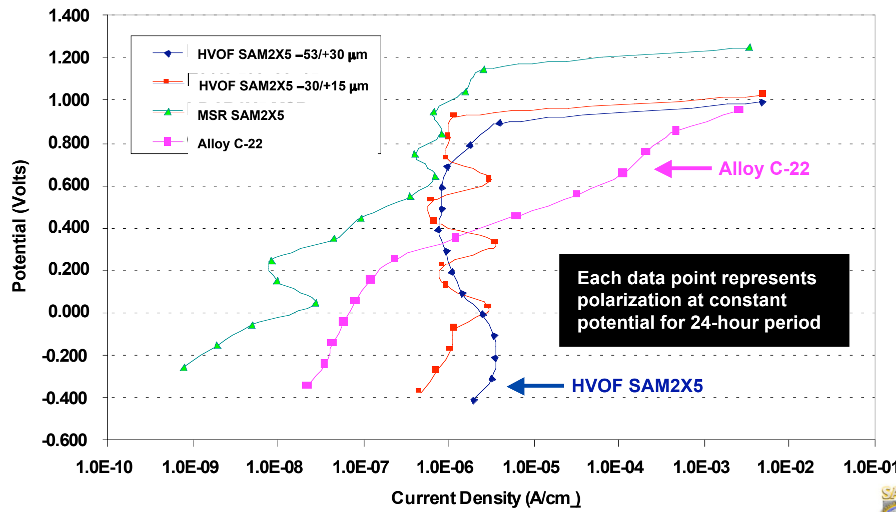


Potentiostatic Polarization of Alloys in Hot Seawater

HVOF & MSR SAM2X5 Fe-Based Amorphous Metal Compared to Wrought Alloy C-22
Potentiostatic 100 mV Step Test in 90ºC Seawater



Backup Slides

Introduction

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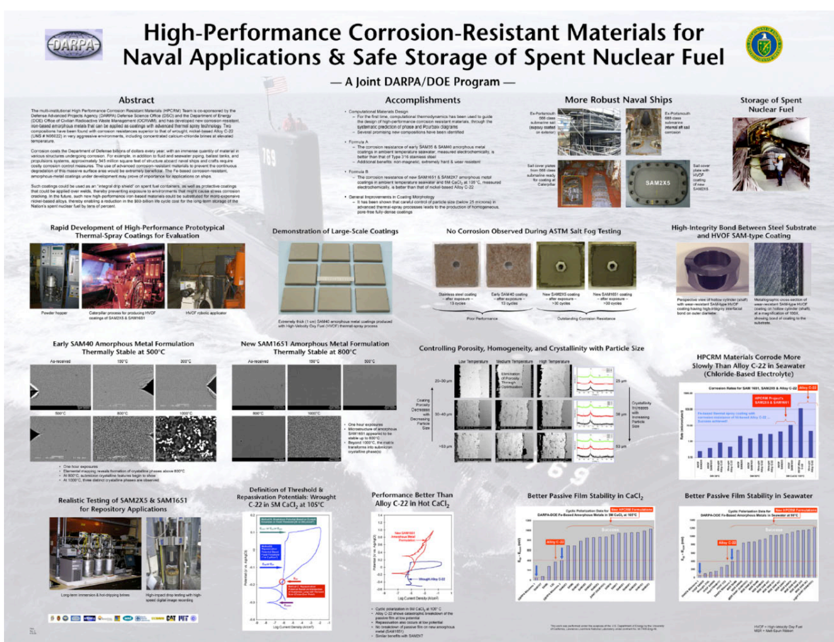

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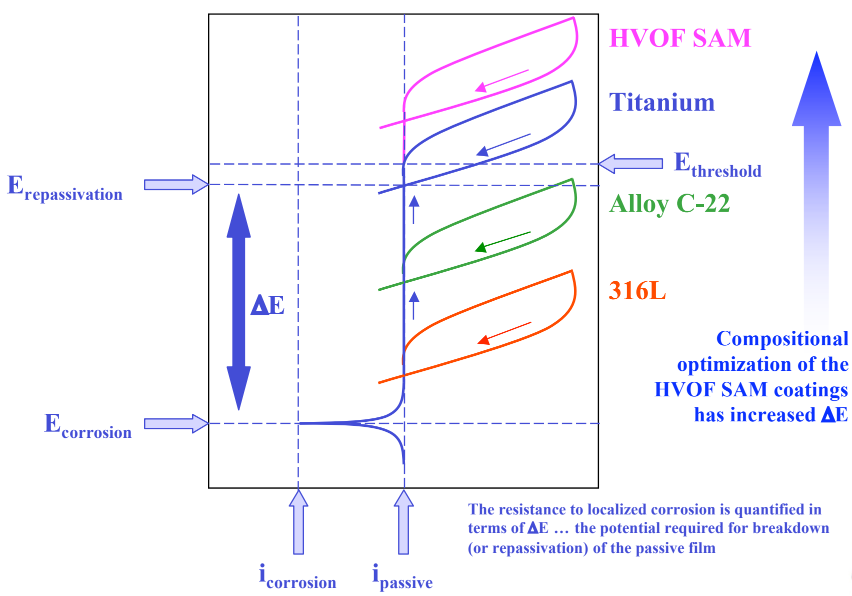
Alloy Screening

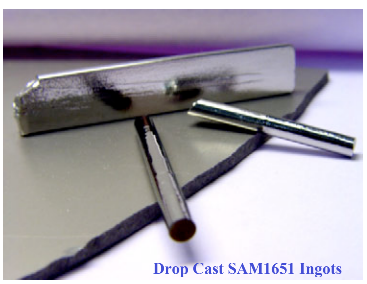
Repassivation Potential as a Quantifiable Metric

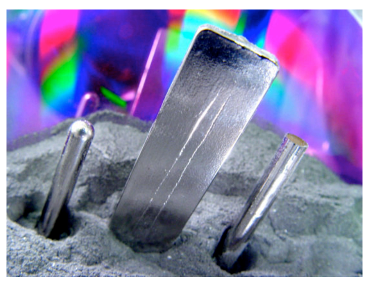


The resistance to localized corrosion is quantified in terms of ΔE … the potential required for breakdown (or repassivation) of the passive film

Ingots & Powder

Amorphous Ingots & Powder – ORNL/Carpenter


Drop Cast SAM1651 Ingots

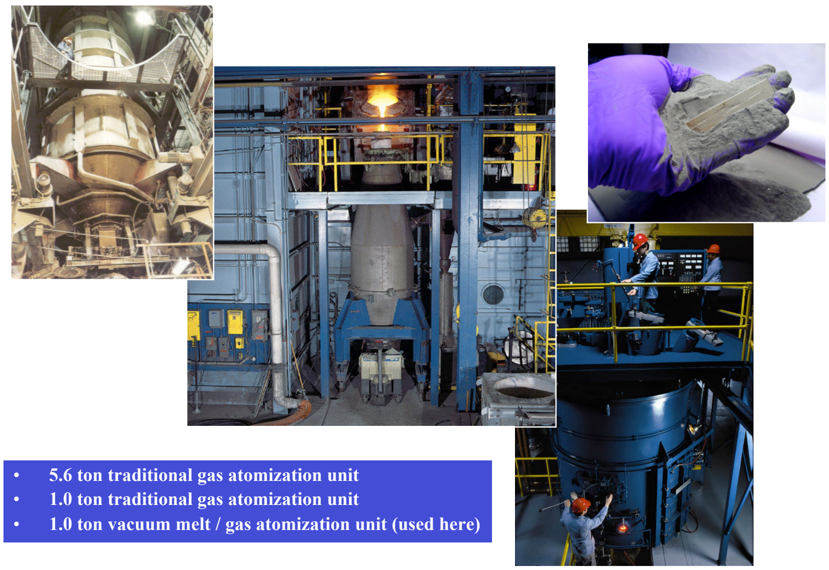



Gas Atomized SAM1651 Powder

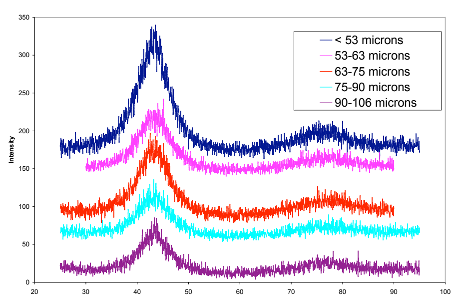
SAM1651 Powder – Four Size Classifications:

{ -16μm } = 100 lb yield
{ -25μm (-500M) +16μm } = 37 lb yield
{ -53μm (-270M) +25μm (+500M) } = 396 lb yield
{ +53μm (+270M) } = 660 lb yield

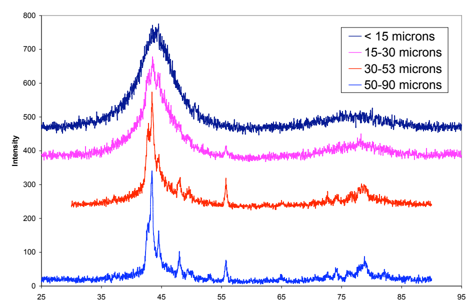
Gas Atomization of Amorphous Powder – ORNL/Carpenter



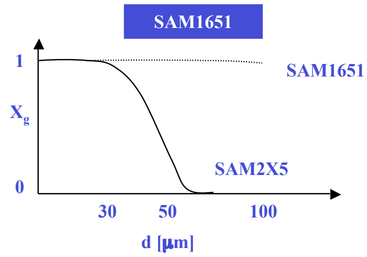
- 5.6 ton traditional gas atomization unit
- 1.0 ton traditional gas atomization unit
- 1.0 ton vacuum melt / gas atomization unit (used here)



SAM1651



SAM2X5



Processes using materials with low critical cooling rates are tolerant of large particle sizes.

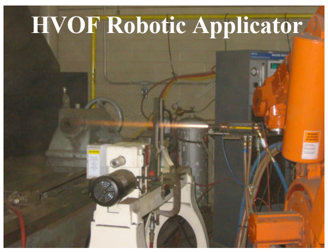
Powder solidification kinetics is being used to optimize gas atomization and thermal spray processes. Fraction amorphous in atomized powder can be approximated by:

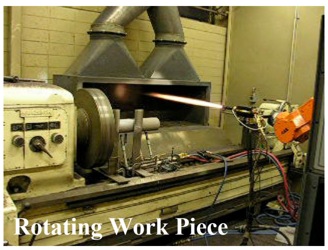
$X_g \sim \exp((-d/d_0)^n)$

where n identifies the crystal nucleation mechanism.

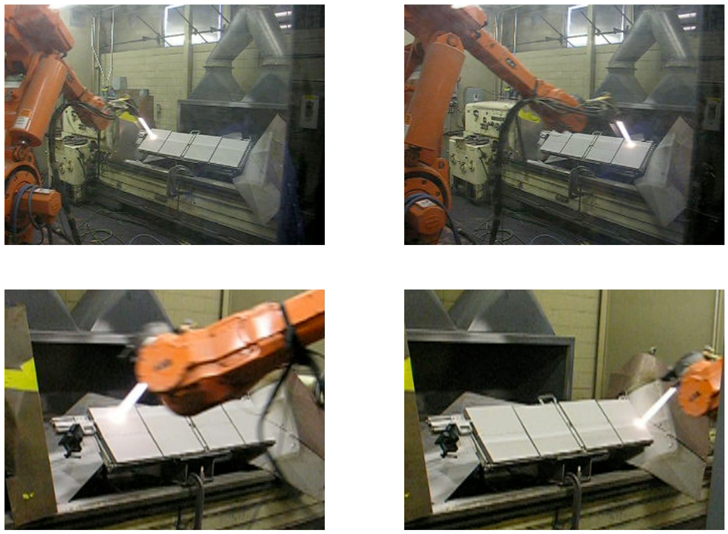
Thermal Spray

Typical Thermal Spray Process at Caterpillar





Coating C-22 Plates with SAM2X5 Using HVOF

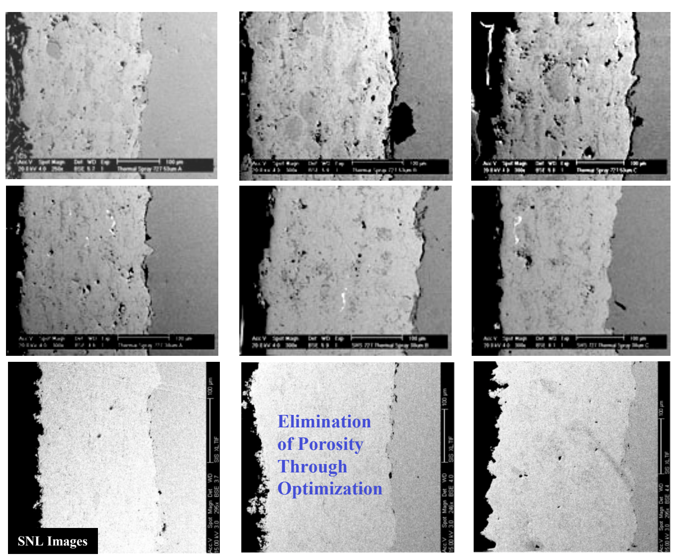


SAM2X5 on Alloy C-22 Plates – HVOF by Caterpillar

SAM2X5 on Alloy C-22 Plates – HVOF by Caterpillar

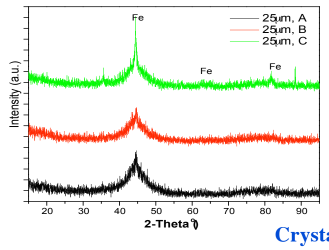
Controlling Porosity & Homogeneity with Particle Size

	Low Temperature	Medium Temperature	High Temperature
> 53 μm			
30-40 μm			
20-30 μm			

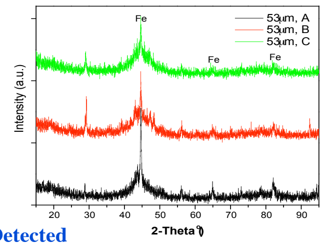


Controlling Crystallinity with Particle Size

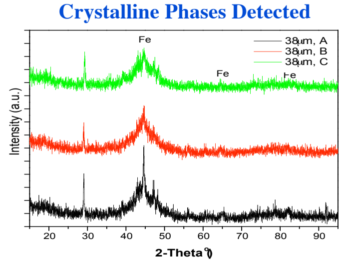
25 µm
High Degree of Amorphous



53 µm
Cystalline Phases Detected



38 µm
Crystalline Phases Detected



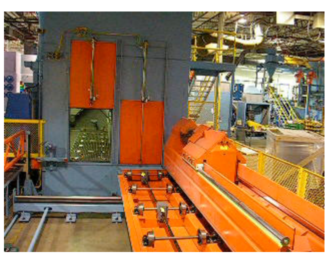
SNL XRD

Degree of Crystallinity Increases with Particle Size

Automated Process

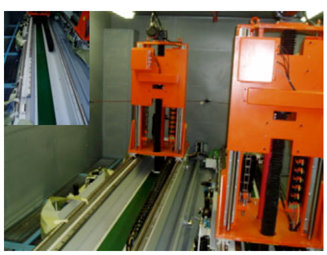
Caterpillar's Robotic Thermal Spray at Progressive Technology

Caterpillar's Robotic Thermal Spray at Progressive Technology

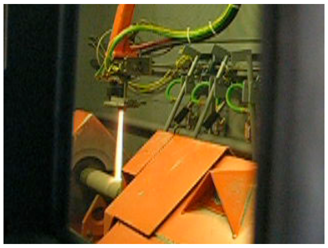
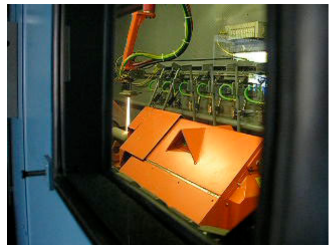
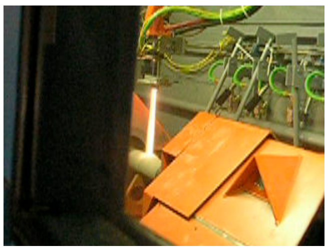
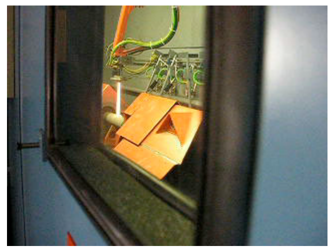


Caterpillar's Robotic Thermal Spray at Progressive Technology

Caterpillar's Robotic Thermal Spray at Progressive Technology



Caterpillar’s Robotic Thermal Spray at Progressive Technology

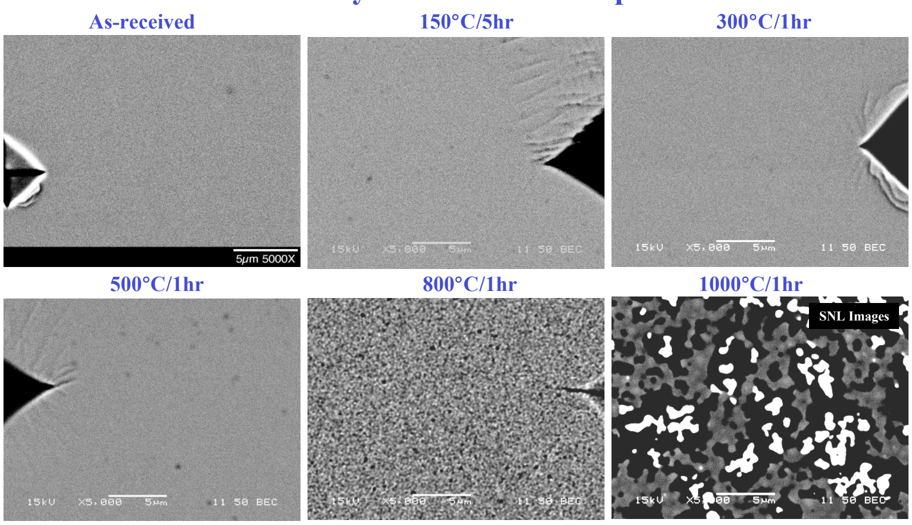
Caterpillar’s Robotic Thermal Spray at Progressive Technology




HPCRM Third Annual Program Meeting & Review – Key West 2006

Phase Stability

Thermal Stability of SAM40 Melt-Spun Ribbons

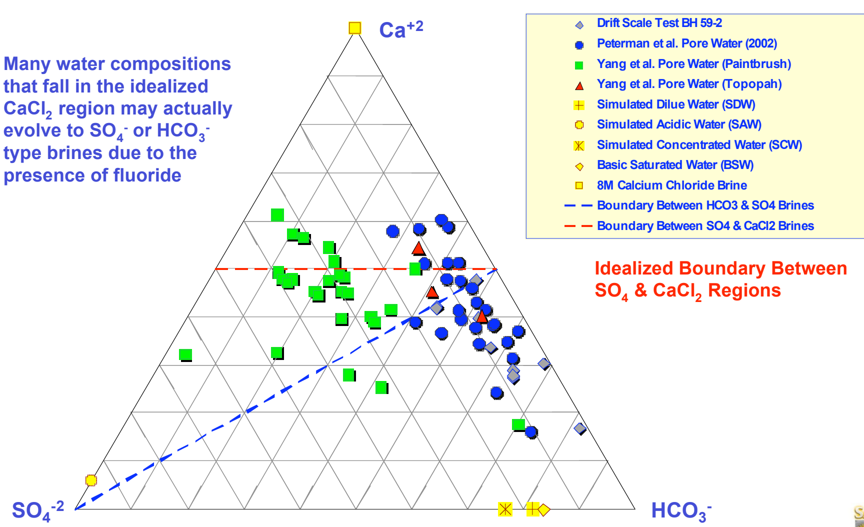


- Elemental mapping reveals formation of crystalline phases above 800°C.
- At 800°C/1hr, submicron crystalline features began to show.
- At 1000°C/2hrs, there were three distinct crystalline phases observed.

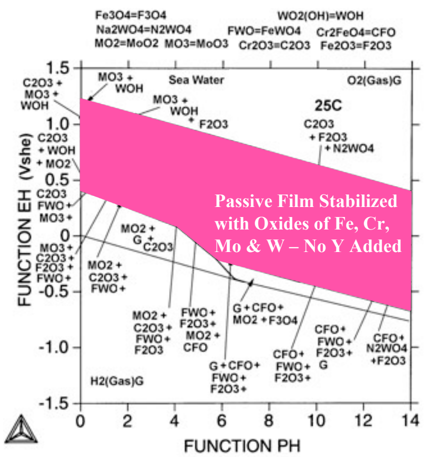
Corrosion & Impact

Classification of Yucca Mountain Brines

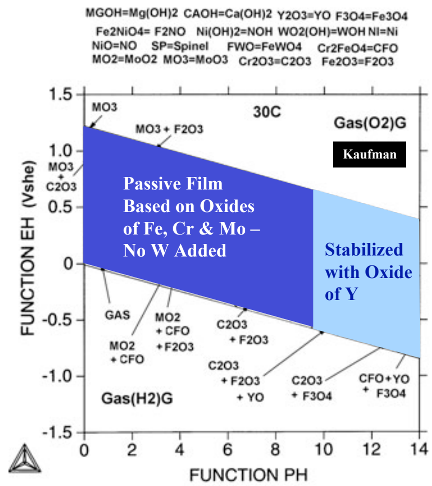
Many water compositions that fall in the idealized $CaCl_2$ region may actually evolve to SO_4^- or HCO_3^- type brines due to the presence of fluoride



Pourbaix Diagrams for SAM40X3 & SAM1651

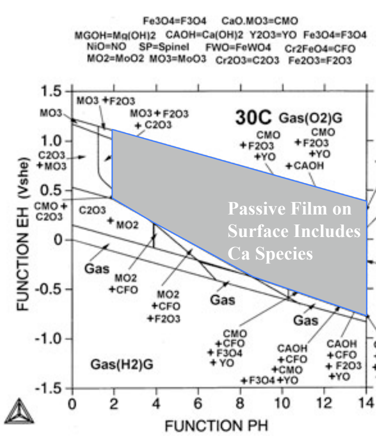


Prediction for early variant of SAM40X3 (Fe – 3Mo – 21Cr – 2W – 17B – atomic percent) in seawater (40 g NaCl + 1000 g H_2O) at 25°C

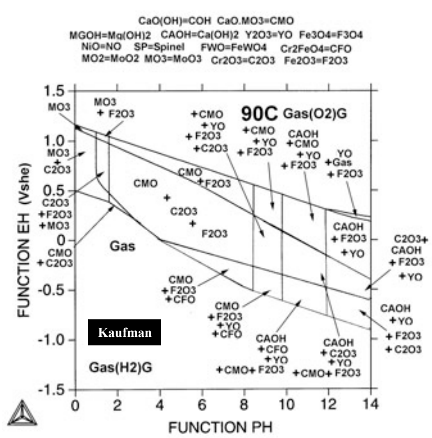


Prediction for SAM1651 (Fe – 14Mo – 15Cr – 2Y – 15C – 6B – atomic percent) in seawater (40 g NaCl + 1000 g H_2O) at 30°C

Calculated Pourbaix Diagram for SAM1651 in Concentrated Calcium Chloride

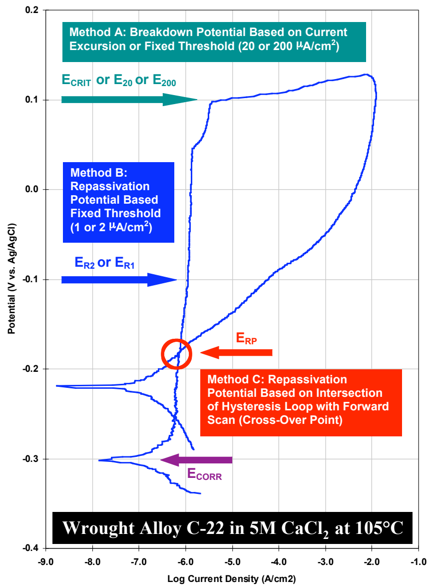


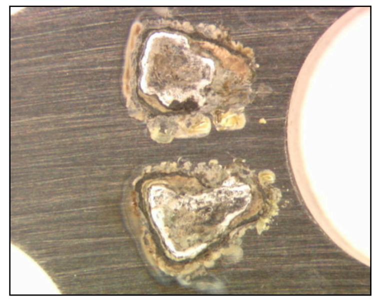
Fe-14Mo-15Cr-2Y-15C-6B
30°C 5M $CaCl_2$



Fe-14Mo-15Cr-2Y-15C-6B
90°C 5M $CaCl_2$

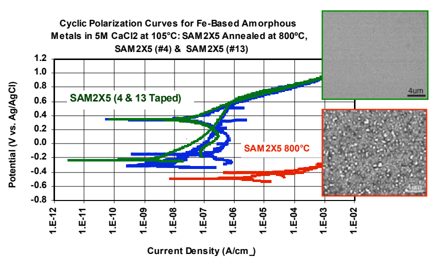
Accelerated Electrochemical Corrosion Testing – LLNL

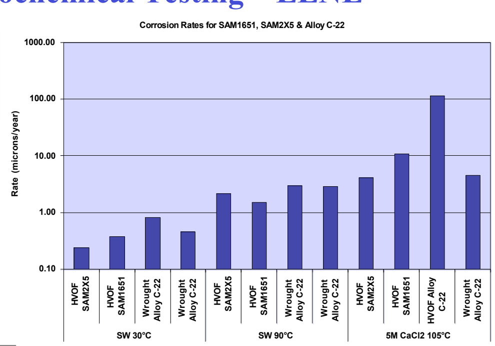


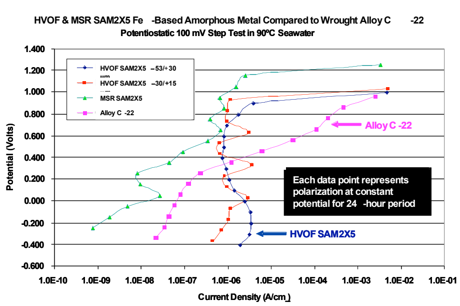


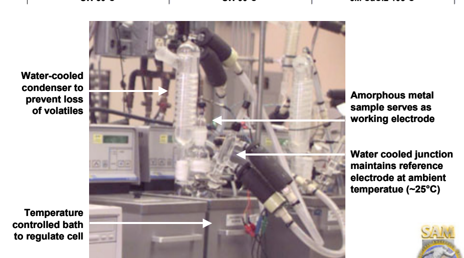
Crevice Attack of Alloy C-22 in Chloride-Nitrate Solution at 90°C

Comprehensive Electrochemical Testing – LLNL

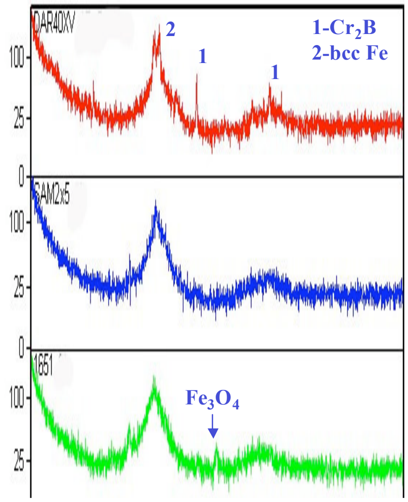


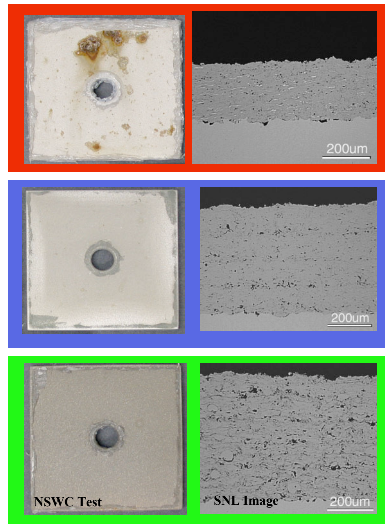






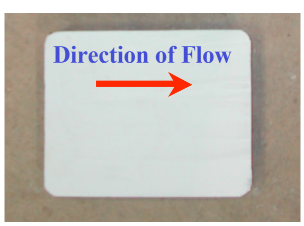
Exceptional Corrosion Resistance of SAM2X5 & SAM1651 Attributed to Sustained Amorphous Structure





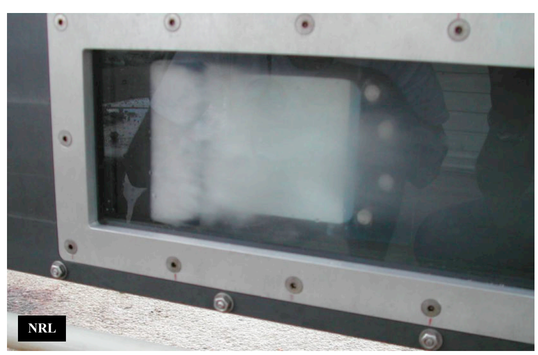
Observed Corrosion of Early SAM40 (DAR40) Coating Due to Devitrification

Naval Vessels – Cavitation Test with Standard Coating



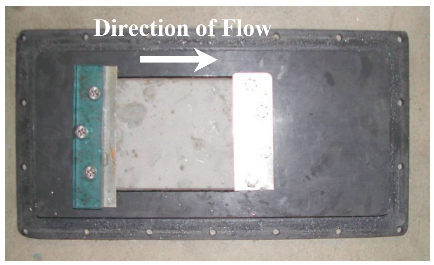
Before

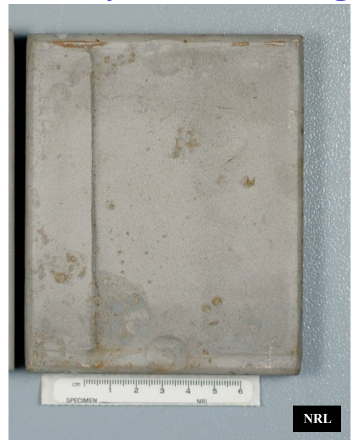
After 24-Hour Exposure



Sample in Flow Trough with Cavitation

Naval Vessels – Cavitation Test with Early SAM40 Coating





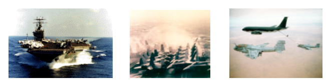
Early SAM40 Coating
After Cavitation Testing

Applications

Department of Defense Interests

- Cost of Corrosion to Department of Defense
  - Helicopter Repair Alone Costs ~ $4 Billion
  - Total Annual Cost ~ $10 Billion to $20 Billion
- Corrosion Effects All Military Assets
  - Ground &Tactical Vehicles ~ 35,000
  - Aircraft & Helicopters ~ 15,000
  - Strategic Missiles ~ 1,000
  - Navy Ships & Boats ~ 300
    - Structure ~ 345M ft^2
    - Propulsion, Fluid & Seawater Piping Systems

- Examples of Corrosion-Related Faults Degrading Mission Readiness
  - Crashes of Several F-16 in 1980s (Corrosion of Electrical Contacts Leading to Uncontrolled Fuel Valve Closure)
  - Collapses of Landing Gear on Navy F-14 and F-18 During Carrier Operations
- Personnel Requirements for Corrosion-Related Maintenance
  - Military & Civilian ~ 700,000
  - Commercial Firms ~ Several Thousand



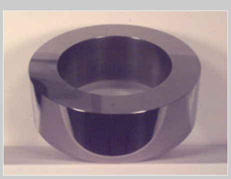
Actual Navy Parts From Puget Sound & Norfolk Naval Shipyards

- Puget Sound Naval Shipyard
  - *Sail Cover Plates from 688 Class Submarines*
    - *Small Size: Approximately 8-inch x 11-inch x 3/8-inch; 18 Parts*
    - *Medium Size: 16-inch x 17-inch x 3/8-inch; 24 Parts*
  - *Availability – July 19th 2005*
- Norfolk Naval Shipyard
  - *10K Low Pressure Brine Pump (Shafts) from 688 Class Submarines*
    - *APL 017030722; NSN 3H4320-01-317-3577*
    - *Manufacturer: Carver Pump Company; Drawing D-NDS-0-98-02*
    - *Shaft has no taper*
  - *1.6K Low Pressure Brine Pump (Shafts) from 688 Class Submarines*
    - *APL 017030383; NSN 2SH4320-01-032-9397-A2*
    - *Manufacturer: Carver Pump Company; Drawing D-NDS-0-98-001*
    - *Shaft has no taper*
  - *1OK & 1.6K Pump Motors*
    - *APL 173870012; NSN 9G6105-01-175-9774*
    - *Manufacturer: Hansome Energy Systems, Incorporated; Drawing A-203-12*
  - *Availability – July 21st 2005*

Actual Navy Parts & Samples Coated by Caterpillar

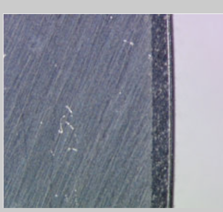
- **HVOF Coating of Actual Navy Parts with SAM1651/SAM2X5**
  - *Small-Size Cover Plates (& Witness Samples) for Sail*
    - *HVOF Coatings of SAM2X5 & SAM1651*
  - *Medium-Size Cover Plates (& Witness Samples) for Sail*
    - *HVOF Coatings of SAM2X5 & SAM1651*
  - *Rotating Shaft (& Witness) from Low-Capacity (1.6K) Brine Pump*
    - *Polished HVOF Coating of SAM2X5*
  - *Rotating Shaft (& Witness) from High-Capacity (10K) Brine Pump*
    - *Polished HVOF Coating of SAM1651*
  - *Prototypical Air-Intake Plenum (& Witness Samples) for Surface Ship*
    - *HVOF Coatings of SAM2X5 & SAM1651*
- **HVOF Coating Navy Test Samples with SAM1651**
  - *Salt Fog Testing – 3000 hour test*
  - *Long-Term Exterior Exposure – 6 months – 1 year*
  - *Alternate Immersion – 6 months – 1 year*
  - *Bend-Over-Mandrel Test – 1year*
  - *Cathodic Disbondment – 90 days*
  - *Galvanic Corrosion – 6 months – 1 year*
  - *Crevice Corrosion (Accelerated) – 1 months*

Rotating Shafts and Other Naval Applications

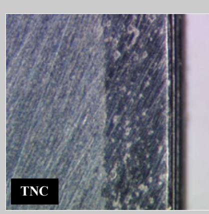


(a) Perspective view of hollow cylinder (shaft) with wear-resistant DAR-type HVOF coating having high-integrity interfacial bond on outer diameter.

(b) Plan view of hollow cylinder (shaft) with wear-resistant DAR-type HVOF coating with high integrity interfacial bond.

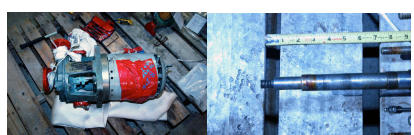


(c) Metallographic cross-section of wear-resistant DAR-type HVOF coating on hollow cylinder (shaft), at a magnification of 20×, showing bond of coating to the substrate.



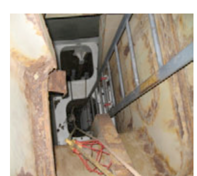
(d) Metallographic cross-section of wear-resistant DAR-type HVOF coating on hollow cylinder (shaft), at a magnification of 100×, showing bond of coating to the substrate.

HVOF SAM-Type Coating on Rotating Shaft with High-Integrity Coating-Substrate Bond



Brine Pump Applications

Pad Eye for Tie Down



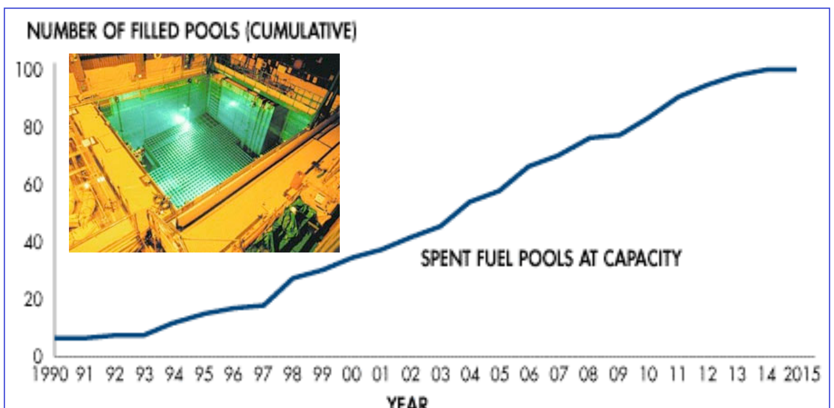
Air Plenum

Surface Ship Landing Ship Dock (LSD) has ~ 300 on deck at a replacement cost of $1000/pad eye.

Shaft with Polished HVOF Amorophous-Metal Coating – TNC

The NanoSteel Company (TNC) has demonstrated that SAM-type coatings can be ground/polished down to 3 micro-inches using diamond impregnated cloth for applications such as replacement for electrolytic hard chrome, or bearing or valve applications. High-quality coatings on cylindrical surfaces demonstrated.

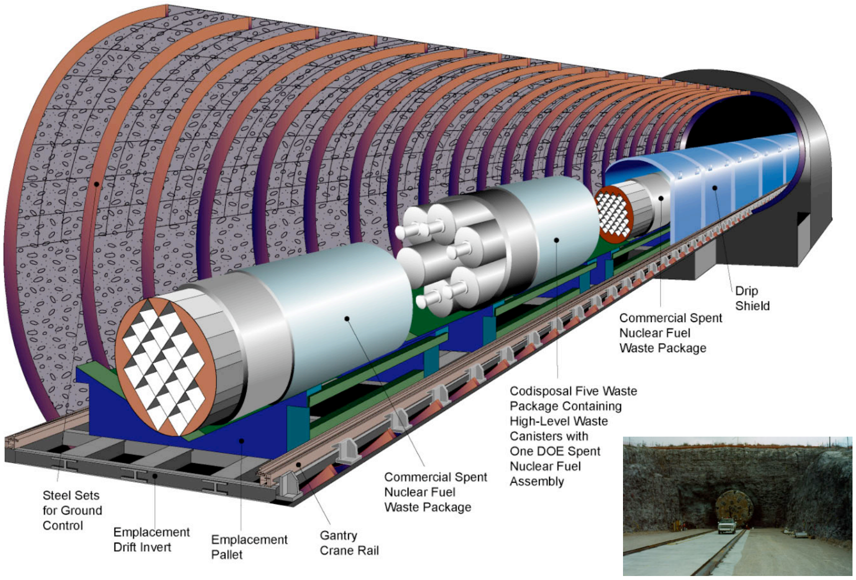
Inventory of Spent Nuclear Fuel in Storage Pools



Note: All operating nuclear power reactors are storing used fuel under NRC license in spent fuel pools. Some operating nuclear reactors are using dry cask storage. Information is based on loss of full-core reserve in the spent fuel pools.

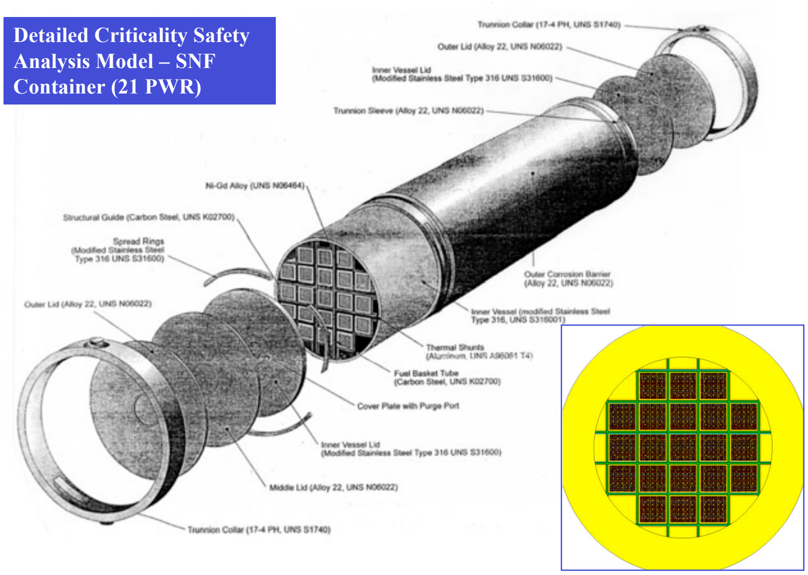
Source: Energy Resources International and DOE/RW-0431 – Revision 1

Underground Repository Needed

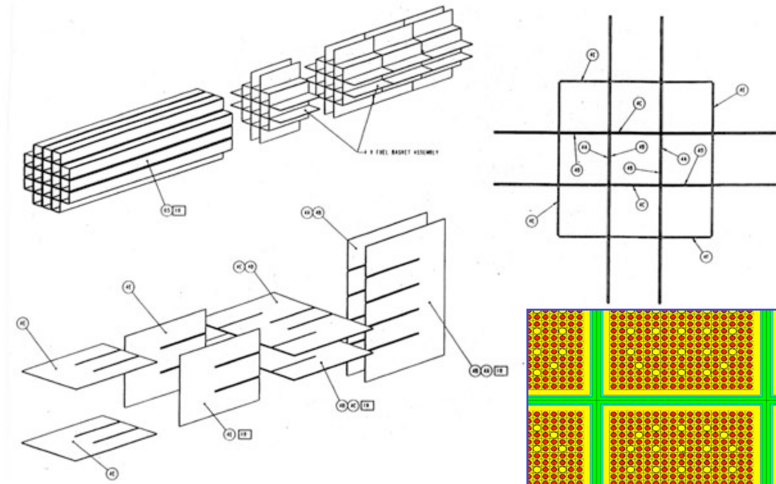


Conceptual SNF Container (21 PWR Assemblies)

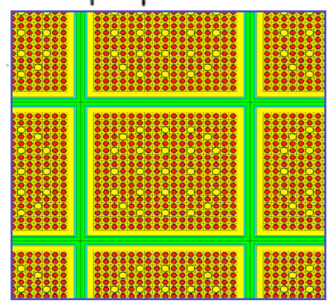
Detailed Criticality Safety Analysis Model – SNF Container (21 PWR)



Conceptual SNF Basket Assembly (21 PWR Assemblies)



Detailed Criticality Safety Analysis Model – Basket Assembly (21 PWR)



Irradiation, Attenuation & Radiography at TRIGA Reactor

Central Irradiation Facility (CIF) [1]
Pneumatic Transfer System (PTS) [2]
Neutron Transmutation Doping (NTD) > 10 Locations

[1] Maximum Value: The active length of TRIGA fuel is 15 inches. Dependent on the control rod elevation, thermal flux could decrease to sixty percent (60%) of 1.5×10^{13} n/cm^2·sec at a distance of 7.5 inches away from the reactor core. The flux/dose information is strongly dependent on water/void volume ratio.

[2] Average Value: Maximum 8.9×10^{12} n/cm^2 at the bottom and minimum 6.0×10^{12} n/cm^2 on the top.

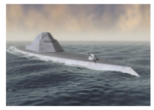
Neutron Irradiation Facility (NIF)

The operating power is 1.5 MW. The usable space is 17 centimeters (7 inches) in diameter and 22 centimeters (9 inches) in length.

$\varphi_{1\text{-MeV eq.}} = 2.3 \times 10^{10}$ n/cm^2·sec $D_{\text{fast neutrons} > 0.1\text{ MeV}}$ (Si) $\cong$ 60 Gy/hr
φ_{thermal} = 1% of $\varphi_{1\text{-MeV eq.}}$ $D_{\text{gamma rays}}$ (Si) $\cong$ 200 Gy/hr

Pulsing

Typical Pulse Reactivity $\cong$ \$1.6 (or \$0.60 prompt reactivity)
Peak Power $\approx$ 400 MW
FWHM $\approx$ 30 milliseconds
Total Energy Release = 14-15 MW·sec

High-Performance Corrosion-Resistant Materials: FY05 Deliverables – Overview & Corrosion

Joe Farmer
Lawrence Livermore National Laboratory
Livermore, California

Time Line for Phase II FY05

- January 10-13, 2005 — Phase I Review in Pearl Harbor – Positive Outcome
- Jan/June 2005 — Parts Obtained from Shipyards Per Phase I Review
- March 25th 2005 — Receipt of Funds at Direct-Funded Institutions
- March/June 2005 — Negotiations for Contract Renewal
- April 12th 2005 — Kaufman – Contract Finalized
- April 22nd 2005 — University of Nevada Reno – Contract Finalized
- April 25th 2005 — Boudreau – Contract Finalized
- April 29th 2005 — University of Wisconsin Madison – Contract Finalized
- May 16th 2005 — Caterpillar – Contract Finalized
- June 7th 2005 — NanoSteel – Contract Finalized
- June/July 2005 — Powder Production & 1st Coating of Sail Cover Plates
- July 12th 2005 — HPCRM Presentation to SUBPAC – Pearl Harbor
- September 7th 2005 — HPCRM Stakeholder's Meeting – Arlington
- September 28th 2005 — HPCRM Presentation to OCRWM – Las Vegas
- November 16th 2005 — HPCRM Presentation to NWTRB – Washington
- Nov/Dec 2005 — Receipt of Samples for Beginning Long-Term Testing
- January 17-20, 2005 — Phase II Review in Key West

Overview of Activities

- The "*Primary Categories of Activities*" within HPCRM are:
  1. Technical Direction;
  2. Powder Atomization & Sizing;
  3. Production of Test Materials;
  4. DOE Prototype Fabrication;
  5. DOE Tunnel Boring & Excavation;
  6. DOD Prototype Fabrication;
  7. Microstructure Characterization;
  8. Thermal Analysis & Process Development;
  9. Corrosion & Mechanical Testing; and
  10. Scientific Foundation

Powder Atomization & Sizing

- The *"Powder Atomization & Sizing"* activity includes:
  1. The atomization of *2000-4000 pounds of SAM2X5*,
  2. The atomization of *2000-4000 pounds of SAM1651*, and
  3. The exploration of *ceramic nanoparticles* that can be dispersed in the amorphous metal for increased hardness and damage tolerance (shear band interruption).
- Deliverables are non-Q and include:
  1. Approx. 2000-4000 pounds of atomized and sized SAM2X5 – completed
  2. Approx. 2000-4000 pounds of atomized and sized SAM1651 – completed
     - *A large portion of this powder will be used by TNC and ORNL (Carpenter) to meet their deliverables, with sufficient powder supplied to other team members, including Caterpillar and UC Davis, so that they can also meet their programmatic deliverables. The balance of unused powder will be shipped to LLNL.*
  3. Synthesis of kilogram quantities of nanometer-sized Y_2O_3 as additive for enhanced damage tolerance – completed

Production of Test Materials

- The "***Production of Test Materials***" activity will yield a large number of samples with thermal-spray coatings of both ***SAM2X5*** and ***SAM1651***:
  - The samples will be prepared to enable a broad range of corrosion testing, microstructure characterization, environmental fracture, and mechanical testing.
  - It is intended to use as much of the current of inventory as possible to produce materials for the Navy applications.
  - Nickel-based ***Alloy C-22 welded and un-welded substrates*** will be used to simulate the waste package surface underlying the integrated drip shield, at the point of contact with the pallet/cradle, and at the final closure weld.
  - Since in these scenarios the waste package will not be completely covered, planar samples will be sprayed on a single side, to simulate the galvanic coupling that may occur between adjacent coated and uncoated surfaces.
  - High-strength ***hy80 steel*** is much less corrosion resistant than Alloy C-22, and will therefore provide a good basis for comparison, revealing areas of substrate attack due to porosity and cracking.

Production of Test Materials

- **Deliverables** are non-Q and include:
  1. Substrate materials for coating with SAM2X5 - **completed**
  2. Rod samples for long-term corrosion potential measurements - **completed**
  3. Lollipop samples for potential-step measurements of the threshold and repassivation potentials - **completed**
  4. Weight-loss samples for determination of corrosion rate - **completed**
  5. Crevice samples for the determining crevice susceptibility - **completed**
  6. Disk samples for galvanic-couple testing – **awaiting samples**
  7. Samples for bond-strength testing; samples for hardness testing; samples for damage tolerance testing – **completed**
  8. Samples with variable thickness coatings with for determination of coating stress from deflection measurement – **awaiting samples**
  9. Witness samples for each milestone – **completed**

DOE Prototype Fabrication

- The ***"DOE Prototype Fabrication"*** activity will yield large-area macroscopically thick coatings for characterization and testing that are deemed to be representative of those that will be deposited on actual waste package surfaces.
- **Deliverables** are non-Q and include:
  1. Large un-welded C-22 plate with HVOF SAM2X5 coating - **completed**
  2. Witness samples from un-welded plate with SAM2X5 coating - **completed**
  3. Large un-welded C-22 plate with HVOF coating of SAM1651 - **completed**
  4. Witness samples from welded plate with SAM1651 coating - **completed**
  5. Large welded C-22 plate with HVOF SAM2X5 coating - **completed**
  6. Witness samples from welded plate with SAM2X5 coating - **completed**
  7. Large welded plate of C-22 with HVOF coating of SAM1651 - **completed**
  8. Witness samples from welded plate with SAM1651 coating – **completed**
- Plates are being used to evaluate (1) heating effects; (2) high-energy impact; (3) hot corrosion by dripping geothermal brines; (4) repair procedures; (5) non-destructive evaluation with radiography & other techniques – **in progress**

DOD Prototype Fabrication

- The ***"DOD Prototype Fabrication"*** activity will yield large-area macroscopically thick coatings for characterization and testing that are deemed to be representative of those that will be deposited on actual naval ships and submarines. These prototypes will be tested under simulated or actual conditions, to provide information the performance of large-scale parts.
- **Deliverables** are non-Q include:
  1. Welded & un-welded plates of (hy80) coated with SAM2X5 – **in progress**
  2. Welded & un-welded plates of (hy80) coated with SAM1651 – **in progress**
  3. Atomized and sized SAM2X5 and SAM1651 powder – **complete**
  4. Cover plates for sail coated with SAM2X5 – **complete**
  5. Cover plates for sail coated with SAM1651 – **requires optimization**
  6. Rotating shaft for Navy coated with SAM2X5 – **in progress**
  7. Rotating shaft for Navy coated with SAM1651 – **requires optimization**
  8. Air-intake plenum for Navy coated with SAM2X5 - **complete**
  9. Air-intake plenum for Navy coated with SAM1651 – **requires optimization**

Tunnel Boring and Excavation (Frank Wong et al.)

- The "*Tunnel Boring and Excavation*" activity will produce prototypical de-vitrified low-carbon SAM40 bits for *Alpine Picks (AP)*, as well as thermally sprayed disc cutters for a *Tunnel Boring Machine (TBM)*. This activity was proposed for FY05 funding. The prototypical bits for the AP will then be tested on a hard-rock cutting machine with serpentine linear tracking, thus simulating impact, abrasion and wear anticipated during the excavation of the repository drifts and alcoves. The prototypical cutting discs for the TBM will be tested on orbital tracks, thereby simulating conditions anticipated during excavation within the repository.
- Deliverables are non-Q and include:
  1. Prototype TBM disc cutters coated with low-carbon SAM40 – complete
  2. Test data for prototypical disc cutters – in progress
  3. Prototypical Alpine Pick bits made from low-carbon SAM40 – complete
  4. Test data for prototypical bits – in progress
  5. Bearings and shafts – under development for Navy brine pumps

Microstructure Characterization

- The *"Microstructure Characterization"* activity will provide detailed insight into the elemental composition, morphology, microstructure, damage tolerance, thermal stability and corrosion resistance of as-sprayed coatings of *SAM2X5* and *SAM1651*. This activity will also be integrated with the *"Production of Test Materials"* activity, so that *Particle Size Optimization (PSO)* can be completed for both formulations. This study will enable those involved in the fabrication of engineered barrier systems for the repository, or involved in the fabrication of naval ships, to produce fully dense, pore-free completely glassy coatings of Fe-based amorphous metals. These attributes are crucial for a high-performance protective coating.
- Deliverables are non-Q and include:
  1. Documented microstructure & thermal properties of SAM2X5 – ongoing
  2. Documented microstructure & thermal properties of SAM1651 – ongoing
  3. Predictions of devitrification & phase formation as a function of time and temperature – initial TTT diagrams complete – ongoing refinement

Corrosion & Mechanical Testing

- The ***"Corrosion & Mechanical Testing"*** activity will provide data for further establish the necessary corrosion resistance and resilience of these thermal spray coatings.
  - ***Potential-step measurements*** with creviced and un-creviced samples will be used to further quantify the threshold and repassivation potentials for SAM2X5 and SAM1651 thermal-spray coatings in relevant environments.
    - *Testing in progress with results presented by LLNL*
  - ***Immersion and salt-fog tests*** will also be conducted.
    - *Testing in progress with initial measurements presented by LLNL*
  - ***Mechanical measurements*** will quantify strength, hardness, toughness and damage-tolerance.
    - *Testing in progress with initial measurements presented by CWRU*
  - ***Galvanic coupling*** between coated and un-coated regions of waste packages with protected welds and integrated drip shields will be simulated with galvanic couples, connected through a standard adder potentiostat, serving as a zero-resistance ammeter.
    - *Awaiting samples from TNC*

Corrosion & Mechanical Testing

- The ***"Corrosion & Mechanical Testing"*** activity will provide data for further establish the necessary corrosion resistance and resilience of these thermal spray coatings.
  - Slow strain rate testing (SSRT) of thermally-sprayed cylindrical dog-bone samples at the CWRU, NRL & LLNL.
    - Fracture in air
    - Susceptibility to stress corrosion cracking (SCC)
    - Susceptibility to hydrogen induced cracking (HIC)
    - Samples Received from TNC
    - *Samples Distributed to Team Members on December 12th 2005*
  - Post-exposure and fracture surfaces will be evaluated at SNL.
    - *Awaiting Samples from Mechanical Testing*

Corrosion & Mechanical Testing

- Deliverables are non-Q and include:
  1. Long-term open circuit corrosion potential
     - Testing initiated & initial measurements presented at KW2006
  2. Corrosion rates of creviced & un-creviced samples during immersion
     - Long-term testing initiated & initial measurements presented at KW2006
  3. Threshold & repassivation potentials for performance assessment model
     - Initial measurements of threshold potential made with long-duration potential-step tests in uncreviced condition & presented at KW2006
     - SAM2X5-coated lollipop (MCA) samples recently received from TNC – shakedown testing initiated at LLNL
  4. Corrosion rates in areas where galvanic coupling may occur, such as the transition between thermal-spray coatings and substrates
     - Test equipment ready & awaiting samples from TNC
  5. Corrosion rates of samples during salt-fog exposure
     - Samples recently received; continue to rely on FY04 results

Availability of SAM2X5 Samples for Testing

Deliverable	Description	Original Date in Contract	Requested Date by TNC	Shipping Date by TNC	Actual Date of Deliverable	Quantity Samples	Witnesses
1	Report on the Technical Merits of SAM2X5	6-Jul-05	Done		Received		
2	HVOF Material of SAM2X5	10-Jun-05	Done	22-Jun-05	Received	Ship Balance to LLNL	
3	Document Receipt of Substrates for Coating	8-Jul-05	Done	28-Jul-05	Received		
4	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified	
5	Coated Rod Samples SAM2X5	22-Jul-05	12-Sep-05	30-Sep-05	5-Oct-05	11	4
6	Coated Corrosion Disk Samples SAM2X5	17-Jun-05	2-Sep-05	None	Waiting	0	0
7	Coated Lollipop Samples SAM2X5	12-Aug-05	7-Sep-05	15-Sep-05	29-Sep-05	22	4
8	Coated Weight Loss Samples SAM2X5	24-Jun-05	16-Sep-05	22-Sep-05	4-Oct-05	22	4
9	Coated Crevice Corrosion Samples SAM2X5	29-Jul-05	22-Sep-05	2-Nov-05	8-Nov-05	22	4
10	Coated Crevice Corrosion Samples SAM2X5	5-Aug-05	16-Sep-05	21-Nov-05	Received	14	4
11	Cyndrical Dog Bone Samples SAM2X5	19-Aug-05	30-Sep-05	8-Nov-05	Received	28	4
12	Cyndrical Dog Bone Samples SAM2X5	26-Aug-05	30-Sep-05	8-Nov-05	Received	6	4
13	Report on Hardness SAM2X5	8-Jul-05	14-Oct-05	None	Waiting	Report Delivered	
14	Tested Bond Strength Samples SAM2X5	11-Jul-05	14-Oct-05	1-Dec-05	Received	6	4
15	Damage Tolerance Samples SAM2X5	15-Jul-05	21-Oct-05	22-Nov-05	23-Nov-05	6	4
16	Variable Coating Thickness Samples SAM2X5	2-Sep-05	28-Oct-05	None	Waiting	0	0
17	Protected Weld Plate Samples SAM2X5	30-Sep-05	11-Nov-05	None	Waiting	TNC Request for Help	
18	Unwelded Plate Samples SAM2X5	14-Oct-05	14-Nov-05	15-Dec-05	15-Dec-05	6	4
19	Coated Samples SAM2X5 to NRL	16-Sep-05	18-Nov-05	None	Waiting	NRL Material Needed	
20	Annual Report	16-Sep-05	18-Nov-05	20-Dec-05	20-Dec-05	Report Delivered	

High-Performance Corrosion-Resistant Materials: FY05 Deliverables – Sandia

Nancy Yang
Sandia National Laboratory
Livermore, California

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FY05 Deliverables – SNL – Characterization

- All the deliverables were completed and the results will be presented in the Key West meeting, Florida, 2006
- Established metallurgical characteristics of feedstock powders, HVOF coatings and their implication to corrosion performaceof SAM formulations developed for HPCRM program
- Identified microstructure features in the coatings affectecting corrosion performance of HVOF coatings
- Established correlation between physical property of feedstock powder and coating morphology that determine corrosion performance of the HVOF coatings
- Provide microstructure characterization supports for all HPCRM team activities

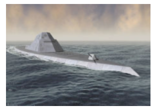
FY05 Deliverables – UCD – Optimization

- Improve Powder Atomization
  - Suitable powder morphology (improved feeding and flight behavior)
  - Narrow particle size distribution (homogeneous and uniform splatting)
  - Maintain amorphous structure (low critical cooling rate)

- HVOF Spray Conditions
  - Control surface and substrate temperature during spray (management of residual stress).
  - Optimize flame and spray conditions for minimal oxidation.
  - Maintain appropriate temperature and velocities for minimum porosity.
  - Minimize the devitrificaion process (CCR and temperature during spray).

FY05 Deliverables – UNR – Nanoparticle Additives

- The tasks in this activity called for the preparation of significant quantities of Y_2O_3 nanoparticles and collaboration with SNL and UC Davis in introducing this nanoparticles into HVOF coatings of the SAM 1651 formulation.
- A 100-g batch of powders treated at 1000°C for two hours was delivered to SNL and a 2-kg batch of powders of the same type as the ones for SNL was delivered to UC Davis.

High-Performance Corrosion-Resistant Materials: FY05 Deliverables – Case Western

John Lewandowski
Case Western Reserve University
Cleveland, Ohio

FY05 Deliverables – CWRU – Mechanical Properties

Item	Status	Value to Team
Initial Hot Hardness Testing - In Vacuum		
Hardness vs Test Temperature	SAM 1651 – Complete	Mechanical Behavior
	Type 316L SS – Complete	Fundamentals
	SAM 2X5 – Ongoing	Link to Processing Studies
	Alloy C-22 – Ongoing	
Hardness Evolution at Fixed Temperature	SAM 1651 – Complete	Mechanical Behavior
(562°C, 575°C, 600°C, 620°C)	SAM 2X5 - Ongoing	Fundamentals
		Link to Processing
		Link to Microstructure Evolution
Notch Toughness Tests	SAM 1651 – Complete	Fracture Fundamentals
	SAM 2X5 – Ongoing	Damage Tolerance
		Effects of Crystallization

FY05 Deliverables – CWRU – Mechanical Properties

Item	Start Date	Finish Date
Hot Hardness Testing - In Vacuum	1/23/06	2/17/06
Slow Strain Rate Testing of Cylindrical	2/20/06	4/14/06
Dog Bone Samples in Air		
Creep Test Under Constant Stress - in Air	4/17/06	6/09/06
Split Hopkinson Bar Test	6/12/06	7/7/06
Data Analysis & Interpretation	8/07/06	9/01/06
Technical Project Review & Report Lab	9/04/06	9/29/06
Scale Test of SAM Coatings		

High-Performance Corrosion-Resistant Materials: FY05 Deliverables – NRL

Bob Brown
Center for Corrosion Science and Engineering
Naval Research Laboratory
Key West, Florida

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FY05 Deliverables – NRL – Submarine Application

- Milestone – Locate Shipboard Parts
  - Located available decommissioned submarines in Norfolk, Virginia and Puget Sound, Washington.
  - Joe Farmer and Bob Brown inspected submarines and identified components for testing.
  - Submitted MIPR's to Puget Sound and Norfolk Naval Shipyards
    - For removal and shipping of:
      - Twenty eight sail cover plates from the 702 (Phoenix) and 693 (Cincinnati).
      - Two Brine Pumps (1.6K & 10K GPD) from the 707 (Portsmouth).
  - Coordinated shipping of plates and pumps received by Caterpillar and NRL.
  - Conducted inventory of sail cover plate materials at Caterpillar.
- Milestone – Determine Parts Engineering Conformance
  - Conducted Engineering Review:
  - Utilized Advanced Technical Information System (ATIS) at Electric Boat, to locate, review and compare active and decommissioned boats drawing for sail cover plates and brine pumps interchangeability.
    - Active Boats and home ports being evaluated for of fit, form and function:
      - Pearl Harbor, Hi.
      - San Diego, Ca.
      - Kings Bay, Ga.

FY05 Deliverables – NRL – Submarine Application

- Milestone – Manage Installation Process
  - Developed Plan of Action & Milestones (POA&M)
  - Provided NAVSEA 07T and NAVSEA 05M with Plan for Qualification of Coating.
  - Sail cover plates to be a Departure From Specification (DFS).
  - Brine pump will be DFS.
  - Met with Pearl Harbor contacts:
    - COMSUBPAC/Type Commander.
    - Pearl Harbor Naval Shipyard (PHNSY).
    - Pearl Harbor Regional Maintenance Center (PHRMC) Business Agent-
      - Establishing MOA between NRL and PHRMC.
    - Squadron 1 Engineering Group.
  - Coordinating with CARVER for the rebuild of both brine pumps incorporating coated shafts.
  - Both pumps have been torn down and the shafts extracted.
  - Located Active Submarine and coordinating available installation.

FY05 Deliverables – NRL – Quality Assurance

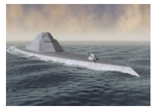
- Milestone – Ensure CATAPILLAR Parts Meet Performance Criteria for Large Scale Application.
  - Flash Rust Revealed After Blasting.
  - Potential Chloride Embedment.
  - Surface Profile
  - Thermal Spray Parameters are Consistent
  - Powder Size and quality

FY05 Deliverables – NRL – Quality Assurance

NRL Quality Assurance at Caterpillar (9/20/05)

Determining Surface Profile

Bresle Patch For Chloride Inspection

High-Performance Corrosion-Resistant Materials: FY05 Deliverables – NRL

Bob Bayles
Naval Research Laboratory
Washington, DC

FY05 Deliverables – NRL – Naval Applications (1)

TASK	SAM 2X5	SAM 1651
1. Application Testing of SAM2X5 & SAM1651 for Sail		
Obtain 4-6 Spare Cover Plates for Sail	COMPLETED	Specimen not received
Coordinate Coating of Inner Surface of Cover Plate (SAM2X5 & SAM1651)	COMPLETED	Specimen not received
Coordinate Coating of Samples Attached with Bolts (SAM2X5 & SAM1651)	COMPLETED	Specimen not received
2. Application Testing of SAM2X5 & SAM1651 for Shafts & Bearings		
Obtain 1-2 Spare Shafts & Bearings	COMPLETED	Specimen not received
Coordinate Coating & Finishing of Shafts & Bearings	In Process	Specimen not received
Monitor Testing & Quantify Results		Specimen not received
3. Laboratory Testing of SAM2X5 & SAM1651 in Ambient Seawater		
Cathodic De-Bonding Testing	COMPLETED	Specimen not received
Slow Strain Rate Testing (SSRT) – Threshold Stress for Stress Corrosion Cracking	Specimens Received December 12th 2005	Specimen not received
Slow Strain Rate Testing (SSRT) – Threshold Stress for Hydrogen Induced Cracking	Specimens Received December 12th 2005	Specimen not received

FY05 Deliverables – NRL – Naval Applications (2)

TASK	SAM 2X5	SAM 1651
1. Data from Sail Application Test – Ambient Seawater	In Test	Specimen not received
2. Data from Shaft & Bearing Application Test – Ambient Seawater	Test Pending	Specimen not received
3. Data from Key West Immersion Test – Ambient Seawater	In Test	Specimen not received
4. Data from Cavitation Test – Ambient Seawater	Specimen not received	Specimen not received
5. Data from Cathodic De-Bonding Test – Ambient Seawater	In Test	Specimen not received
6. Data from SSRT Stress Corrosion Cracking (SCC) Test – Ambient Seawater	Specimen not received	Specimen not received
7. Data from SSRT Hydrogen Induced Cracking (HIC) Test – Ambient Seawater	Specimen not received	Specimen not received
8. Data from Fracture Toughness Test	Specimen not received	Specimen not received
9. Data from B117 Salt Fog Test	Test Start Pending Successful Cyclic Polarization Results	Specimen not received

FY05 Deliverables – NRL – Naval Applications (3)

TASK	SAM 2X5	SAM 1651
10. Data from KISCC & KIHIC Tests – Yucca Mountain Brines		
a. SAM2X5 Ingot	Specimen not received	Specimen not received
b. SAM2X5/C-22	Specimen not received	Specimen not received
c. SAM2X5/316L	Specimen not received	Specimen not received
d. SAM1651 Ingot	Specimen not received	Specimen not received
e. SAM1651/C-22	Specimen not received	Specimen not received
f. SAM1651/316L	Specimen not received	Specimen not received
11. Final Report	COMPLETED	COMPLETED
a. Description of Specific Navy Applications	Pending receipt of all specimens and completion of all tests	Pending receipt of all specimens and completion of all tests
b. Compilation of All Results for Application Testing	Pending receipt of all specimens and completion of all tests	Pending receipt of all specimens and completion of all tests
c. Documentation of Anticipated Benefits	Pending receipt of all specimens and completion of all tests	Pending receipt of all specimens and completion of all tests

FY05 Deliverables – NRL – Actual Work Performed (4)

TASK	SAM 2X5	SAM 1651
B117 Salt Fog: 4-inch by 6-inch by 1/8-inch (Scribed and unscribed)	Test start pending successful cyclic polarization results	Specimen not received
Long-Term Exterior: 4-inch by 6-inch by 1/8-inch (Scribed and unscribed)	In Process	Specimen not received
Alternate Immersion: 6-inch by 12-inch by 1/8-inch	In Process	Specimen not received
Bend-Over-Mandrel: 4-inch by 6-inch by 22 gauge	Specimen not received	Specimen not received
Cathodic Disbondment: 6-inch by 12-inch by 1/8-inch	In Process	Specimen not received
Galvanic Corrosion: 2-inch by 2-inch by 1/8-inch	Test start pending successful cyclic polarization results	Specimen not received
Long Term Crevice Corrosion: 2-inch by 2-inch by 1/8-inch	In Process	Specimen not received
Accelerated Crevice Corrosion	Completed on all samples received	Specimen not received
Edge Retention	Completed on all samples received	Specimen not received

High-Performance Corrosion-Resistant Materials: FY05 Deliverables – BLE

Jay Boudreau
BLE
Los Alamos, New Mexico

FY05 Milestones – BLE – Management Support

- Co-Chair Weekly HPCRM Team Meetings Conducted via Teleconference
- Co-Chair Resultant Side Bar Teleconferences

FY05 Deliverables – BLE – Management Support

- Co-Chair of HPCRM Teleconference & Videoconference Meetings
- Written Documentation of All Teleconferences
- Concise Streamlined Requirements Document for DOE Applications
- Documentation of Successful FY04 Salt Fog Testing & Sample Analysis
- Formal Document Review with Written Comments
- Comment Resolution Satisfactory to Reviewer & Authors

Contribution to the HPCRM Project

- **Helped the HPCRM Team Leader maintain continuity and frequent interaction amongst Team Members**
- **Identified "Action Items" for each weekly teleconference and followed up with Team members to ensure closure**
- **Provided thorough, clear, and accurate documentation of each meeting and distributed minutes to all appropriate HPCRM Team members to enable all participants to focus on evolving project goals**
- **Provided formal documentation for "Salt Fog" specimens and for DOE Requirements**

Backup Slides

High-Performance Corrosion-Resistant Materials: Originally Proposed FY05 SOW – TNC

Dan Branagan
The NanoSteel Company
Idaho Falls, Idaho

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Originally Proposed FY05 SOW – TNC (1)

- Non-Q Fractionated SAM2X5 Powder (2000-4000 lb): Produced Powder
  - Fines (< 15 microns) – Not Fractionated in FY05
  - Small (15-30 microns) – Not Fractionated in FY05
  - HVOF Cut (30-50 microns) – Not Fractionated in FY05
  - Large (> 50 microns) – Not Fractionated in FY05
- *Non-Q Fractionated Powder from Atomization of SAM1651 (Not Produced)*
  - *Fines (< 15 microns)*
  - *Small (15-30 microns)*
  - *HVOF Cut (30-50 microns)*
  - *Large (> 50 microns)*
- Non-Q Substrate Materials for Coating & Baseline Testing: Completed
  - Nickel-Based Alloy C-22 Rods & Plates (Only Combination Produced)
  - *Stainless Steel Type 316L Rods & Plates (Not Produced)*
  - *High Strength Steel hy80 Rods & Plates (Not Produced)*

Originally Proposed FY05 SOW – TNC (2)

- **Non-Q Rod Samples : Completed**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*
- **Non-Q Lollipop Samples : Completed**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*

Originally Proposed FY05 SOW – TNC (3)

- **Non-Q Weight Loss Samples**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*
- **Non-Q Crevice Corrosion Samples**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*

Originally Proposed FY05 SOW – TNC (4)

- **Non-Q Traceable Samples for Instrumented Crevice Corrosion: Completed**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*

Originally Proposed FY05 SOW – TNC (5)

- **Cylindrical Dog-Bone Samples for SSRT – SCC Susceptibility: Completed**
  - **SAM2X5/C-22**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - **SAM2X5/hy80**
  - *SAM1651/hy80 (Not Produced)*
- **Cylindrical Dog-Bone Samples for SSRT – Modulus & Strength: Completed**
  - **SAM2X5/C-22**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - **SAM2X5/hy80**
  - *SAM1651/hy80 (Not Produced)*

Originally Proposed FY05 SOW – TNC (6)

- **Samples for Bond Strength Measurements: Completed**
  - **SAM2X5/C-22: (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced)*
  - *SAM1651/hy80 (Not Produced)*
- **Samples for Hardness Measurements: Completed**
  - **SAM2X5/C-22: (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced)*
  - *SAM1651/hy80 (Not Produced)*

Originally Proposed FY05 SOW – TNC (7)

- **Coatings of Variable Thickness on Deflection Samples: Completed**
  - **SAM2X5/C-22**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced)*
  - *SAM1651/hy80 (Not Produced)*
- **Large-Scale "Protected-Weld" Prototypical Plates for DOE: Completed**
  - **Welded Plate of Alloy C-22 with SAM2X5 Coating**
  - *Welded Plate of Alloy C-22 with SAM1651 Coating (Not Produced)*
- **Large-Scale "Integrated Drip Shield" Prototypical Plates for DOE: Completed**
  - **Plate of Alloy C-22 Base Metal with SAM2X5 Coating**
  - *Plate of Alloy C-22 Base Metal with SAM1651 Coating (Not Produced)*

Originally Proposed FY05 SOW – TNC (8)

- **Produce Large-Scale "Pallet" Prototypical Plates for DOE: Completed**
  - **Plate of hy80 Weld/Base Metal with SAM2X5 Coating**
  - ***Plate of hy80 Weld/Base Metal with SAM1651 Coating (Not Produced)***

High-Performance Corrosion-Resistant Materials: Original FY05 Contractual Deliverables – TNC

Dan Branagan
The NanoSteel Company
Idaho Falls, Idaho

Original FY05 Contractractual Deliverables (#1 & #2) - TNC

- Task # 1
  - Prepare Technical Document on Technical Merits of SAM2X5 Formulation.
- Deliverable # 1 – Delivered
  - Technical Document on Merits of SAM2X5 Formulation. This document may be used on behalf of HPCRM Team as the basis for an RD100 Award submittal.
- Task # 2
  - Production of SAM2X5 HVOF Powder. Atomization of up to 4000 pounds of SAM2X5 alloy, with necessary sieving and air classification, to deliver necessary SAM2X5 powder for estimated NanoSteel and Caterpillar needs. The powder for NanoSteel and Caterpillar will be a nominal "HVOF Cut" with particle sizes in the 15 to 53 micron range.
- Deliverable # 2 – Final Shipment to Caterpillar on June 22, 2005 – Balance Due LLNL
  - Produce sufficient HVOF material of SAM2X5 composition (verified by Sandia National Laboratory) to spray all coupons in subsequent Tasks and demonstration plates being prepared by Caterpillar Corporation. Deliver HVOF powder of SAM2X5 from stock to Caterpillar Corporation as needed. Deliver balance of HVOF powder of SAM2X5 (powder not used in preparation of HPCRM samples by NanoSteel and Caterpillar) to the prime contractor (LLNL).

Original FY05 Contractractual Deliverables (#3) - TNC

- Task # 3
  - Procure/Obtain/Prepare Substrates for Coating
  - Nickel-Based Alloy C-22 Rods, Discs, Lollipops & Plates
- Deliverable # 3 – July 28, 2005
  - Procure substrates in sufficient quantity to perform spray studies listed in subsequent Tasks and forward documentation from the manufacturers to LLNL.
  - For Tasks #5 through #18, the SAM2X5 coating will be sprayed using the optimized parameters developed during Phase 1 of FY05.
- Task #4
  - Unspecified/Reserved/Contract Negotiation
- Deliverable # 4 – Eliminated During Contract Revision
  - Unspecified/Reserved/Contract Negotiation

Original FY05 Contractractual Deliverables (#5) - TNC

- Task # 5
  - HVOF Coating of Rod Samples with High-Density Low-Permeability SAM2X5
    - Rod Samples for Corrosion Potential Measurements
      - Geometry:
        - » Diameter = 3/4 inch; Length = 8 inches
      - Coating Details:
        - » Cylindrical surface & hemispherical ends of rods will be coated
        - » 11 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 Coatings onto C-22 substrates at 0.030-inch thick
- Deliverable # 5 – Shipped September 30, 2005
  - Deliver coated Rod Samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#6) - TNC

- Task # 6
  - HVOF Coating of Electrochemical Disk Samples with High-Density Low-Permeability SAM2X5
    - Electrochemical Disk Samples for Galvanic Coupling Test
      - Geometry:
        - » Diameter = 5/8 inch; Thickness = 1/8 inches
      - Coating Details:
        - » The front surfaces of electrochemical disk samples will be coated
        - » 22 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick
- Deliverable # 6 – Not Delivered
  - Deliver coated corrosion disk samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#7) - TNC

- Task # 7
  - HVOF Coating of Lollipop Samples for Crevice Corrosion Testing with High-Density Low-Permeability SAM2X5
    - Flat Electrochemical Lollipop Sample for Crevice Testing
      - Geometry:
        - » Diameter = 0.787 inches; Thickness = 0.125 inches; Hole = 0.276 inches; Stem Length = 8 inches; Stem Width = 0.197 inches
      - Coating Details:
        - » Single side of standard flat lollipop samples will be coated
        - » 22 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 7 – Shipped September 15, 2005
  - Deliver coated lollipop samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#8) - TNC

- Task # 8
  - HVOF Coating of Weight Loss Samples with High-Density Low-Permeability SAM2X5
    - Flat Weight Loss Samples
      - Geometry:
        - » Width = 4 inches; Length = 4 inches; Thickness = 1/4 inch
      - Coating Details:
        - » The front surfaces of standard weight loss samples will be coated
        - » 22 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 8 – Shipped September 22, 2005
  - Deliver coated weight loss samples and witness samples to LLNL.

Original FY05 Contractractual Deliverables (#9) - TNC

- Task # 9
  - HVOF Coating of Crevice Corrosion Samples with High-Density Low-Permeability SAM2X5
    - Flat Crevice Corrosion Samples
      - Geometry:
        - » Width = 4 inches; Length = 4 inches; Thickness = 1/4 inch; Center Hole for Crevice Former Bolt = 5/16 inch
      - Coating Details:
        - » Front surfaces of standard weight loss samples will be coated
        - » 22 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 9 – Shipped November 2, 2005
  - Deliver coated crevice resistance samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#10) - TNC

- Task # 10
  - HVOF Coating of Samples for Instrumented Crevice Corrosion with High-Density Low-Permeability SAM2X5
    - Flat Instrumented Crevice-Corrosion Cell Samples
      - Geometry:
        - » Width = 4 inches; Length = 4 inches; Thickness = 1/4 inch
      - Coating Details:
        - » Front surfaces of standard weight loss samples will be coated
        - » 14 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 10 – Shipped November 2, 2005
  - Deliver coated flat samples for instrumented crevice corrosion cell and witness samples to LLNL

Original FY05 Contractractual Deliverables (#11) - TNC

- Task # 11
  - HVOF Coating of Slow Strain Rate Testing (SSRT) Samples for Stress Corrosion Cracking (SCC) & Hydrogen Induced Cracking (HIC) Susceptibility Studies with High-Density Low-Permeability SAM2X5
    - Cylindrical Dog-Bone Samples for SSRT – SCC & HIC Susceptibility
      - Fabricate Per ASTM E8 Procedure
      - Gauge Length:
        - » Dia. = ½ inch; Length = 2 inches
      - Terminals:
        - » Dia. = ¾ inch, Length = 5-1/2 inches, Threads = ¾ inch 10 UNC
      - Coating Details:
        - » Cylindrical outer surface of gauge length & transition will be coated
        - » 22 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
        - » 6 pieces: SAM2X5 coating on hy80 substrate at ~ 0.030-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick
- Deliverable # 11 – Shipped November 8, 2005
  - Deliver cylindrical dog bone samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#12) - TNC

- Task # 12
  - HVOF Coating of Slow Strain Rate Testing (SSRT) Samples for Mechanical Property & Fracture Mechanics Studies with High-Density Low-Permeability SAM2X5
    - Cylindrical Dog-Bone Samples for SSRT – Mechanical Properties
      - Fabricate Per ASTM E8 Procedure
      - Gauge Length:
        - » Dia. = ½ inch; Length = 2 inches
      - Terminals:
        - » Dia. = ¾ inch, Length = 5 ½ inches, Threads = ¾ inch 10 UNC
      - Coating Details:
        - » Cylindrical outer surface of gauge length & transition will be coated 3 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
        - » 3 pieces: SAM2X5 coating on hy80 substrate at ~ 0.030-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick
- Deliverable # 12 – Shipped November 8, 2005
  - Deliver cylindrical dog bone samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#13) - TNC

- Task# 13
  - HVOF Coating of Bond-Strength Specimens with High-Density Low-Permeability SAM2X5
    - Samples for Bond Strength Measurements
      - Geometry:
        - » Width = 1 inches; Length = 1 inches; Thickness = 1 inch; Threaded Hole for Tensile Testing Bolt = 3/8 inch
      - One flat end of cylindrical specimen will be coated as follows:
        - » 3 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
        - » 3 pieces: SAM2X5 coating on hy80 substrate at ~ 0.030-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 13 – Not Delivered
  - Measure Bond Strength Per ASTM Standard Procedure C633. Tally bond strength results and supply report. Deliver tested bond strength samples and witness samples to LLNL. Prepare article for possible publication, with submission to appropriate journal or conference.

Original FY05 Contractractual Deliverables (#14) - TNC

- Task # 14
  - HVOF Coating of Standard Hardness Specimens with High-Density Low-Permeability SAM2X5
    - Samples for Hardness Measurements
      - Geometry:
        - » Width = 4 inches; Length = 4 inches; Thickness = 1 inch
      - One flat side of the plates will be coated as follows:
        - » 3 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
        - » 3 pieces: SAM2X5 coating on hy80 substrate at ~ 0.030-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 14 – Shipped December 1, 2005
  - Metallographically mount sample cut from each coupon. Measure hardness at both 300 gram and 100 gram loads (5 measurements of each). Measure porosity of samples. Write-up and send in report on hardness and porosity for each sample. Deliver the remaining unmounted hardness samples and witness samples to LLNL.

Original FY05 Contractractual Deliverables (#15) - TNC

- Task # 15
  - HVOF Coating of Damage Tolerance Specimens with High-Density Low-Permeability HVOF Coating of SAM2X5
    - Samples for Damage Tolerance Testing
      - Geometry:
        - » Width = 4 inches; Length = 4 inches; Thickness = 1 inch
      - One flat side of the plates will be coated as follows:
        - » 3 pieces: SAM2X5 coating on C-22 substrate at ~ 0.040-inch thick
        - » 3 pieces: SAM2X5 coating on hy80 substrate at ~ 0.030-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 15 – Shipped November 22, 2005
  - Deliver damage tolerance samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#16) - TNC

- Task # 16
  - HVOF Coating of Specimens with Variable-Thickness for Determination of Coating Stress with High-Density Low-Permeability SAM2X5
    - Samples with Variable Thickness Coating for Stress Measurement
      - Geometry:
        - » Width = 2 inches; Length = 4 inches; Thickness = 1/8 inch
      - One side of substrate will be coated as follows:
        - » SAM2X5 coatings onto C-22: 30 mil, 1 mm, 3.5 mm, 5 mm & 7.5 mm thickness (two samples each)
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 16 – Not Delivered
  - Deliver variable coating thickness samples and witness samples to LLNL. Since alterations in parameter development may be necessary, coating quality may degrade with increasing thickness.

Original FY05 Contractractual Deliverables (#17) - TNC

- Task # 17
  - HVOF Coating of "Protected-Weld" Prototypical Plate with High-Density Low-Permeability SAM2X5
    - Large-Scale "Protected-Weld" Prototypical Plates for DOE
      - Geometry:
        - » Width = 12 inches; Length = 12 inches; Thickness = 1 inch
      - Coat one side of each prototypical welded plate as follows:
        - » 6 SAM2X5 HVOF coatings sprayed onto a welded plate of Alloy C-22 at 0.04" thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 17 – Not Delivered – Request for Assistance with Weld from LLNL
  - Deliver plate samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#18) - TNC

- Task # 18
  - HVOF Coating of "Un-Welded" Prototypical Plates for "Integrated Drip Shield" Concept with High-Density Low-Permeability SAM2X5
    - Produce Large-Scale "Integrated Drip Shield" Prototypical Plates for DOE
      - Geometry:
        - » Width = 12 inches; Length = 12 inches; Thickness = 1 inch
      - Coat one side of each prototypical un-welded plate as follows:
        - » 6 SAM2X5 HVOF coatings sprayed onto a welded Plate of Alloy C-22 at 0.04-inch thick
    - Witness Samples:
      - Standard Alloy C-22 Weight Loss Samples:
        - » 4-inch × 4-inch × 1/8-inch thick
      - Coating Details:
        - » 4 SAM2X5 coatings onto C-22 substrates at 0.030-inch thick

- Deliverable # 18 – Shipped December 15, 2005
  - Deliver plate samples and witness samples to LLNL

Original FY05 Contractractual Deliverables (#19) - TNC

- Task # 19
  - HVOF Coating of Navy Samples with High-Density Low-Permeability HVOF Coating of SAM2X5 (Coating will Approach Full-Density & Pore-Free Microstructure to Extent Possible):
    - (1) Salt Fog Testing; (2) Long-Term Exterior Exposure; (3) Alternate Immersion; (4) Bend-Over-Mandrel Test; (5) Cathodic Disbondment; (6) Galvanic Corrosion; (7) Crevice Corrosion.
  - Supply of Substrates:
    - All substrates (hy80) will be supplied to NanoSteel Company by the Naval Research Laboratory without cost (no cost). Operating parameters used during the thermal spray process will be documented as shown in Figure 3 of MIL-STD-1687A (Thermal Spray Processes for Naval Ship Machinaery Applications).
    - Number of Samples: The number samples to be produced in each sample geometry category will be three (3) so that material performance can be established with replicates.
    - Coating Details: The front surfaces of standard weight loss samples will be coated. SAM2X5 coating on each hy80 sample geometry at ~ 0.030-inches thick.
    - Witness Samples: Standard Alloy C-22 Weight Loss Samples: 4-inch × 4-inch × 1/8-inch thick. 4 SAM2X5 coatings onto hy80 substrates at 0.030" thick.

- Deliverable # 19 – Not Delivered – Waiting for NRL to Send Substrates
  - Deliver coated samples and witness samples to NRL

Original FY05 Contractractual Deliverables (#20) - TNC

- Task # 20
  - Final Report Summary

- Deliverable # 20
  - Final Report

Availability of SAM2X5 Samples for Testing

Deliverable	Description	Original Date in Contract	Requested Date by TNC	Shipping Date by TNC	Actual Date of Deliverable	Quantity Samples	Witnesses
1	Report on the Technical Merits of SAM2X5	6-Jul-05	Done		Received		
2	HVOF Material of SAM2X5	10-Jun-05	Done	22-Jun-05	Received	Ship Balance to LLNL	
3	Document Receipt of Substrates for Coating	8-Jul-05	Done	28-Jul-05	Received		
4	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified	Unspecified	
5	Coated Rod Samples SAM2X5	22-Jul-05	12-Sep-05	30-Sep-05	5-Oct-05	11	4
6	Coated Corrosion Disk Samples SAM2X5	17-Jun-05	2-Sep-05	None	Waiting	0	0
7	Coated Lollipop Samples SAM2X5	12-Aug-05	7-Sep-05	15-Sep-05	29-Sep-05	22	4
8	Coated Weight Loss Samples SAM2X5	24-Jun-05	16-Sep-05	22-Sep-05	4-Oct-05	22	4
9	Coated Crevice Corrosion Samples SAM2X5	29-Jul-05	22-Sep-05	2-Nov-05	8-Nov-05	22	4
10	Coated Crevice Corrosion Samples SAM2X5	5-Aug-05	16-Sep-05	21-Nov-05	Received	14	4
11	Cyndrical Dog Bone Samples SAM2X5	19-Aug-05	30-Sep-05	8-Nov-05	Received	28	4
12	Cyndrical Dog Bone Samples SAM2X5	26-Aug-05	30-Sep-05	8-Nov-05	Received	6	4
13	Report on Hardness SAM2X5	8-Jul-05	14-Oct-05	None	Waiting	Report Delivered	
14	Tested Bond Strength Samples SAM2X5	11-Jul-05	14-Oct-05	1-Dec-05	Received	6	4
15	Damage Tolerance Samples SAM2X5	15-Jul-05	21-Oct-05	22-Nov-05	23-Nov-05	6	4
16	Variable Coating Thickness Samples SAM2X5	2-Sep-05	28-Oct-05	None	Waiting	0	0
17	Protected Weld Plate Samples SAM2X5	30-Sep-05	11-Nov-05	None	Waiting	TNC Request for Help	
18	Unwelded Plate Samples SAM2X5	14-Oct-05	14-Nov-05	15-Dec-05	15-Dec-05	6	4
19	Coated Samples SAM2X5 to NRL	16-Sep-05	18-Nov-05	None	Waiting	NRL Material Needed	
20	Annual Report	16-Sep-05	18-Nov-05	20-Dec-05	20-Dec-05	Report Delivered	

High-Performance Corrosion-Resistant Materials: Originally Proposed FY05 SOW – Caterpillar

M. Brad Beardsley
Technology & Solutions Division – Caterpillar Inc.
Peoria, Illinois

FY05 Deliverables – Caterpillar – Prototypical Coatings

- Full-Scale HVOF-Coated Parts for Navy: Completed
  - Cover Plates for Sail with HVOF Coating of SAM2X5
  - *Cover Plates for Sail with HVOF Coating of SAM1651 (Not Produced)*
  - Rotating Shaft with Polished HVOF Coating of SAM2X5
  - *Rotating Shaft with Polished HVOF Coating of SAM1651(Not Produced)*
  - Air-Intake Plenum with HVOF Coating of SAM2X5
  - *Air-Intake Plenum with HVOF Coating of SAM1651 (Not Produced)*
- Large-Scale "Protected-Weld" Prototypical Plates for DOE: Completed
  - Welded Plate of Alloy C-22 with SAM2X5 HVOF Coating
  - Welded Plate of Alloy C-22 with SAM1651 HVOF Coating
- Large-Scale "Integrated Drip Shield" Prototypical Plates for DOE: Completed
  - Plate of Alloy C-22 Base Metal with SAM2X5 HVOF Coating
  - Plate of Alloy C-22 Base Metal with SAM1651 HVOF Coating
- Report Documenting Work: Completed
  - Navy & DOE




High-Performance Corrosion-Resistant Materials: Originally Proposed FY05 SOW – Wisconsin

John Perepezko
University of Wisconsin – Madison
Madison, Wisconsin

Originally Proposed FY05 SOW – Wisconsin

- **Documented Microstructure & Thermal Properties: <u>Completed</u>**
- **Documented Kinetic Model for Phase Stability: <u>Completed</u>**
- **Determination of Diffusivities & Rate Constants: <u>Completed</u>**
- **Predictions of Phases Formed at Various Times & Temperatures: <u>Completed</u>**




High-Performance Corrosion-Resistant Materials: Originally Proposed FY05 SOW – Kaufman

Larry Kaufman
CALPHAD – MIT
Brookline, Massachusetts

Originally Proposed FY05 SOW – Kaufman

- Calibrated/Validated THERMOCALC Code for Phase Diagram Prediction
- Calibrated/Validated THERMOCALC Code for Pourbaix Diagram Prediction

High-Performance Corrosion-Resistant Materials: Originally Proposed FY05 SOW – LLNL

Joe Farmer
Lawrence Livermore National Laboratory
Livermore, California

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Originally Proposed FY05 SOW – LLNL (1)

- **Program Management: Completed**
  - **Technical Plan & Budget**
  - **Contracts & Weekly Teleconferences**
  - **Presentations**
  - **Annual Report**
- **Preliminary Documents: Completed**
  - **White Paper Describing Specific Yucca Mountain Applications**
  - **Description of Anticipated Environment & Relevant Test Conditions**
  - **Description of Formal Requirements for Yucca Mountain**
- **Testing in Relevant Test Environments: Test in Progress**
  - *Bicarbonate = SAW, SCW, BSW (Testing in FY03 & FY04)*
  - *Calcium-Chloride = Calcium Chloride (Previous Tests, No Longer Relevant)*
  - **Chloride-Nitrate = Seawater, SSW, NaCl + KNO_3 (Current Emphasis)**

Originally Proposed FY05 SOW – LLNL (2)

- **Data from Rod Samples with Long-Term OCP Monitoring: Test in Progress**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*
- **Data from Galvanic Coupling with Disk Samples: Preparing for Test**
  - **SAM2X5/C-22 vs. Ti Grade 7 Counter Electrode**
  - *SAM1651/C-22 vs. Ti Grade 7 Counter Electrode (Not Produced)*
  - **SAM2X5/C-22 vs. Alloy C-22 Counter Electrode**
  - *SAM1651/C-22 vs. Alloy C-22 Counter Electrode (Not Produced)*
  - **SAM2X5/C-22 vs. Type 316L Counter Electrode**
  - *SAM1651/C-22 vs. Type 316L Counter Electrode (Not Produced)*

Originally Proposed FY05 SOW – LLNL (3)

- **Data from Lollipop Samples with Potential Stepping: <u>Test in Progress</u>**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*
- **Data from Instrumented Crevice Corrosion Cell: <u>Preparing for Test</u>**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*

Originally Proposed FY05 SOW – LLNL (4)

- **Immersion Testing of Weight Loss & Crevice Samples: <u>Test in Progress</u>**
  - **SAM2X5/C-22 (Only Combination Produced)**
  - *SAM1651/C-22 (Not Produced)*
  - *SAM2X5/316L (Not Produced)*
  - *SAM1651/316L (Not Produced)*
  - *SAM2X5/hy80 (Not Produced*
  - *SAM1651/hy80 (Not Produced)*
- **Data Feed for Economic Analysis: <u>Completed</u>**
  - **Yucca Mountain & Navy Applications**
- **Final Report: <u>Completed</u>**
  - **Description of Specific Yucca Mountain Applications**
  - **Summary of Requirements Document**
  - **Compilation of All Test Results**
  - **Documentation of Anticipated Benefit**

High-Performance Corrosion-Resistant Materials: Originally Proposed FY05 SOW – NRL

Bob Bayles
Naval Research Laboratory
Washington, DC

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ADC Signature

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Sensitive Subject Reviewer Signature

Originally Proposed FY05 SOW – NRL (1)

- **Data from Sail Application Test – Ambient Seawater**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***
- **Data from Shaft & Bearing Application Test – Ambient Seawater**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***
- **Data from Key West Immersion Test – Ambient Seawater**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***
- **Data from Cavitation Test – Ambient Seawater**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***

Originally Proposed FY05 SOW – NRL (2)

- **Data from Cathodic De-Bonding Test – Ambient Seawater**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***
- **Data from SSRT Stress Corrosion Cracking (SCC) Test – Ambient Seawater**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***
- **Data from SSRT Hydrogen Induced Cracking (HIC) Test – Ambient Seawater**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***
- **Data from Fracture Toughness Test**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***

Originally Proposed FY05 SOW – NRL (3)

- **Data from B117 Salt Fog Test**
  - **SAM2X5/hy80**
  - ***SAM1651/hy80***
- **Data from KISCC & KIHIC Tests – Yucca Mountain Brines**
  - **SAM2X5 Ingot**
  - **SAM2X5/C-22**
  - ***SAM2X5/316L***
  - ***SAM1651 Ingot***
  - ***SAM1651/C-22***
  - ***SAM1651/316L***
- **Final Report: Completed**
  - **Description of Specific Navy Applications**
  - **Compilation of All Results for Application Testing**
  - **Documentation of Anticipated Benefits**

Originally Proposed FY05 SOW – CWRU

- Publication of FY04 Results on Carburization (Heuer – LLNL/DARPA Procurement)
  - Carburization of Type 316L Stainless Steel & Nickel-Based Alloy 22
  - Carburization of Fe-Based Amorphous Metals
- Measurements of Strength & Hardness (Lewandowski)
  - Hardness Indentation Testing
  - Hot Hardness Testing
  - Tension Testing
- Measurements of Toughness
  - Bend Testing
  - Notched Toughness Testing
  - Fatigue Pre-Cracked Fracture Toughness Testing
  - Cyclic Fatigue Testing
- Impact Testing
  - Charpy
  - Split-Hopkinson Pressure Bar

Originally Proposed FY05 SOW – SNL

- Data from Characterization of Powders & Coatings: Completed
  - Macro Structural & Morphological Data
    - Optical Microscopy
    - SEM & TEM Images
  - Micro Structural & Compositional Data
    - SEM & TEM Images
    - WDS, AES & XPS Spectra
    - XRD Data
  - Damage Tolerance
    - Vickers Hardness
    - Corrosion/Cracking Damage & Products

Originally Proposed FY05 SOW – UCD

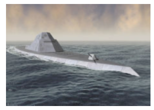
- Non-Porous Fully-Dense HVOF Coatings of Amorphous SAM2X5
  - Thickness ~ 200 to 400 Microns
- Non-Porous Fully-Dense Fe-Based Amorphous Metal Coatings for Testing
  - Two (2) Materials
    - SAM2X5/C-22
    - SAM1651/C-22
  - Six (6) Test Samples for Each Material
    - Two (2) Particle Size Distributions
      - 16-25 microns
      - 25-53 microns
    - Three (3) Configurations
      - 5/8" Discs for Electrochemical Testing
      - 2" x 2" Plates for Crevice Corrosion Testing
      - 1" x 2" Plates for Weight Loss Testing
- Written Monthly Progress Reports
  - Direct Delivery to SNL & LLNL

High-Performance Corrosion-Resistant Materials: Originally Proposed FY05 SOW – CSM

Levent Ozdemir
Colorado School of Mines
Boulder, Colorado

Originally Proposed FY05 SOW – Colorado School of Mines

- **Data from Abrasion & Wear Testing of Bearings/Shafts: ORNL et al.**
  - **Low-Carbon SAM40 Coating**
- **Data from Abrasion & Wear Testing of Disc Cutters: ORNL et al.**
  - **Low-Carbon SAM40 Coating**
- **Data from Abrasion & Wear Testing of Alpine Pick: ORNL et al.**
  - **Low-Carbon SAM40 Bulk Material**

High-Performance Corrosion-Resistant Materials: Submarine Applications

Bob Brown
Naval Research Laboratory
Key West, Florida

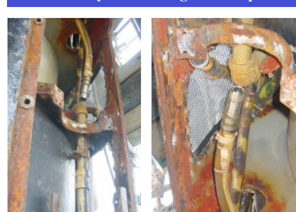
Ex-Portsmouth 688 Class Submarine Sail


Sail of Ex-Portsmouth 688 Class Submarine with Black Epoxy Coating on Exterior Surface

Sail Cover Plates Ready for Coating at Caterpillar


Exterior Forward Sail Cover Plates Removed


Internal Aft Sail Corrosion


Internal Forward Sail Corrosion

NRL FY05 Submarine Application Accomplishments (1)

- Milestone: Locate Shipboard Parts
  - Located available decommissioned submarines in Norfolk, VA. and Puget Sound WA.
  - Joe Farmer and Bob Brown inspected submarines and identified components for testing.
  - Submitted MIPR's to Puget Sound and Norfolk Naval Shipyards
    - For removal and shipping of:
      - Twenty eight sail cover plates from the 702 (Phoenix) and 693 (Cincinnati).
      - Two Brine Pumps (1.6K & 10K GPD) from the 707 (Portsmouth).
  - Coordinated shipping of plates and pumps received by Caterpillar and NRL.
  - Conducted inventory of sail cover plate materials at Caterpillar.
- Milestone: Determine Parts Engineering Conformance:
  - Conducted Engineering Review:
  - Utilized Advanced Technical Information System (ATIS) at Electric Boat, to locate, review and compare active and decommissioned boats drawing for sail cover plates and brine pumps interchangeability.
    - Active Boats and home ports being evaluated for of fit, form and function:
      - Pearl Harbor, Hi.
      - San Diego, Ca.
      - Kings Bay, Ga.

NRL FY05 Submarine Application Accomplishments (2)

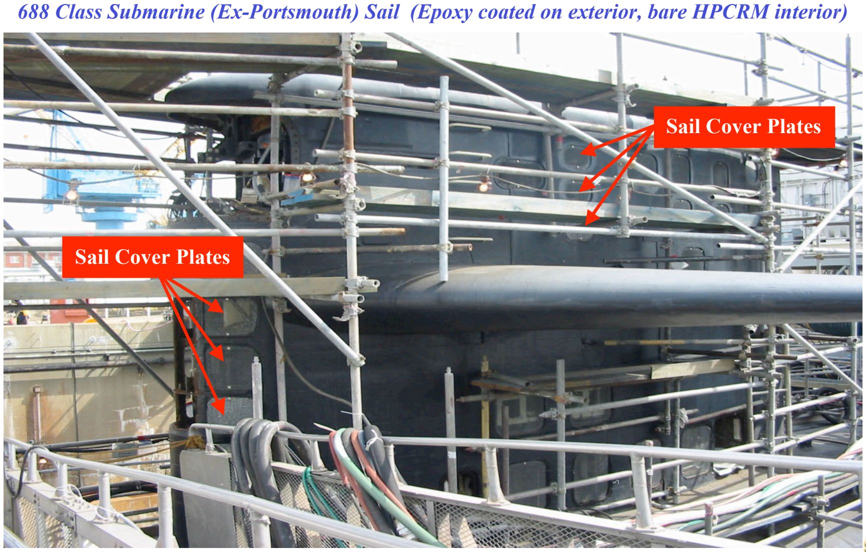
- Milestone: Manage Installation Process:
  - Developed Plan of Action & Milestones (POA&M)
  - Provided NAVSEA 07T and NAVSEA 05M with Plan for Qualification of Coating.
  - Sail cover plates to be a Departure From Specification (DFS).
  - Brine pump will be DFS.
  - Met with Pearl Harbor contacts:
    - COMSUBPAC/Type Commander.
    - Pearl Harbor Naval Shipyard (PHNSY).
    - Pearl Harbor Regional Maintenance Center (PHRMC) Business Agent-
      - Establishing MOA between NRL and PHRMC.
    - Squadron 1 Engineering Group.
  - Coordinating with CARVER for the rebuild of both brine pumps incorporating coated shafts.
  - Both pumps have been torn down and the shafts extracted.
  - Located Active Submarine and coordinating available installation.

Parts From Puget Sound & Norfolk Naval Shipyards

- **Puget Sound Naval Shipyard:**
  - Sail Cover Plates from 688 Class Submarines
    - **Small Size: Approximately 11-inch x 15-inch x 3/8-inch; 12 Parts**
    - **Medium Size: 16-inch x 17-inch x 3/8-inch; 16 Parts**
  - Plates arrived at Caterpillar – June 28th 2005
- **Norfolk Naval Shipyard:**
  - 10K Low Pressure Brine Pump (Shafts) from 688 Class Submarines
    - **APL 017030722; NSN 3H4320-01-317-3577**
    - **Manufacturer: Carver Pump Company; Drawing D-NDS-0-98-02**
    - **Shaft has no taper**
  - 1.6K Low Pressure Brine Pump (Shafts) from 688 Class Submarines
    - **APL 017030383; NSN 2SH4320-01-032-9397-A2**
    - **Manufacturer: Carver Pump Company; Drawing D-NDS-0-98-001**
    - **Shaft has no taper**
  - 1OK & 1.6K Pump Motors
    - **APL 173870012; NSN 9G6105-01-175-9774**
    - **Manufacturer: Hansome Energy Systems, Incorporated; Drawing A-203-12**
  - Received at NRL August 2nd 2005

Application #1: 688 Class Sail Cover Plate

688 Class Submarine (Ex-Portsmouth) Sail (Epoxy coated on exterior, bare HPCRM interior)



Application #1: 688 Class Sail Cover Plate

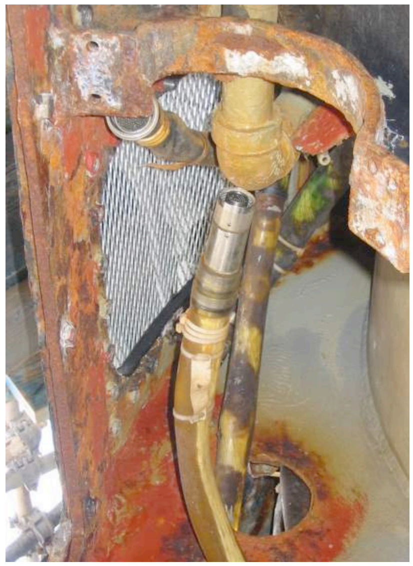
688 Class Submarine (Ex-Portsmouth) Exterior Sail forward (cover plates removed)

Application #1: Corrosion Near Sail Cover Plates

688 Class Submarine (Ex-Portsmouth) Internal Sail (Aft) Corrosion

Application #1: Corrosion Near Sail Cover Plates

688 Class Submarine (Ex-Portsmouth) Internal (Forward) Sail Corrosion



Application #1: Sail Cover Plate
- Coating Qualification and Application -

- Sail cover plates need to been removed from an active 688-class submarine in a major availability (overhaul). These will be shipped to Caterpillar Inc. who will apply the High Velocity Oxy Fuel (HVOF) HPCRM thermal spray at a thickness of 30 mils on all surfaces of the sail cover plate.
- The substrate will be prepared by the contractor to a SP-5 surface.
- The exterior of the sail cover plate will also be coated with conventional black MIL-P-24441 epoxy polyamide coating to maintain the exterior appearance of the sail, while the inside of the respective plate remains without a topcoat. Application of the MIL-P-2441 will be done in accordance with a Preservation Process Instruction (PPI).
- A NACE and NAVY Certified Coatings' Inspector will be onsite during each coatings application and will be responsible for final QA.

Application #1: Sail Cover Plate
- Coating Qualification and Application -

- Corrosion Tests
  - Accelerated Crevice Corrosion Conditions: with comparison to WROUGHT C-22 & 316L
  - ASTM G31-72 Long Term Immersions with and Without Scribe
  - ASTM G71-81 Galvanic Corrosion Testing: Carbon Steel, and Zinc anodes
- Coatings Tests
  - ASTM G8-96 Cathodic Disbondment (ASTM G8-96, modified; required by MIL-PRF-23236)
  - Alternate Seawater Immersion encased in black box exposed to outside elements in Key West FL (1 year)
  - Edge Retention (70% on a 1mm radii)
  - Removability Index

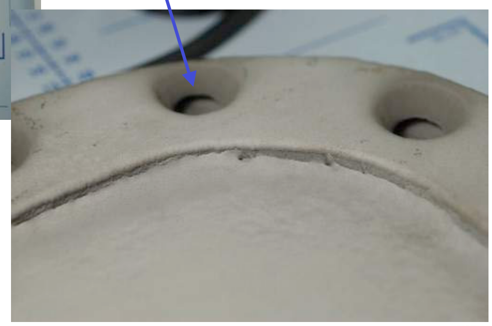
Application #1: Sail Cover Plate
- Installation Process -

- Identification and Ship check of Submarine.
- Submission and Approval of DFS by NAVSEA 07T.
- NRL will supply:
  - Relevant and required Test Data.
  - Coated Sail Cover Plates.
  - Supporting DFS documents.
  - NAVSEA liaison.
  - On-site technical support.
  - Replacement Sail Cover Plates.
  - Requisite Preservation Process Instructions (PPI).
- Test fitting of sail cover plates on Submarine during availability.
- Machining & Refitting of Sail Cover Plates to Fit in Existing Sail.
- Coating of Sail Cover Plate (Offsite, Private Contractor).
- Installation of Sail Cover Plates.
- Removal of Sail Cover Plates after at least 1 year of installation.
- Replacement with Standard Sail Cover Plates.

Application #1: Sail Cover Plates
- Process Control -

Sail Cover Plate from 702 Boat (11"x15")

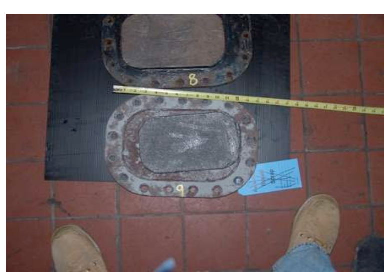
Complex Geometry on Outer & Inner Edges, and Holes.



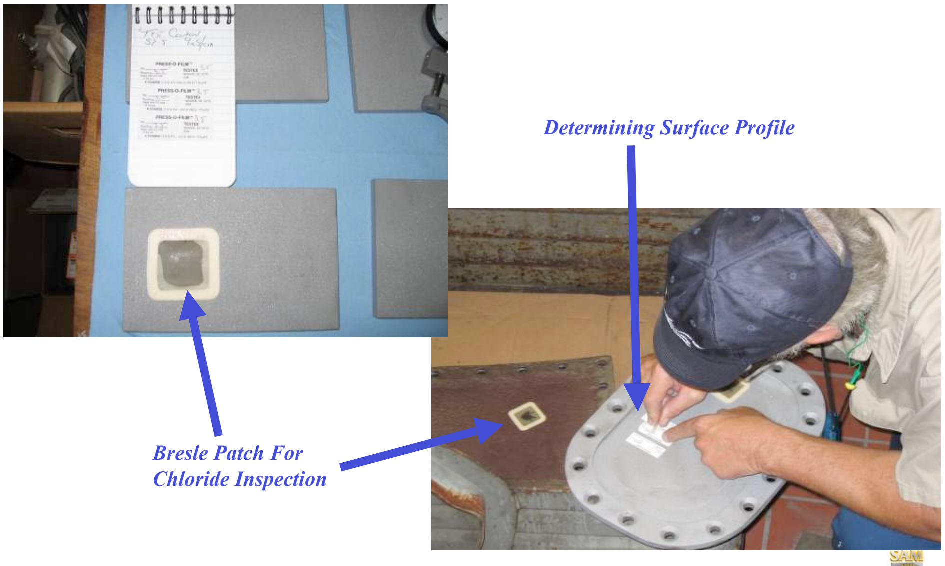
Coated With SAM2X5

NRL Quality Assurance

- Milestone: Ensure CATAPILLAR Parts meet Performance Criteria for Large scale application.
  - Flash Rust Revealed After Blasting
  - Potential Chloride Embedment
  - Surface Profile
  - Thermal Spray Parameters are Consistent
  - Powder Size and Quality

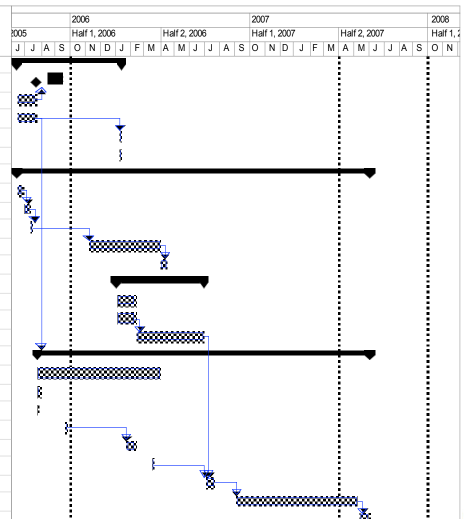


Application of NRL Quality Assurance at Caterpillar (9/20/05)



Application #1: Sail Cover Plate

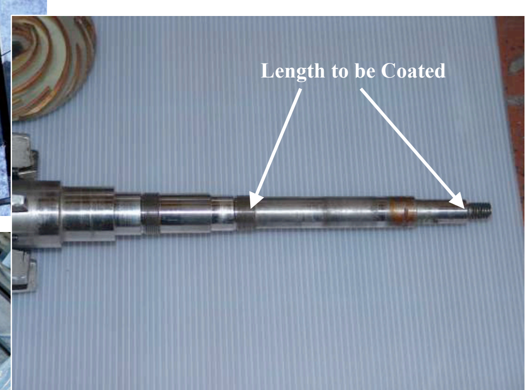
ID	Task Name	Start	Finish
1	**Sail Cover Plates Demo**	**Mon 6/13/05**	**Thu 1/12/06**
2	Submission of POA&M to SEA07T	Fri 7/22/05	Fri 7/22/05
3	Receipt of Concurrance from SEA07T	Mon 6/13/05	Fri 7/22/05
4	Identification of submarine platform	Mon 6/13/05	Fri 7/22/05
5	Brief TYCOM, PMT, CHENG for each platform	Mon 1/9/06	Thu 1/12/06
6	Perform a ship check for each platform	Mon 1/9/06	Thu 1/12/06
7	**Assemble detailed test plan and technical data package**	**Mon 6/13/05**	**Fri 6/1/07**
8	Ship specific drawing of individual sail cover plates.	Mon 6/13/05	Fri 6/24/05
9	Submission of drawings to Caterpillar Inc.	Mon 6/27/05	Fri 7/8/05
10	Specification for final component weight, dimensions and tolerances	Mon 7/11/05	Fri 7/15/05
11	Assemble lab test data to support DFS	Mon 11/7/05	Fri 3/31/06
12	Identification of performance & financial metrics	Mon 4/3/06	Fri 4/14/06
13	**DFS Approval Process**	**Mon 1/2/06**	**Fri 6/30/06**
14	Submission of proposed DFS to SEA07T	Mon 1/2/06	Fri 2/10/06
15	Submission of DFS to SEA05M	Mon 1/2/06	Fri 2/10/06
16	DFS Approval	Mon 2/13/06	Fri 6/30/06
17	**Shipboard demonstration Platform #1**	**Mon 7/25/05**	**Fri 6/1/07**
18	Inclusion of removal/installation in Work Package	Mon 7/25/05	Fri 3/31/06
19	Removal of Sail cover plates	Mon 7/25/05	Fri 7/29/05
20	Shipping of Sail Cover Plates to Caterpillar	Mon 7/25/05	Tue 7/26/05
21	Surface preparation of sail cover plates	Mon 9/19/05	Fri 9/23/05
22	Coating of sail cover plates with HPCRM	Mon 1/23/06	Fri 2/10/06
23	Shipping of sail cover plates to shipyard	Mon 3/13/06	Mon 3/20/06
24	Installation and documentation of sail cover plates	Mon 7/3/06	Fri 7/21/06
25	Test & Evaluation Period	Mon 9/4/06	Fri 5/11/07
26	Removal and repair to original condition	Mon 5/14/07	Fri 6/1/07



Application #2
Brine Pump Testing & Installation Process

- **Pump tear down**
  - **Remove shaft from pump & motor.**
  - **Shafts to be turned down 10 mils.**
  - **Shafts to be coated at Caterpillar.**
  - **Shafts to be machined at step and polished to RMS specification.**
- **Send 1.6K & 10K GPD pumps to CARVER for rebuild to like new condition.**
  - **Megger test and rotor evaluation.**
  - **New mechanical seals.**
  - **New impeller.**
- **Establish baseline criteria of pump performance to monitor/trend operation.**
  - **Baseline vibration analysis.**
  - **Flow rate vs. pressure and amps.**
- **NRL Key West to set up mock-up with 1.6K GPD pump**
  - **Land based testing.**
  - **Monitor performance while routing package for approval.**
    - **This will provide additional qualification testing information to NAVSEA.**

Applications #2: Brine Pump Shaft

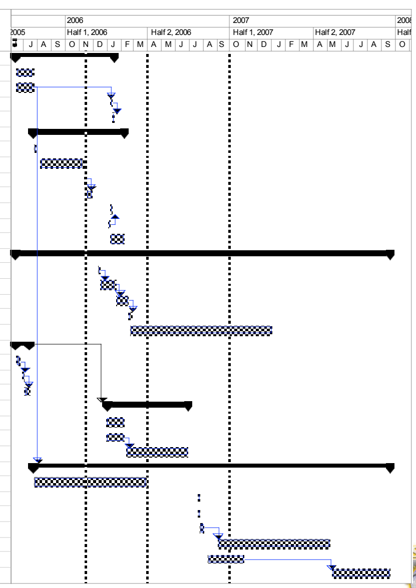


Application #2: Brine Pump
- Installation Process -

- Identification and Ship check of Submarine.
- Submission and Approval of DFS by NAVSEA 07T.
- NRL will supply.
  - Relevant and required Test Data.
  - Requisite Preservation Process Instructions (PPI).
  - Coated shaft with rebuilt pump/motor.
  - Supporting DFS documents.
  - NAVSEA liaison.
  - On-site technical support.
  - Rebuilt pump to specifications (10K GPD brine pump).
  - Installation of 10K GPD Brine Pump.
- Removal of HPCRM coated 10K GPD pump after at least 1 year of installation.
  - Replacement with original 10K GPD Brine pump.

Application # 2: Brine Pump

ID	Task Name	Start	Finish
1	**Brine Pump Demo**	**Mon 6/13/05**	**Wed 1/18/06**
2	Receipt of Concurrance from SEA07T	Mon 6/13/05	Fri 7/22/05
3	Identification of submarine platform	Mon 6/13/05	Fri 7/22/05
4	Brief TYCOM, PMT, CHENG for each platform	Mon 1/9/06	Thu 1/12/06
5	Perform a ship check for each platform	Fri 1/13/06	Wed 1/18/06
6	**Preparation of Pumps**	**Fri 7/22/05**	**Thu 2/9/06**
7	Receive pumps from Puget Sound NSY	Fri 7/22/05	Fri 7/29/05
8	Disassembly of Pumps	Mon 8/8/05	Thu 11/10/05
9	Shafts shipped to Caterpilar	Mon 11/14/05	Wed 11/16/05
10	Shafts machined & Coated by Caterpilar	Thu 11/17/05	Wed 11/30/05
11	Shafts shipped to NRLKW	Tue 1/10/06	Fri 1/13/06
12	Pumps crated and shipped to Carver	Tue 1/3/06	Mon 1/9/06
13	Pumps rebuilt by Carver	Mon 1/9/06	Thu 2/9/06
14	**Land Based Pump Demonstration**	**Mon 6/13/05**	**Fri 9/21/07**
15	Fabricate pump foundation	Mon 12/12/05	Fri 12/16/05
16	Install seawater piping	Mon 12/19/05	Fri 1/20/06
17	Install electrical	Mon 1/23/06	Fri 2/17/06
18	PMT's to get baseline vibration readings	Mon 2/20/06	Fri 2/24/06
19	Run/test and aquire DFS data	Fri 2/24/06	Mon 1/1/07
20	**Assemble detailed test plan and technical data package**	**Mon 6/13/05**	**Fri 7/15/05**
21	Ship specific drawing of individual brine pumps.	Mon 6/13/05	Fri 6/24/05
22	Specification for final component weight, dimensions and tolerances	Mon 6/27/05	Fri 7/1/05
23	Identification of performance & financial metrics	Mon 7/4/05	Fri 7/15/05
24	**DFS Approval Process**	**Mon 1/2/06**	**Fri 6/30/06**
25	Submission of proposed DFS to SEA07T	Mon 1/2/06	Fri 2/10/06
26	Submission of DFS to SEA05M	Mon 1/2/06	Fri 2/10/06
27	DFS Approval	Mon 2/13/06	Fri 6/30/06
28	**Shipboard demonstration Platform #1**	**Mon 7/25/05**	**Fri 9/21/07**
29	Inclusion of removal/instalation in Work Package	Mon 7/25/05	Fri 3/31/06
30	Shipping of Brine pump to PHNSY	Fri 7/21/06	Thu 7/27/06
31	Removal of brine pump	Fri 7/21/06	Wed 7/26/06
32	Installation and documentation of brine pump	Wed 7/26/06	Fri 8/4/06
33	Test & Evaluation Period	Mon 9/4/06	Fri 5/11/07
34	Rebuild old pump (Carver)	Mon 8/14/06	Tue 10/31/06
35	Removal and reinstall to original condition	Mon 5/14/07	Fri 9/21/07



Turtleback Test Panels

- SSBN Submarine: Location to be determined:
  - Four (4) test panels to be installed under turtleback
    - Awaiting TEMPALT approval for installation
    - TEMPALT prepared by NSWC Carderock
  - Panels provide for additional real-world exposure
  - Direct comparison with other coating systems on same test rack

Application PHNSY Support
Brine Pump and Sail Cover Plate Installation

- NRL Briefing at Pearl Harbor Naval Shipyard
- Pearl Harbor-Regional Maintenance Center Support (PHRMC)
  - Not expected to be simultaneous installations.
    - Important from a scheduling and budget standpoint.
  - MOA's (DARPA & COMSUBPAC) and (NRL & PHRMC)
  - Identification of Platform (boat) & Availability.
  - Issuance of DFS from Boat/Boats.
  - Inclusion of all work in Availability Work Packages as necessary.
  - Scaffolding/Staging.
  - Shop Support for Machining/Adjustment of Sail Cover Plates.
  - Personnel to support one initial test fit of prototype sail cover plates into identified locations.
  - Personnel to support installation, removal and replacement of coated sail cover plates.
  - Brine Pump
    - Pipe fitters, electrician, riggers

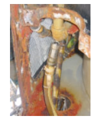
Tentative Planning for HPCRM Thermal Spray Facility at PHNSY

- NRL Briefing at Pearl Harbor Naval Shipyard
- Plan is for a facility modeled after the new CAT Joliet plant
- Several operational modes under consideration
  - Shipyard facility using shipyard personnel
    - Limited Caterpillar involvement
  - Shipyard facility operated by Caterpillar
    - Caterpillar expertise, leveraging on other installations
  - Caterpillar facility
    - Primarily local labor
    - Portable, modular (may move to another shipyard, as needed)
- Intention to be installed and operational in FY2008

Backup Slides

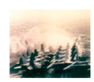
Department of Defense Interests

- Cost of Corrosion to Department of Defense
  - Helicopter Repair Alone Costs ~ $4 Billion
  - Total Annual Cost ~ $10 Billion to $20 Billion

- Corrosion Effects All Military Assets
  - Ground &Tactical Vehicles ~ 35,000
  - Aircraft & Helicopters ~ 15,000
  - Strategic Missiles ~ 1,000
  - Navy Ships & Boats ~ 300
    - Structure ~ 345M ft^2
    - Propulsion, Fluid & Seawater Piping Systems


- Examples of Corrosion-Related Faults Degrading Mission Readiness
  - Crashes of Several F-16 in 1980s (Corrosion of Electrical Contacts Leading to Uncontrolled Fuel Valve Closure)
  - Collapses of Landing Gear on Navy F-14 and F-18 During Carrier Operations
- Personnel Requirements for Corrosion-Related Maintenance
  - Military & Civilian ~ 700,000
  - Commercial Firms ~ Several Thousand

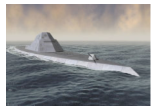




Actual Navy Parts From Puget Sound & Norfolk Naval Shipyards

- Puget Sound Naval Shipyard
  - *Sail Cover Plates from 688 Class Submarines*
    - *Small Size: Approximately 8-inch x 11-inch x 3/8-inch; 18 Parts*
    - *Medium Size: 16-inch x 17-inch x 3/8-inch; 24 Parts*
  - *Availability – July 19th 2005*
- Norfolk Naval Shipyard
  - *10K Low Pressure Brine Pump (Shafts) from 688 Class Submarines*
    - *APL 017030722; NSN 3H4320-01-317-3577*
    - *Manufacturer: Carver Pump Company; Drawing D-NDS-0-98-02*
    - *Shaft has no taper*
  - *1.6K Low Pressure Brine Pump (Shafts) from 688 Class Submarines*
    - *APL 017030383; NSN 2SH4320-01-032-9397-A2*
    - *Manufacturer: Carver Pump Company; Drawing D-NDS-0-98-001*
    - *Shaft has no taper*
  - *1OK & 1.6K Pump Motors*
    - *APL 173870012; NSN 9G6105-01-175-9774*
    - *Manufacturer: Hansome Energy Systems, Incorporated; Drawing A-203-12*
  - *Availability – July 21st 2005*

Actual Navy Parts & Samples Coated by Caterpillar

- **HVOF Coating of Actual Navy Parts with SAM1651/SAM2X5**
  - *Small-Size Cover Plates (& Witness Samples) for Sail*
    - *HVOF Coatings of SAM2X5 & SAM1651*
  - *Medium-Size Cover Plates (& Witness Samples) for Sail*
    - *HVOF Coatings of SAM2X5 & SAM1651*
  - *Rotating Shaft (& Witness) from Low-Capacity (1.6K) Brine Pump*
    - *Polished HVOF Coating of SAM2X5*
  - *Rotating Shaft (& Witness) from High-Capacity (10K) Brine Pump*
    - *Polished HVOF Coating of SAM1651*
  - *Prototypical Air-Intake Plenum (& Witness Samples) for Surface Ship*
    - *HVOF Coatings of SAM2X5 & SAM1651*
- **HVOF Coating Navy Test Samples with SAM1651**
  - *Salt Fog Testing – 3000 hour test*
  - *Long-Term Exterior Exposure – 6 months – 1 year*
  - *Alternate Immersion – 6 months – 1 year*
  - *Bend-Over-Mandrel Test – 1year*
  - *Cathodic Disbondment – 90 days*
  - *Galvanic Corrosion – 6 months – 1 year*
  - *Crevice Corrosion (Accelerated) – 1 months*

High-Performance Corrosion-Resistant Materials: Broad Perspective of Navy & Defense Applications

Bob Bayles & Ted Lemieux
Naval Research Laboratory
Washington DC & Key West FL

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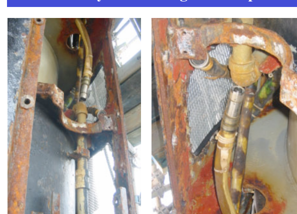
Ex-Portsmouth 688 Class Submarine Sail

Sail of Ex-Portsmouth 688 Class Submarine with Black Epoxy Coating on Exterior Surface

Sail Cover Plates Ready for Coating at Caterpillar

Exterior Forward Sail Cover Plates Removed

Internal Aft Sail Corrosion



Internal Forward Sail Corrosion

NRL Applications

- Introduction
- Technical Authority
- Submarine Applications – Bob Brown
- Pearl Harbor Facility
- Amphib Components
- Fracture and Damage Tolerance
- Coating Process
- Quality Assurance
- Non-Destructive Inspection
- Wire HVOF
- Conclusions

Introduction

- Identifying and arranging for coating of parts from submarines, surface ships, and Marine artillery, and installation of these parts. This includes interaction with the approval authority, port engineers, ships, shipyards, and preparation of the necessary Departure From Specification (DFS) documents.
- We are in the preliminary stages of planning for installation of an HPCRM thermal spray facility at Pearl Harbor Naval Shipyard.
- Evaluating fracture and damage tolerance under corrosion conditions
- Evaluating non-destructive inspection methods to supplement present process control and inspections
- Performing research to enable the use of wire-fed HVOF systems which show great promise for improved economics and versatility
- Contributing to all aspects of the HPCRM program by participating in the weekly conference calls on both science and applications

NRL Staff Involved in Naval Applications of HPCRM Materials

- Dr. Bob Bayles – Principal Investigator
  - Alteration and Installation Team Lead for Amphibious Ships
  - Mechanical & Non-Destructive Testing
- Ted Lemieux – Co Investigator
  - Alteration and Installation Team (AIT) Lead for Submarine Applications
- Bob Brown – Expertise in Submarine
- AIT Support Personnel
  - Paul Slebodnick: Technical & Administrative Waterfront Guidance, Paint Preservation Process Instruction (PPI) Lead
  - Dave Zuskin: CES, NACE Level III Certified Coatings Inspector
  - Bill Groeninger: NACE Level III Certified Coatings Inspector
  - Bucky Glenn: Former SURFLANT TYCOM, DFS & NAVMESSAGE Writing
  - Theresa Newbauer: Corrosion Engineer, Parts/Components Land-Based Testing

What is Technical Authority?

- "The exercise of Technical Authority is a process that establishes and assures adherence to technical standards and policy…a range of technically acceptable alternatives with risk and value assessments…."
- Responsibilities of Technical Authority
  - Setting and enforcing technical standards
  - Maintaining subject matter expertise
  - Assuring safe and reliable operations
  - Ensuring effective and efficient systems engineering
  - Making unbiased independent technical decisions
  - Providing stewardship of technical and engineering capabilities
  - Being held accountable

NAVSEA NAVAL SEA SYSTEMS COMMAND

"I believe Technical Authority is the most important thing we do."
VADM Phillip M. Balisle, COMNAVSEA

Applications and the Navy Approval Process

As Presented in Pearl Harbor Annual Review Meeting:

- "Road to Transition" Requires a Management of Change:
  - Technical Authority approval of DFSs
- POA&M spells out the qualification testing NRL recommends
  - Incorporates a schedule for testing and component installation
- Type Commander and respective boat involved

Applications and the Navy Approval Process

- Brief NAVSEA Tech Authority on POA&M and Qualification Test Outcome.
- Departure from Specification (Waiver)
  - DFS requirements & procedures well defined by the Joint Fleet Maintenance Manual (JFMM Vol 5., COMFLTFORCOMINST 4790.3 REV A).
  - Work with the Regional Maintenance Center (RMC) (e.g. PHSNY & IMF) to submit DFS NAVMESSAGE from the boat to NAVSEA.
  - NAVSEA Technical Warrant Holder provide approval of DFS
    - Sail Cover Plates: NAVSEA 07T – Karen Poole
      - NAVSEA05M1(Thomas) expects to comment but no further role at this time
    - Brine Pump: NAVSEA 07T – Rich Kurz
    - Test items on USS PONCE: Port Engineer, MARMC – Bud Hubert
    - Marine Corps howitzer components: Fire Support Systems Program Manager, MARCORSYSCOM – James Ripley

Example DFS – NAVMESSAGE

ATTENTION INVITED TO ADMINISTRATIVE MESSAGE

ROUTINE

R APRIL 05 ZYB PSN

FM USS ROSS

TO COMNAVSURFLANT NORFOLK VA//N434A41/N434//

INFO COMLANTFLT NORFOLK VA//N43//
COMNAVSURPAC SAN DIEGO CA//N43//
PEO SHIPS WASHINGTON DC//SHIPS-F/PMS400D/SHIPS-FT//
COMNAVSEASYSCOM WASHINGTON DC//05//
COMNAVSEASYSCOM WASHINGTON DC//05M1//
NRL WASHINGTON DC//6130//
NAVSURFWARCEN CARDEROCK DIV BETHESDA MD//613//
COMDESRON TWENTY EIGHT
RSO NORFOLK VA
MIDLANT RMC NORFOLK VA//N43/N43A/PORTENG/200/300/900/980//

BT
UNCLAS //N04790//

MSGID/GENADMIN/ROSS//

SUBJ/DFS REQUEST TEST INITIATIVE FOR INTERNATIONAL INTERGARD 783 (3-COAT UHS SYSTEM) IN COMP FUEL TANKS//
POC//D. ELMER/MPA/TEL: (757) 445-6274/EMAIL:MPA@ROSS.NAVY.MIL//

REF/A/MTG/NRL/07APR05/-/NOTAL//

REF/B/DOC/COMFLTFORCOMINST 4790.3/REV A CH 2//

NARR/REF A IS MEETING BET NRL/PAUL SLEBONICK (CODE 6130) AND SHIPS FORCE MPA ENS D. ELMER ON 7 APRIL 05. REF B IS JOINT FLEET MAINTENANCE MANUAL//

RMKS/1. DFS NUMBER

2. SHIP: USS ROSS

3. HULL NUMBER: DDG-71

4. JCN:

5. CWP NUMBER: **N/A**

6. DATE OF DFS: **TBD**

7. ORIGINATOR AND ADDRESS: USS ROSS (DDG-71) **FPO-AE-09586-1288**

8. DEPARTURE TYPE: **MAJOR-TEMPORARY**

9. SYSTEM/COMPONENT/LOCATION/ESWBS: **CF 4-370-3-F, CF 4-370-4-F**

10. NAVSEA DRAWING/PLAN NUMBER/PIECE NUMBER: **N/A**

11. REFERENCES: **APPLICATION REQUIREMENTS (PPI), TEST AND EVALUATION PROVIDED BY NRL-CODE 6130 /NSWCCD-CODE 613.**

12. APPLICABLE SPECIFICATIONS: N/A

13. SITUATION/DEGREE OF NON-COMPLIANCE:
(EXAMPLE) APPLY INTERNATIONAL 783 (3-COAT SYSTEM) IN COMP FUEL TANKS CF 4-370-3-F/CF 4-370-4-F TO SUPPORT IN-SERVICE TESTING, EVALUATION AND QUALIFICATION OF PRODUCT FOR FUTURE COMP FUEL TANK APPLICATIONS.

14. COMMENTS/RECOMMENDATION:
(PLEASE PROVIDE INFO FOR THE FOLLOWING ELEMENTS WHERE APPLICABLE)
A. DESCRIBE TEST INITIATIVE
B. PRODUCT & MANUFACTURER
C. SPONSORING AGENCIES (I,E., NAVSEA, SPAWAR, PEO, FTSCLANT, PORT ENGINEER, DEPOT FACILITY, ETC.)
D. TECHNICAL AND OTHER ASSISTS IF REQUIRED (SF, SIME, RRC, ETC.)
E. ESTIMATED DATE OF INSTALLATION
F. BRIEFLY DESCRIBE "TEST INITIATIVE PROCESS" (INCLUDE ANTICIPATED BENEFIT AND PROBLEM DESIGNED TO FIX)
G. DEFINE TEST INITIATIVE AND COMPARTMENT LOCATION (SYSTEM, EQUIPMENT, COMPONENT, HULL STRUCTURE, ETC.)
H. PROVIDE "FEEDBACK REPORT" TO ORIGINATOR AND ALCON (REPORT SHOULD ASSESS MATERIAL CONDITION/STATUS OF TEST INITIATIVE AND REQUEST TECHNICAL FOLLOW-UP IF REQUIRED).
I. DESCRIBE LONG/SHORT TERM ILS PLANS, IF AVAILABLE (FOR PARTS SUPPORT, OTHER NEW MAINTENANCE REQUIREMENTS (I,E, PMS AND TECHNICAL DOCUMENTATION)
J. PROVIDE TEST COMPLETION DATE AND SPONSORING AGENCY EVALUATION POA&M.
15. DATE ANSWER REQUESTED:

Metrics for Successful Coating: Large-Scale Navy Application & Chrome Plating Alternative (1)

- **Large Applications are defined by:**
  - > 100 ft^2 in application
  - Tanks and voids, well deck overheads, and freeboards
  - Bulkheads, flight and all weather decks, and underwater hull
  - VLS & Hanger Bays
- **Metrics (to compete with State-of-the-Art Coatings being implemented now)**
  - **Epoxy Coating Cost:** < $27 to $35/$ft^2$ (cost includes, surface preparation, reduction of substrate chlorides, coatings application, stripe coats on all edges and welds, touch-up of coating, environmental controls during application and capitalization of equipment) Surface prep and application IAW:
    - Navy Document Requirements;
      - Preservation Process Instruction (PPI) CORE, PPI NRB: 63101-000 (Rev 17) or better.
      - NAVSEA Standard Items, 009-32, 24 MAR 2005 or better.
      - Naval Ships Technical Manual, Chapter 631. Preservation of Ships in Service, Surface Preparation and Painting Vol 1, General. Section 1 General Information. S9086-VD-STM-020/CH-631V1R3 (DRAFT) or better.
      - Naval Ships Technical Manual, Chapter 631. Preservation of Ships in Service, Surface Preparation and Painting Vol 2, Section 5 Surface Preparation. S9086-VD-STM-020/CH-631V2R2 (DRAFT) or better.
      - Naval Ships Technical Manual, Chapter 631. Preservation of Ships in Service, Vol 3. Surface Ship/Submarine Applications, Section 8 Shipboard Paint Application. S9086-VD-STM-020/CH-631V3R3 (DRAFT) or better.
  - **Chrome Plating Costs:** $0.65-$1.00 per Square Inch on average within continental U.S.
    - Environmental Regulations in the Hawaiian Islands precludes the Industrial Chrome plating process.
    - All materials are shipped to the Continental United States.
    - Turnaround time is approximately 2-5 days within the U.S. 3-4 additional days can be added to this if shipped form the Hawaiian Islands to the mainland.
    - Shipping costs are approximately $500-$1000 for 200-500 lbs respectively.

Metrics for Successful Coating: Large-Scale Navy Application & Chrome Plating Alternative (2)

- Epoxy Coating Performance
  - Up to 20 year life time with cathodic protection (to >3% damage, localized or overall)
  - Environmental Sensitivity during spray (RH < 50% Temperatures between 50 to 115°F)
  - Surface Preparation: SSPC-SP-10
  - Surface Tolerance: Surfaces prepared to a minimum of SP11/SP15 (power tool cleaned)
  - Edge Retention: 70% on a 1 mm radii
  - Removability Index: Rate at which coating can be removed and at what cost compared to a traditional coating system used for the same application.
  - Over-coat-ability: Repairs or applications over new and aged systems, other coatings, and compatibility with metal containing AF paints.
  - Reduced signature from non skid decks and superstructures using LSA or state of the art coatings.
- Safety, Environmental & Ergonomics
  - Applicator Health & Safety (NEHAC Approval)
  - Ship Safety
  - Hydraulic oil is disposed of as a D7 waste oil IAW CFR part 260 due to chrome deposition (>5 mg/l). Waste Oil requires administrative & physical environmental controls. Satellite storage areas, waste tracking, shipping and final disposition.

Applicator Standards and Inspection Protocols

- Validate preparation/application standards and inspection protocols
  - Identify inspection checkpoints
    - Instrumented inspection
    - Process control
- 100% third party inspection of all steps in coating process
  - Essential to ensure coating performance
  - Never allow applicator to cut corners
  - You get what you inspect, not what you specify
- Evaluate non-destructive, on-part inspection methods for thermal spray coating
  - Conventional NDI/NDE techniques ineffective for thermal spray
    - Reliance on test coupon coated in parallel
    - Uncertainty that coupon was processed identically
  - Novel methods
    - ESPI (Electronic Speckle Pattern Interferometer) with acoustic excitation
    - Porosity & material characterization
      - X-Ray Tomography
      - Ultrasonic/Laser Ultrasonic Shear Wave
  - Verify amorphous condition
- "Design for inspectability" should be a goal

Navy Parts & Samples To Be Coated by Caterpillar

- **Milestone 1: HVOF Coating of Actual Navy Parts with SAM2X5/1651**
  - Small-Size Cover Plates (& Witness Samples) for Sail
    - HVOF Coating of SAM2X5
    - HVOF Coating of SAM1651
  - Medium-Size Cover Plates (& Witness Samples) for Sail
    - HVOF Coating of SAM2X5
    - HVOF Coating of SAM1651
  - Rotating Shaft (& Witness) from Low-Capacity (1.6K) Brine Pump
    - Polished HVOF Coating of SAM2X5-Landbased Tested
  - Rotating Shaft (& Witness) from High-Capacity (10K) Brine Pump
    - Polished HVOF Coating of SAM2X5
  - Prototypical Air-Intake Plenum (& Witness Samples) for Surface Ship
    - HVOF Coating of SAM2X5
    - HVOF Coating of SAM1651

Navy Parts & Samples To Be Coated by Caterpillar

- **Milestone 2: HVOF Coating Navy Test Samples with SAM1651 (time from receiving samples)**
  - Salt Fog Testing: 3000 hr test
  - Long-Term Exterior Exposure: 6 months & 1 year
  - Alternate Immersion: 6 months & 1 year
  - Bend-Over-Mandrel Test: 1 year
  - Cathodic Disbondment: 90 days
  - Galvanic Corrosion: 6 months & 1 year
  - Crevice Corrosion (Accelerated): 1 month

Navy Samples to be Coated by NanoSteel

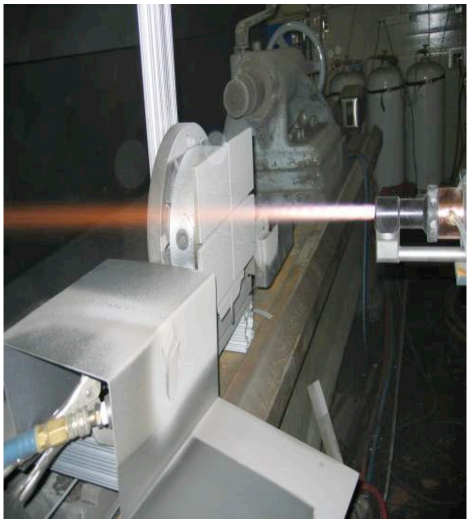
- Milestone 2: HVOF Coating Navy Test Samples with SAM2X5 (time from receiving samples)
  - Salt Fog Testing: 3000 hr test
  - Long-Term Exterior Exposure: 6 months & 1 year
  - Alternate Immersion: 6 months & 1 year
  - Bend-Over-Mandrel Test: 1 year
  - Cathodic Disbondment: 90 days
  - Galvanic Corrosion: 6 months & 1 year
  - Crevice Corrosion (Accelerated): 1 month

Application Process

- Substrate Preparation Preservation Process Instruction (PPI)
  - Need to ensure contaminants have been removed from substrate
    - SSPC SP-5
    - NRL provided Level III NACE Certified Coating Inspector at Caterpillar for substrate testing and coating application as 3rd party QA oversight.

Assessment of Caterpillar HVOF Application for Potential Navy Mobile/Portable Application

HVOF Robotic Applicator



Powder Hopper

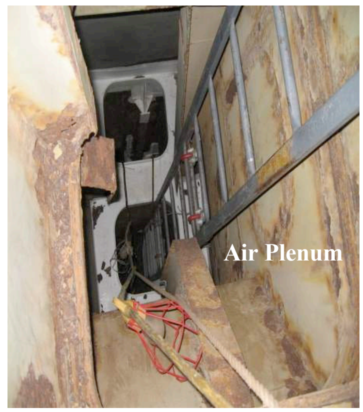
NRL Test Samples Sprayed at Caterpillar

(Click to View Video)

Amphibious Ship: USS PONCE, LPD 15
Installation of Test Items

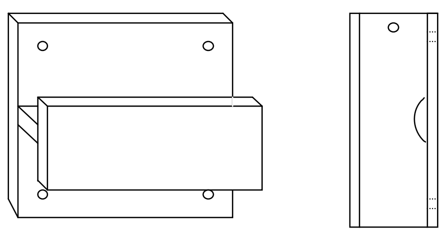
- **Air plenum brings air into the ship's ventilation system**
  - Salt-laden, moist air
- **Representative Structures**
  - Box shape
  - Confined space
  - T-stiffeners

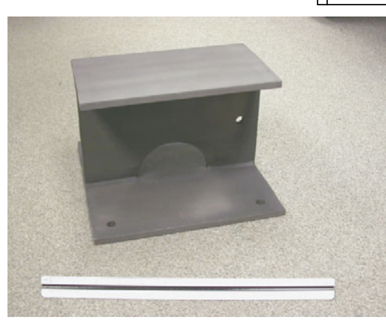
- **Cooperation from Port Engineer & Private Shipyard**
  - Bud Hubert, MARMC
  - Bob Crosby, Metro Machine Corp



Amphibious Ship: USS PONCE, LPD 15
Air Plenum Inserts for Testing

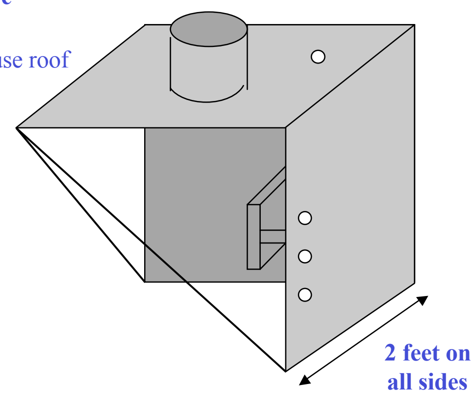
- **HPCRM Plenum Inserts**
  - Small 10"x10"
  - Welded T-stiffener
  - Mouse hole
  - Various coating configurations
- **Installation in Plenum's Fan Room**
  - Up to 6 inserts
  - Fan room at Frame 120
  - 4 studs welded to bulkhead
- **Variety of Coatings**
  - Plasma SAM2X5 by A&A
    - Conventional & 90 degree
    - Sealed and unsealed
  - HVOF SAM2X5 & SAM1651 by Caterpillar





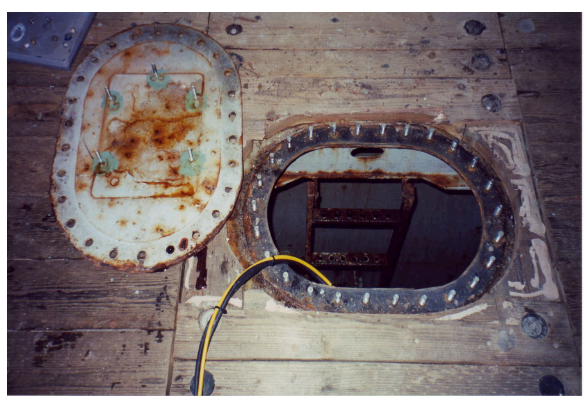
Amphibious Ship: USS PONCE, LPD 15
Simulated Air Plenum

- **More Complex Representative Structure**
  - For installation on pilothouse roof
  - Weather exposure
- **Coating by Caterpillar**
  - SAM2X5
  - SAM1651



Amphibious Ship: USS PONCE, LPD 15
Well Deck Ballast and Fuel Tank Hatch Covers

- **Well deck almost always wet, especially under the wood flooring**
- **Tank hatch cover plates suffer severe corrosion**
- **Coat with HPCRM and install**
- **Accessible for inspection by removing a square access panel in the flooring.**

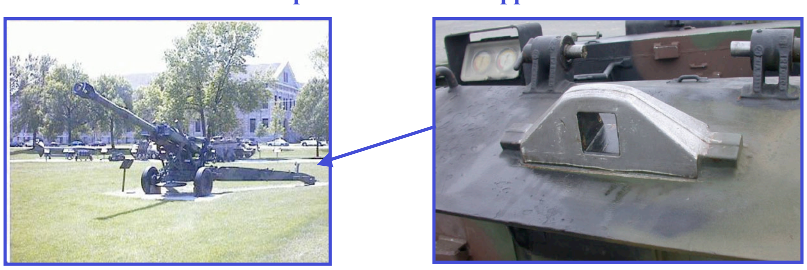


Surface Ship Components – Accomplishments

- Cooperation established for installations on USS PONCE
  - Liaison by Bucky Glenn, NRL contractor (former TYCOM) at Norfolk
  - Port Engineer very supportive of NRL’s T&E efforts
  - NACE Certified NRL inspectors on site full time
- Designed “plenum inserts”
  - Challenging structure
  - Fabricated and coated by A&A
  - More planned by Caterpillar
- Designed “simulated plenum”
  - More complex, representative structure
  - To be sprayed by Caterpillar
- Identified well deck tank hatch covers, anchor chain covers and mooring line port covers for harsh environment and handling abuse

Marine Corps Artillery Components – Accomplishments

- Identified recoil reaction spades on M198 Howitzer as challenging the applicator, operating in a harsh environment, exposed to handling abuse
- Spades sent from Rock Island to Caterpillar
- HPCRM-coated spade will be paired with conventional spade on same gun
- Quantico guns have higher firing rate; convenient for monitoring
- Spades dig into ground when gun is fired
- Spades are removed to bracket on frame for transit/storage
- Although M198 is being phased out, Program Manager is enthusiastic for material with HPCRM’s promise for future applications



Sealer Evaluations

- Thermal spray coatings are usually treated with a sealer to prevent moisture from entering the pores and causing corrosion.
- In the case of HPCRM, the material itself is corrosion resistant and the intended density is high to prevent the environment from accessing the substrate through continuous pores.
- Nevertheless, additional protection will be afforded by sealing the HPCRM coatings.
- In many applications the coating must also be painted. In order to ensure that the sealer does not jeopardize paint adhesion, a set of ASTM D-4541 paddy pull tests was performed on two submarine sail cover plates. Each cover plate was sprayed with SAM2X5 by Caterpillar and then dipped halfway into Microseal DS-AC sealer.
- At NRL, one plate was grit blasted to remove excess sealer and the other was left with a noticeable amount of sealer on the surface of the half that was immersed. The cover plates were then painted with Mare Island F-150, a typical primer for the submarine sail.
- After the paint was cured, the pull tests were performed and no substrate/sealer or sealer/paint failures were observed, indicating very good adhesion whether the surface was original HPCRM, sealer blasted to remove excess, or thick sealer. Based on these results it is recommended to seal all HPCRM coatings. Note that the HPCRM coatings, as applied, have an appropriate surface profile (approximately 0.002 inch) for ideal paint adhesion.
- The test plates were repaired at the pull test sites and painted with the final coat of Mare Island epoxy and put into alternate immersion testing.

Fracture and Damage Tolerance

- Slow strain rate experiments
  - Evaluate coatings and bulk
  - Corrosion conditions (with and without cathodic protection)
  - Comparison with bare substrate
- Impact experiments
  - Simulate real-life damage scenarios, followed by corrosion conditions
  - Characterize "depth" of damage and relate to extent of corrosion
- Scribe experiments
  - Perform corrosion experiments on scribed specimens
  - Crevice experiments have shown some propensity for undercutting
  - Testing so far only on fully encapsulated specimens

NRL Quality Assurance

- **Milestone: Ensure CATAPILLAR Parts meet Performance Criteria for Large scale application.**
  - **Flash Rust Revealed After Blasting**
  - **Potential Chloride Embedment**
  - **Surface Profile**
  - **Thermal Spray Parameters are Consistent**
  - **Powder Size and Quality**

Application of NRL Quality Assurance at Caterpillar (9/20/05)

Determining Surface Profile

Bresle Patch for Chloride Inspection

Non-Destructive Inspection Methods (1)

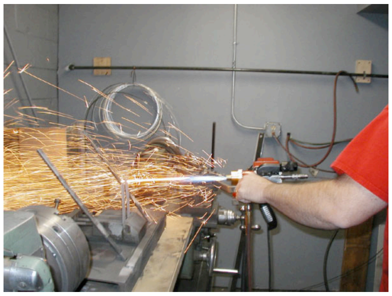
- Capability is critical to assuring a high quality coating every time
- Bond between substrate and coating difficult to inspect
  - Thousands of boundaries between splats
    - Spurious signals confuse ultrasonic measurements
    - Conductivity variations disrupt eddy current measurements
    - NDI equipment manufacturers are not optimistic
- Conventional Approach
  - Destructive examination of witness plates
  - Strict process control

Non-Destructive Inspection Methods (2)

- Concepts from HPCRM Program
  - Sonalysts
    - Adaptation of Ultrasonics with Laser Excitation
  - NRL
    - ESPI (Electronic Speckle Pattern Interferometer) with Acoustic Excitation
    - Flash and Observe Using High-Speed Infrared Camera
  - Intelligent Automation and Luna Innovations
    - Exploring Capabilities
- Assurance of Amorphous Condition Needed

Proposed Development of Wire HVOF Process

- Wire HVOF has many potential advantages (economic, versatile) that the HPCRM team will exploit.
- Present difficulty is in obtaining homogeneous wire so each droplet has appropriate composition.
- Two systems being evaluated:
  - A&A/Plasma Powders (right)
  - Gehring LP (GM-developed)
- Both TNC (NanoSteel) and PRAXAIR working on a system.
- Wire may be extruded by ORNL.
- Higher ductility of SHS727 may be useful for extrusion.
- Nanosteel has produced wire in the past, but need economic incentive before supporting HPCRM team.



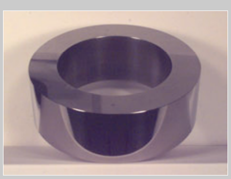
Conclusions

- Technical authority defined
- Submarine components (sail plates, brine pumps) – Bob Brown
- Pearl Harbor facility preliminary planning
- Amphibious ship components (inserts, tank hatches, anchor chain and mooring port covers)
- Fracture and damage tolerance
- Coating Process
- Quality Assurance
- Non-destructive inspection
- Wire HVOF

NRL is actively getting quality HPCRM components ready for the Fleet for real-world testing, and exposing the Fleet to the capabilities of HPCRM

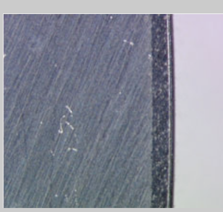
Backup Slides

Rotating Shafts and Other Naval Applications

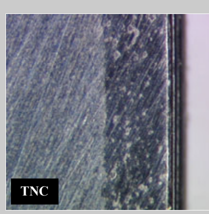


(a) Perspective view of hollow cylinder (shaft) with wear-resistant DAR-type HVOF coating having high-integrity interfacial bond on outer diameter.

(b) Plan view of hollow cylinder (shaft) with wear-resistant DAR-type HVOF coating with high integrity interfacial bond.



(c) Metallographic cross-section of wear-resistant DAR-type HVOF coating on hollow cylinder (shaft), at a magnification of 20×, showing bond of coating to the substrate.

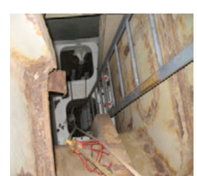


(d) Metallographic cross-section of wear-resistant DAR-type HVOF coating on hollow cylinder (shaft), at a magnification of 100×, showing bond of coating to the substrate.

HVOF SAM-Type Coating on Rotating Shaft with High-Integrity Coating-Substrate Bond

Brine Pump Applications

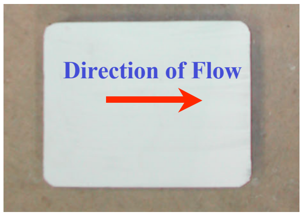
Pad Eye for Tie Down



Air Plenum

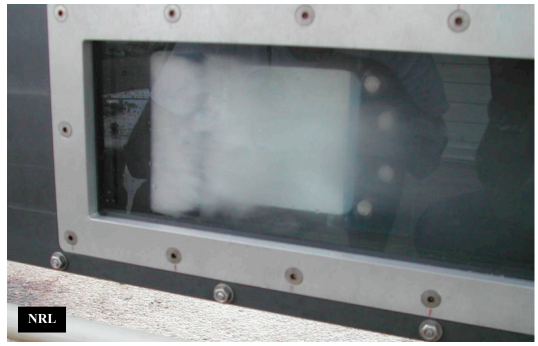
Surface Ship Landing Ship Dock (LSD) has ~ 300 on deck at a replacement cost of $1000/pad eye.

Naval Vessels – Cavitation Test with Standard Coating



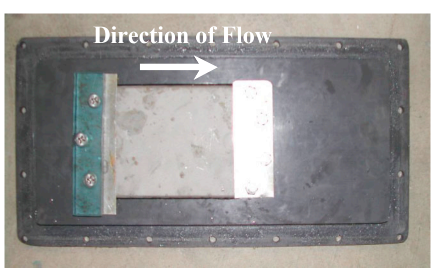
Before

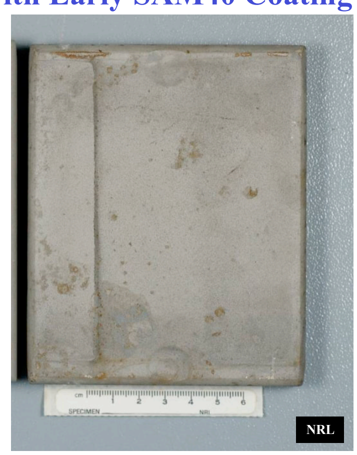
After 24-Hour Exposure



Sample in Flow Trough with Cavitation

Naval Vessels – Cavitation Test with Early SAM40 Coating





Early SAM40 Coating
After Cavitation Testing

High-Performance Corrosion-Resistant Materials for Naval Warfare & Safe Storage of Spent Nuclear Fuel

– Criticality Safety & Radiation Effects –

Jor-Shan Choi, PhD, PE
Physicist/Nuclear Engineer
Energy and Environment Directorate
Lawrence Livermore National laboratory

Presentation Outline

Criticality Safety

- Applications to Spent Fuel Support Baskets
- Issues Driven by Regulatory Considerations
- Development of New Neutron Absorbers
- Use of HPCRM-SAM as Neutron-Absorbing Material

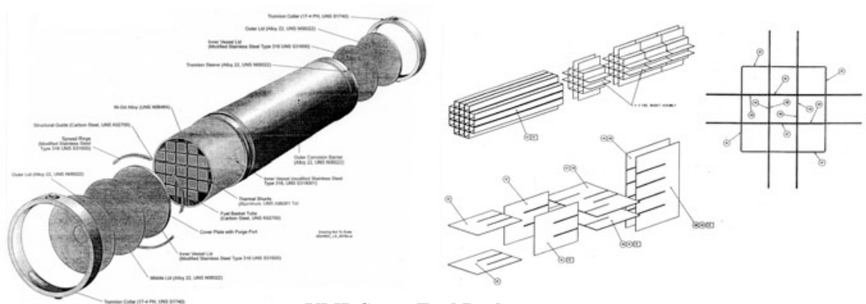
Radiation Effects

- Damage due to Neutron and Gamma Radiation
- Radiation Testing Facilities

Paths Forward

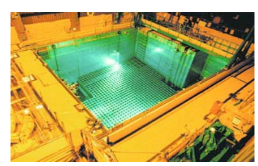
Criticality Safety - Applications to Spent Fuel Support Baskets

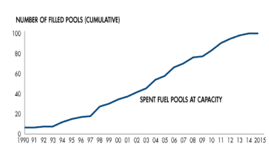
- Spent fuel contains fissionable materials (^{235}U, ^{239}Pu, ^{241}Pu, etc.)
- Criticality safety is significant in the presence of a moderator, such as during loading of spent fuel under water, or water incursion which could occur under accidents conditions in transport or in long-term storage



YMP Spent Fuel Package

Criticality Safety - Issues Driven by Regulatory Considerations





Note: All operating nuclear power reactors are storing used fuel under NRC license in spent fuel pools. Some operating nuclear reactors are using dry cask storage. Information is based on loss of full-core reserve in the spent fuel pools.

Source: Energy Resources International and DOE/RW-0431 – Revision 1

Key issues during regulatory period:

- **Loss of configuration control**
  - damaged spent fuel
  - spent fuel in prolonged storage (>20 years)
- **Higher burn-up spent fuel and burn-up credit**
  - from 35 GWD/t to 60 GWD/t
  - no burn-up credit for fission products allowed
  - confirm reactor records with burn-up measurements
- **Degradation of neutron absorbers in wet storage**
  - degraded Boraflex™ affects reactivity control, dissolved silica affects operation of interconnection plant systems
  - Boral™ blistering has been observed

The growing global and US commercial spent fuel inventories have reached 180,000 and 50,000 MTU in 2005, respectively

Development of New Neutron Absorbers

- **Metamic™**
  - A fully dense aluminum/boron carbide composite - an industrial development
- **Qualification for Metamic™**
  - hydrogen generation
  - corrosion testing
  - radiation testing
  - elevated temperature
- **Application**
  - used in Southern Nuclear's Farley Plant
  - significantly improve cask drying time
- **Ni-Gd™**
  - A Ni-Cr-Mo-Gd alloy developed by INL/SNL for USDOE's spent HEU fuel
- **Qualification for Ni-Gd™**
  - determine corrosion, mechanical and thermal neutron absorption properties
  - develop an ASTM standard
  - perform criticality experiment at LANL
- **Application**
  - intended for use as basket material in YMP waste packages

Use of HPCRM-SAM as Neutron Absorbing Material

- **Appreciable boron content in the SAM materials**:

	Atom %	Weight %
SAM2X5	15.2	3.4
SAM1651	6.0	1.3

Coating the spent fuel basket materials (e.g., borated stainless steel, Boraflex™, Boral™, Metamic™ , Ni-Gd™, etc.) with the boron-containing structural amorphous metal (SAM) would:

- **Enhance criticality safety of spent fuel in wet storage pools, in dry storage containers, and in transportation casks**
- **Prevent the preferential leaching of boron from basket materials and improve long-term criticality safety for waste package in repository**

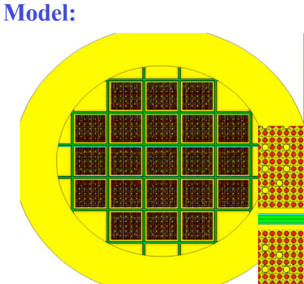
The use of SAM as a neutron absorber is evaluated

A Criticality Evaluation for a Disposal Container holding 21 PWR spent fuel assemblies

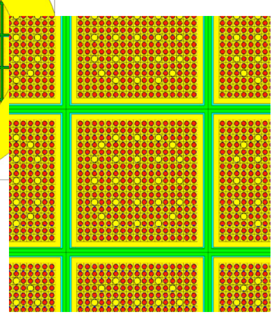
- Each 17x17 PWR assembly was exposed to 33 GWD/t and with 10-year decay
- The stainless steel support basket contains boron, varying from 0.12 to 2 wt%
- A 1 mm coating of SAM materials (SAM 2X5 or SAM1651) is applied to the stainless steel support with either no boron or 0.12 wt% of Boron
- A case with ¼" (6.4 mm) Ni-Gd™ support basket is also evaluated

Criticality Safety Analysis

Model:



Disposal Container

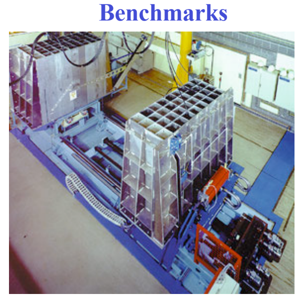


Support Basket

Computer Code:
MCNP5, COG, KENO5,

Libraries:
ENDF libraries, ENDL

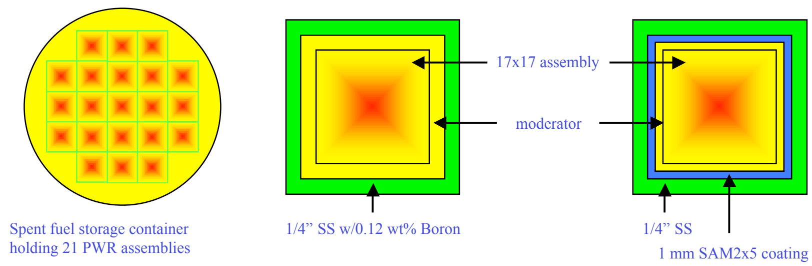
Critical Experiment Benchmarks



Validation
Bias and uncertainty
Range of applicability

Double Contingency Principle

Criticality Safety Analysis Results



	¼'' (6.4 mm) stainless steel basket								
	No Boron	0.12 wt% B	1 wt% B	2 wt% B	No Boron 1mm SAM2X5	0.12% B 1mm SAM2X5	No Boron 1mm SAM1651	0.12%B 1mm SAM1651	¼" Ni-Gd basket material
k_{eff}	0.96	0.91	0.85	0.83	0.87	0.86	0.90	0.88	0.86
del k_{eff}	0.0	0.05	0.11	0.13	0.09	0.10	0.06	0.08	0.10

Neutron attenuation effectiveness of SAM will be tested at radiation facilities

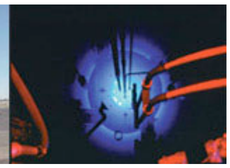
Radiation Effects

- The SAM-coated baskets and containers will be subjected to high gamma and neutron radiation from the stored radioactive materials
- Gamma radiation, with peak flux above 1000 rad/h after 10-y decay from spent fuel of 33 GWD/t can cause radiolysis of the surrounding environment, potentially altering the OCP of C-22 container and adversely affecting the corrosion properties of C-22
- Gamma flux could potentially trigger the on set of re-crystallization of SAM at a lower ambient temperature and adversely affect its corrosion properties
- Energetic neutron, with peak flux reaching 5 x 10^4 n/cm^2-s from spent fuel of 33 GWD/t can cause atomic displacement damage in SAM
- Higher burn-up spent fuel has higher neutron and gamma flux

Radiation effects on SAM will be tested at radiation facilities

Radiation Testing Facilities

Neutron Radiation

UC Davis's McClellan Nuclear Radiation Center (UCD/MNRC)

A 2-MW TRIGA™ Research Reactor

Gamma Radiation

Gamma Radiation Testing Facility at DOD's Defense Microelectronic Activity (DOD/DEMA) in McClellan

Co-60 and Cs-137 sources with exposure rates from 40 to 4000 Rad/h

Gamma Exposure Facility at LLNL's Radiation Detection Center (LLNL/RDC)

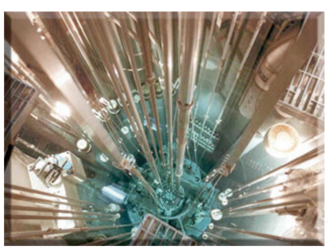
Co-60 and Cs-137 sources with exposure rates from 10mR/h to 400 Rad/h

Paths Forward

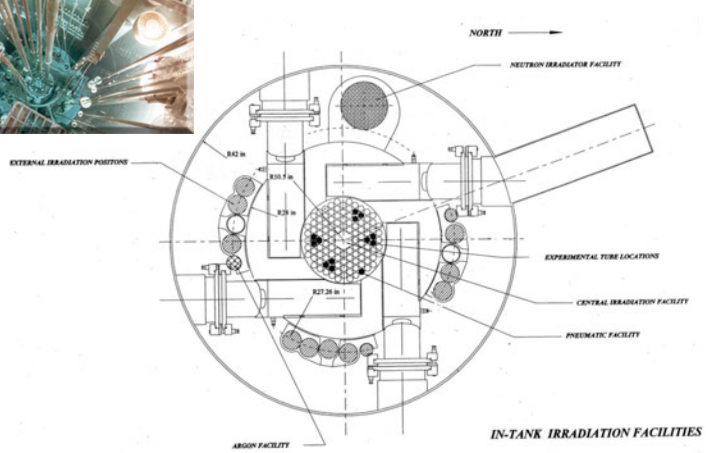
- **Radiation testing of the SAM-coated specimen at UC Davis's McClellan Nuclear Radiation Center (MNRC), and DOD's Defense Microelectronic Activity (DMEA) / LLNL's Radiation Detection Center (RDC)**
  - radiation effects, neutron and gamma
  - neutron attenuation
- **Qualification and Acceptance Testing of HPCRM-SAM**
  - service conditions and design requirements
  - qualification testing (corrosion, thermal and radiation) on durability and physical characteristics
  - neutron attenuation testing on absorber's effectiveness
  - key manufacturing processes and controls
  - acceptance testing program (statistical sampling plan, test locations, etc.)
- **Criticality safety evaluation for long-term performance of the HPCRM-SAM in a repository environment**

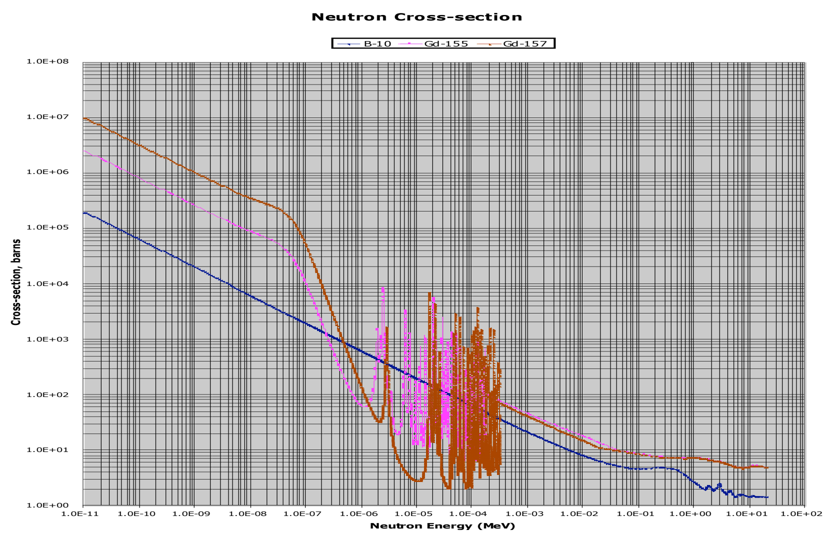
Back-up Slides

MNRC Facility



Reactor Core




Neutron Cross-section
B-10
Gd-155
Gd-157
Cross-section, barns
Neutron Energy (MeV)
1.0E+08
1.0E+07
1.0E+06
1.0E+05
1.0E+04
1.0E+03
1.0E+02
1.0E+01
1.0E+00
1.0E-11
1.0E-10
1.0E-09
1.0E-08
1.0E-07
1.0E-06
1.0E-05
1.0E-04
1.0E-03
1.0E-02
1.0E-01
1.0E+00
1.0E+01
1.0E+02

High-Performance Corrosion-Resistant Materials: Process Optimization Studies

Leonardo Ajdelsztajn, Jon Dannenberg, Enrique Lavernia & Julie Schoenung
University of California, Davis
Davis, California

Optimization of Atomization & Thermal Spray Processes

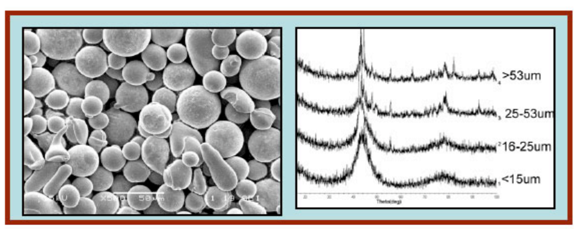
- **Improve Powder Atomization**
  - Suitable powder morphology (improved feeding and flight behavior)
  - Narrow particle size distribution (homogeneous and uniform splatting)
  - Maintain amorphous structure (low critical cooling rate)
- **HVOF Spray Conditions**
  - Control surface and substrate temperature during spray (management of residual stress).
  - Optimize flame and spray conditions for minimal oxidation.
  - Maintain appropriate temperature and velocities for minimum porosity.
  - Minimize the devitrificaion process (CCR and temperature during spray).

Optimization of HVOF Amorphous Metal Coatings

- **Powder**
  - Powder particle size
  - Powder morphology
  - Powder composition
  - Critical cooling rate
- **Spray Conditions**
  - Flame conditions
  - Nozzle geometry
  - Spray distance
  - Spray gun transversal speed
  - Feeding rate
  - Cooling of the sample during spray

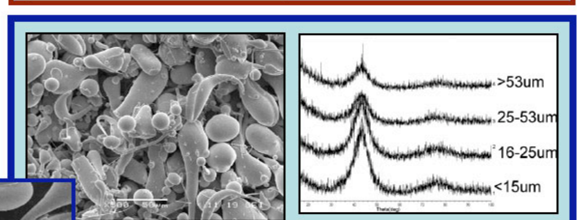
SAM2x5

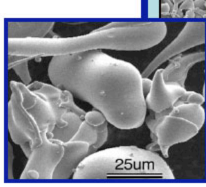
Large powders devitrified



SAM1651

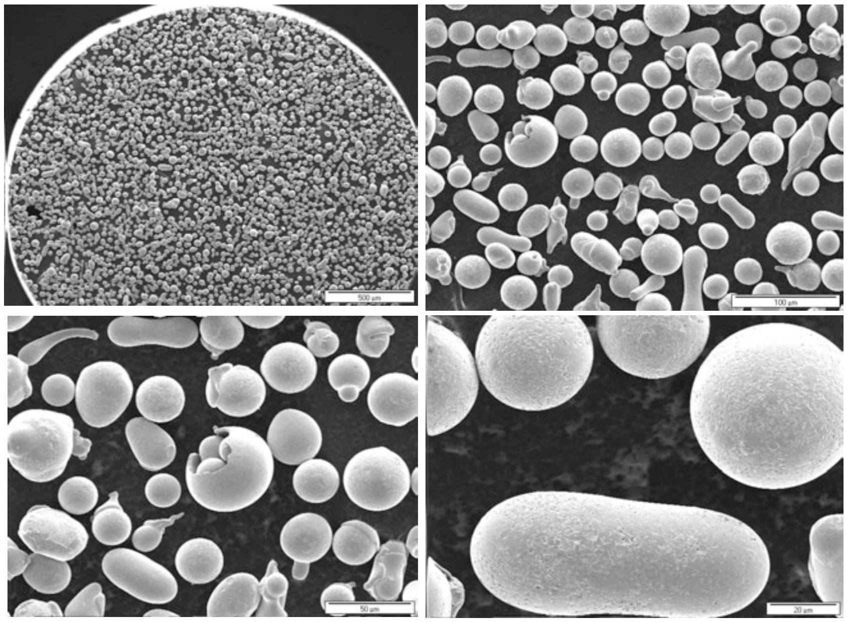
Amorphous structure for all sizes



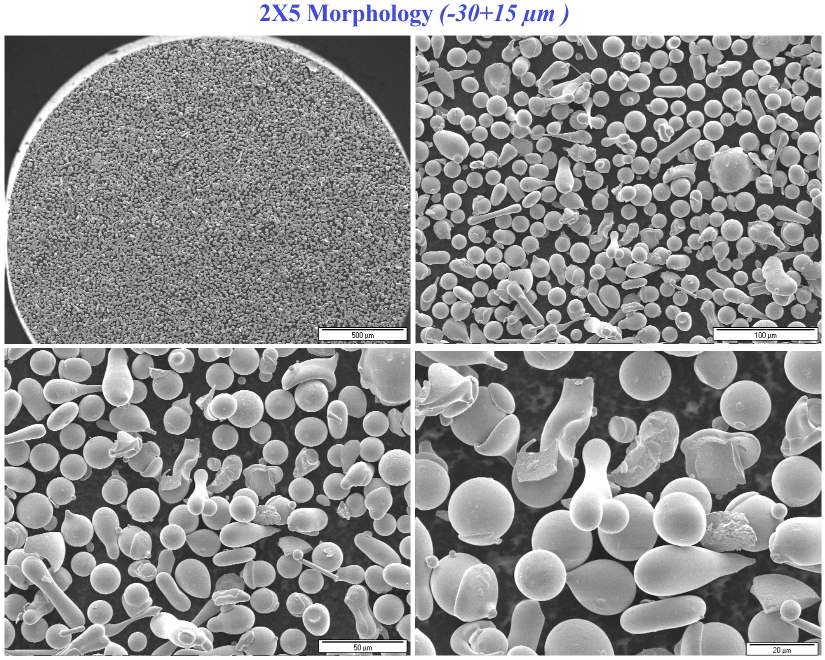


Irregular shaped SAM 1651 powders often exhibit multiple layers

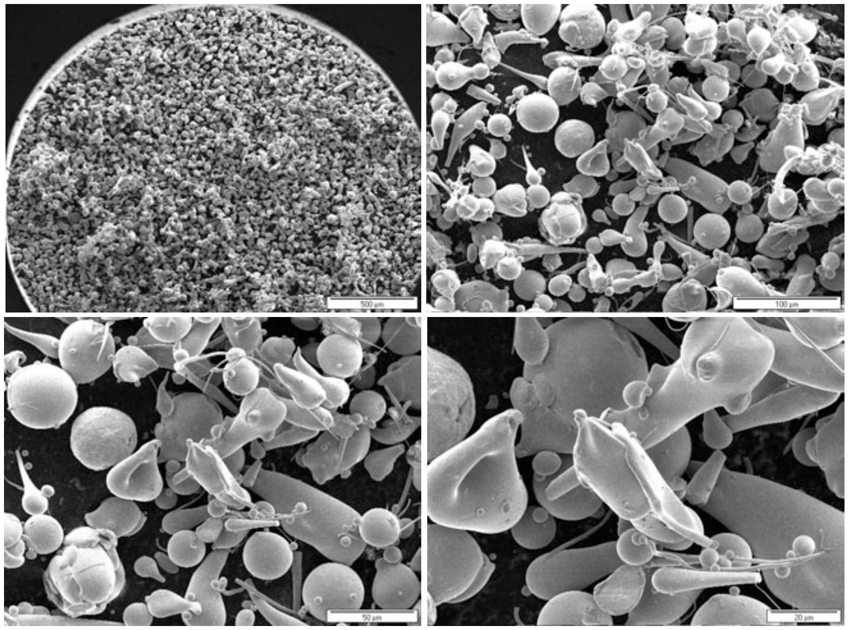
2X5 Morphology (*-53+30 µm)*



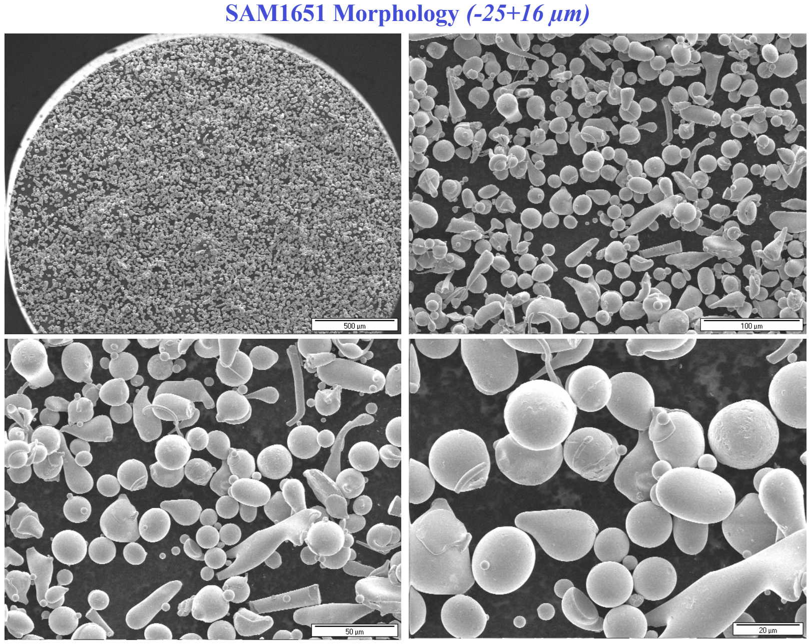
2X5 Morphology *(-30+15 µm)*



SAM1651 Morphology (*-53+25 µm)*

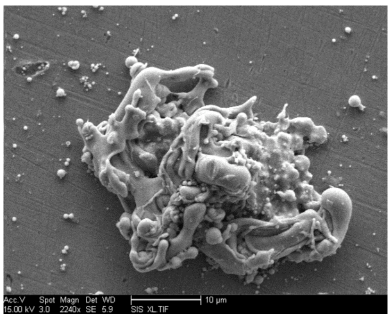


SAM1651 Morphology *(-25+16 µm)*

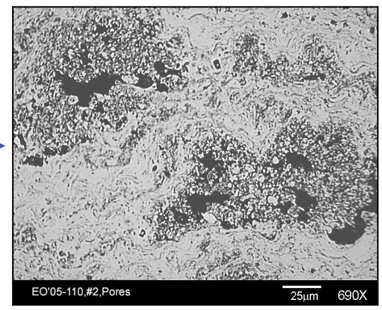


Why powder morphology is important?

- **Flowability – Maintain stable feeding rates during spray**
- **Particle behavior during flight affects coating microstructure:**
  - **Powder velocity and temperature**
  - **Surface area - oxidation**
  - **Agglomeration during flight due to irregular morphology (fine ligaments)**



Single splat analysis (agglomeration during flight, severe oxidation of the fine ligaments)



Oxidation pockets in the coating due to particle agglomeration

How to improve powder morphology?

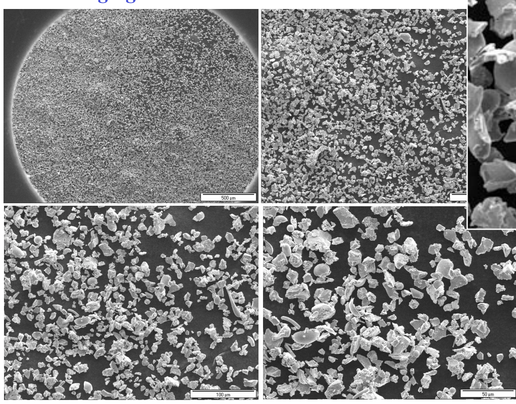
- **Mechanical Milling of Powder (cryogenic/room temperatures)**
  - **Cryomilling (UCD facilities)**
  - **Improved morphology (preliminary results)**

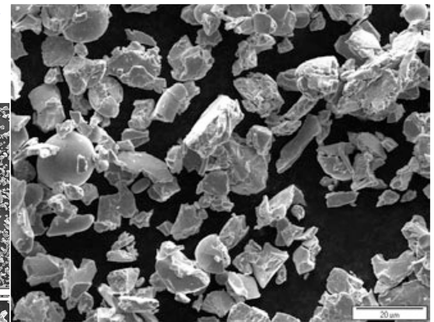
- **Optimization of the Atomization Process**
  - **UCD facilities and experience**

Mechanical Milling

Test run was conducted using 1196 g of SAM1651 -53+25 powder,
30 kg of 0.25” stainless steel balls.
The run was conducted under liquid nitrogen for 45 minutes with an armature speed of 180 rpm, producing a final yield of 1132 g of milled powder.

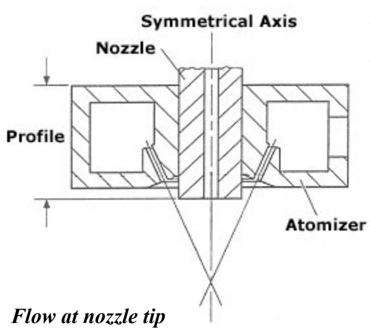
“rocky” morphology, with few remaining ligaments

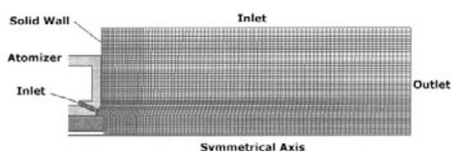


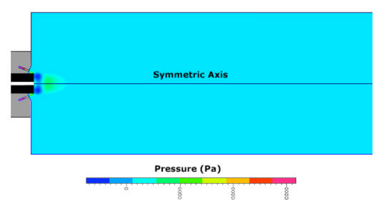


- **Homogeneous Distribution**
- **Refinement of Particles Size**
  - **Spray parameters need to be adjusted**
- **High Yield**
- ***Stable feeding during spray***

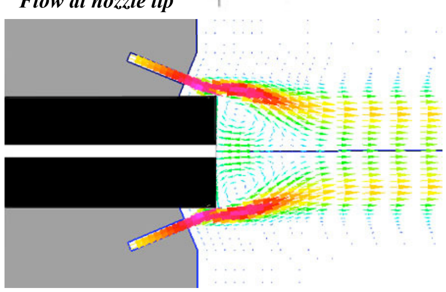
Optimization of the Atomization Process

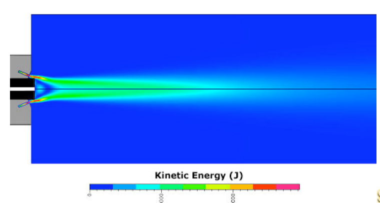






Flow at nozzle tip





Optimization of the Atomization process

$$d_{50} = k_d \left[\frac{\eta_m d_0 \sigma_m}{\eta_g v_{ge}^2 \rho_m} \left(1 + \frac{J_{melt}}{J_{gas}} \right) \right]^{1/2}$$

k_d is an empirically determined coefficient whose value ranges between 40 and 400.
k_d takes into consideration the parameters which cannot be readily incorporated into the calculation, such as the geometry of the atomizer and nozzle. d_0 is the diameter of the melt stream;
η_m is the kinematic viscosity; σ_m is the surface tension; ρ_m is the density;
and J_{melt} is the mass flow rate of the melt.
η_g, v_{ge}, and J_{gas} are the kinematic viscosity, velocity, and the mass flow rate of the atomization gas.

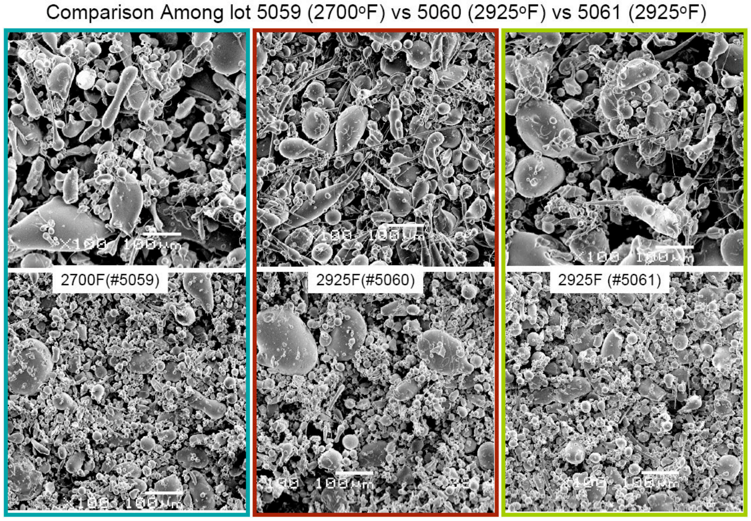
$$t_{sph} = \frac{3\pi^2 \eta_1}{4 V_{lig} \sigma_1} \left(r^4 - r_{lig}^4 \right)$$

t_{sph} is the spheroidization time; η_1 and σ_1 are viscosity and surface tension of the liquid; V_{lig} is the volume of the ligament; r_{lig} and r are the diameters of the ligament prior to and after spheroidization, respectively.

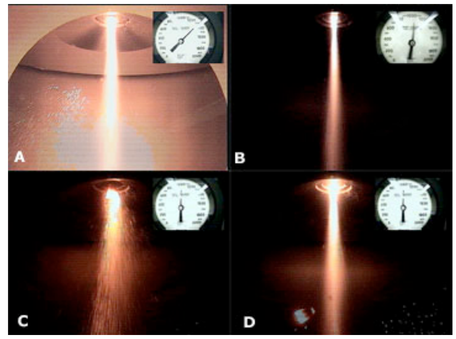
Viscosity will play a significant role on the particle size and morphology.
Atomization should be conducted considering the highly viscous behavior of the 1651 alloy.

Superheating Alone
Does Not Improve Particle Morphology (ORNL)

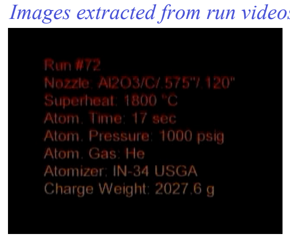
Comparison Among lot 5059 (2700°F) vs 5060 (2925°F) vs 5061 (2925°F)



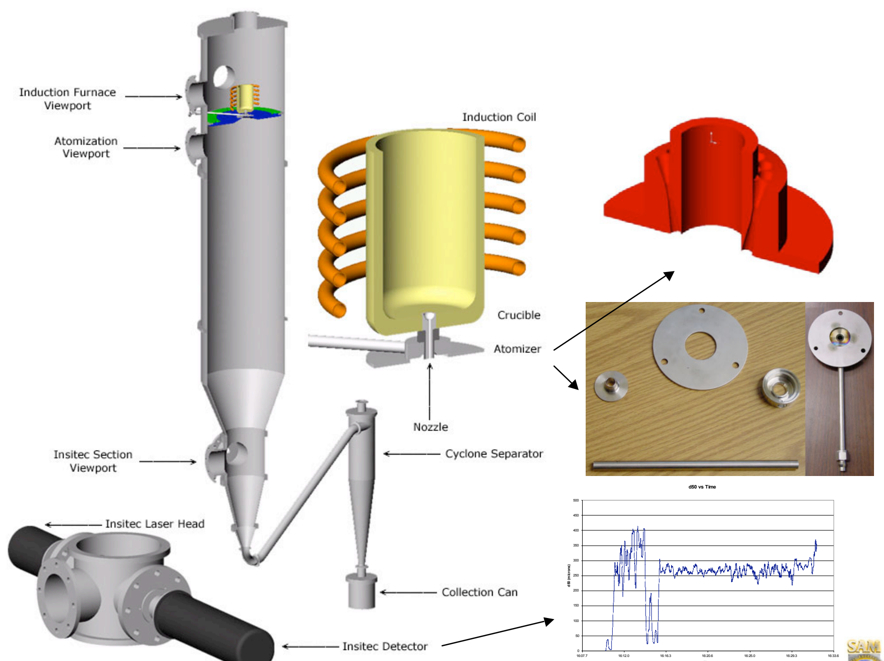
University of California Davis (UCD) Facilities



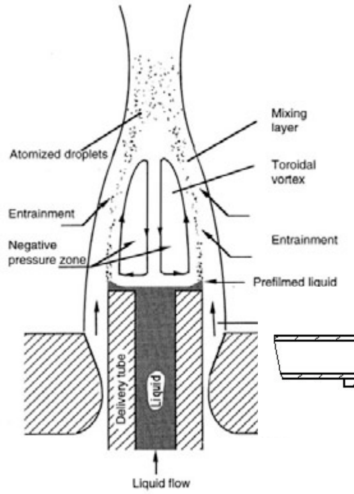
Images extracted from run videos



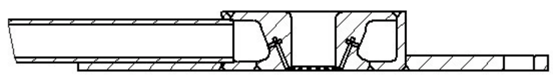
Atomization facility at UCD



Atomization Methods



- Sonic
- Supersonic
- UCD (improve efficiency)

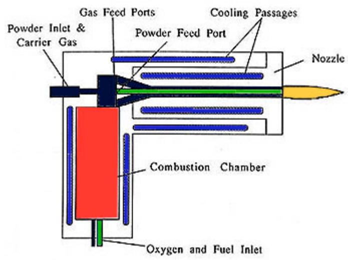


UCD Atomizer

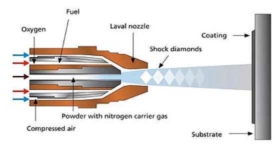
HVOF Spray Conditions

- Different guns
  - nozzle geometry and length
- Flame conditions
  - different fuel (H_2 and C_3H_6)
  - oxygen/fuel ratio
- Spray distance
- Spray gun transverse speed
- Feeding rate
- Cooling of the sample during spray

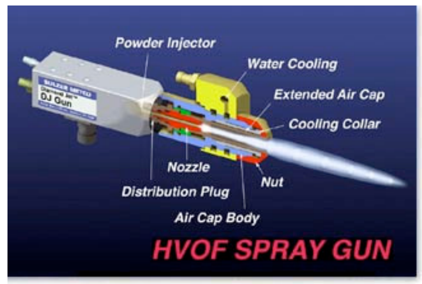
Most Popular HVOF Systems



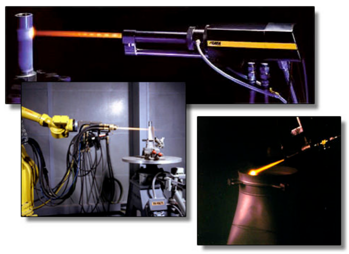
Jet Kote



*DJ2600 (air cooled nozzle)

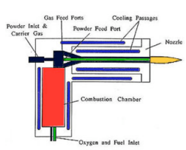


*DJ2700 (water cooled nozzle)

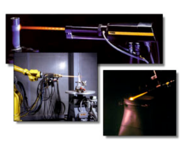


JP 5000

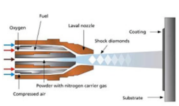
* Available at UCD



Jet Kote gun – Combustion chamber (combustion take place before the powder injection). Long nozzle and duel time (9''). Fuel: Hydrogen

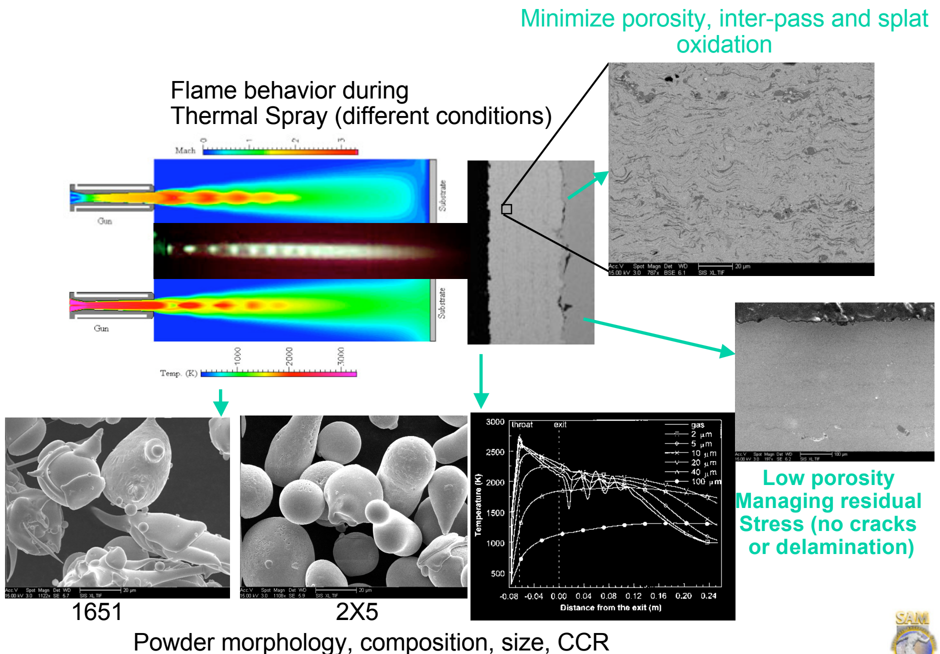


JP 5000 – High velocity and moderate temperatures.
Superior velocities compared to Jet Kote and DJ Sulzer Metco.
Fuel: Liquid, Kerosene



Diamond Jet 2600 (DJ2600) Sulzer Metco.
Air cooled nozzle. Short nozzle and duel time.
Ideal for fine powder (less oxidation and reasonable velocities)

Diamond Jet 2700 (DJ2700) Sulzer Metco.
Water cooled nozzle. Intermediary nozzle length.
Fuel: Propylene, Propane, Natural Gas, Hydrogen.
Good combination of temperature and velocities.



Numerical Modeling

- **Mass:** $\frac{\partial \rho}{\partial t} + \frac{\partial}{\partial x_j}(\rho u_j) = 0$
- **Momentum:** $\frac{\partial}{\partial t}(\rho u_i) + \frac{\partial}{\partial_j}(\rho u_i u_j) = -\frac{\partial p}{\partial x_i} + \frac{\partial}{\partial x_j}\left[\mu\left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i} - \frac{2}{3}\frac{u_k}{\partial x_k}\delta_{ij}\right)\right]$
- **Energy:** $\frac{\partial}{\partial t}(\rho H) + \frac{\partial}{\partial x_j}(\rho u_i H) =$

$$\frac{\partial}{\partial x_j}\left(k\frac{\partial T}{\partial x_j}\right) + \frac{\partial p}{\partial t} + \frac{\partial}{\partial x_j}\left[\mu\left(\frac{\partial u_i}{\partial x_j} + \frac{\partial u_j}{\partial x_i} - \frac{2}{3}\frac{\partial u_k}{\partial x_k}\delta_{ij}\right)\right] - \frac{\partial}{\partial x_j}(J_{ij}h_i) + S_a$$

- **Favre (or density) approach is used to average conservation equations.**
- **RNG κ-ε model is applied to estimating turbulent eddy viscosity.**
- **Semi-empirical wall functions are used in cells adjacent to walls because RNG κ-ε model is high Reynolds model and is not intended to be used in near-wall regions.**

Momentum Transfer

Motion of a particle is governed by:

$$m_p \frac{dV_p}{dt} = \frac{1}{2}\rho A_p C_{drag}(V - V_p)\left|V - V_p\right| - V_p \nabla p$$

where C_{drag} is determined by using the following drag correlation for non-spherical particles:

$$\frac{C_D}{K_2} = \frac{24}{\mathrm{Re}\, K_1 K_2}\left[1 + 0.1118(\mathrm{Re}\, K_1 K_2)^{0.6567}\right] + \frac{0.4305}{1 + \frac{3305}{\mathrm{Re}\, K_1 K_2}}$$

where C_D and Re are based on the equal volume sphere diameter, and K_1 and K_2 are unique functions of particle sphericity.

Heat Transfer

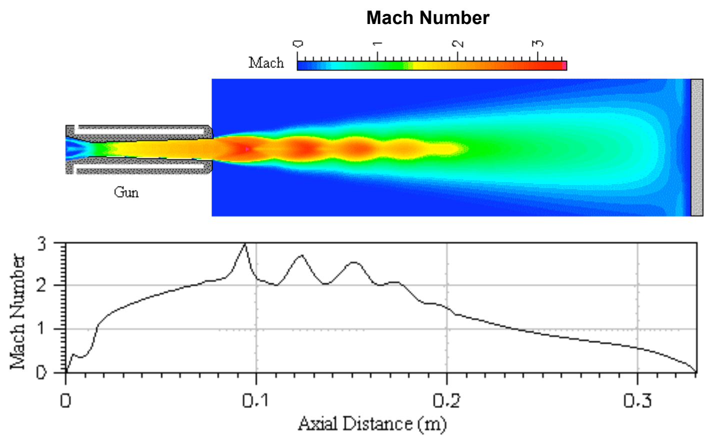
Newtonian cooling is assumed for cooling process of particles.
By applying energy conservation to a particle:

$$m_p c_p \frac{dT_p}{dt} + \Delta H_m \frac{dm_p}{dt} = hA_p(T - T_p)$$

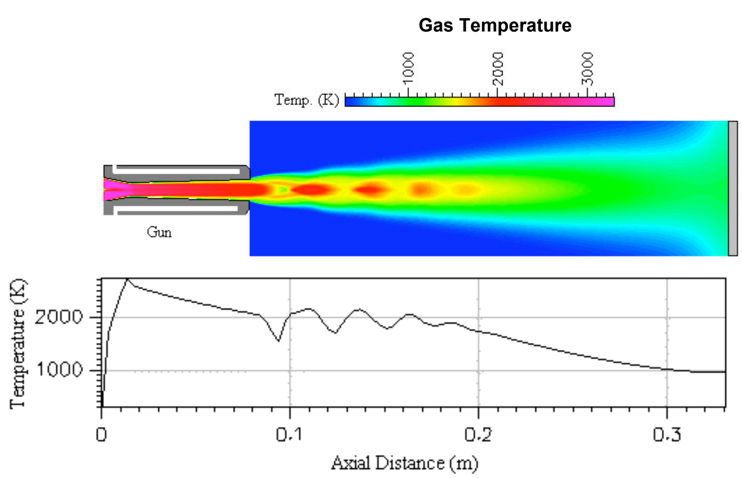
where surface heat transfer coefficient, h, is determined by:

$$Nu = 2 + 0.6\sqrt{\mathrm{Re}}\sqrt[3]{\mathrm{Pr}}$$

Flame Behavior



Flame Behavior



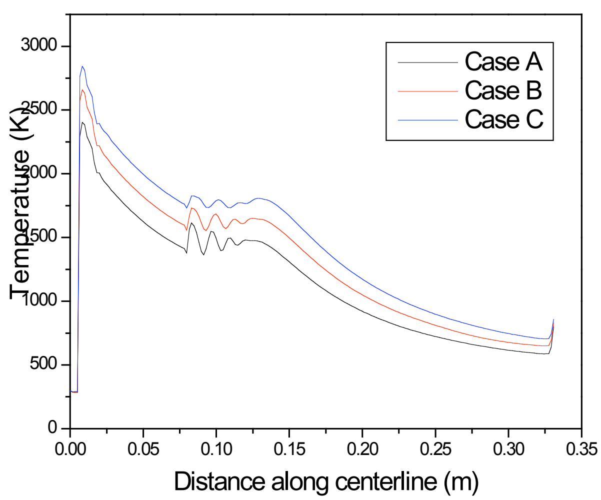
Spray Conditions

Flame conditions for the DJ 2701 gun (water cooled nozzle)

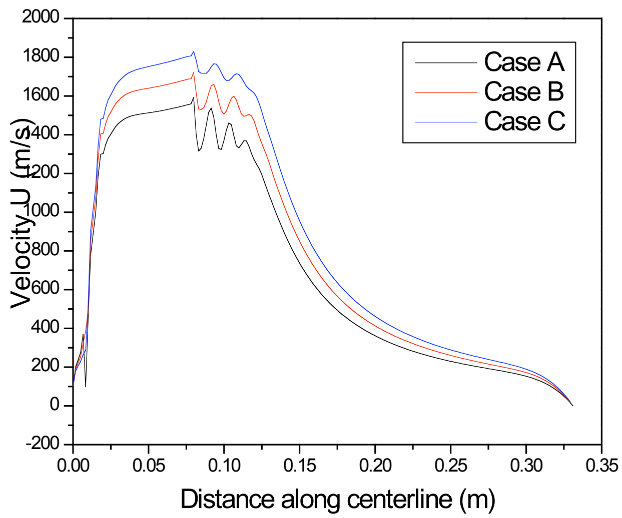
Case	Oxygen		Propylene		Air	
	Mass flow rate (SCFH)	Pressure (psi)	Mass flow rate (SCFH)	Pressure (psi)	Mass flow rate (SCFH)	Pressure (psi)
A	578	150	144	105	857	100
B	578	150	180	105	857	100
C	578	150	216	105	857	100

Spray distance: 8 inches.
Gun transverse speed: 1m/s
Powder feeding rate: 6 lb/hr

Gas Temperatures



Gas Velocities

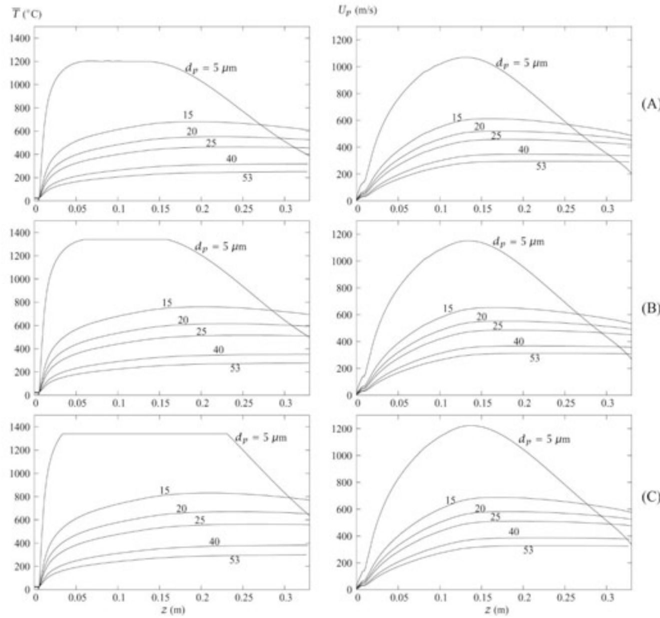


Material Properties Used in Numerical Model

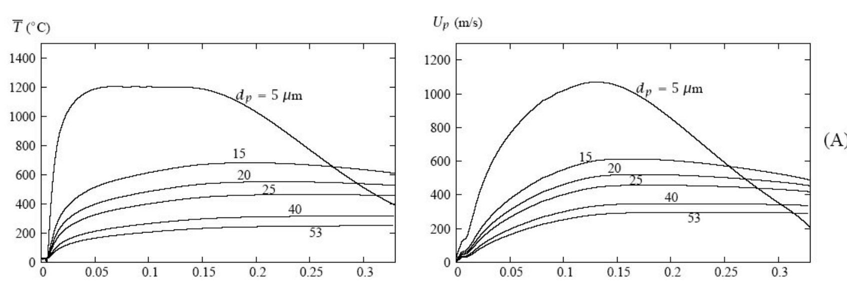
Density: 7500 kg m^{-3}
Melting Temperature: 1610 K
Latent Heat of Fusion: 2.47×10^5 J/kg
Specific Heat: 680.5 J/kg/K
Thermal Conductivity: 40.0 W/m/K

- **Properties should be checked and changed accordingly to each alloy (SAM2X5 and SAM1651)**
- **Model can be adjusted for better accuracy on predicting particle behavior during flight.**

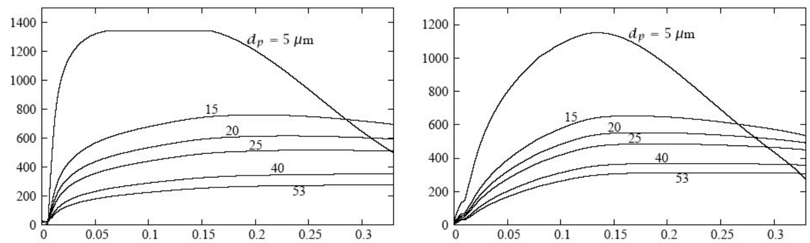
Particles Behavior During Spray (SAM1651)



Particles Behavior During Spray (SAM1651)

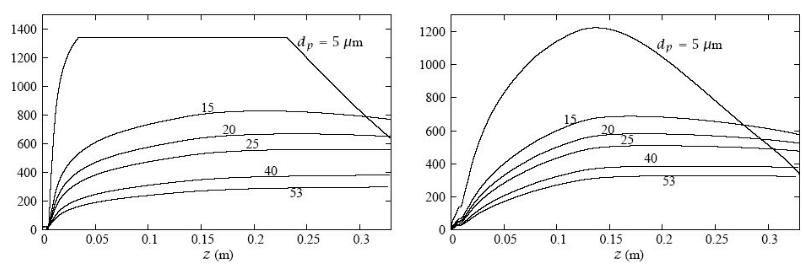


Particles Behavior During Spray (SAM1651)



(B)

Particles Behavior During Spray (SAM1651)

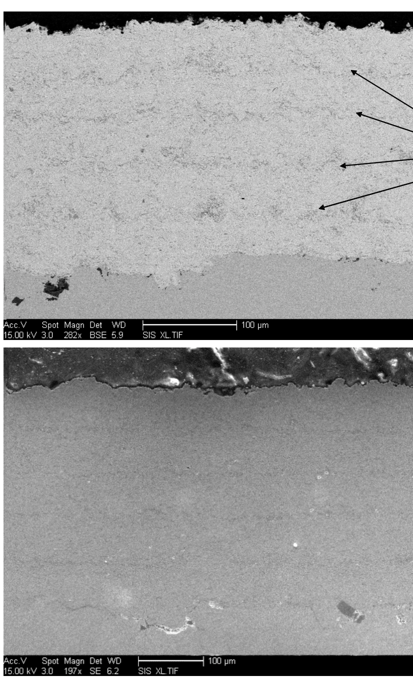


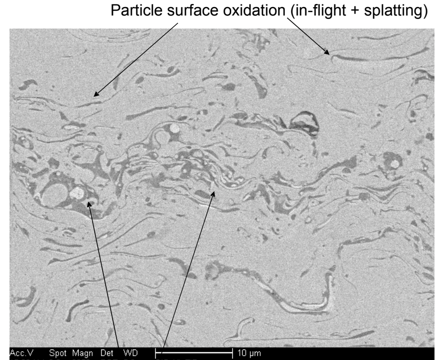
(C)

Major Conclusions Based on Numerical Modeling

- **Fine particles (< 25 microns) will impact the substrate at temperatures close to or above the Tg.**
- **Impacting at this range of temperatures will promote extensive deformation of the particles resulting in very dense coatings.**
- **These results show that if a temperature close to the Tg is achieved for large particles before impacting the substrate, high density coatings can be obtained.**
- **Smaller particles will achieve even higher temperatures which will lead to dense coatings but will also increase oxidation during flight.**
- **Melting was not achieved in any conditions tested in this work.**

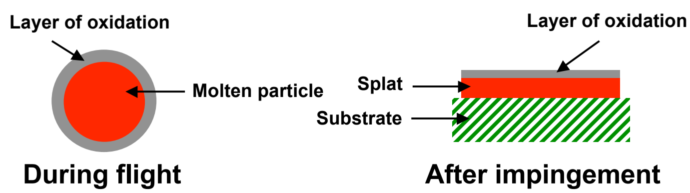
Splat vs. Inter-pass Oxidation





Oxidation During Flight

- **Oxidation Kinetics**
  - **Presence of oxygen in entrained air**
  - **Excess oxygen in products of combustion during HVOF spraying**
  - **Molten or semi-molten particles can be oxidized during flight and after impingement onto the substrate surface**



Oxidation During Flight

- The mass of oxidation layer developed m_{ox} can be presented in a form:

  $m_{ox} = q_{ms} S_p t_{ox}$

- Where, q_{ms} is the mass flux of oxide, S_p is the surface area of the particle subjected to oxidation and t_{ox} is the characteristic time of oxidation. The ratio Z of m_{ox} to particle mass $m_p = 4\pi R_p^3 \rho/3$ is as follows:

  $Z = 3\, q_{ms}\, t_{ox}\, (\rho R_p)^{-1}$

- The value of Z may be considered as the relative mass of oxidation giving the level of oxidation. The thickness δ_{ox} of the oxidized layer which is equal to the difference between particle radius R_p and the radius of the interior boundary of the oxidized region R_{ox} can be presented as:

  $\delta_{ox} = R_p\, [1-(1-Z\rho\rho_{ox}^{-1})^{1/3}]$

- where, ρ_{ox} is the oxide density. When $Z<<1$ the thickness δ_{ox} can be estimated by:

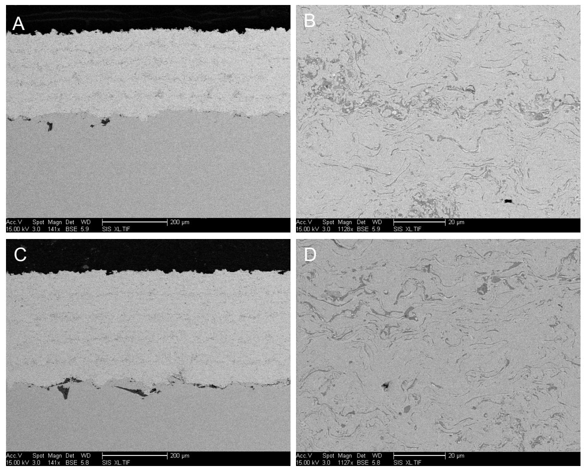
  $\delta_{ox} = Z\rho R_p\, (3\rho_{ox})^{-1}$

- Considering the case of Fe coatings, the oxide mass flux q_{ms} can be taken as 3.6 kg/(m_2s) (oxidized Fe droplet [1], time of oxidation t_{ox} can approximately 1ms [1], ρ= 7874kgm^{-3}, ρ_{ox} = 5210 kgm^{-3} and R_p = 20 μm, we obtain Z= 0.072 (7.2%) and δ_{ox} = 716 nm. When R_p = 10 μm, Z= 0.144 (14.4%) and δ_{ox} = 745 nm.

So when spraying larger particles size lower level of oxidation Z and thinner oxidation layer thickness δ_{ox} will be observed. When increasing the particle size from 10 to 40 μm the volume fraction of oxidation can vary from 22 to 5%.

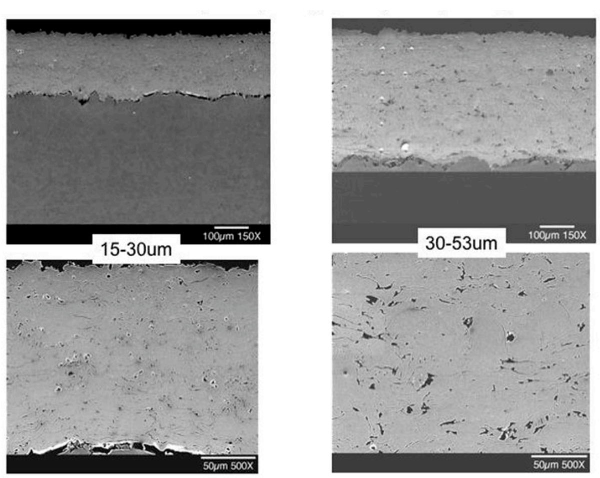
Reference: A. VArdelle, P. Fauchais and N.J. Themelis: Advances in Thermal Spray Science and Technology, CC. Berndt and S.Sampath, eds., ASM International, Materials Park, OH, USA, 1995, pp. 175-180.

Spray Distance (SAM1651)



Slightly lower inter-pass oxidation, without compromising coating density,
when the spray distance is increased from (A and B) 8.5 inches to (C and D) 10 inches.
Results corroborate numerical modeling

Nozzle Geometry vs. Particle Size (SAM1651)

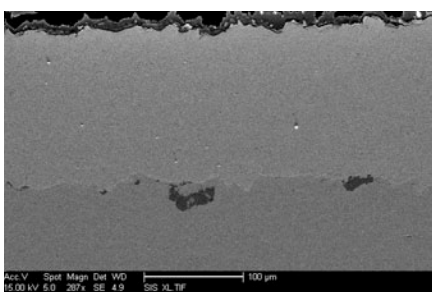


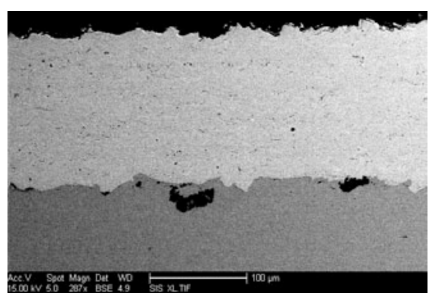
SAM1651 coatings sprayed at UCD with the air-cooled H2 nozzle.

Nozzle Geometry vs. Particle Size (SAM1651)

- The air-cooled H_2 nozzle used here has a diversion section one order of magnitude shorter that the water-cooled nozzle, which will lead to a shorter dwell time and consequently lower particle temperatures and velocities.
- The air-cooled H_2 nozzle is well suited for the finer particles size distribution as seen in the porosity level of the coating.
- Longer water-cooled nozzle is better suited for the larger particles (long dwell time – higher temperatures and velocities). Nevertheless, all the coatings sprayed with the air-cooled nozzle presented very limited oxidation and there is no evidence of undesired interconnected porosity.

SAM2X5 Coating Sprayed with H_2 Jet Kote Gun

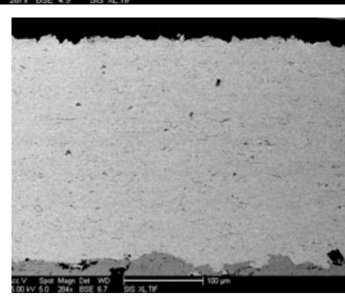




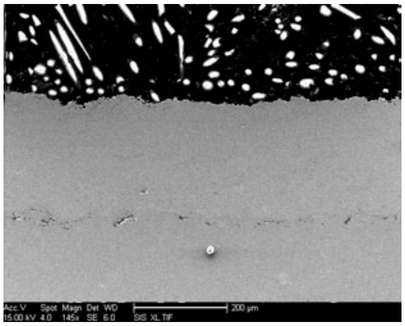
Gun: JK-2000
nozzle: 1/4" x 9"
Spray distance: 9.5"
Powder feeder: 3 RPM (Praxair powder feeder)
Gun speed 180 ft/sec
5 passes

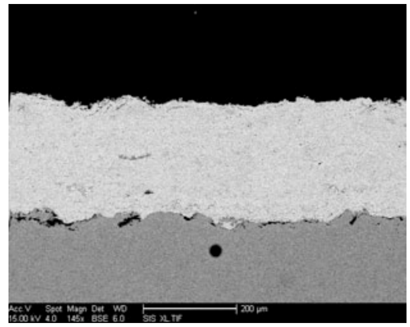
High density
Low oxidation content
No cracking

	Oxygen		Hydrogen	
	FMR*	psi	FMR*	psi
	86	120	40	120



SAM2X5 *(-53+30 µm)* Sprayed with C_3H_6 at UCD





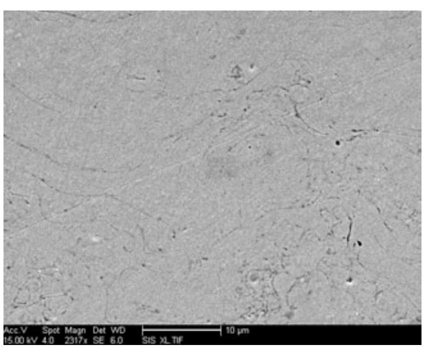
Powder size: (30-53 microns)

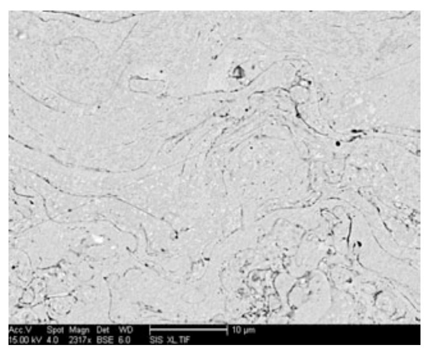
Condition	Oxygen		Propylene		Air	
	FMR*	psi	FMR*	psi	FMR*	psi
B	40	150	40	105	48	100

*Flow Meter Reading
8 passes
Metco DJ2701 - water cooled propylene
Spray distance: 9 inches.
Gun speed 300 ft/sec
Powder feeding rate: 5.5 lb/hr

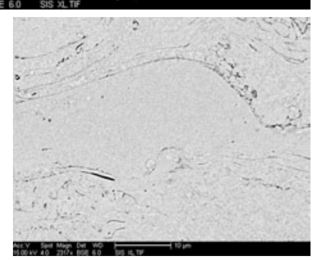
Same condition used for the numerical simulation – flame condition (B)

SAM2X5 *(-53+30 µm)* Sprayed with C_3H_6 at UCD





- **Particle temperature close to T_g**
- **No cracks**
- **Low porosity**
- **Low oxidation**



Summary (1)

- **Numerous spray conditions were tested in this work to study the formation of amorphous coating using HVOF thermal spray techniques.**
- **Due to different critical cooling rate (CCR), different powder composition will promote different degrees of devitrification of the amorphous powder during spraying and resulting in different coating microstructures. However, larger particles sizes *(-53+25 μm)* are preferable in terms of in-flight oxidation (provided that density is not compromised, in other words, optimum spray conditions are used). The SAM2X5 powder presented a better powder morphology (more suited for thermal spray) when compared to the morphology of the SAM1651 powder.**

Summary (2)

- **The SAM1651 fine particle distribution (16-25 *μm*) did not present any feeding problem in the new powder feeder system installed at UCD. The large powder size distribution (25-53 *μm*) was very unstable during feeding and it is clear that more emphasis should be devoted to the optimization of the powder atomization process in order to produce amorphous structure and better suited powder morphology for thermal spray.**
- **Preliminary test on the coarse SAM1651 powder have shown that cryomilling for a short period of time (45 min) promoted a dramatic change in powder size distribution and morphology, improving the powder flowability during spray. Spray conditions need to be adjusted for the new powder size and morphology.**

Summary (3)

- First test runs of the atomization experiments on the SAM1651 powder are currently being conducted at UCD facilities.

Backup Slides

High-Performance Corrosion-Resistant Materials: SAM40 Modified Alloys

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Idaho Falls, Idaho

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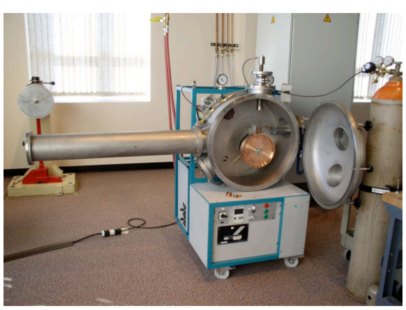
Table of Contents

Design of Experiment

- Broken-up into two areas; intrinsic / extrinsic studies
- Alloy Design Studies to Optimize Intrinsic Properties
- Properties related to the composition /microstructure
  - Alloy Modifications to SAM40
  - Alloy Modification Based on Modeling
- HVOF Studies to Optimize Coating Density
  - Spray studies focused on coating macrostructure
  - 3rd Generation HVOF Studies with SAM2X5
  - 4th Generation XVHVOF Studies with SAM40

Alloy Design Approach: Melt-Spinning

- The overriding advantage is that almost perfect 100% amorphous structures can be obtained which contain essentially no defects and these structures can be devitrified to form 100% nanocomposite structures
- Note that this is key since these 'ideal structures' establish a beacon for alloy design and development



Alloy Design

- Alloy design in Phase 1 focused on modifying the existing SAM40 stoichiometry since the coatings
- Appear to be close to exhibiting necessary damage tolerance:
  - Bond strength: > 13,000 psi
  - Impact resistance: ability to survive drop impact energy of at least 480 inch/lbs
  - Wear and erosion resistant: hardness > 1000 kg/mm^2
  - Corrosion resistance: roughly equivalent to a 316L but inferior to C-22

SAM Series Alloys Phase 1

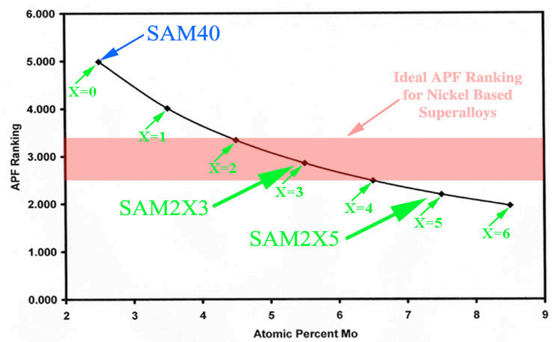
Alloy Designation	Stoichiometry (Y)	Additions (x)
SAM40	$Fe_{52.3}Mn_2Cr_{19}Mo_{2.5}W_{1.7}B_{16}C_4Si_{2.5}$	0
SAM1	$(DAR40)_{100-x} + Ni_x$	1,3,5,7
SAM2	$(DAR40)_{100-x} + Mo_x$	1,3,5,7
SAM3	$(DAR40)_{100-x} + Y_x$	1,3,5,7
SAM4	$(DAR40)_{100-x} + Ti_x$	1,3,5,7
SAM5	$(DAR40)_{100-x} + Zr_x$	1,3,5,7

To show the influence of key elements (Ni, Mo, Y, Ti and Zr) on alloying behavior, processability, hardness,glass forming ability, and corrosion

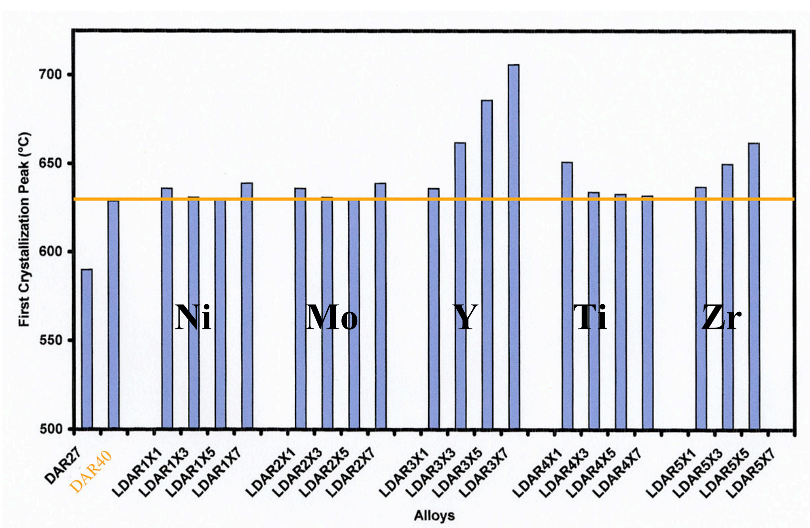
LDAR2 Series History – APF Ranking Factors

Concept developed for nickel-based superalloys

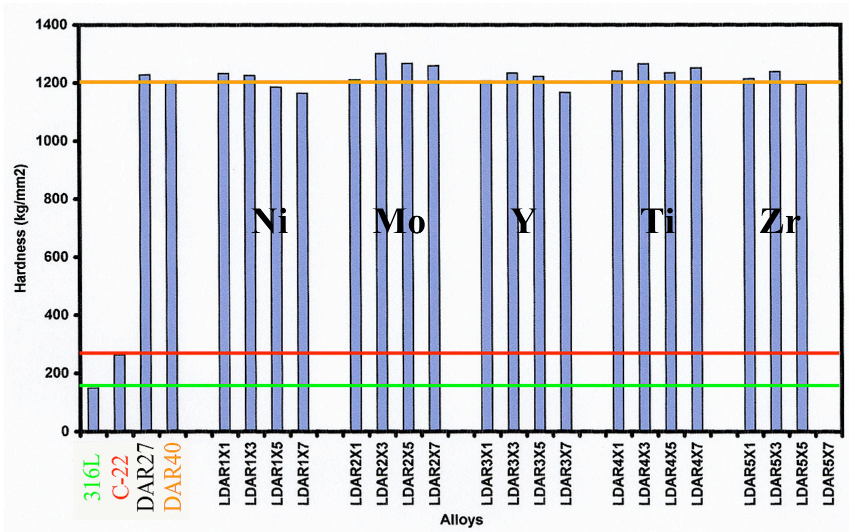
- At the start of the program, it was believed to be an important concept for iron based systems
  - Especially useful for determining optimum Mo, W, and Cr ratios
- $(DAR40)_{100-x} + Mo_x$ where DAR40 has the stoichiometry of; $Fe_{52.3}Mn_2Cr_{19}Mo_{2.5}W_{1.7}B_{16}C_4Si_{2.5}$



DTA Analysis
First Crystallization Peak



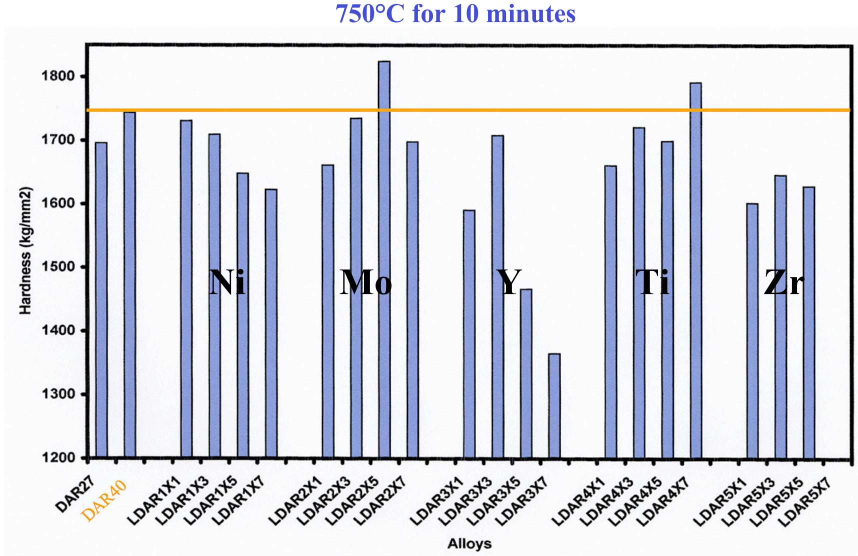
As-Solidified Hardness (Glass)



Note hardness of conventional materials (red and green lines)

Devitrified Nanocomposite Hardness

750°C for 10 minutes



SAM2X5 HVOF Spray Parameter Development

- Commercial atomization run of SAM2X5
- Air classified to yield three powder cuts
  +15 to -53 µm (conventional sizing cut for HVOF)
- +15 to -30 µm and +30 to -53 µm cuts
- Used a proprietary approach which we have developed for commercial alloys to find the optimum spray parameters
- Using the Tafa JP5000 gun system, 9 spray conditions were sprayed
- Focus was on achieving high coating density

SAM2X5 HVOF Spray Parameter Development

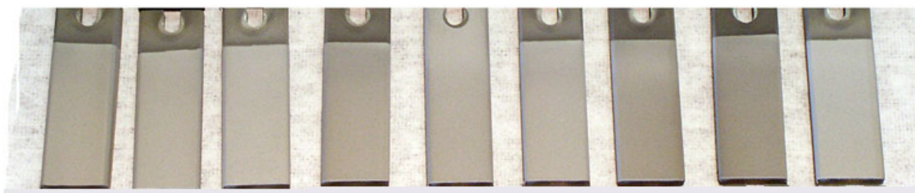
Utilize existing 3rd generation HVOF technology with no changes to gun design and with parameter sets which are within the range of existing commercial spray operations to allow rapid transition to military depots

Experimental Procedure	Parameter Used
HVOF Gun	Tafa JP5000
Barrel Length	4”
Combustion Feedstock	Kerosene / Oxygen
Powder Feeder	Miller / Praxair model 1270
Powder Feed RPM	4 with a 6 slm argon carrier gas
Coupon Stand-Off	350 mm
Coating Thickness	Nominally 15 mils (375 µm)
Combustion Pressure	Variable (80-100 psig)
Stoichiometry Ratio	Variable (105-125%)

SAM2X5 HVOF Spray Parameter Development

Combustion Pressure (psig)	Stoichiometric Ratio		
	105%	115%	125%
80	HV1	HV2	HV3
90	HV4	HV5	HV6
100	HV7	HV8	HV9

HV1 HV2 HV3 HV4 HV5 HV6 HV7 HV8 HV9

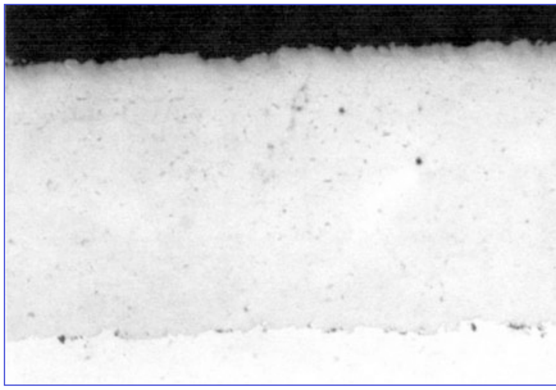


Spray Optimization Results – SAM2X5 Hardness

Hardness (kg/mm^2)	HV1	HV2	HV3	HV4	HV5	HV6	HV7	HV8	HV9
HV100	1032	926	1022	1203	1228	1063	1166	1161	1250
HV100	1016	932	1183	1081	957	1189	1108	1121	1138
HV100	1177	1151	1170	1094	1124	1194	1204	1161	1010
HV100	1093	1208	1137	1086	864	1065	1104	1153	1174
HV100	955	1206	1002	1206	1261	1228	1141	1189	1154
Average	1055	1085	1103	1134	1087	1148	1145	1157	1145
HV300	993	949	981	1015	1029	1076	1014	1066	1005
HV300	950	934	1000	1047	980	939	1141	1095	1100
HV300	893	1116	920	1062	990	1109	1062	1074	1054
HV300	1006	957	839	981	983	914	1044	1073	1001
HV300	1074	910	1012	1093	895	972	1052	1085	1088
Average	983	973	950	1040	975	1002	1063	1079	1050

Spray Optimization Results – SAM2X5 Density

Coating Density	HV1	HV2	HV3	HV4	HV5	HV6	HV7	HV8	HV9
Porosity (%)	1.46	1.54	0.96	0.32	1.05	2.50	5.09	2.07	3.49
Coating Density (%)	98.54	98.46	99.04	99.68	98.95	97.50	94.91	97.93	96.51



SAM2X5
HV4 Spray Condition
99.7% Dense

<<< Coating / Substrate Interface

SAM Coupon Delivery

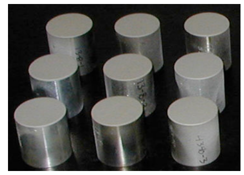
- Delivered SAM2X5 Coatings using optimum spray parameters (HV4)
  - 40 electrochemical disks sprayed onto 316L substrates
  - 5 crevice corrosion plates sprayed onto 316L substrates

Example Corrosion Disks

Example Crevice Corrosion Plate

SAM2X5 Bond Strength

ASTMC633-01 Bond-Pull Testing



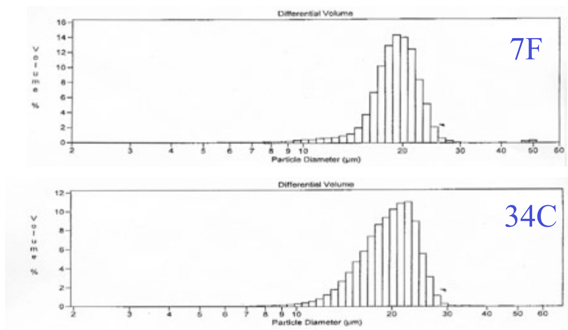
Alloy	Spray Condition	Lbs	PSI	Failure Mode
SAM2X5	HV4	6784	8642	100% coating break at bottom
SAM2X5	HV4	5553	7074	100% coating break at bottom
SAM2X5	HV4	5490	6994	100% coating break at bottom
Average		5942	7570	

4th Generation HVOF Systems

- XVHVOF: 4th generation HVOF systems
  - Much higher operating gun pressure (400 psi) and higher particle velocity
  - Promise to produce fully dense coatings in one step from impact fusion – achieved for soft metallic coatings
  - Utilized SAM40 for coating trials to compare with previous results on 3rd generation systems
- Special sizing was needed
  - 8 Parameters were sprayed using two powder sizes
- Using optimum parameters:
  - 40 electrochemical disks sprayed onto 316L substrates
  - 5 crevice corrosion plates sprayed onto 316L substrates

XVHVOF – Powder Sizing

- **SAM40 was atomized**
  - **Two special cuts (34C and 7F) of powder were classified in order to spray with XVHVOF since non-standard and finer cuts are necessary with this system**



XVHVOF – Spray Parameters

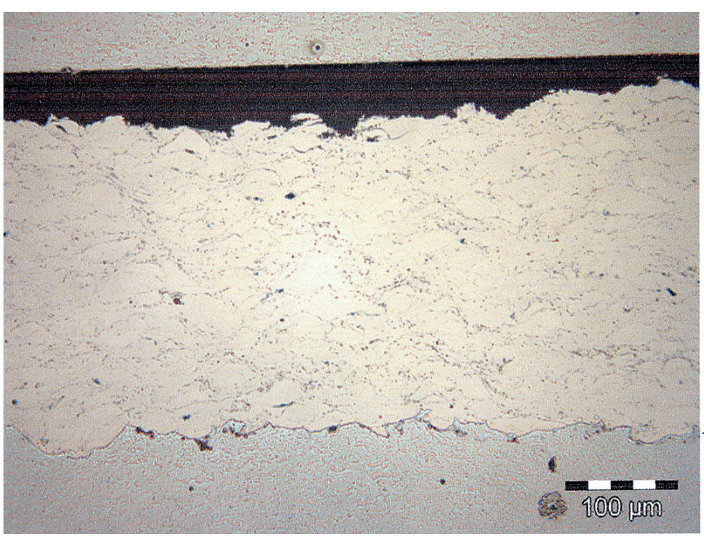
Spray Parameters XV1 > XV8	XV2	XV4
Powder Lot #	34C	7F
Chamber / throat	.156	.156
Barrel (inch)	8	8
Nitrogen Pressure [psi]		500
Nitrogen Flow scfh	300	0
Oxygen Pressure [psi]		550
Oxygen Flow scfh	1200	1400
Fuel (GasType) Pressure [psi]		500
Fuel (GasType) Flow scfh	500	500
N_2Carrier Pressure [psi]	140	140
N_2Carrier Flow [FMR]	25	25
Back psi:	375	350
Substrate Temperature [°F]	< 250	< 250
No. of Passes	32	38
Coating Thickness [Inches]	.011	.011

XVHVOF Spray Results

- Results of the spray parameter trials
  - Very good coatings were obtained which were much better than other hard alloys in this spray system

Spray Parameter	Macrohardness (R_C)	Microhardness (HV300)	Porosity
XV1	54	539	1.20
XV2	52	509	1.00
XV3	66	1066	1.50
XV4	67	1038	1.25
XV5	62	1030	1.75
XV6	62	1029	2.50
XV7	63	942	2.00
XV8	61	843	1.75

XVHVOF Coupon Structure / Density



SAM40
XV2 Spray Condition
99% Dense

Coating/ Substrate Interface

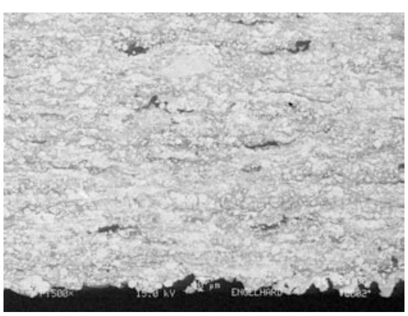
Polished HVOF Coatings

Coatings can be ground/polished down to 3 micro-inches using diamond impregnated cloth for applications such as replacement for electrolytic hard chrome, or bearing or valve applications

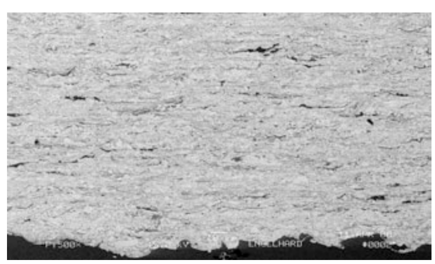
HVOF Coatings – State of the Art

- **Examples are shown of approved HVOF coatings for landing gear applications**
  - **One of the most difficult applications for coatings**
- **Coatings are currently being transitioned to Tinker, Hill and NIP Jacksonville Air Force Bases**

HVOF WC-Co



HVOF WC-CoCr



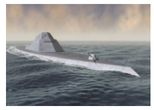
Conclusions

- Coating properties are a compendium of the microstructure and macrostructure. To continue to make progress, research was focused on separating out extrinsic and intrinsic property effects in order to optimize in parallel the composition and the ability to spray with high density and low permeability
- To address this, 'ideal' glasses were produced by melt-spinning which were additionally heat treated to produce 'ideal' nanocomposite structures
- These 'ideal' samples acted as a beacon in order to develop new alloy compositions which are intrinsically more resistant to electrochemical attack in the targeted environments

Conclusions (Cont'd.)

- The downselected SAM2X5 alloy has been shown to have very good corrosion resistance in a wide variety of environments
- 3rd generation HVOF gun system was used to spray the SAM2X5 alloy and after optimizing spray parameters very high coating densities up to 99.7% were obtained
- Spray work using the 4th generation XVHVOF system showed that high density coatings could be obtained using the SAM40 alloy with densities up to 99% achieved
- Further optimization of the spray process (i.e. spray parameters, feedstock condition, sizing etc.) will be necessary to produce denser coatings which may be necessary to meet the DOE goals of this Program

Backup Slides



High-Performance Corrosion-Resistant Materials: Synthesis of SAM1651 Powder & Ingots

Craig Blue & Bill Peter
Oak Ridge National Laboratory
Oak Ridge, Tennessee

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Introduction

- The electrochemical, mechanical, and thermodynamical behaviors of high-Mo Fe-based structurally amorphous metals (SAM) have been encouraging.
  - Excellent corrosion resistance in various electrolytes (outperformed 316 and C-22 in corrosion testing)
  - High hardness/strength measurements
  - Good glass forming abilities and low critical cooling rates for several compositions allowing retention of glassy phase through processing
- Processing continues to be a critical point of interest for structural amorphous material applications and for technology transfers.
- The roles of Oak Ridge National Laboratory (ORNL) in the DARPA-DOE HPCRM Program
  - Provide materials processing knowledge
  - Continue to oversee fabrication of amorphous powders for spraying and production
  - Supply the scientific collaborative group with the fabricated amorphous samples as needed

Accomplishments

- Powder Fabrication of SAM1651.
  - Two – one ton gas atomized batches of SAM1651 powder were sprayed in association with Carpenter Technologies (Pittsburgh, PA). Also, further refinement and development of induction melting capabilities for large quantities.
  - Gas atomized and size classified approximately 2800lbs of fully amorphous SAM1651 powder.
  - Improved the sprayability of SAM 7 through adjustments of the atomization melt temperature. More work is needed.
- Drop Cast Specimens Were Provided to the HPCRM Team for Electrochemical, Thermodynamical, Microstructural, and Mechanical Evaluation.
  - Vacuum arc melting and drop casting of SAM7(1651), SAM8(SAM1651+W), SAM2X5 alloys.
  - Near one hundred (100) - ingots cast for analysis by the HPCRM team
  - SAM7 cast fully amorphous in bulk form with ingot diameters up to 10.0mm
  - Drop cast ingots of SAM1651 alloys exhibited high hardness and compressive strength values.
- Successful Machining of SAM7 and SAM8 in the Amorphous State.
  - Drop cast ingots of SAM7 were machined using a rotary lathe and grinding attachment to produce button head tensile specimens for mechanical evaluation.
- Vacuum Hot Pressing of SAM2X5, SAM7, and SAM8 Bulk Specimens.
- Laser Fusing of SAM2X5 and SAM7 to H13 Steel Coupons and Herrenknecht Disc Cutters (To Be Discussed in Subsequent Presentation)

Accomplishments

- Pearl Harbor 2005 meeting
  - Drop cast ingots of SAM6, SAM1651, SAM1651-116, and SAM1651-117.
  - Gas atomized and size classified approximately 2000lbs of fully amorphous SAM1651 powder.
  - Laser fused HVOF sprayed SAM1651 coatings.
- Drop cast ingots of SAM1651 alloys perform better than 316 and C-22 in corrosion testing.
- Gas atomized SAM1651 powder fully amorphous in all powder size classifications, including large particles.
- Amorphous, sprayed SAM1651 coating remained amorphous after laser fusing with reduction in porosity.

- Key West 2006 meeting
  - ~90 samples were fabricated and supplied of SAM7(1651), SAM8(SAM1651+W), SAM2X5.
  - Gas atomized and size classified approximately 3,000 lbs of fully amorphous SAM1651 powder.
  - Successful machining of amorphous, cast ingots (SAM7 and SAM8).
  - VHP with 90% consolidation, devitrification with retained hardness/strength and introduction of toughness.
- Drop cast ingots of SAM1651 alloys exhibited high hardness and compressive strength values.
- Drop cast ingots of SAM1651 continued to show excellent corrosion properties.
- Improved the sprayability of SAM 7 through adjustments of the atomization melt temperature. More work is needed.

Alloy Compositions

- Compositions considered in FY05 ORNL-HPCRM work.

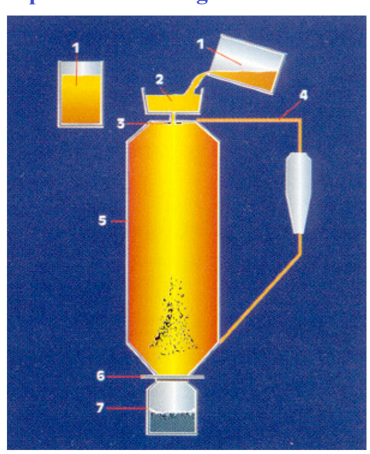
Name	Formula	Fe	Cr	Mo	W	B	C	Y	Mn	Si	Total
SAM2X5	$Fe_{49.7}Cr_{18}Mo_{7.4}W_{1.6}B_{15.2}C_{3.8}Mn_{1.9}Si_{2.4}$	49.7	18.0	7.4	1.6	15.2	3.8		1.9	2.4	100.0
SAM7 (1651)	$Fe_{48}Mo_{14}Cr_{15}Y_2C_{15}B_6$	48.0	15.0	14.0		6.0	15.0	2.0			100.0
SAM8 (1651 -116)	$Fe_{46}Mo_{14}Cr_{15}Y_2C_{15}B_6W_2$	46.0	15.0	14.0	2.0	6.0	15.0	2.0			100.0
SAM10 + 1 %C (Mining Bits)	$Fe_{56.7}Cr_{21.2}Mo_{2.6}W_{1.8}B_{16.7}C_1$	56.7	21.2	2.6	1.8	16.7	1.0				100.0

- Compositions previously considered in ORNL-HPCRM work (FY03 & FY04).

Name	Formula	Fe	Cr	Mo	W	B	C	Y	Zr	Co	Al	P	Total
SAM6	$Fe_{43}Cr_{16}Mo_{16}B_5C_{10}P_{10}$	43.0	16.0	16.0		5.0	10.0					10.0	100.0
SAM9 (1651 -117)	$Fe_{48}Mo_{12}Cr_{14}Y_2C_{15}B_6W_2$	48.0	15.0	12.0	2.0	6.0	15.0	2.0					100.0
SAMFeZr	$Fe_{61}Y_2Zr_8Co_6AlMo_7B_{15}$	61.0		7.0		15.0		2.0	8.0	6.0	1.0		100.0

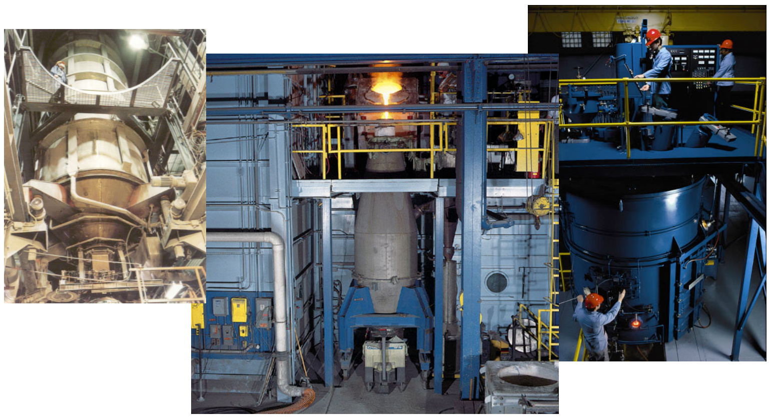
Gas Atomization of Amorphous Powder (Review)

- Based on its corrosion performance and the successful synthesis of exceeding 316 and C-22, SAM1651 was gas atomized for use in coating applications.
- Gas atomization was performed by Carpenter Technologies in partnership with ORNL.
- ORNL responsible for distribution within the HPCRM team
- Schematic shows gas atomization setup.
  - 1. 5.6 metric ton furnace.
  - 2. Tundish.
  - 3. Atomization nozzle.
  - 4. Gas recirculation system.
  - 5. Atomization tower.
  - 6. Sealing mechanism.
  - 7. Powder collection vessel.



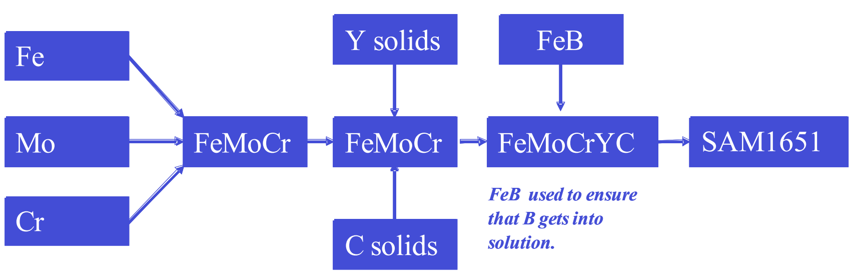
Gas Atomization of Amorphous Powder (Review)

- Three spray apparatus are available for use at Carpenter,
  - 5.6 ton traditional gas atomization unit.
  - 1.0 ton traditional gas atomization unit.
  - 1.0 ton vacuum melt, gas atomization unit (used in this work).



Gas Atomization of Amorphous Powder (Review)

- Vacuum melt setup used to prevent oxidation during alloy additions.
- Alloy additions combined in specific order to prevent boride and carbide phase developing that might clog the spray nozzle.

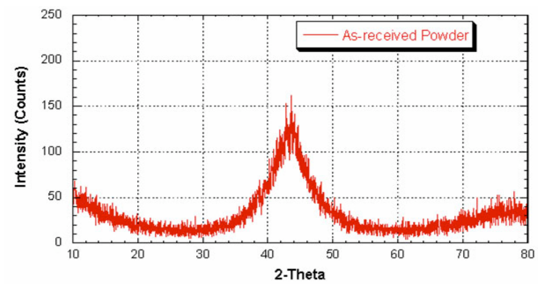


Fe, Mo, and Cr melted and combined to form FeMoCr bath. Homogenized at 1570 °C to 1590 °C

Y and C solids added to FeMoCr bath.

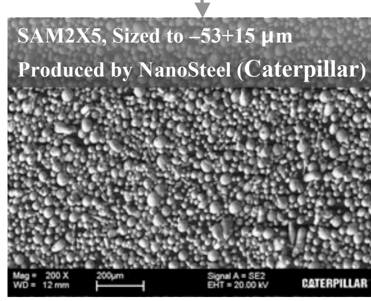
Review of FY04 SAM7(1651) 1-Ton Heat

- 1-ton heat of SAM1651 powder was gas atomized, superheat 1538°C(2800°F).
- SAM1651 powder was classified into 4 size cuts.
  - -16µm, 100-lb yield.
  - -500M (-25µm) +16µm, 37-lb yield.
  - -270M (-53µm) +500M (+25µm), 396-lb yield.
  - +270M (+53µm), 660-lb yield.
- Chemical analysis for each cut indicated that target compositions were achieved (atomic %).
- XRD analysis of as-gas atomized SAM1651 powder illustrates amorphous halo (SNL).
- However, powder morphology was problematic for HVOF and cold spraying of SAM7(1651) powder. Powder would become lodged in the feeder bin (Caterpillar).



Development of FY05 Gas Atomization Parameters

- Two (2) 1-ton heats scheduled for FY05
- Parametric study of SAM7(1651) gas atomization not in work scope for FY05
- HPCRM Atomization Committee: LLNL, UCD SNL, ORNL (Nancy Yang, Enrique Lavernia, Jeff Haslam, Craig Blue, Bill Peter, etc.)
- Recommendations from Carpenter Powder
- Develop similar morphology to 2X5 to eliminate issues with feeder bin
  - Resolve some of the issues with ligament/stringer morphology
  - Do not make extreme change in batch temperature
    - Continue to keep amorphous nature
    - Maintain low oxidation
- HPCRM Atomization Committee agreed increasing the superheat temperature by 50°C would be advantageous
- Carpenter recommended running one - heat ~ 50°C above and one heat ~ 50°C below FY04 heat



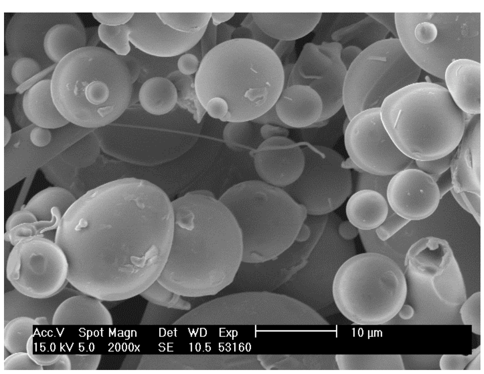
Gas Atomization of Amorphous Powder

- Three (3) 1-ton gas atomization heats conducted in FY 2005 (low temperature heat not used).
- Approximately 2800 lbs of SAM1651 powder was gas atomized.
- Chemical analysis for each indicated that target compositions were achieved atomic % (weight %). Atomic percentages within +/- 1.3 for individual elements for all lot numbers.

Lot #	Fe	B	C	Mo	Y	Cr	Total
V5059 1482 °C(2700 °F)	Low Induction Temperature Resulted in Molten Alloy Freezing in the Nozzle						
V5060 1607 °C(2925 °F)	47.8 (50.4)	6.1 (1.2)	14.1 (3.2)	13.9 (25.2)	3.2 (5.4)	14.9 (14.6)	100.0
V5061 1607 °C(2925 °F)	48.3 (50.8)	6.2 (1.3)	13.7 (3.1)	14.2 (25.7)	2.7 (4.5)	14.9 (14.6)	100.0
V5060B (5060+5061)	48.1 (50.6)	6.1 (1.3)	13.9 (3.1)	14.1 (25.5)	2.9 (4.9)	14.9 (14.6)	100.0
Target -1651 at% (wt%)	48.0 (51.3)	6.0 (1.2)	15.0 (3.5)	14.0 (25.7)	2.0 (3.4)	15.0 (14.9)	100.0

Gas Atomization of Amorphous Powder

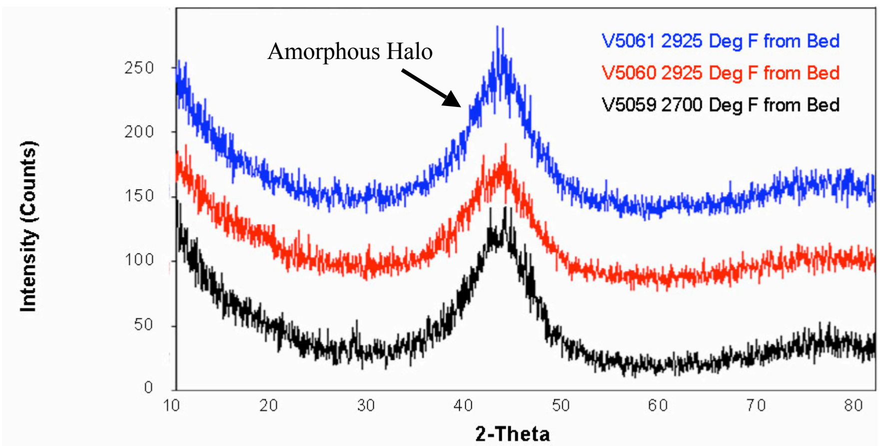
- SAM7(1651) Powder was classified into 4 size cuts. Total 2,771 lbs from
  - -16µm, 361-lb yield = 13%
  - - 45µm (-325M) +16µm / 532-lb yield (prime cut) = 19%
  - -425 µm (-40M) +45 µm (+325M) / 1,672-lb yield = 60%
  - +425 µm (+40M) / 206 lbs yield = 8%



- SE Image of V5060 Dust at 2,000X Uncut (SNL)

Gas Atomization of Amorphous powder

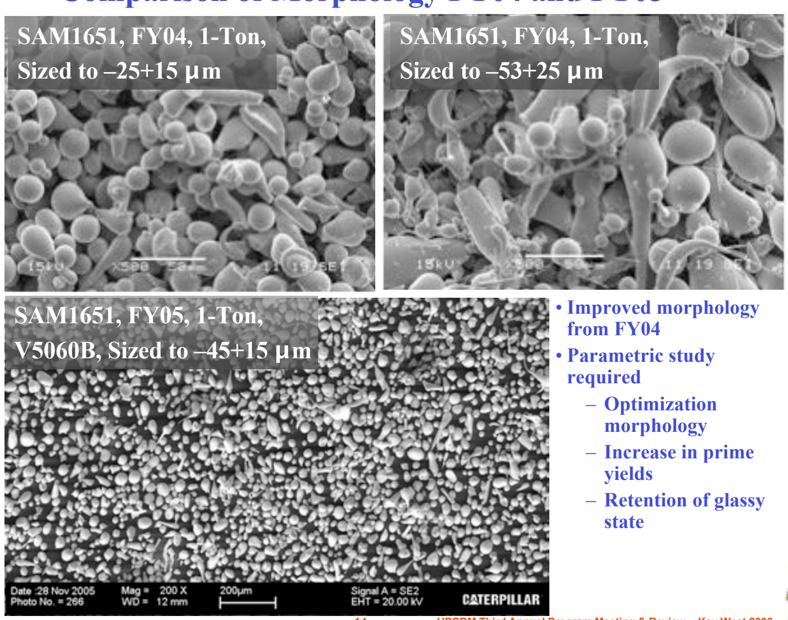
- XRD analysis of as-gas atomized SAM1651 powder (no cuts in the powder) exhibits broad halo characteristic of amorphous materials, and no Bragg's Peaks characteristic of crystalline materials.



Comparison of Morphology FY04 and FY05

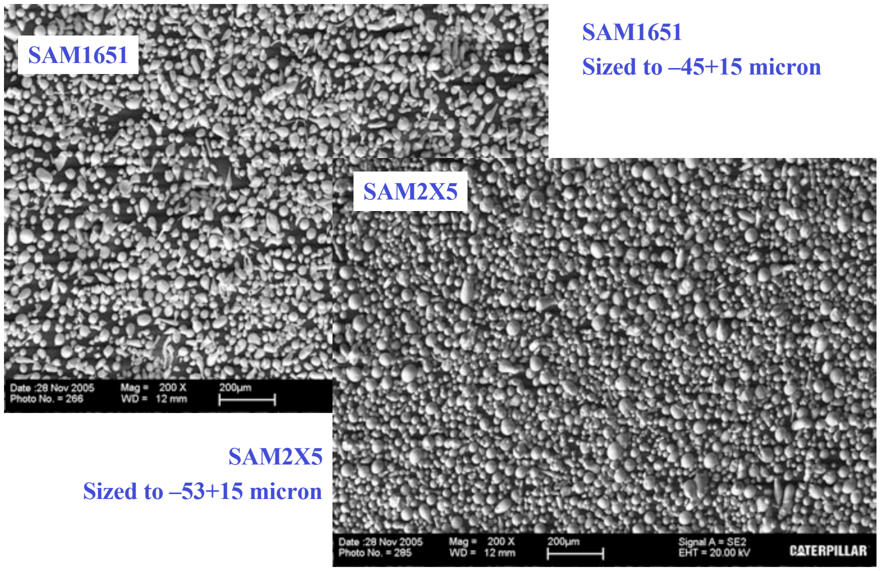
FY04, 2800°F (1538°C)

F05, 2925°F (1538°C)



- Improved morphology from FY04
- Parametric study required
  - Optimization morphology
  - Increase in prime yields
  - Retention of glassy state

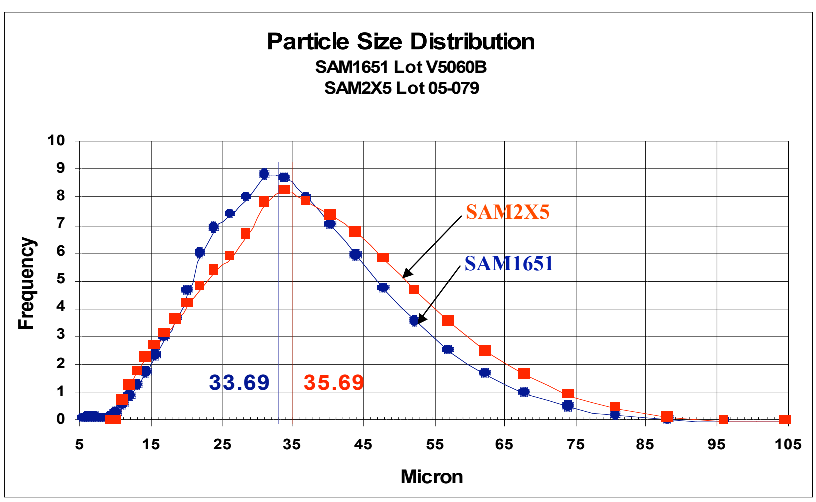
Comparison of Powder Morphology Between SAM7(1651) & SAM2X5 (Caterpillar)



SAM1651
Sized to –45+15 micron

SAM2X5
Sized to –53+15 micron

Comparison of Particle Size Distribution for Prime Cut –45+15 micron (Caterpillar)



Supply HPCRM Team with Samples for Evaluation

- Joe Farmer and Jeff Haslam, Lawrence Livermore National Laboratory (LLNL)
  - Electrochemical behavior: dynamic polarization, and potentiostatic stepping
- John Lewandowski, Case Western University (CWU)
  - Mechanical behavior: including tensile/compressive testing, hot hardness data, flexural testing, Charpy impact, and fracture toughness
- Nancy Yang, Sandia National Laboratory (SNL)
  - Microstructural evaluation: including XRD examination and chemical analysis
- John Perpezko and Kjetl Hildal, University of Wisconsin, Madison (UWM)
  - Thermal evaluation: including differential scanning calorimetry and differential thermal analysis
  - Fabrication of ribbons
- Rest of HPCRM team for various efforts
- Near one hundred cast specimens were fabricated for the HPCRM team

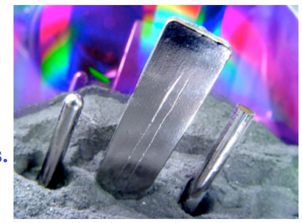
- Drop Casting of Amorphous SAM1651
  - 45 – rods, 4.8 mm dia. x 76.2 mm length
  - 28 – slabs, 4.8 mm x 12.7 mm x 44.5 mm
  - 1 – rods, 6.4 mm dia. x 152.4 mm length
  - 5 – tensile specimens
- Drop Casting of Crystalline SAM2X5
  - 10 - ingots/rods, 4.8 mm diameter x 76.2 mm length

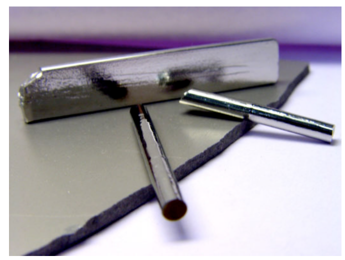
Procedure for Drop Casting Amorphous Ingots from Amorphous Powder

- Previous drop castings of SAM1651 (from pure elements) requiring sequential additions for proper chemistry
- Method to drop cast amorphous powders
  - Composition homogeneous throughout powder
  - Compressed into pellets
  - Sintered at 500°C in vacuum furnace
- The arc melting facilities at ORNL operate at a maximum current of 1500A with voltages of 15 to 20V.
- The water cooled chamber is equipped with a diffusion pump capable of 10^{-6} Torr.
- The evacuated chamber is backfilled with ultra high-purity Ar gas in order to facilitate a plasma arc.
- The arc is struck between a 0.5-inch thoriated tungsten electrode and a copper base plate.
- The intense heat of the arc was used to melt the consolidated powder pellet. When melted, the liquid was dropped through a hole in the bottom of the chamber and the molten alloy is cast in a water cooled copper mold.
- Ability to drop cast net shape components.

Drop Casting of Amorphous Ingots

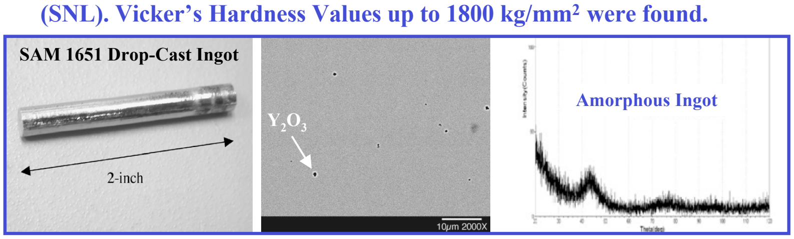
- SAM2X5 was vacuum drop cast in ingot form.
  - Vacuum drop cast ingots were crystalline.
    - Ingots, 4.8 mm diameter x 76.2 mm.
- SAM1651 was drop cast in ingot form of up to 10mm-diameter.
  - Drop cast ingots were completely amorphous.
    - Slab, 4.8 mm x 12.7 mm x 44.5 mm.
    - Ingots, 4.8 mm diameter x 76.2 mm.
  - Critical cooling rate of 85ºC/s allows for processing of glasses
- SAM1651-116 were drop cast in ingot form.
  - Drop cast ingots were largely amorphous.
    - Ingots, 4.8 mm diameter x 76.2 mm length
    - 116 composition sometimes contained micro-crystalline phases.
- Ingots and slabs were used for electrochemical, mechanical and thermodynamical evaluation.



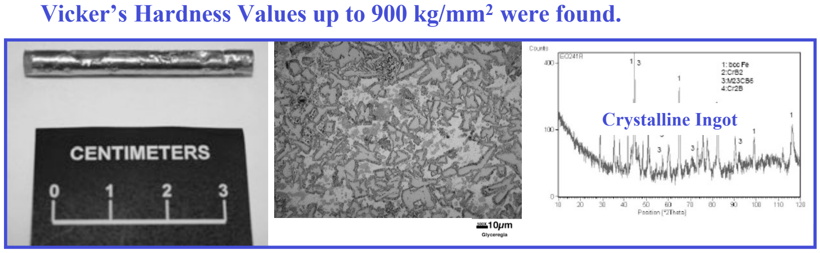


Drop Casting of Amorphous Ingots

- XRD analysis of SAM 1651 shows characteristic featureless pattern (SNL). Vicker's Hardness Values up to 1800 kg/mm² were found.

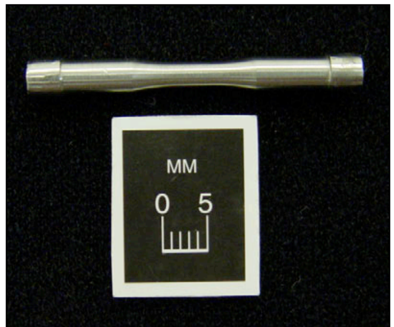


- XRD analysis of SAM 2X5 shows characteristic crystalline peaks (SNL). Vicker's Hardness Values up to 900 kg/mm² were found.



Successful Machining of Fe-Based Structural Amorphous Materials

- Fe-based structural amorphous materials can be machined into intricate geometries
- Drop cast ingots of SAM7 and SAM8 were machined using a rotary lathe and grinding attachment to produce button head tensile specimens for mechanical evaluation.
- Tensile specimen with reduced testing gauge section (eliminate failure at the grips)
- 50 to 60 % return for this geometry.
  - Yield could be much better with simplified geometries.
  - Failure during machining occurs in the gauge section (2 mm)
- Presently evaluating the possibility of suction casting tensile specimens
  - Glasses have benefit of net shape fabrication



Machined Button-Head SAM7 (SAM1651)
Fe-Based Bulk Metallic Glass

VHP of Devitrified SAM Material with High Hardness

- Vacuum hot pressing (VHP) SAM2X5, SAM7, SAM8, and SAM10+1%C indicative of other work
- Bulk of consolidation in the amorphous state within the super-cooled liquid region
- Processing of amorphous and crystalline materials with Vicker's Hardness Values up to 1800 kg/mm^2
- Fracture toughness values up to 18 MPa√m in as-VHP Condition (CWU) - Potential to increase with annealing treatment (in progress)
- Possibility of controlled crystal grain size growth with crystallites in the "nanometer scale" for up to 2 hours or more
- Potential of bulk nanocrystalline composites with significant engineering properties

Conclusions

- **Powder fabrication of SAM1651.**
  - **Two (2) 1-ton gas atomized batches of SAM1651 powder were successfully sprayed and fully amorphous with improved morphology for spraying.**
  - **Target compositions were achieved.**
  - **XRD and SEM analysis performed.**
  - **Classified to specific particle size for optimization of spray coatings.**
  - **Problems were encountered with powder morphology.**
  - **Process development by HPCRM Team (SNL, UCD, ORNL, etc.) directed at overcoming problems.**
  - **Parametric study will further improve the morphology and maximize yield of prime cut.**
- **Vacuum arc melting and drop casting of SAM7(1651), SAM8(SAM1651+W), SAM2X5 alloys.**
  - **Nearly one hundred (100) - ingots were provided to the HPCRM team for chemical, thermodynamical, microstructural, and mechanical evaluation.**
  - **SAM7 cast fully amorphous in bulk form with ingot diameters up to 10.0mm due to low critical cooling rate of 85^{o}C.**
  - **Drop cast ingots of SAM1651 alloys exhibited high hardness values.**
- **Successful machining of SAM7 and SAM8 in the amorphous state.**
- **Vacuum hot pressing of SAM2X5, SAM7, SAM8 and SAM10+1%C bulk specimens with potentially exciting mechanical benefits.**

Backup Slides

High-Performance Corrosion-Resistant Materials: Industrial Scale Processing

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Peoria, Illinois

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Thermal Spray at Caterpillar

Advanced Materials Technology
Flame spray since the 1960's
Plasma spray since the 1960's
(major research effort since 1984)
HVOF since 1992
Production use of HVOF for chrome plate replacement being evaluated

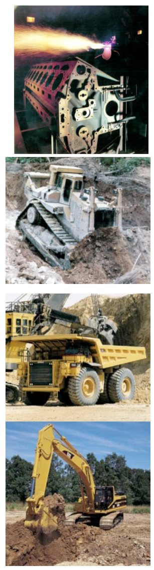
Corinth, MS Remanufacturing
Flame spray since 1986
- cylinder heads
- engine block decks
- engine covers
- connecting rods

Caterpillar Dealers
Flame spray since 1981
HVOF since 1998
92 applications
- oil pump shafts
- water pump shafts
- housings

Solar Turbines
TBC's since 1985
- fuel injectors
- combustion liners
Wear coatings since 1990
- power turbine shafts

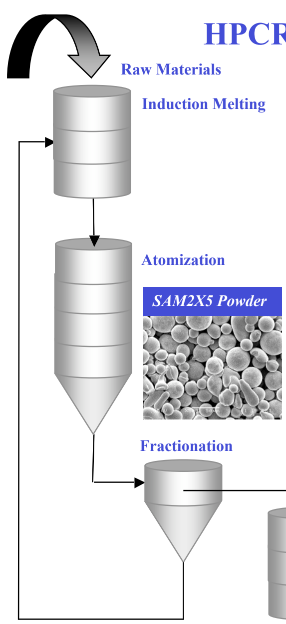
- Production HVOF Facility for Chrome Plate Replacement in Joliet, IL – Fall 2005
- Production HVOF Facility for Undercarriage in Danville, KY – Spring 2006

Caterpillar Thermal Spray Process Capability

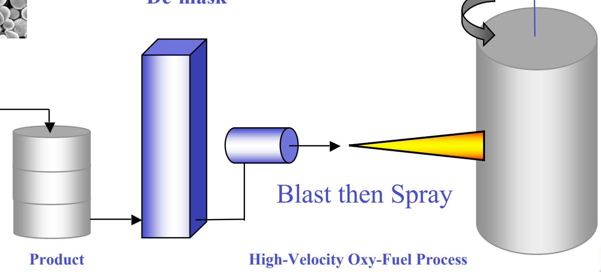


- Flame Spray Process
  - 6P Combustion Powder Spray
- Wire-Arc Process
  - BP 400
  - Praxair
- Plasma Spray Process
  - Sulzer Metco 9MB
  - Praxair SG-100
  - Water Stabilized Plasma (high throughput)
- High Velocity Oxygen-Fuel (HVOF) Process
  - Sulzer Metco DJ2700
  - Praxair TAFA JP5000/JP8000
  - GTV System
    - K2 Torch
    - SLM XV Torch
    - Jet Kote Torch
    - HV2000 Torch
- Detonation Process
  - Demeton Detonation Spray System

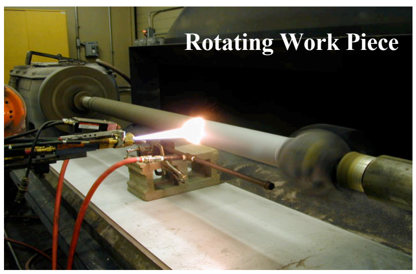
HPCRM Thermal Spray Process



- Machining for Coating Application
  - Radius corners
- Masking
  - If required for areas not to be sprayed (area to be welded)
- Surface Preparation for Bonding
  - Grit blast to roughen and clean
  - Laser treatment
- Spray Application
  - HVOF
- De-mask



Typical Thermal Spray Process at Caterpillar



Corrosion Problems in Mexico Salt Mine

Laboratory Data Demonstrated Superior Corrosion Resistance of Caterpillar Developed Low Cost Chrome Carbide HVOF Coatings for Salt Mine Application

Corrosion testing of ~ 0.0035 inches (90 μm) thick HVOF coatings

Ground HVOF Coating	Tested for 72 hrs ASTM B117 Salt Fog	Tested for 243 hrs ASTM B117 Salt Fog

Corrosion of Hydraulic Lift Cylinder in Salt Mine

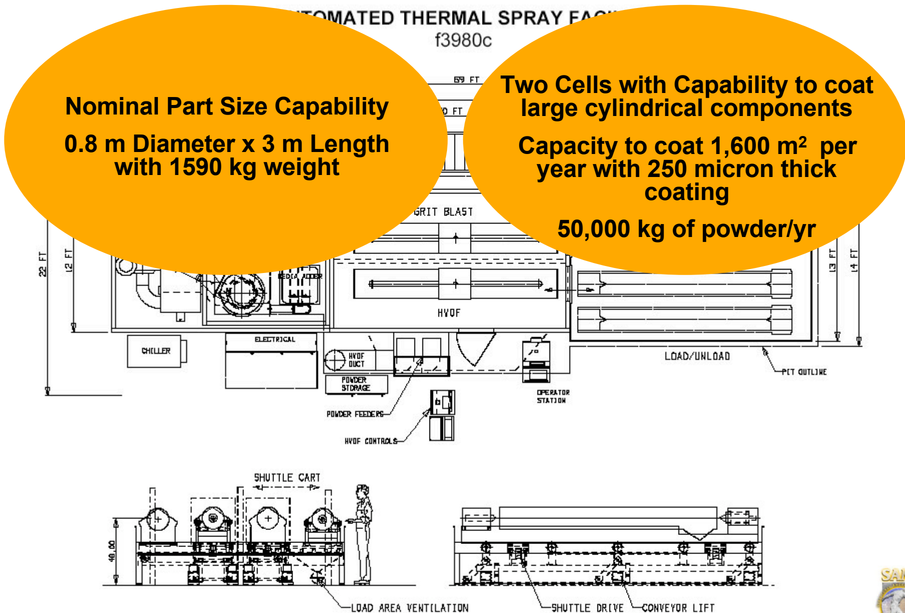
Standard Cr-plate after 682 hrs

HVOF coating after 3200 hrs

Chrome Carbide HVOF Coating Provides 10X Improvement in Corrosion Resistance of Hydraulic Lift Cylinder in Salt Mine

HVOF Application to Large Off-Highway Truck Components

New Large-Scale Automated HVOF Facility for Truck Components



New Large-Scale Automated HVOF Facility for Truck Components

Runoff at Supplier - Progressive Technology

New Large-Scale Automated HVOF Facility for Truck Components

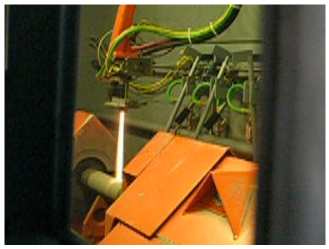
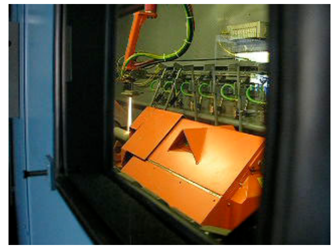
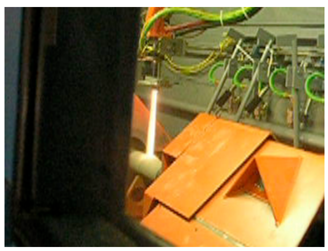
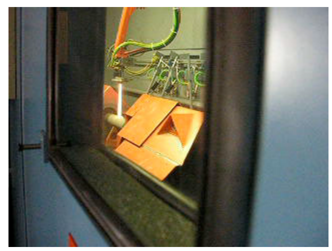
Runoff at Supplier - Progressive Technology

New Large-Scale Automated HVOF Facility for Truck Components

Runoff at Supplier - Progressive Technology

New Large-Scale Automated HVOF Facility for Truck Components

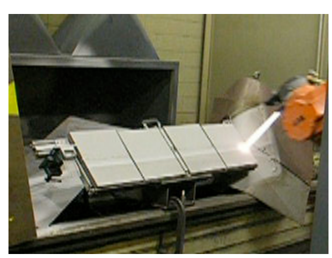
Runoff at Supplier - Progressive Technology

Coating C-22 Plates with SAM2X5/SAM1651 Using HVOF

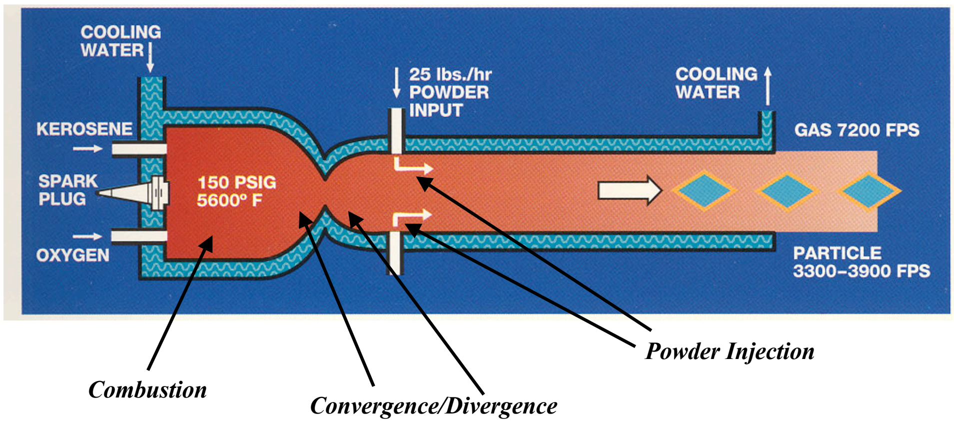
Coating Alloy C-22 Plates with SAM2X5 Using JP HVOF



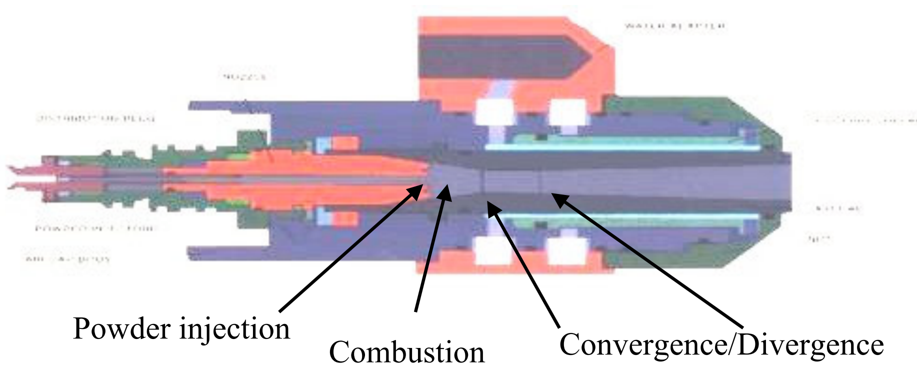
Spray Parameter Development for SAM1651

Praxair JP High Velocity Oxygen-Fueled (HVOF) Torch
(Kerosene Fueled)



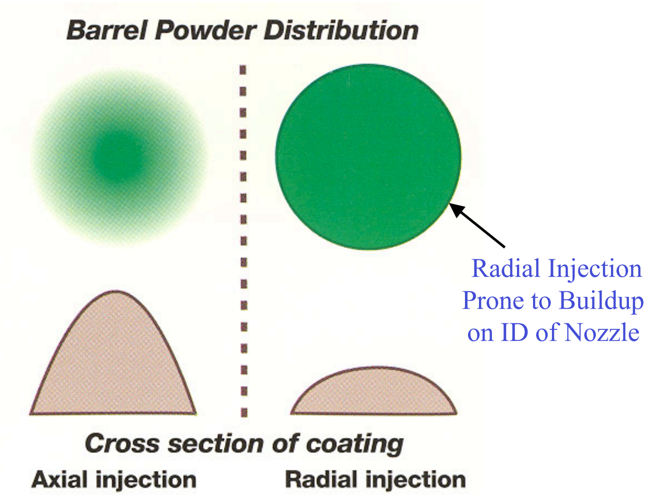
Spray Parameter Development for SAM1651

Sulzer Metco DJ High Velocity Oxygen-Fueled (HVOF) Torch
(Gas Fueled)



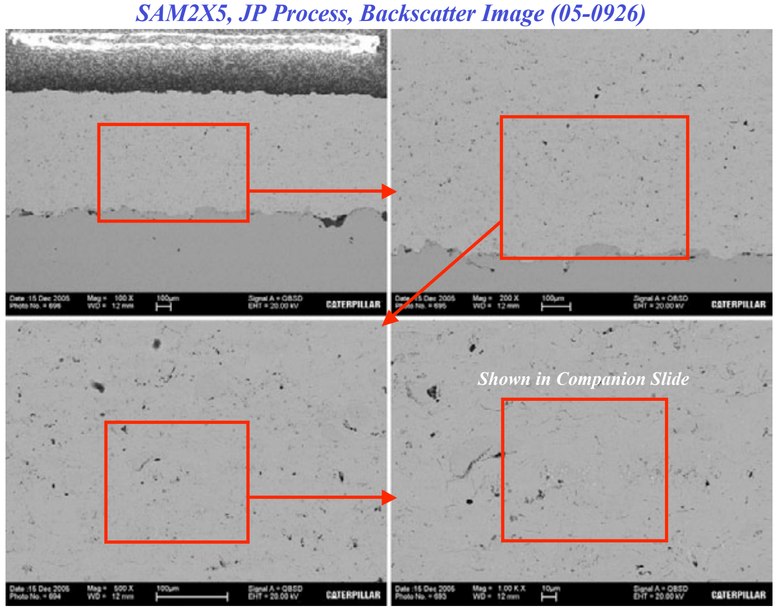
Spray Parameter Development for SAM1651

Powder Injection Method Impacts Ability to Spray Powders



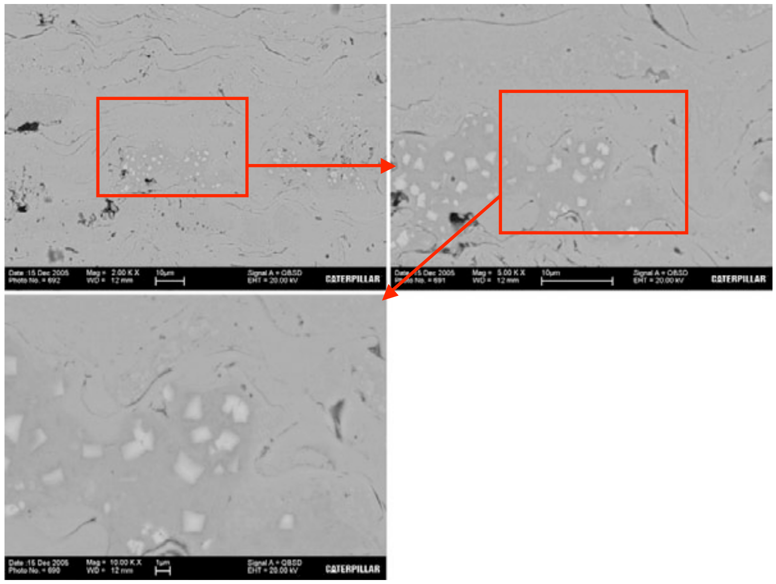
HY80 Witness Sample: SAM2X5 & JP Process

SAM2X5, JP Process, Backscatter Image (05-0926)



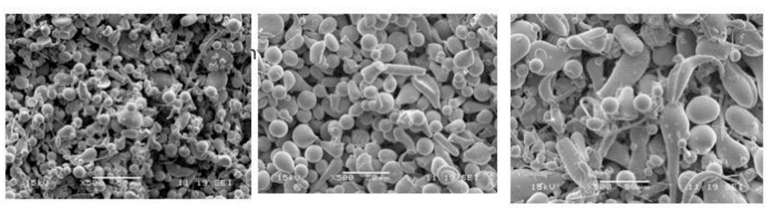
HY80 Witness Sample: SAM2X5 & JP Process

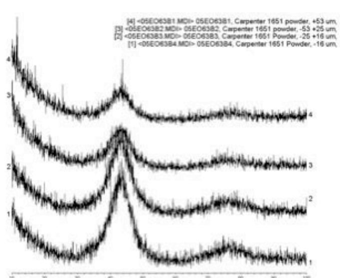
SAM2X5, JP Process, Backscatter Image (05-0926)



Problematic Morphology of SAM1651 Powder

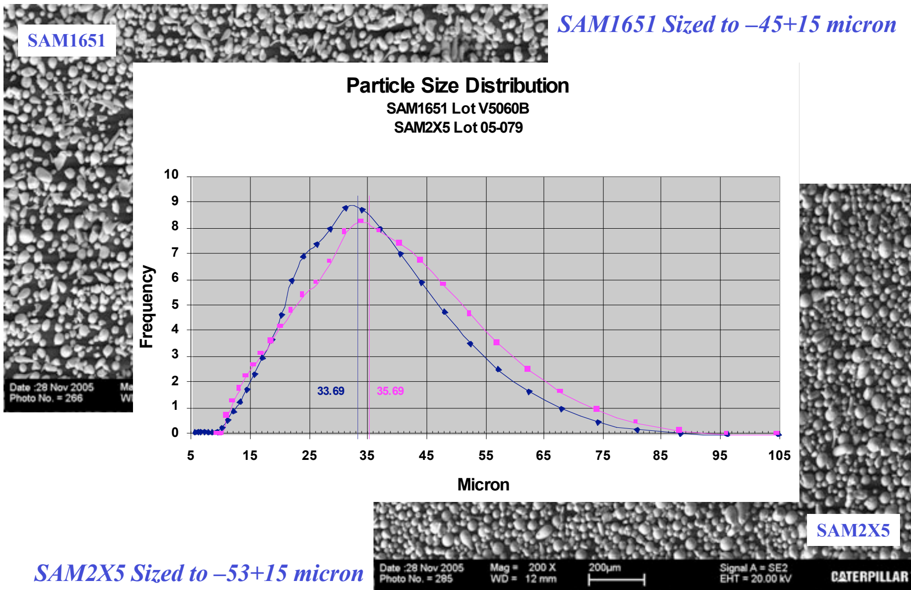
Prior Powder Lot Produced in 2004 - Poor Morphology for Flow





Spray Parameter Development: SAM1651 & SAM2X5 Powders

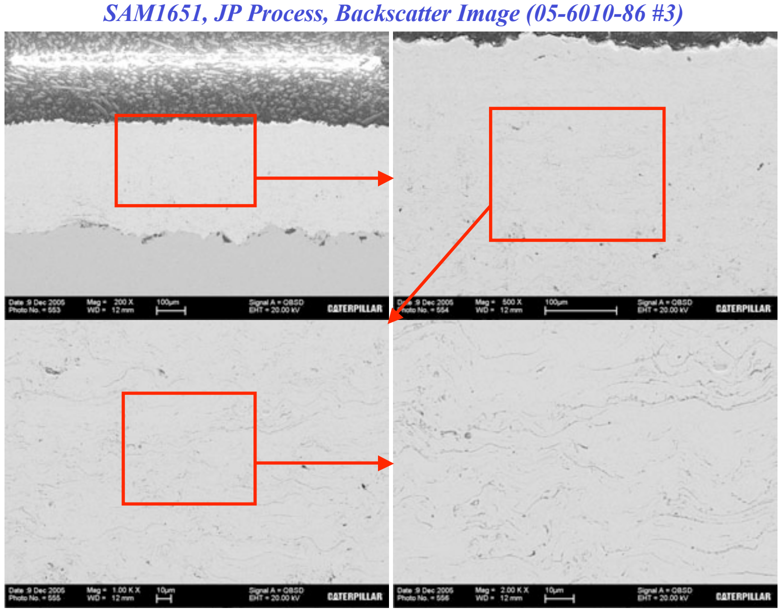
SAM1651 Sized to –45+15 micron



SAM2X5 Sized to –53+15 micron

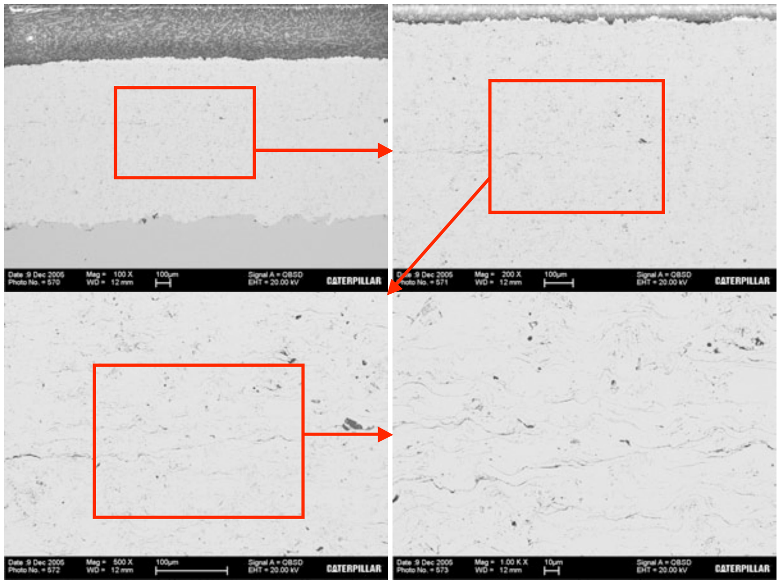
Spray Parameter Development: SAM1651 & JP Process

SAM1651, JP Process, Backscatter Image (05-6010-86 #3)



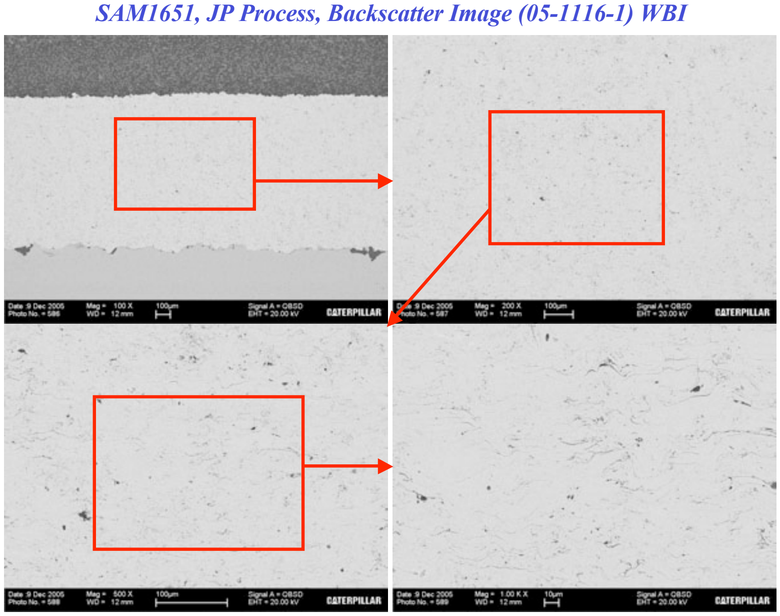
Witness Sample for Alloy C-22 Plate: SAM1651 & JP Process

SAM1651, JP Process, Backscatter Image (05-1116-1) WAI



Witness Sample for Alloy C-22 Plate: SAM1651 & JP Process

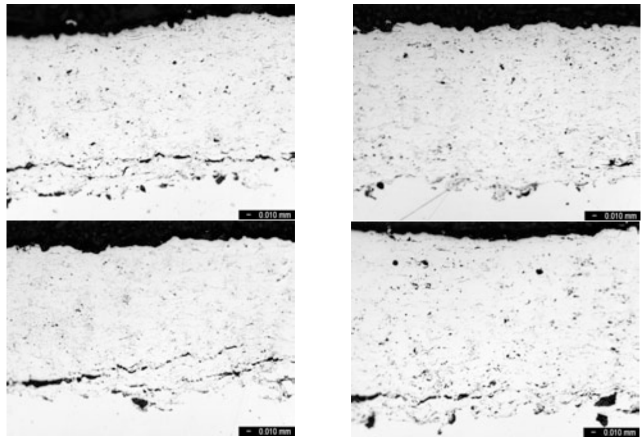
SAM1651, JP Process, Backscatter Image (05-1116-1) WBI



Spray Parameter Development: SAM1651 & JP Process

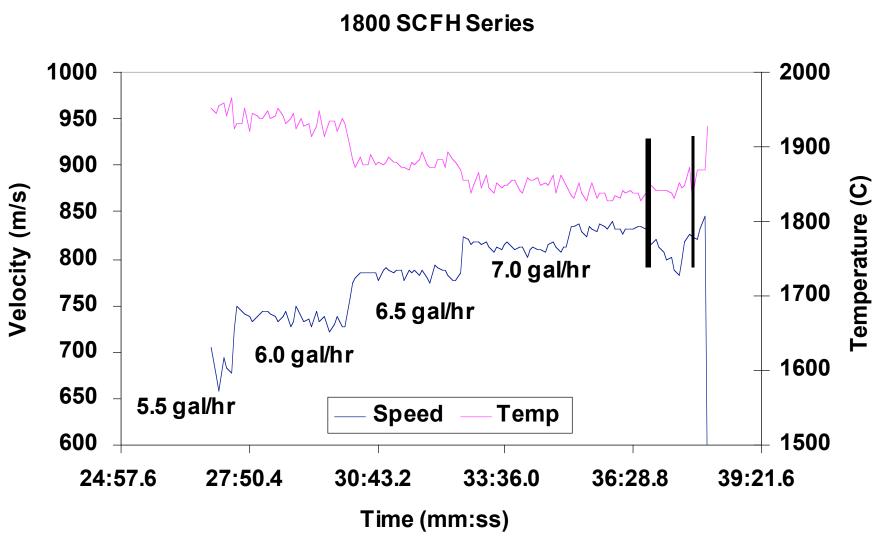
Sample 1 *Sample 2*

Sample 3 *Sample 4*



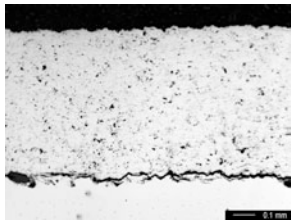
Sample	Oxygen Flow, SCFH	Kerosene Flow, Gal/hr
1	1900	7.0
2	1900	6.5
3	1900	7.5
4	1900	5.0

Spray Parameter Development: SAM1651 & JP Process

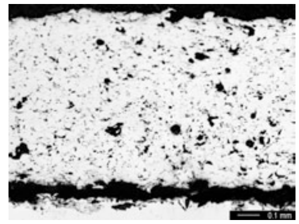


Spray Parameter Development: SAM1651 & DJ Process

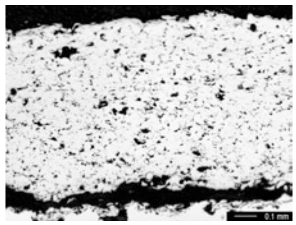
Sample 020
Set 1



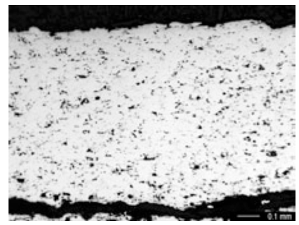
Sample 021
Set 2



Sample 022
Set 3



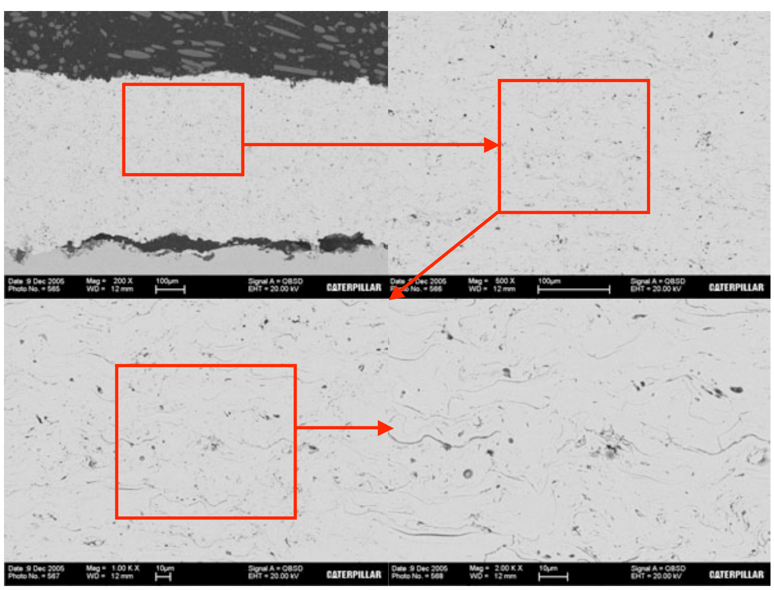
Sample 023
Set 4



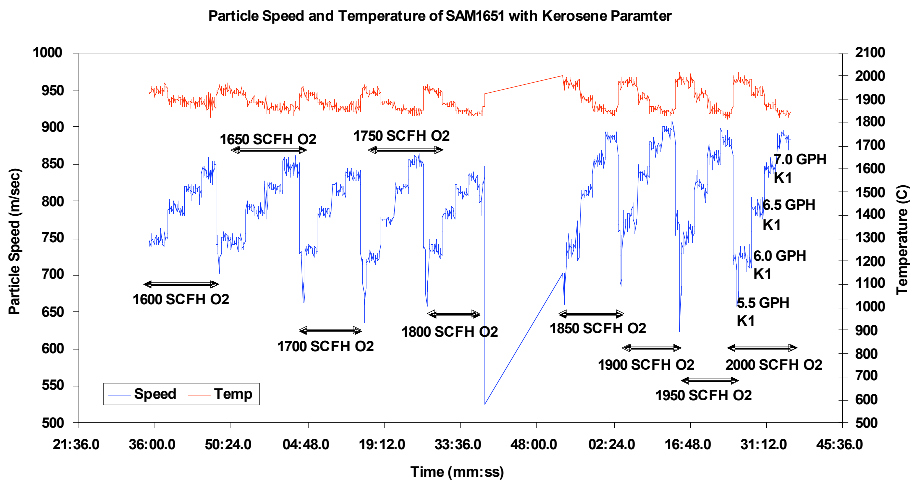
Parameter Set	Air Flow, FMR	Oxygen Flow, FMR	Propylene Flow, FMR
1	46	40	40
2	48	22	46
3	58	20	40
4	58	24	40

Spray Parameter Development: SAM1651 & DJ Process

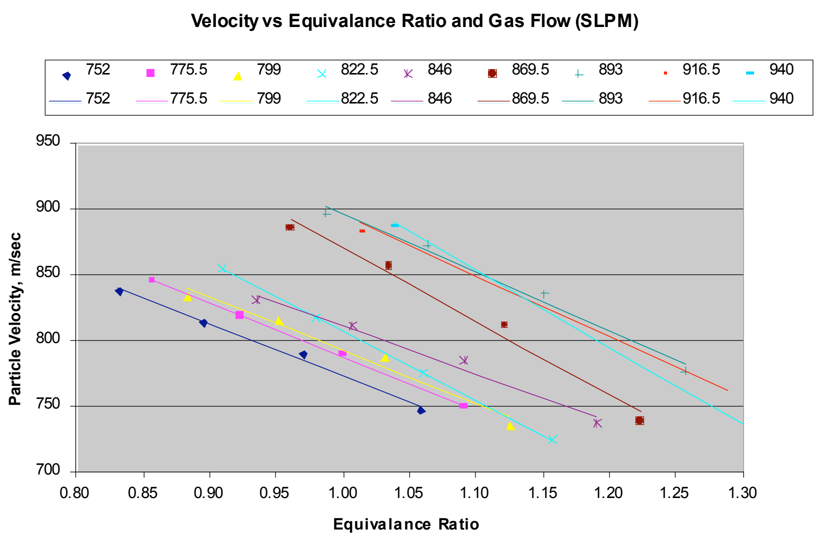
SAM1651, DJ Process, Backscattered Image (Sample 002)



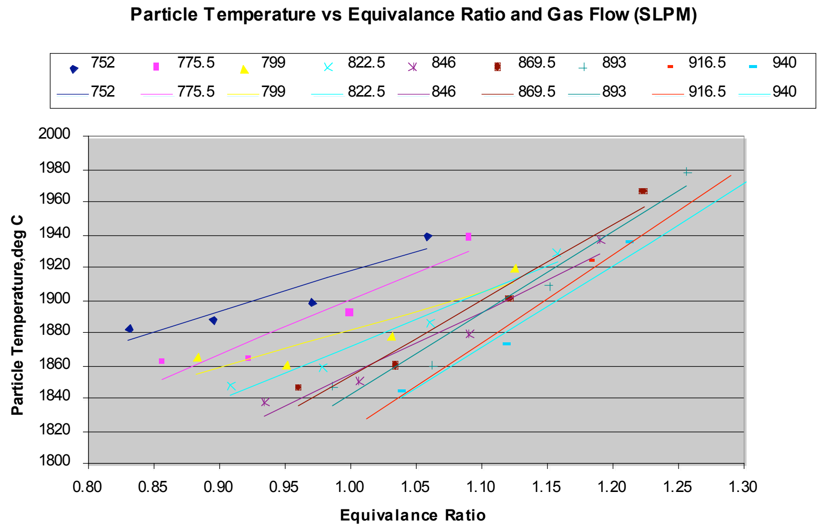
Spray Parameter Development: SAM1651 & DJ Process



Spray Parameter Development: SAM1651 & DJ Process

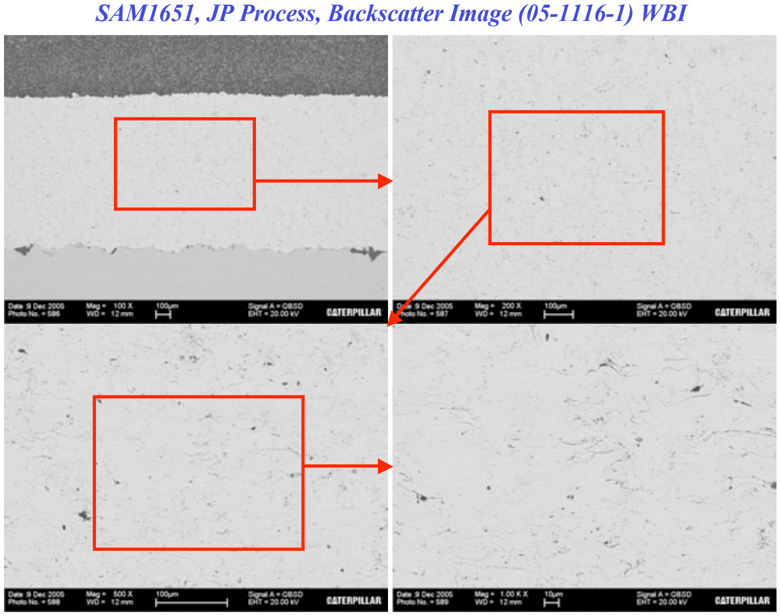


Spray Parameter Development: SAM1651 & DJ Process



Witness Sample for Alloy C-22 Plate: SAM1651 & JP Process

SAM1651, JP Process, Backscatter Image (05-1116-1) WBI



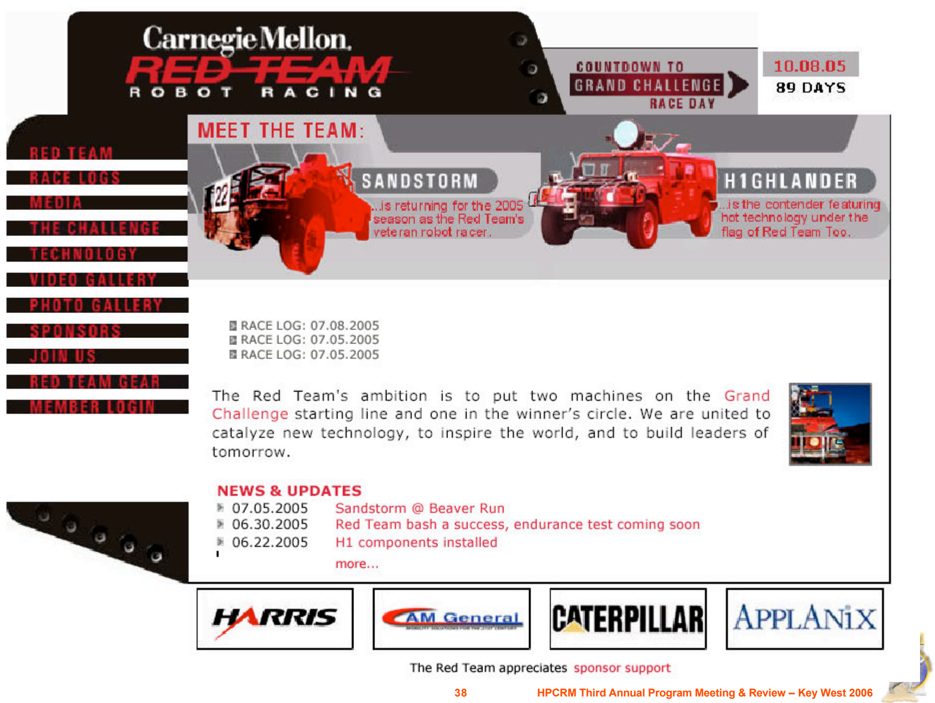
FY05 Accomplishments & FY06 Future Work

- Initial Industrial-Scale Coating of Large Prototypical Samples with Iron-Based Amorphous Metals for HPCRM Program
  - SAM2X5
    - Good Powder Morphology / High CCR / Partially Devitrified
  - SAM1651
    - Poor Powder Morphology / Low CCR / Fully Amorphous
- Response to Request for Proposal
  - Further Development of Robust Industrial HVOF Process
    - Surface Preparation
    - Spray Parameters
  - Demonstration of Coating for Prototype MPC & Basket
    - FY06 – Sub Scale
    - FY07 – Full Size

Backup Slides

Caterpillar is…

- World's largest manufacturer of construction and mining equipment, diesel and natural gas engines and industrial gas turbines.
- Technology leader in construction, transportation, mining, forestry, energy, logistics and electric power generation.



Technical Center

19 acres under roof

6 major buildings

6 SIGMA

Robust Engineering of HVOF Coatings

DMEDI Training

November 14, 2005

CATERPILLAR®

Process Parameters

- Surface preparation
  - Determine Grit Blasting Process Capability for Ra
  - Process Capability – Variables
  - Substrate Hardness
  - Blast nozzle wear
  - Media mix/size
  - Grit quality (supplier)
  - Blast air pressure and flow
  - Blast standoff
  - Blast nozzle speed
  - Blast nozzle overlap
  - One pass blasting vs multiple pass
  - Coverage of complete surface
  - Alternate Prep Methods
  - PROTAL™ Processing
  - Machined Surface
- Spray Process
  - Current Thermal Spray Process – TAFA JP
  - Optimum Particle Size Distribution
  - Critical Thickness Requirement
  - Layered Structure
  - Variation in Spray Parameters
  - Thickness per pass
  - Traverse speed
  - Overlap of torch footprint
  - Powder feedrate
  - Fuel/Oxygen Flow and Ratio
  - Fuel/Oxygen quality/type
  - Powder Injection
  - Powder Feedrate
  - Limitations due to robot speeds
  - Spray Distance
  - Torch Degradation
  - Barrel Wear
  - Rotated Barrel
  - Interconnector (combustion pressure)
  - Powder injector wear
  - Time Between Spray and Blast
  - Laser Cleaning (PROTAL™ Processing)

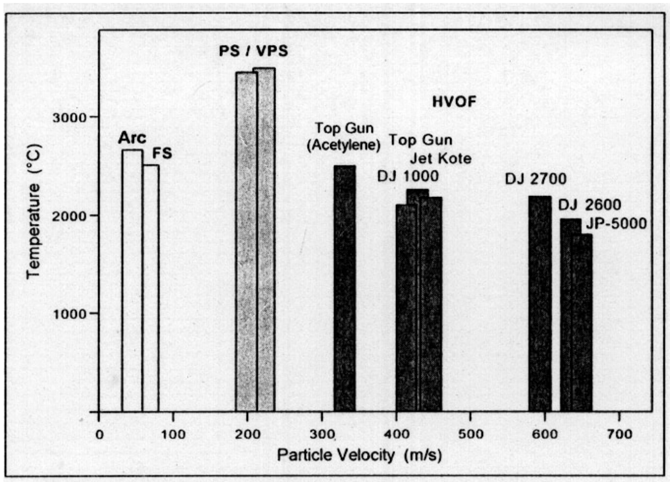
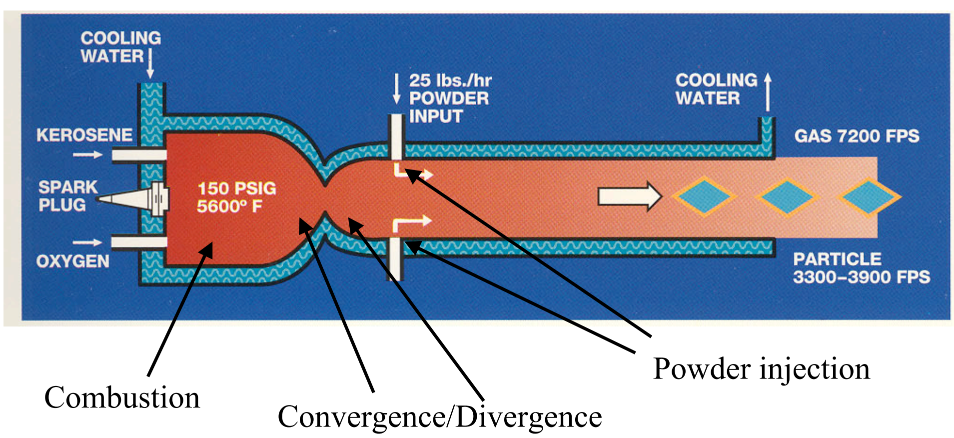


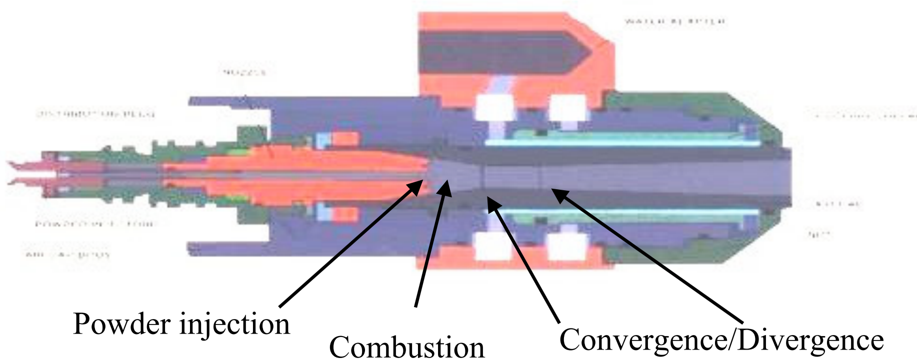
Bild 1: Erwärmung und Geschwindigkeit der Partikel bei verschiedenen Spritzverfahren (v-Messung mit WC-Co 83-17, -45+10 μm)

Figure 1. Particle temperatures and velocities for various spray processes (Velocity Measurements with WC-Co 83-17, -45+10 μm)

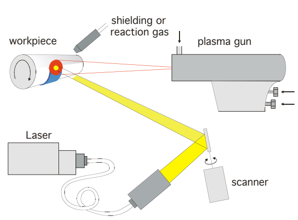
Praxair JP High Velocity Oxygen-Fueled (HVOF) Torch (Kerosene Fueled)

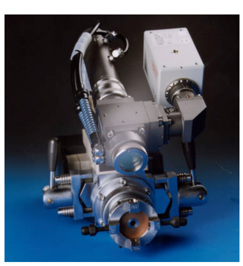


Sulzer Metco DJ High Velocity Oxygen-Fueled (HVOF) Torch (Gas Fueled)



Laser-Assisted Plasma Spray





- Direct interaction of the laser beam with the liquid spray particles and the substrate

⇒ one-step coating process

Plasma Assisted Laser Cladding

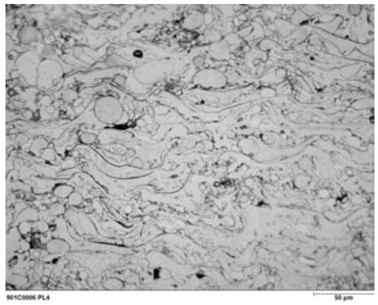
- Function sharing of the heat sources:

		process energy
- plasma torch:	melting the spray powder	90 – 95%
- laser beam:	fusing the spray particles and melting the substrate	5 – 10%

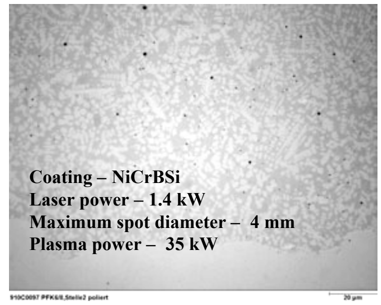
⇒ High efficiency for large-area coatings

Fraunhofer IWS Institut Werkstoff- und Strahltechnik

Laser-Assisted Plasma Spray



As-sprayed coating



Laser/plasma coating

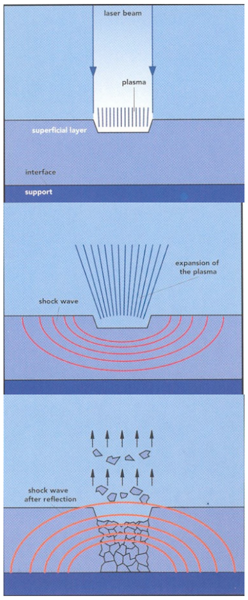
Results:
- Completely dense structure
- Metallurgical bonding to the substrate
- Thickness of a single layer: 50 … 150µm
- Minimum beam intensity: 8kW/cm²

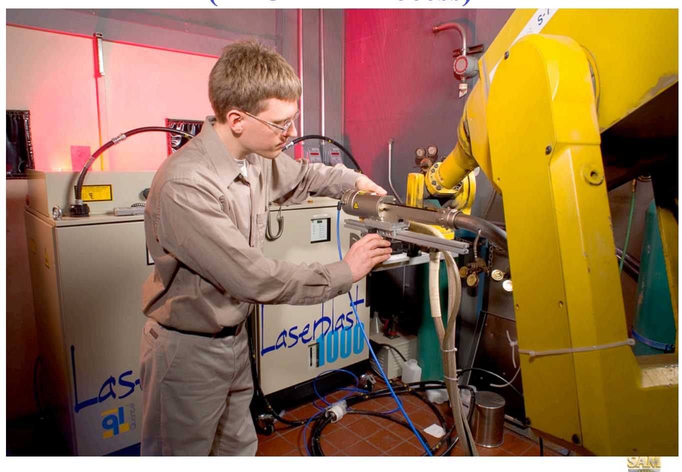
Fraunhofer IWS
Institut Werkstoff- und Strahltechnik

Caterpillar Laser-Assisted Plasma Spray Capability

4 kW YAG laser with fiber optic beam deliver system now installed in Caterpillar's Thermal Spray Laboratory to be used for LAPS

Future Work Will Involve Laser Surface Cleaning To Replace/Improve Grit Blast Surface Preparation (PROTAL Process)





	Jet Kote Top Gun DJ	JP-5000 DJ 2600 DJ 2700	Jet Kote Top Gun DJ	JP-5000 DJ 2600 DJ 2700
Porengehalt (%) Porosity (%)	≈ 0,5	≈ 0,1	≈ 1,0	≈ 0,8
Haftzugfestigkeit (MPa) Bond Strength (MPa)	75	80	60	80
Härte HV 0,3 Microhardness (VHN 0.3)	1050 – 1250	1200 – 1500	850 – 1050	1050 – 1250
Abrasivverschleiß (mg) *) Abrasive Wear *)	3 – 10	2 – 6	35 – 50	30 – 35
C-Abbrand (%) Decarburization (%)	40 – 60 **)	30 – 40	18 – 50	13 – 18
	WC-Co 83-17		Cr_3C_2-NiCr 75-25	

**) JIS H 8615, Abrieb nach 1200 DS **) bei Jet Kote (Wasserstoff) Abbrand nur 25 %*
**) JIS H 8615, mass loss after 1200 DS **) for Jet Kote (Hydrogen), carbon loss is only 25%*

Tabelle 1: Eigenschaften von Cermetschichten, gespritzt mit WC-Co 83-17 aggl. ges. -45+10 µm, Cr_3C_2-NiCr 75-25 aggl. ges. -45+10/15 µm

Table 1. Properties of cermet coatings sprayed with WC-Co 83-17 aggl. sintered -45+10 µm, Cr_3C_2-NiCr 75-25 aggl. sintered -45+10/15 µm

High-Performance Corrosion-Resistant Materials: Synthesis of Y_2O_3 Nanopowders

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Department of Materials Science
University of Nevada – Reno
Reno, Nevada

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ADC Signature

This document contains no sensitive subjects:

Sensitive Subject Reviewer Signature

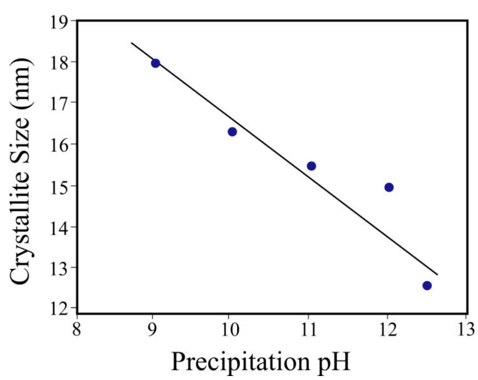
Introduction

- Sandia National Laboratory has observed a dispersion of Y_2O_3 nanoparticles in the SAM 1651 formulation, which is probably formed by the reaction of yttrium with oxygen.
- It is believed that this dispersion of nanoparticles can enhance both hardness and fracture toughness of these materials.
- The ***"Production of Test Materials"*** activity will enable further enhancement of the SAM1651 formulation, with the intentional introduction of Y_2O_3 nanoparticles to control hardness and fracture toughness, and achieve overall enhancement of the material properties.

Objectives

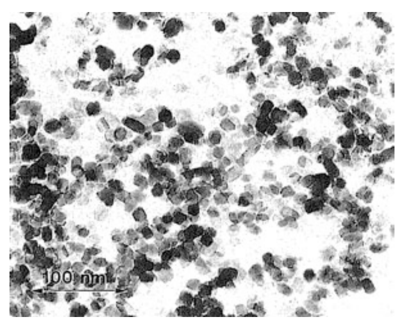
- The present investigation has been undertaken with the following specific objectives:
  - to synthesize nanostructured powders of Y_2O_3 using the reverse micelle synthesis process.
  - to characterize the process of reverse micelle synthesis over a wide range of experimental conditions in order to optimize and scale-up the process.

Solution Synthesis



$$M^{x+} + x(OH^-) \rightarrow M(OH)_x$$

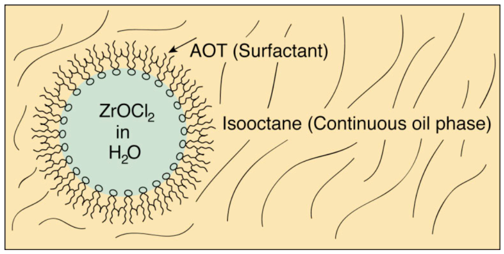
M.J. Mayo, D.C. Hague, and D.-J. Chen, "Processing nanocrystalline ceramics for applications in superplasticity," *Materials Science and Engineering A*, **166** (1993) 145-159.



Nanocrystalline ZrO_2-3 mol%Y_2O_3
Average Particle Diameter = 13 nm

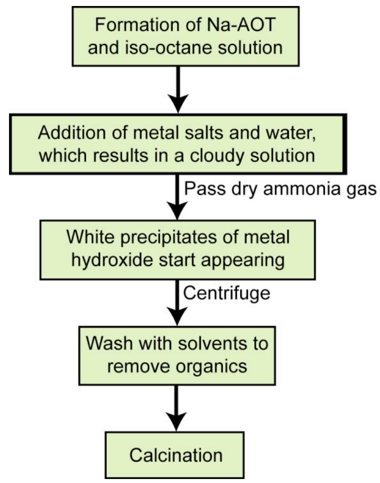
If the metal hydroxide $M(OH)_x$, has a moderate-to-low solubility product, it will precipitate out of the newly combined solution as a fine powder.

Reverse Micelle Synthesis

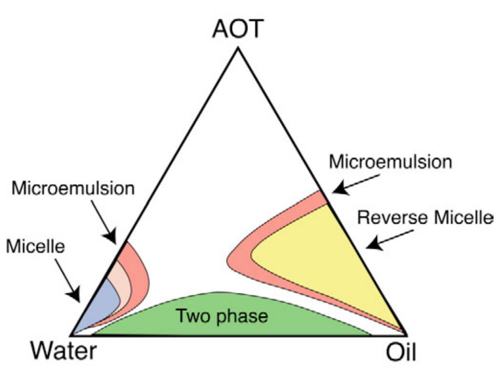


An aqueous phase is dispersed in an oil phase. The droplets of the aqueous phase are coated with a surfactant and they serve as chemical reactors for the production of nanocrystalline materials.

$$NH_3 + H_2O \rightarrow NH_4^+ + OH^-$$

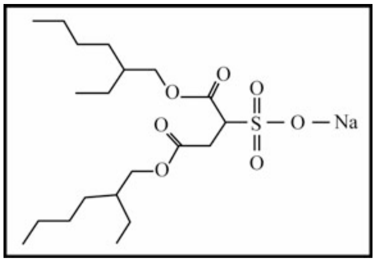


Why Reverse Micelle Synthesis?



- All precursor materials are commercially available.
- Offers high level of chemical homogeneity.
- Mixing is achieved at molecular levels.
- Ability to effect particle size and morphology.

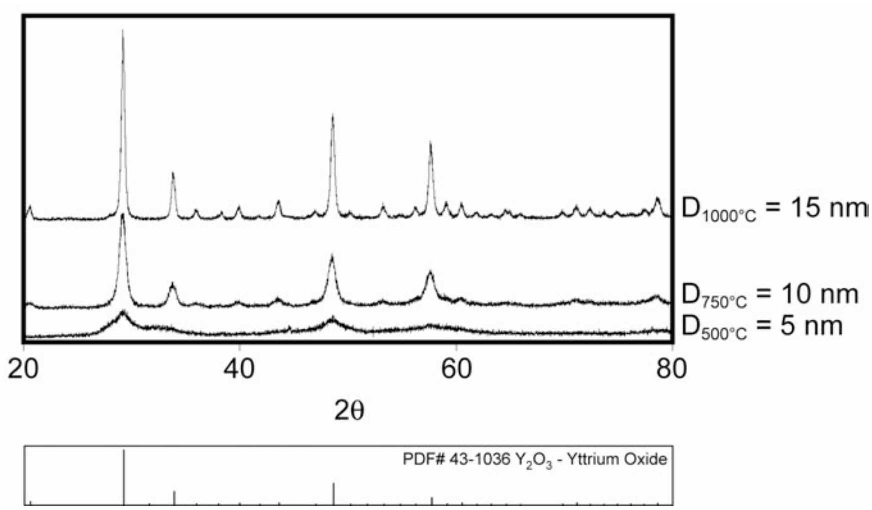
Reactants Used



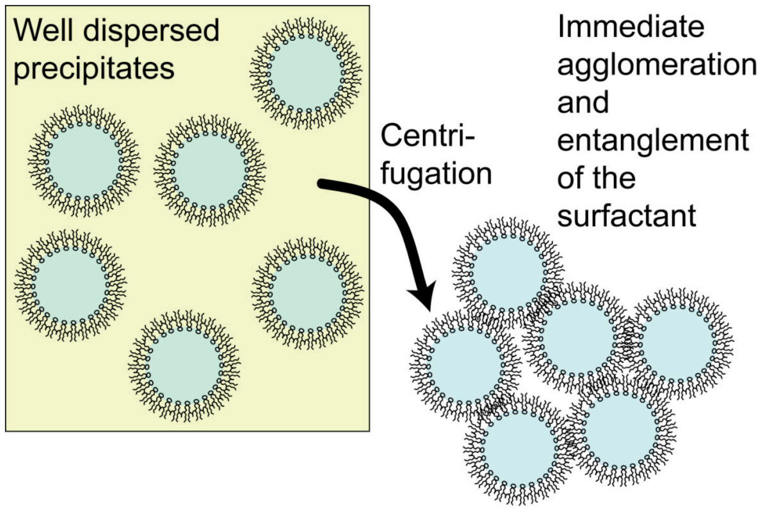
Na-AOT

Chemicals	Purpose
Yttrium Nitrate	Yttrium Ion Source
Isooctane	Non-Polar Solvent
AOT	Surfactant
De-Ionized water	Water Domains
Dry Ammonia Gas	Formation of Metal Hydroxide
Ethanol	Washing of Nanopowders

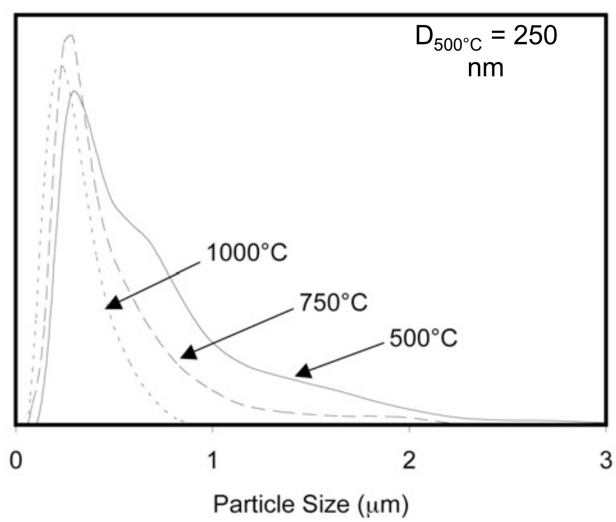
X-Ray Diffraction



Agglomeration Problem



Dynamic Light Scattering



Conclusions

- The tasks in this activity called for the preparation of significant quantities of Y_2O_3 nanoparticles and collaboration with SNL and UC Davis in introducing this nanoparticles into HVOF coatings of the SAM 1651 formulation.
- A 100-g batch of powders treated at 1000°C for two hours was delivered to SNL and a 2-kg batch of powders of the same type as the ones for SNL was delivered to UC Davis.

Future Work

- Scanning electron and transmission electron microscopy of the powders.
- FTIR for determination of organic impurities.
- Formation of Y2O3/SAM 1621 mixture via ball milling.
- Manufacture of coatings at UC Davis.

Backup Slides

High-Performance Corrosion-Resistant Materials: Corrosion Testing

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ADC Signature

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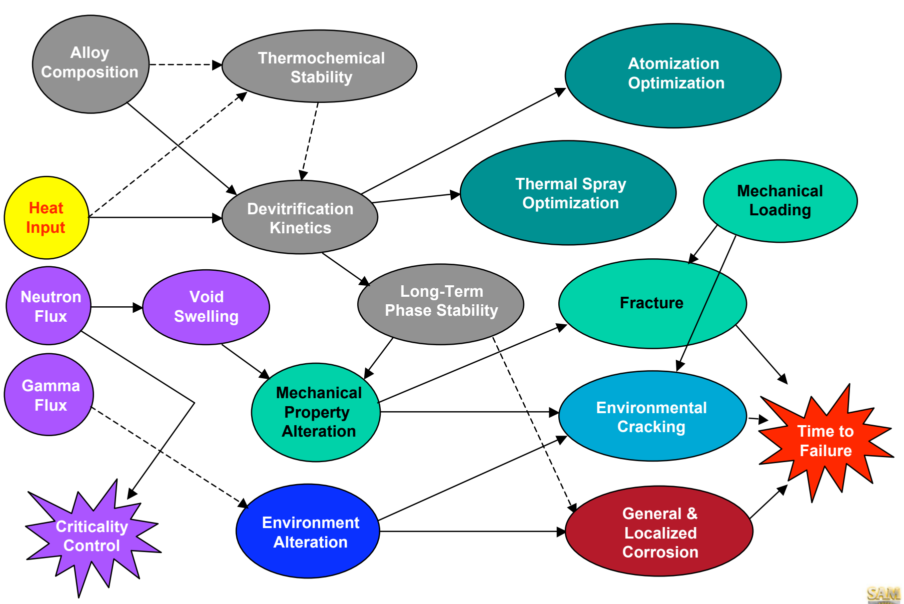
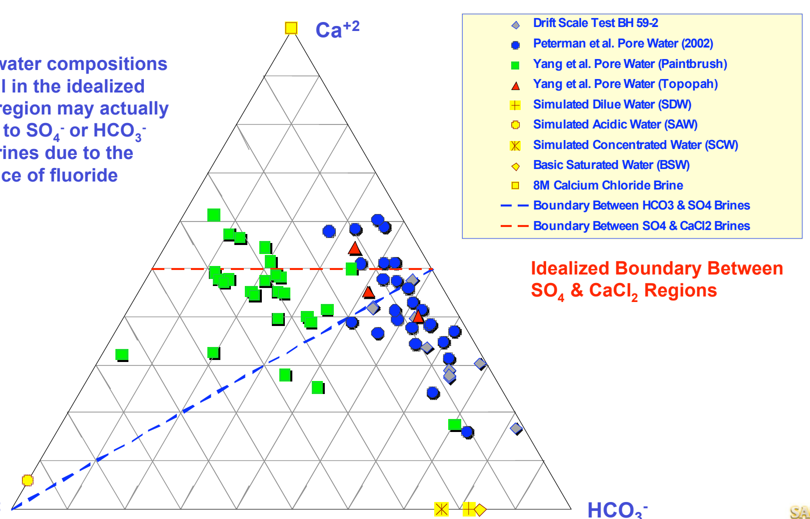
Sensitive Subject Reviewer Signature



Test Solutions

Classification of Yucca Mountain Brines

Many water compositions that fall in the idealized $CaCl_2$ region may actually evolve to SO_4^- or HCO_3^- type brines due to the presence of fluoride

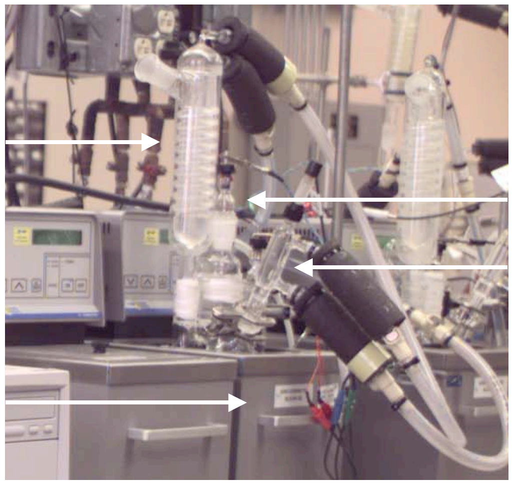


Corrosion Test Matrix at LLNL

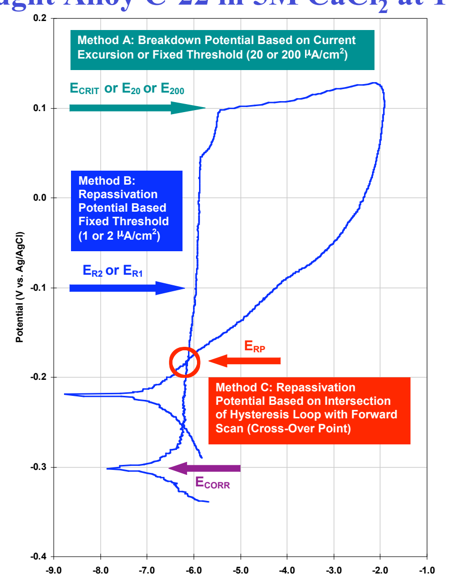
Test Matrix at Lawrence Livermore National Laboratory						
Test Solution Type	NaCl	KNO_3	T	$CaCl_2$	$Ca(NO_3)_2$	T
	M or m	M or m	°C	M or m	M or m	°C
Half Moon Bay SW			30, 90			
Half Moon Bay SW			30, 90			
Chloride-Nitrate	1 M	None	30, 90			
Chloride-Nitrate	3.5 m	None	30, 90			
Chloride-Nitrate	3.5 m	0.175 m	30, 90			
Chloride-Nitrate	3.5 m	0.525 m	30, 90			
Chloride-Nitrate	6.0 m	None	30, 90			
Chloride-Nitrate	6.0 m	0.300 m	30, 90			
Chloride-Nitrate	6.0 m	0.900 m	30, 90			
Calcium Chloride				5 M	None	105
Calcium Chloride				12 m	None	130
Calcium Chloride				12 m	6 m	130
Published References: PVP 2005 -71173; 71174; 71175; 71176.						

Quantifiable Metrics

Electrochemical Measurements in Temperature Controlled Cells at LLNL

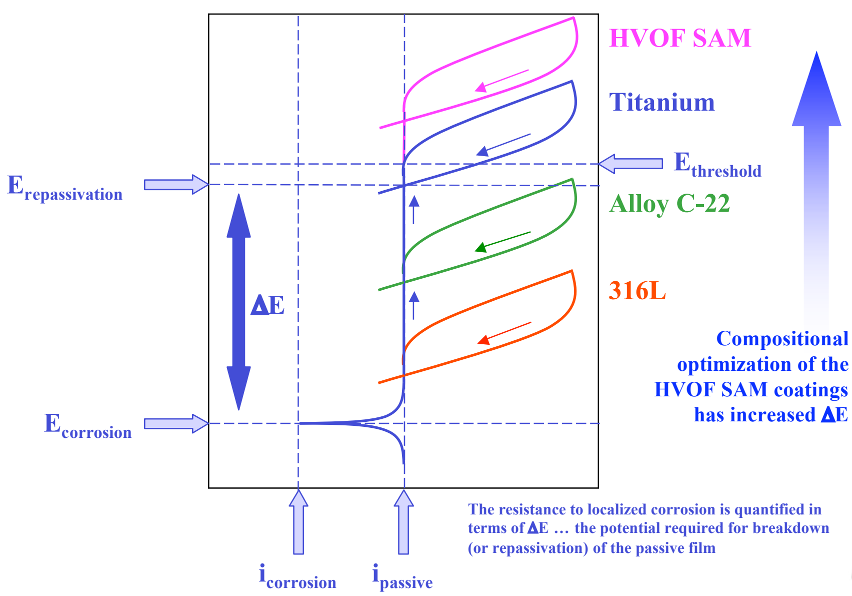


Definition of Critical & Repassivation Potentials Wrought Alloy C-22 in 5M $CaCl_2$ at 105°C



Alloy Screening

Repassivation Potential as a Quantifiable Metric



The resistance to localized corrosion is quantified in terms of ΔE … the potential required for breakdown (or repassivation) of the passive film

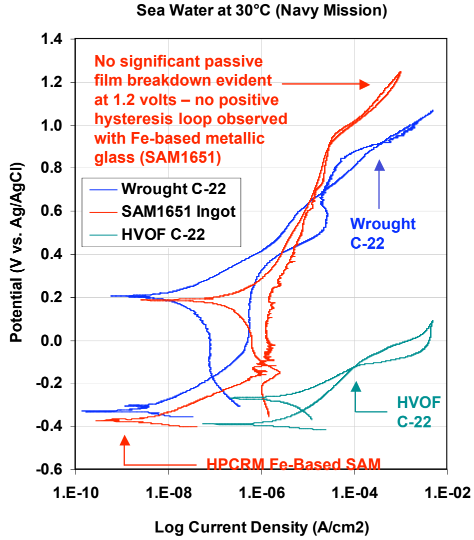
Synthesis & Screening of More Than Forty Candidates

Formulation	Formula	Fe	Cr	Mn	Mo	W	B	C	Si	Y	Zr	Ti	Co	Ni	Al	P	Other	Total
SAM27	$(Fe_{0.8}Cr_{0.2})_{73}Mo_2W_2B_{16}C_4Si_1Mn_2$	58.4	14.6	2.0	2.0	2.0	16.0	4.0	1.0									100.0
SAM35	$Fe_{54.5}Mn_2Cr_{15}Mo_2W_{1.5}B_{16}C_4Si_5$	54.2	15.0	2.0	2.0	1.5	16.0	4.0	5.0								0.3	100.0
SAM40	$Fe_{52.3}Mn_2Cr_{19}Mo_{2.5}W_{1.7}B_{16}C_4Si_{2.5}$	52.3	19.0	2.0	2.5	1.7	16.0	4.0	2.5									100.0
SAM40X3	$Fe_{50.7}Mn_{1.9}Cr_{18.4}Mo_{5.4}W_{1.6}B_{15.5}C_{3.9}Si_{2.4}$	50.7	18.4	1.9	5.4	1.6	15.5	3.9	2.4								0.2	100.0
SAM1	$(SAM40)_{100-x}+Ni_x$																	
SAM1X1	$(SAM40)_{99}+Ni_1$	51.8	18.8	2.0	2.5	1.7	15.8	4.0	2.5					1.0				100.0
SAM1X3	$(SAM40)_{97}+Ni_3$	50.7	18.4	1.9	2.4	1.6	15.5	3.9	2.4					3.0				100.0
SAM1X5	$(SAM40)_{95}+Ni_5$	49.7	18.1	1.9	2.4	1.6	15.2	3.8	2.4					5.0				100.0
SAM1X7	$(SAM40)_{93}+Ni_7$	48.6	17.7	1.9	2.3	1.6	14.9	3.7	2.3					7.0				100.0
SAM2	$(SAM40)_{100-x}+Mo_x$																	
SAM2X1	$(SAM40)_{99}+Mo_1$	51.8	18.8	2.0	3.5	1.7	15.8	4.0	2.5									100.0
SAM2X3	$(SAM40)_{97}+Mo_3$	50.7	18.4	1.9	5.4	1.6	15.5	3.9	2.4									100.0
SAM2X5	$(SAM40)_{95}+Mo_5$	49.7	18.1	1.9	7.4	1.6	15.2	3.8	2.4									100.0
SAM2X7	$(SAM40)_{93}+Mo_7$	48.6	17.7	1.9	9.3	1.6	14.9	3.7	2.3									100.0
SAM3	$(SAM40)_{100-x}+Y_x$																	
SAM4	$(SAM40)_{100-x}+Ti_x$																	
SAM5	$(SAM40)_{100-x}+Zr_x$																	
SAM6	$Fe_{43}Cr_{16}Mo_{16}B_5C_{10}P_{10}$	43.0	16.0		16.0		5.0	10.0								10.0		100.0
SAM7 (SAM1651)	$Fe_{48}Mo_{14}Cr_{15}Y_2C_{15}B_6$	48.0	15.0		14.0		6.0	15.0		2.0								100.0
SAM8	$(Fe_{48}Mo_{14}Cr_{15}Y_2C_{15}B_6)_{97}+W_3$	46.6	14.6		13.6	3.0	5.8	14.6		1.9								100.0
SAM9	$(SAM40)_{90}+Mo_7+Y_3$	47.1	17.1	1.8	9.3	1.5	14.4	3.6	2.3	3.0								100.0
SAM10	$Fe_{57.3}Cr_{21.4}Mo_{2.6}W_{1.8}B_{16.9}$	57.3	21.4		2.6	1.8	16.9											100.0

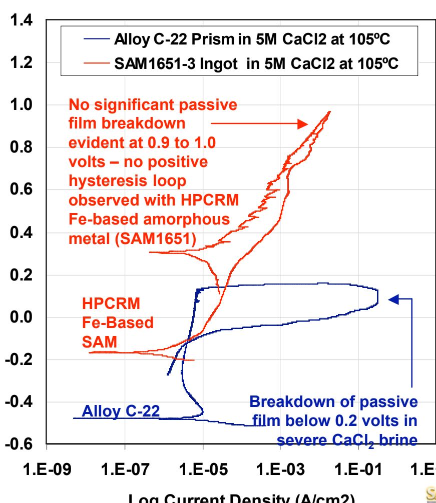
HPCRM MSR Samples by TNC

Thermally Sprayed Fe-Based Amorphous Metal Better Than C-22 in Seawater & Hot $CaCl_2$ Brines

Cyclic Polarization of HPCRM Fe-Based SAM in Sea Water at 30°C (Navy Mission)

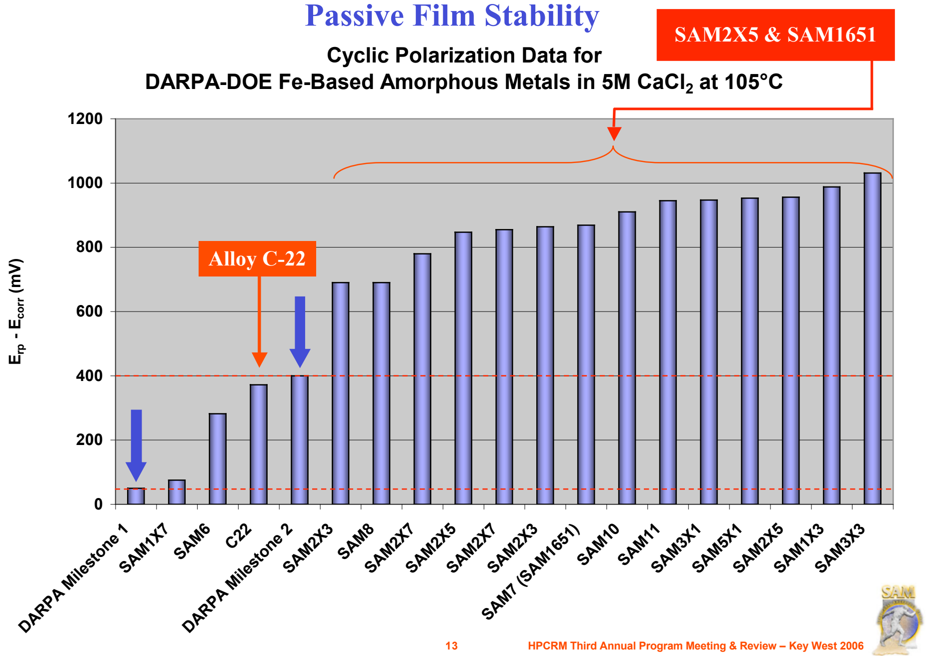


Cyclic Polarization of Alloy C-22 and HPCRM Fe-Based SAM in 5M CaCl2 at 105°C (DOE Mission)



Passive Film Stability

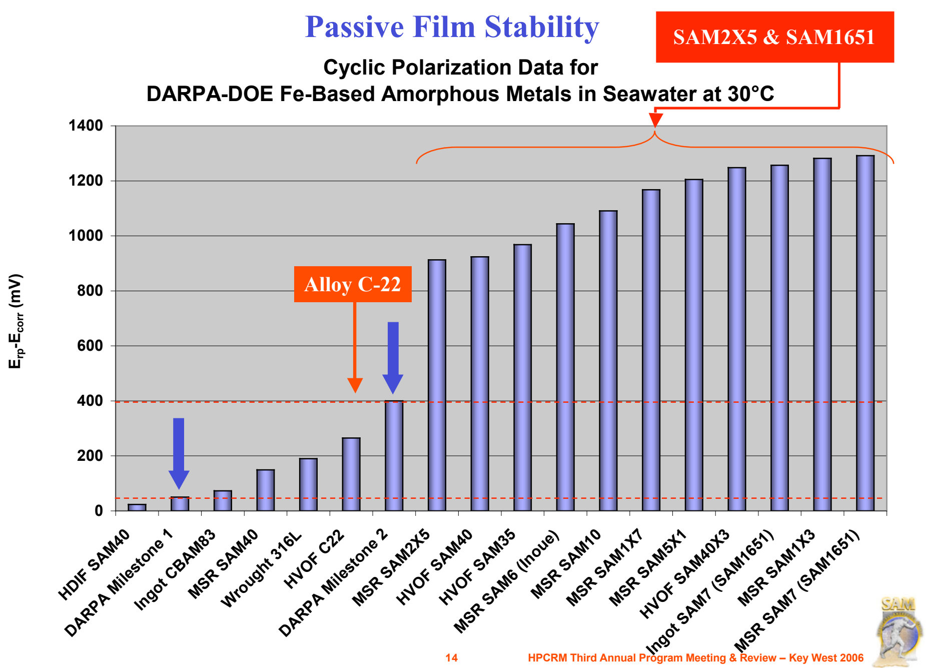
Cyclic Polarization Data for
DARPA-DOE Fe-Based Amorphous Metals in 5M $CaCl_2$ at 105°C



13 HPCRM Third Annual Program Meeting & Review – Key West 2006

Passive Film Stability

Cyclic Polarization Data for
DARPA-DOE Fe-Based Amorphous Metals in Seawater at 30°C



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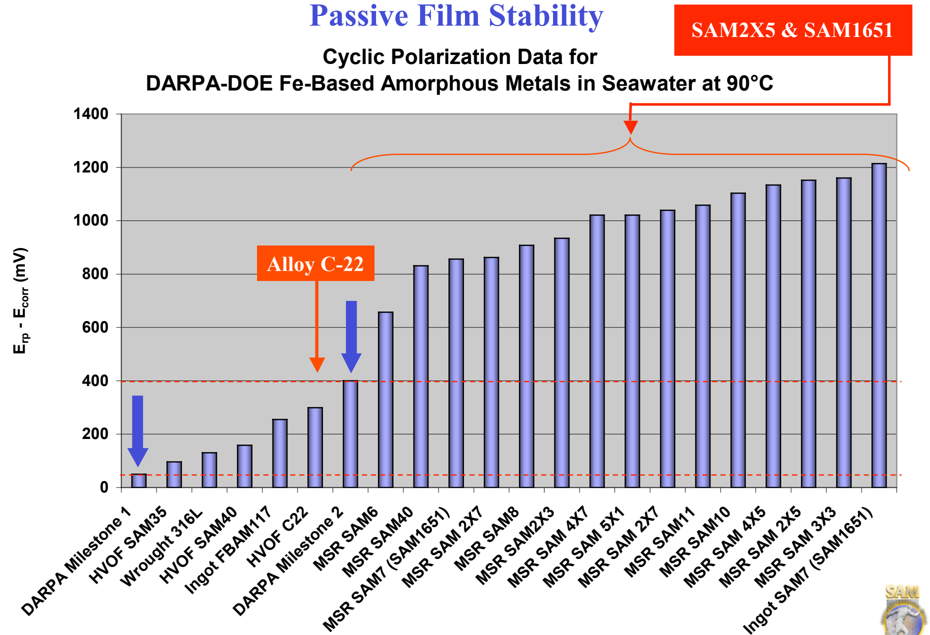
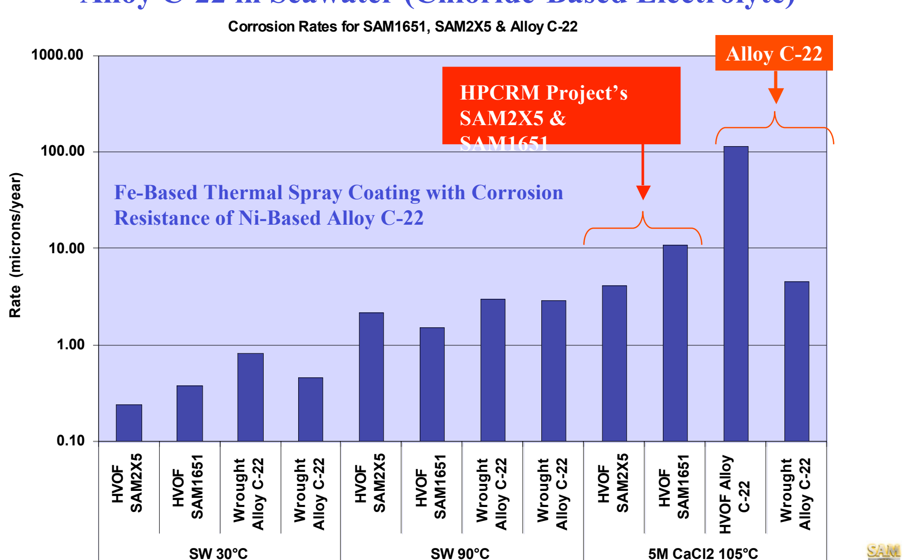
Passive Film Stability



HPCRM Materials Corrode More Slowly Than Alloy C-22 in Seawater (Chloride-Based Electrolyte)



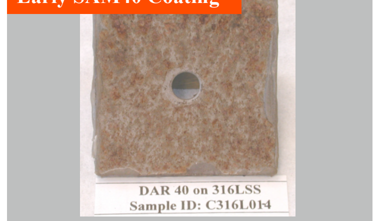
Salt Fog Test

No Corrosion Observed During Salt Fog Testing

Stainless Steel Coating

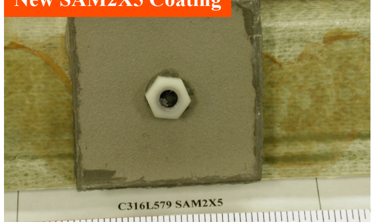
Type 316L Stainless – After Exposure – 13 Cycles

Early SAM40 Coating



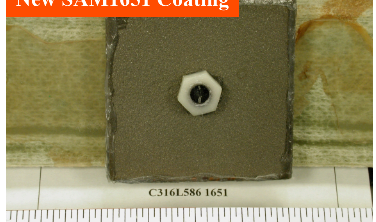
Original DAR40 – After Exposure – 13 Cycles

New SAM2X5 Coating



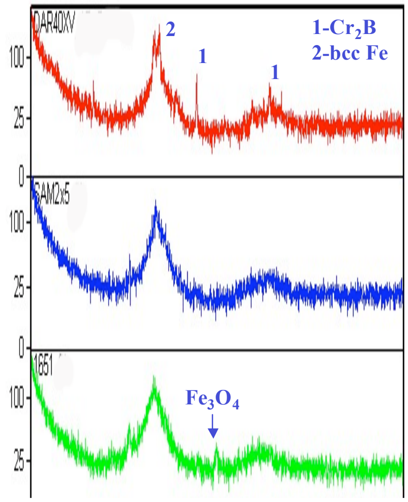
HPCRM SAM2X5 – After Exposure – 30 Cycles

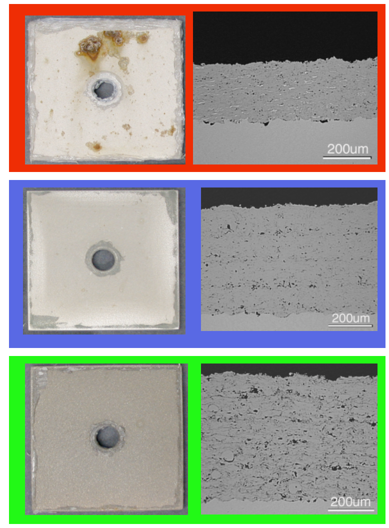
New SAM1651 Coating



HPCRM SAM1651 – After Exposure – 30 Cycles

Exceptional Corrosion Resistance of SAM2X5 & SAM1651 Attributed to Sustained Amorphous Structure

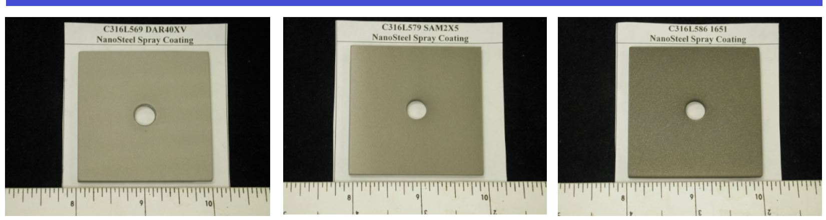




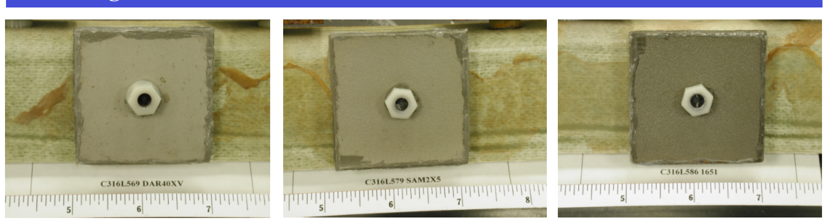
Observed Corrosion of Early SAM40 (DAR40) Coating Due to Devitrification

Standard Salt Fog Testing – NSWC

As-Received HVOF SAM40XV, SAM2X5 & SAM1651 on 316L Substrates

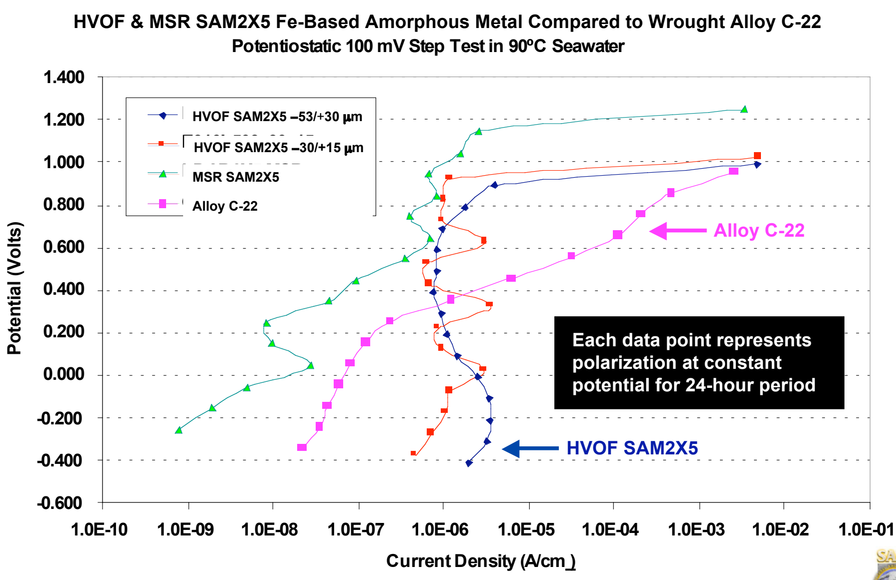


30-60 Cycles in Salt Fog:
Slight Attack of SAM40XV / No Attack of SAM2X5 or SAM1651

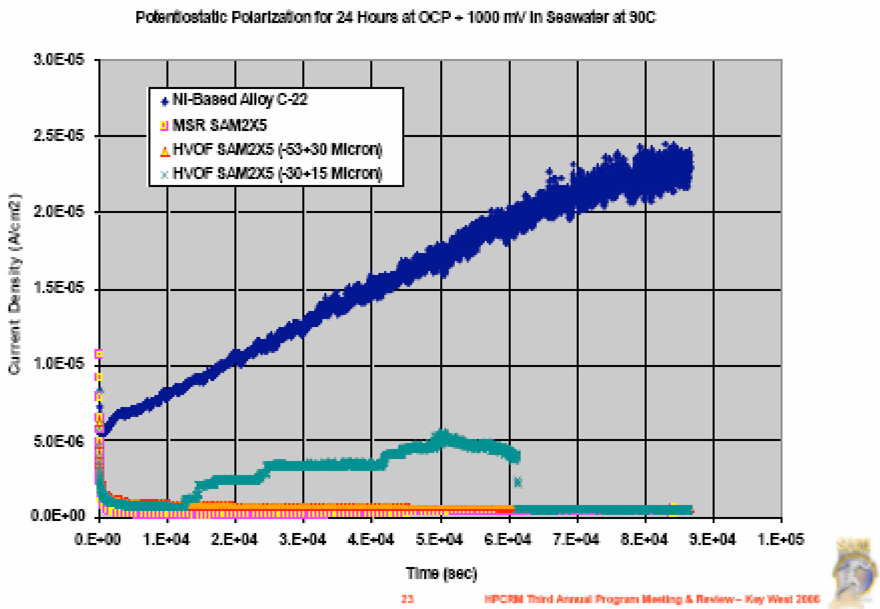


Potentiostatic Step Test

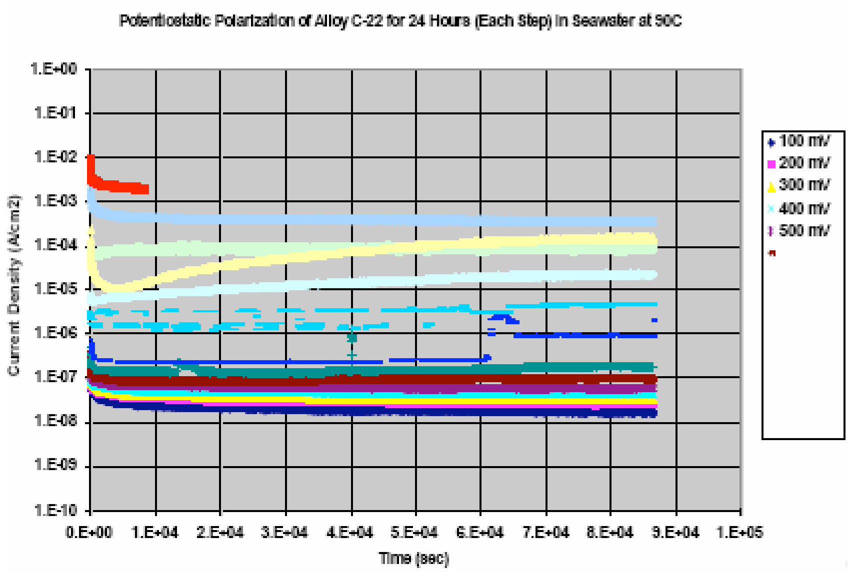
Potentiostatic Polarization of SAM2X5 in Hot Seawater



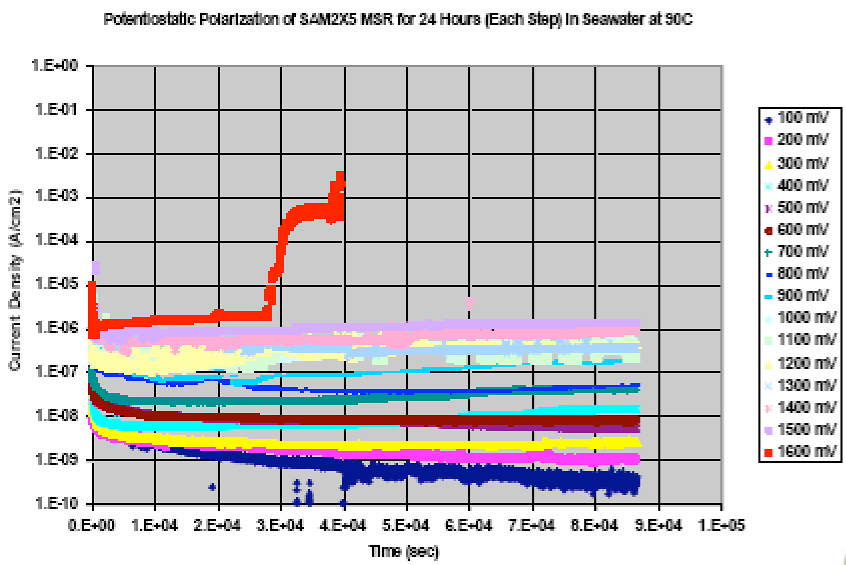
Comparison of Alloy C-22 & SAM2X5 in Hot Seawater



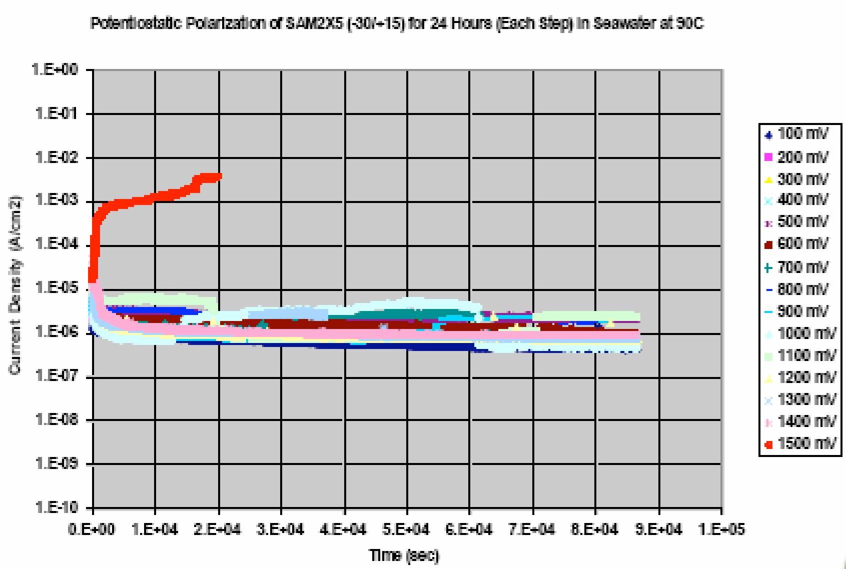
Potentiostatic Polarization of Wrought Alloy C-22 in 90°C Seawater



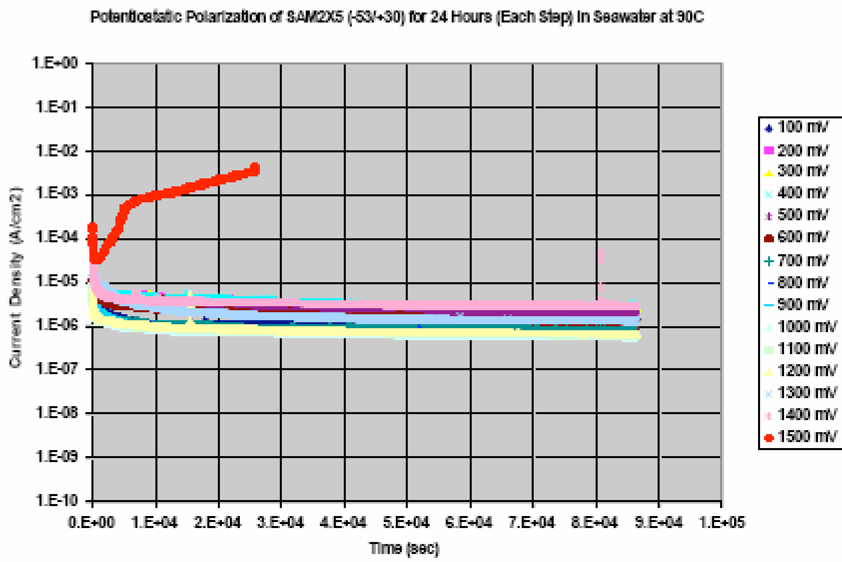
Potentiostatic Polarization of Melt Spun Ribbon of SAM2X5 in 90°C Seawater



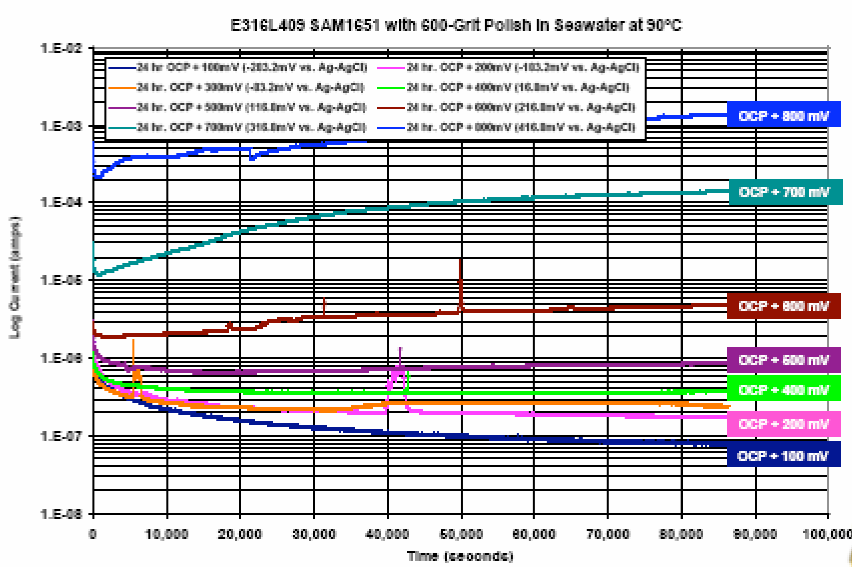
Potentiostatic Polarization of Polished HVOF SAM2X5 (-30/+15 μm) in 90°C Seawater



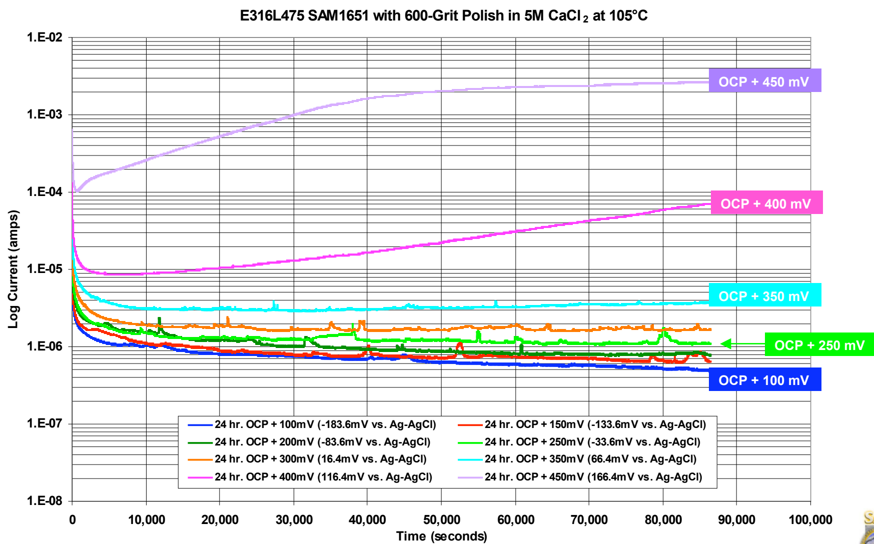
Potentiostatic Polarization of Polished HVOF SAM2X5 (-53/+30 µm) in 90°C Seawater



Potentiostatic Polarization of Polished SAM1651 HVOF Coating in 90°C Seawater

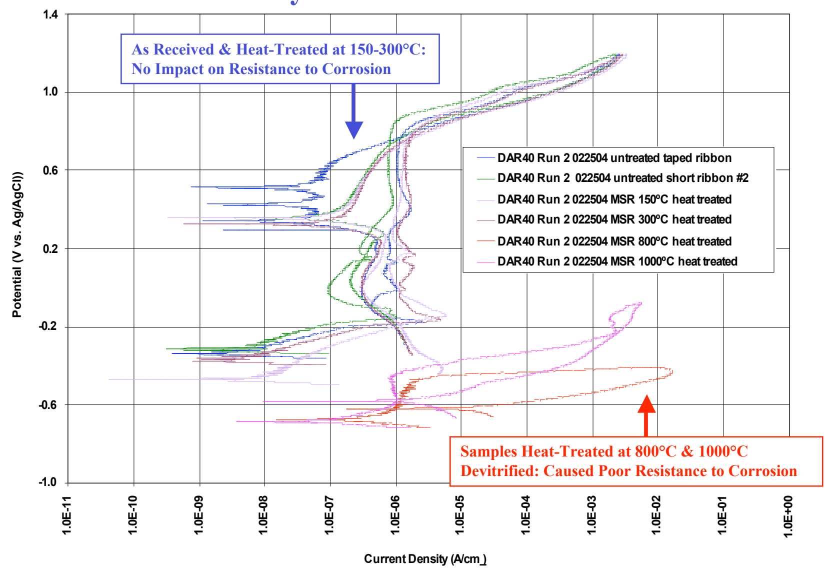


Potentiostatic Polarization of SAM1651
Polished SAM1651 HVOF Coating in 105°C 5M $CaCl_2$

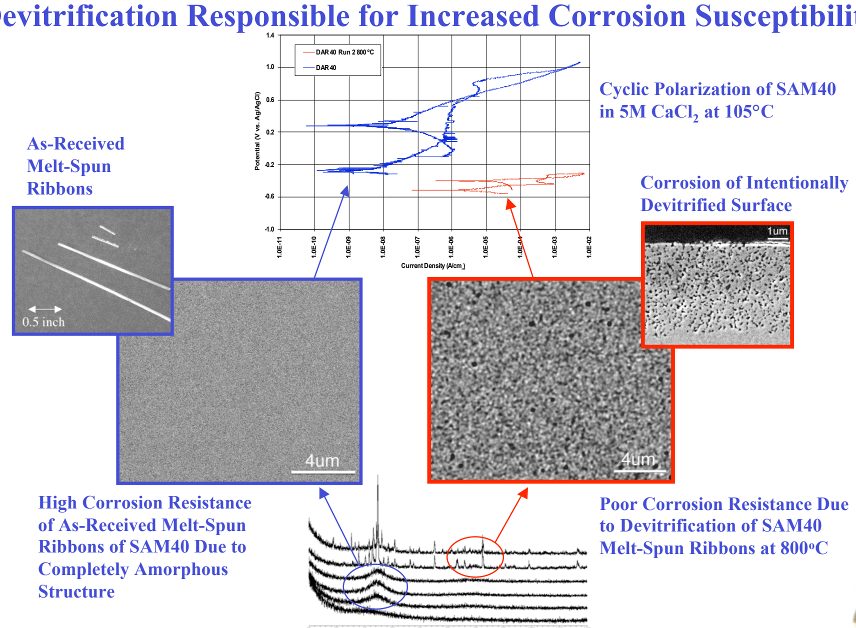


Effects of Devitrification

Effect of High Temperature on Corrosion Resistance of Early SAM40 Formulation

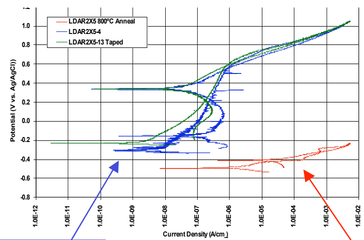


SAM40 (DAR40) Devitrifies After 1 Hour at 800°C: Devitrification Responsible for Increased Corrosion Susceptibility



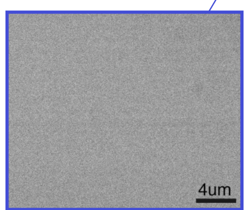
SAM2X5 (LDAR2X5) Devitrifies After 1 Hour at 800°C: Devitrification Responsible for Increased Corrosion Susceptibility

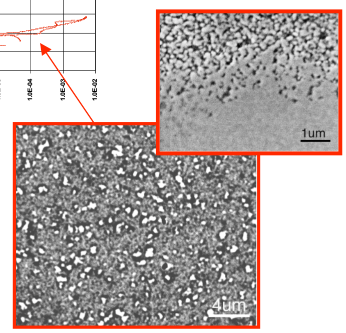
Cyclic Polarization in 5M $CaCl_2$ at 105°C



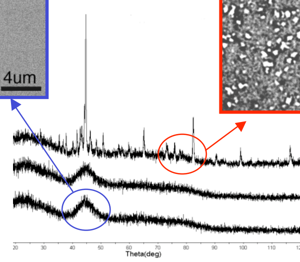
Corrosion of Intentionally Devitrified Surface

As-Received Melt-Spun Ribbons





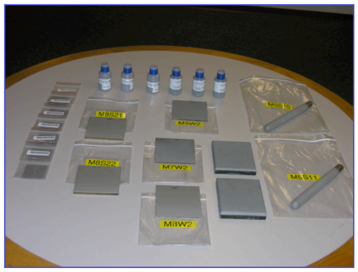
High Corrosion Resistance of As-Received Melt-Spun Ribbons of SAM2X5 Due to Completely Amorphous Structure

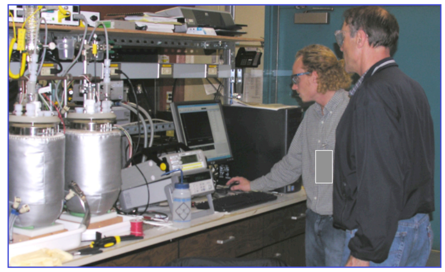


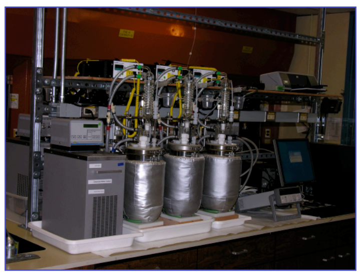
Poor Corrosion Resistance Due to Devitrification of SAM2X5 Melt-Spun Ribbons at 800°C

Long-Term Corrosion Tests

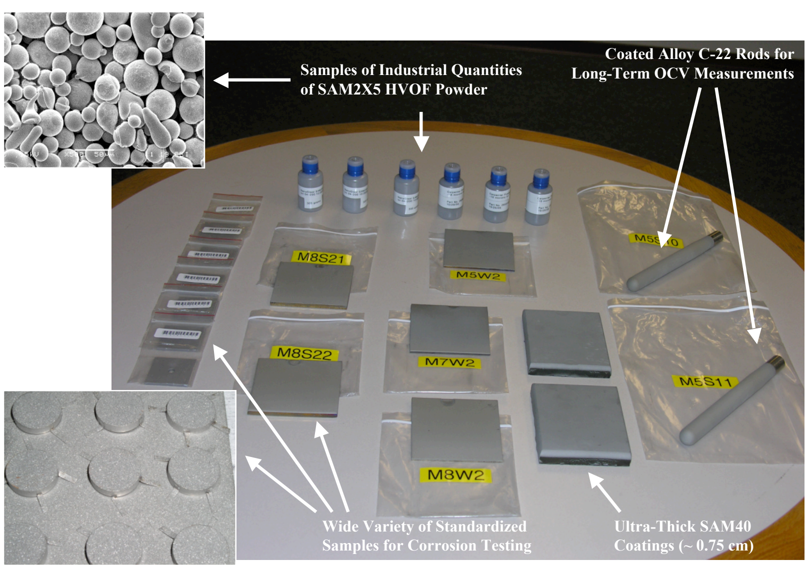
Long-Term Immersion Testing of SAM2X5 & SAM1651 Corrosion Potential, Weight Loss & Crevice Corrosion – LLNL



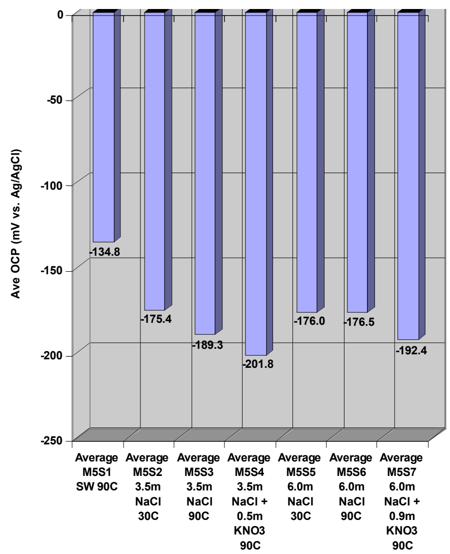


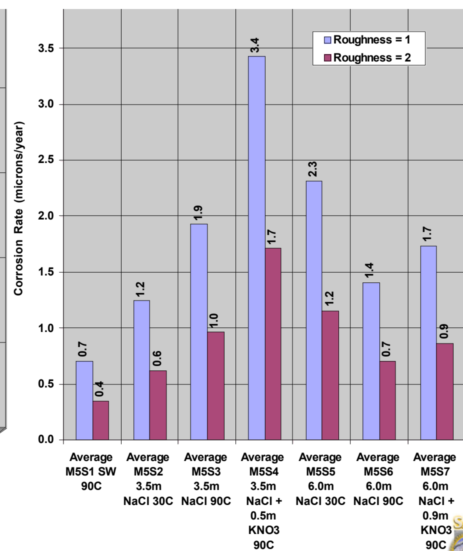


SAM2X5 Powder & HVOF Samples – TNC/INL/Caterpillar

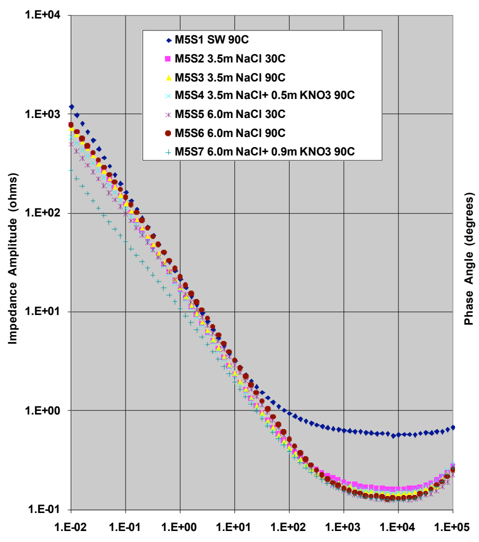


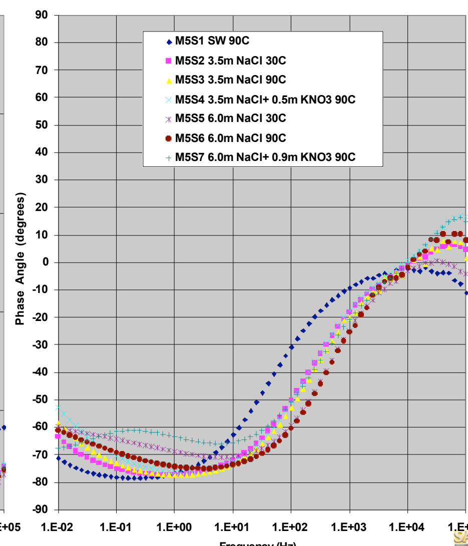
Long-Term Testing of SAM2X5: Initial Corrosion Potential & Rates





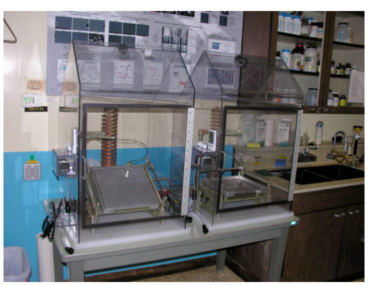
Long-Term Testing of SAM2X5: Initial Electrochemical Impedance Spectra

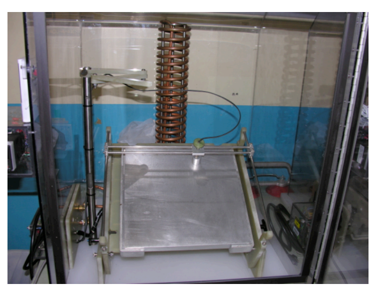


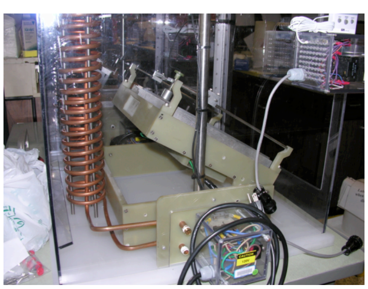


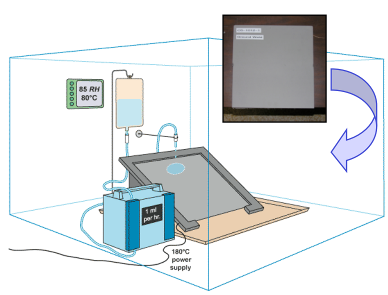
Testing Large Coated Alloy C-22 Plates

Exposure of Hot Plate to Dripping Geothermal Brines – LLNL

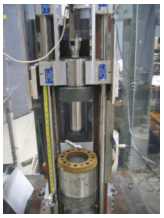


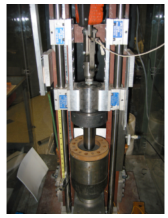


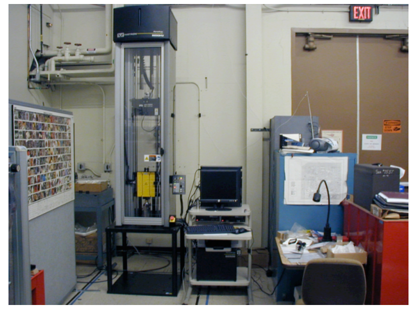




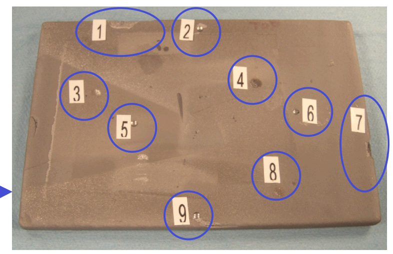
Impact Testing with Fully Instrumented Drop Towers – LLNL





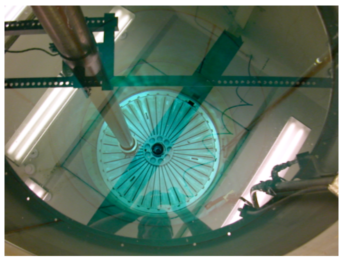


Preliminary Damage Testing of HY80 Plate:
- Impact from Side: 1
- Impact Directly on Top: 2, 5, 6, 9
- Ball Peen Hammer Top: 3, 4, 8
- Ball Peen Hammer on Edge: 7



Effects of Radiation on Corrosion Resistance

Gamma Pit at Lawrence Livermore National Laboratory



Irradiation, Attenuation & Radiography at TRIGA Reactor

Unperturbed Neutron Fluxes & Heating at 1.5 MW Operating Power in MNRC's TRIGA Reactor						
Facility	Thermal < 0.1 eV	Fast > 1 MeV	$\frac{\varphi_{fast}}{\varphi_{thermal}}$	Heating in Aluminum	Diameter	Length
	n cm^{-2} sec^{-1}	n cm^{-2} sec^{-1}	%	W g^{-1}	cm	cm
Central Irradiation Facility (CIF)	1.5×10^{13}	7.6×10^{12}	50	0.16	4.4	38
Pneumatic Transfer System (PTS)	7.6×10^{12}	3.7×10^{12}	50	0.084	1.5	10
Neutron Transmutation Doping (NTD)	4.4×10^{11}	1.1×10^{13}	2.5	0.0027	8.8	22

Summary

Summary

- Two Fe-based amorphous metal formulations have been found that appear to have corrosion resistance comparable to (or better than) that of Ni-based Alloy C-22 (based on breakdown potential and corrosion rate)
  - Cr & Mo provide corrosion resistance
  - B enables glass formation
  - Y lowers critical cooling rate
    - SAM1651 = 80 K/s (yttrium added)
    - SAM2X7 = 610 K/s (no yttrium)
- Both amorphous metal formulations have strengths and weaknesses
  - SAM1651 (yttrium added)
    - Low critical cooling rate (CCR) = amorphous in 'as sprayed' condition
    - Irregular powder = difficult to atomize and spray
    - Possible need for cryogenic milling of powder
  - SAM2X5 (no yttrium)
    - High critical cooling rate = potential problem with devitrification
    - Spherical powder = more easily atomized and thermally sprayed

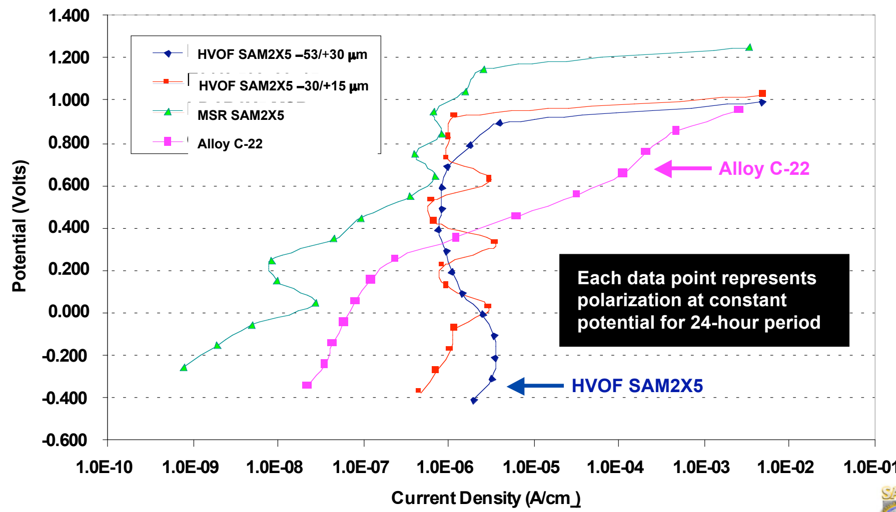
Summary

- Alloy C-22 is an outstanding corrosion-resistant engineering material
  - Even so … crevice corrosion has been observed with C-22 in hot sodium chloride environments without buffer or inhibitor
  - Comparable metallic alloys such as SAM2X5 and SAM1651 may also experience crevice corrosion under sufficiently harsh conditions
  - Accelerated crevice corrosion tests are now being conducted to intentionally induce crevice corrosion … and to determine those environmental conditions where such localized attack occurs
- Such 'super hard steels' provide enhanced resistance to abrasion and gouges (stress risers) from backfill operations … and possibly even tunnel boring
  - Type 316L Stainless Steel = 150 VHN
  - Alloy C-22 = 250 VHN
  - HVOF SAM2X5 = 1100-1300 VHN

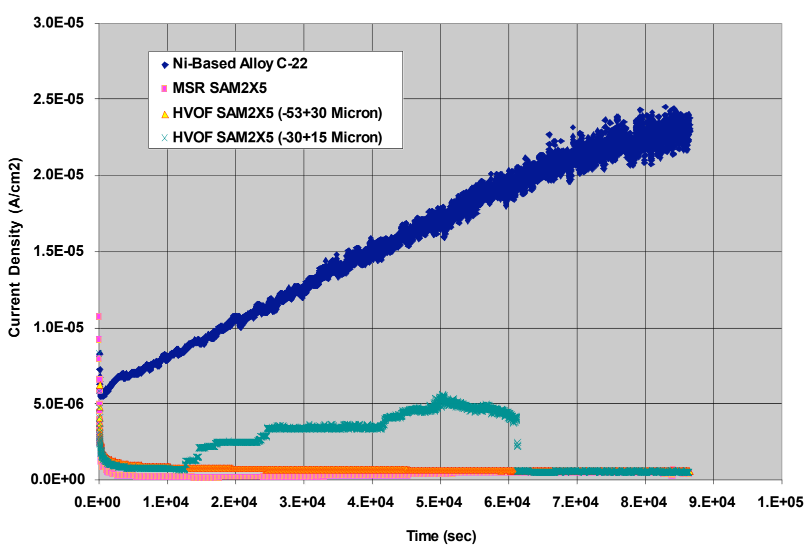
Summary

- These new materials provide a viable coating option for repository engineers
  - SAM2X5 & SAM1651 coatings can be applied with thermal spray processes without any significant loss of corrosion resistance
  - Both Alloy C-22 and Type 316L stainless lose their resistance to corrosion during thermal spraying
- SNF/HLW containers with corrosion resistant coatings are envisioned
  - Enhanced multi-purpose container (MPC) … leverage existing capability
  - Protected closure weld … eliminate need for stress mitigation
  - Integral drip shield … elimination of titanium drip shield
  - Thicken areas where greater corrosion is expected (crevices)
- Both SAM2X5 & SAM1651 have high boron content which enable them to absorb neutrons and therefore be used for criticality control in baskets
  - Alloy C-22 and 316L have no neutron absorber
  - Borated stainless steel and Gd-doped Ni-Cr-Mo alloys have relatively poor corrosion performance
  - Boron is believed to be a better neutron absorber than gadolinium

Summary

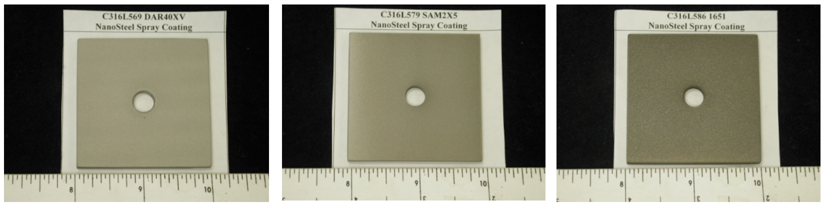


Summary

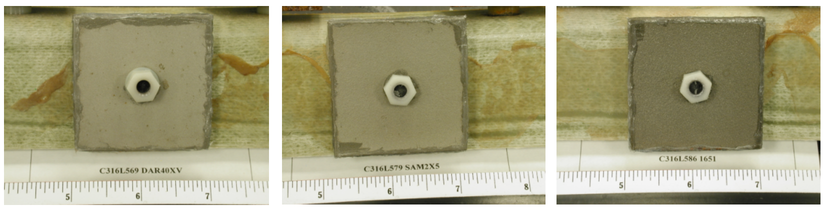


Summary

As-Received HVOF SAM40XV, SAM2X5 & SAM1651 on 316L Substrates

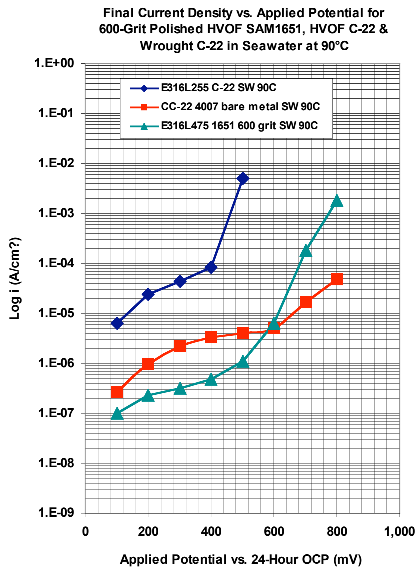


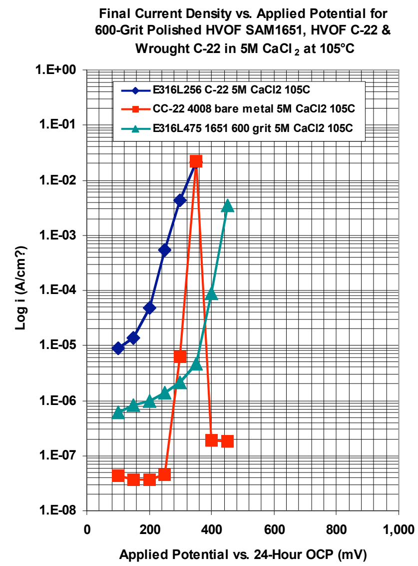
30-60 Cycles in Salt Fog:
Slight Attack of SAM40XV / No Attack of SAM2X5 or SAM1651



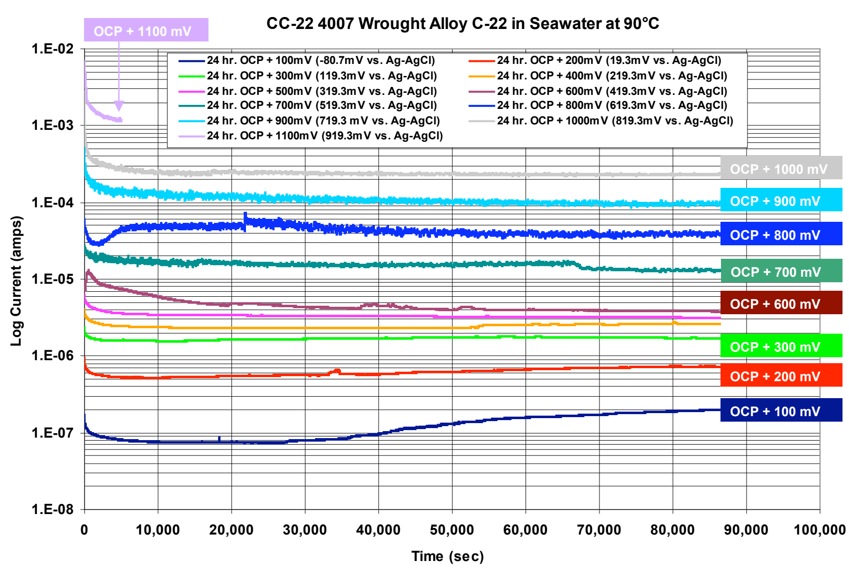
Backup Slides

Potentiostatic Polarization of Polished HVOF SAM1651 Coatings

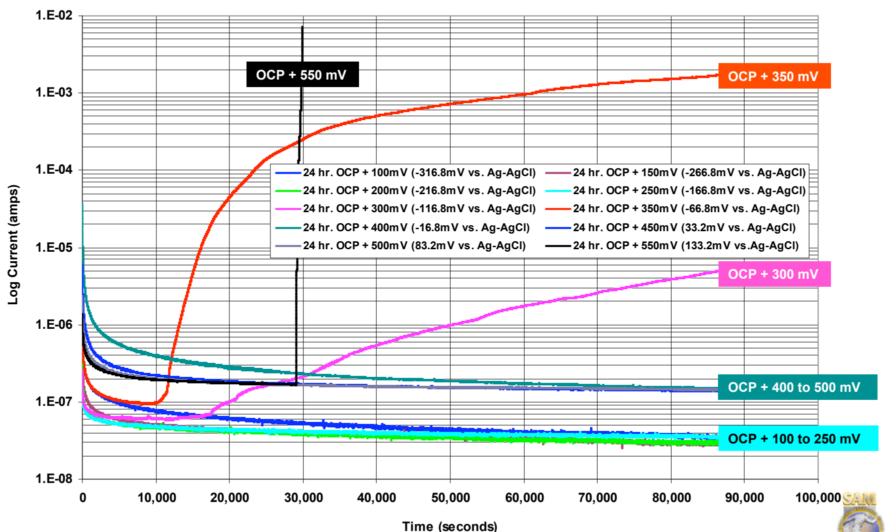




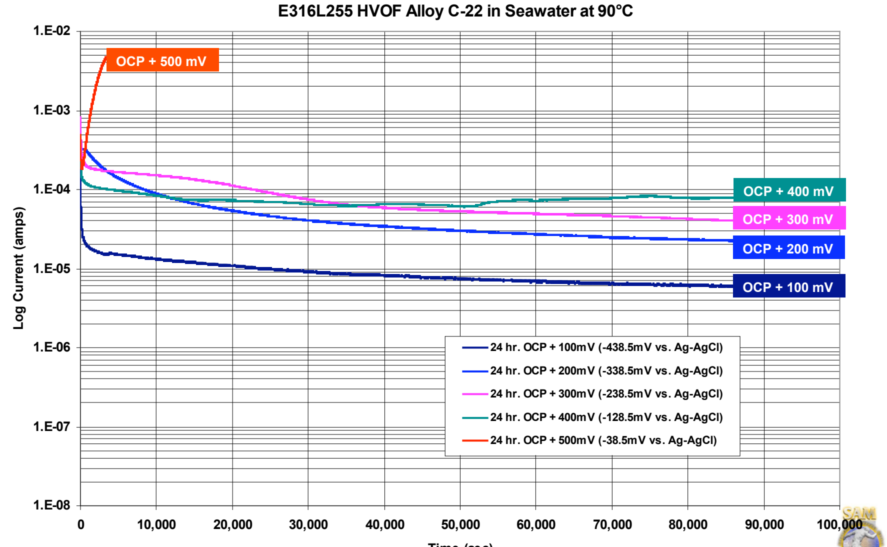
Potentiostatic Polarization of Polished Alloy C-22 in 90°C Seawater



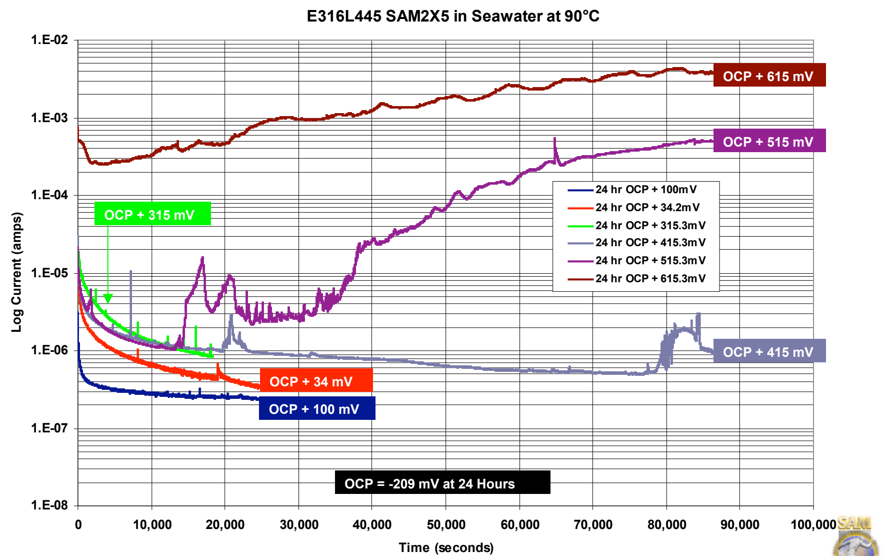
Potentiostatic Polarization of Polished Alloy C-22 in 105°C 5M $CaCl_2$



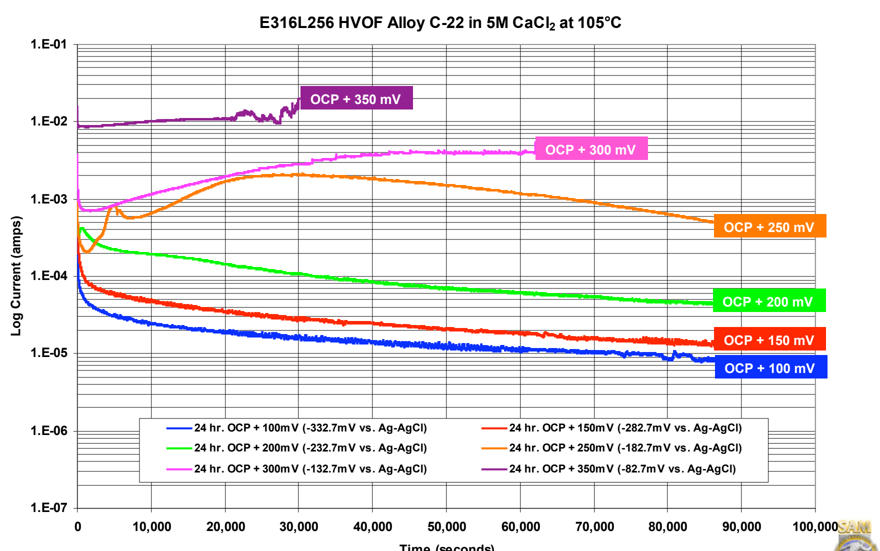
Potentiostatic Polarization of Unpolished Alloy C-22 HVOF Coating in 90°C Seawater



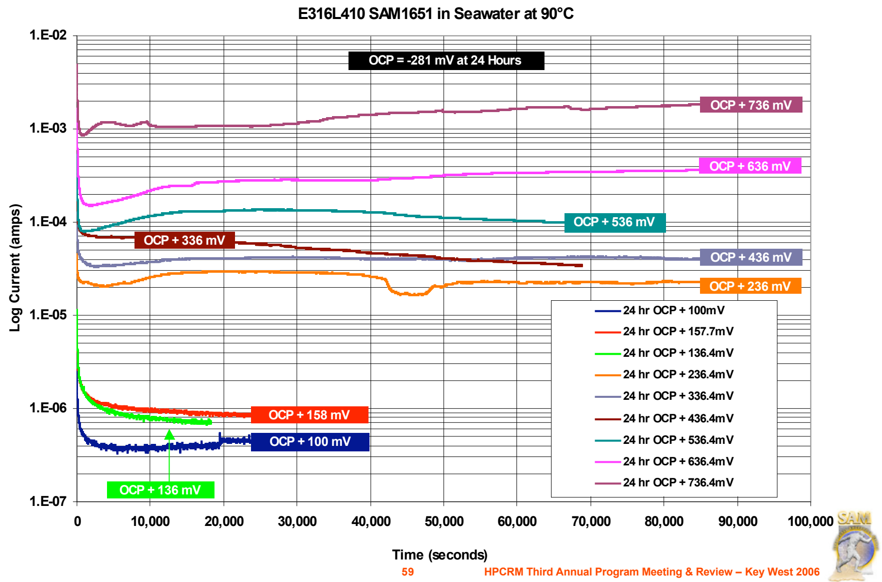
Potentiostatic Polarization
Unpolished SAM2X5 HVOF Coating in 90°C Seawater



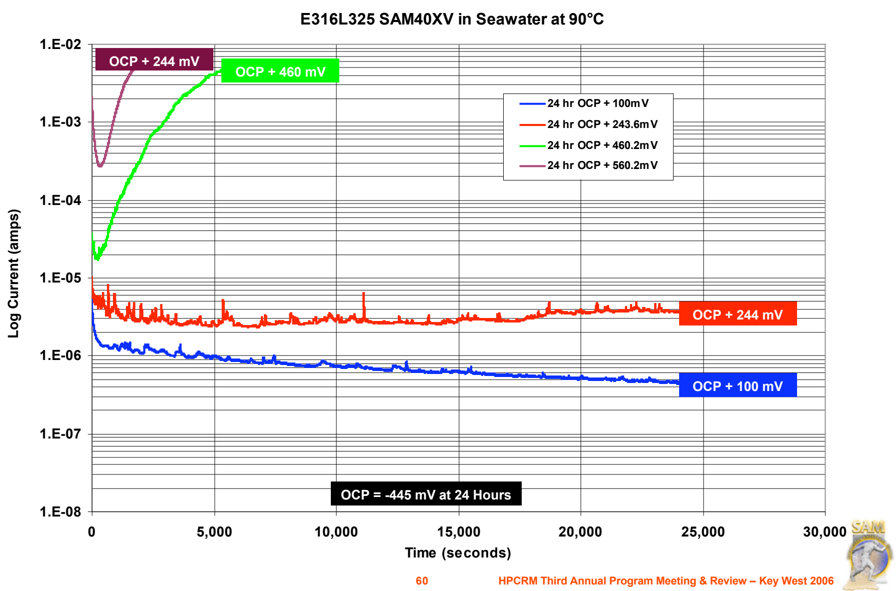
Potentiostatic Polarization of Alloy C-22
Unpolished Alloy C-22 HVOF Coating in 105°C 5M $CaCl_2$



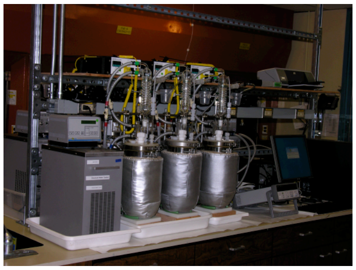
Potentiostatic Polarization of Unpolished SAM1651 HVOF Coating in 90°C Seawater

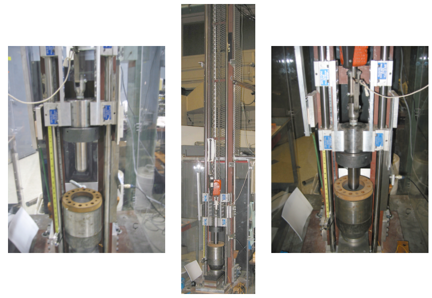


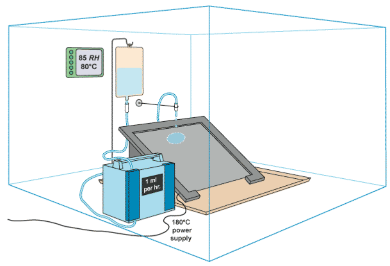
Potentiostatic Polarization Unpolished SAM40XV HVOF Coating in 90°C Seawater



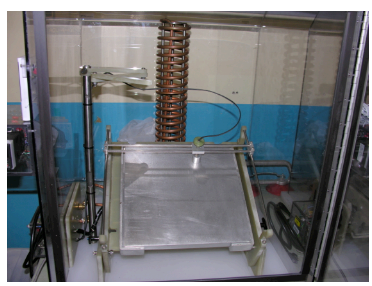
SAM2X5 & SAM1651 Subjected to Long-Term Immersion, Hot Dripping Brines & High-Impact Drop Testing

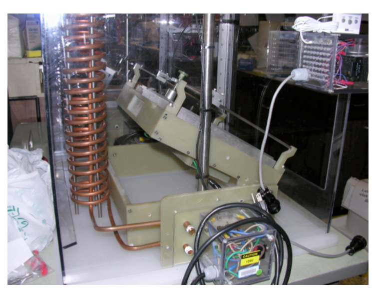




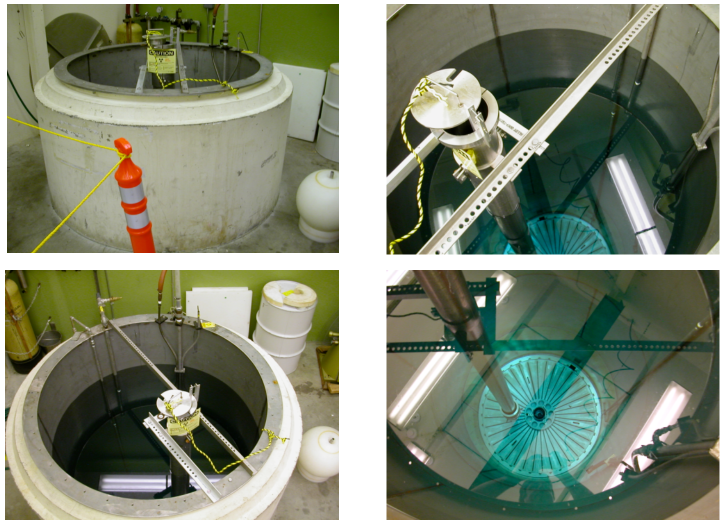


Hot Drip Test Apparatus





Gamma Pit at Lawrence Livermore National Laboratory



Irradiation, Attenuation & Radiography at TRIGA Reactor

Central Irradiation Facility (CIF) [1]
Pneumatic Transfer System (PTS) [2]
Neutron Transmutation Doping (NTD) > 10 Locations

[1] Maximum Value: The active length of TRIGA fuel is 15 inches. Dependent on the control rod elevation, thermal flux could decrease to sixty percent (60%) of 1.5×10^{13} n/cm^2·sec at a distance of 7.5 inches away from the reactor core. The flux/dose information is strongly dependent on water/void volume ratio.

[2] Average Value: Maximum 8.9×10^{12} n/cm^2 at the bottom and minimum 6.0×10^{12} n/cm^2 on the top.

Neutron Irradiation Facility (NIF)

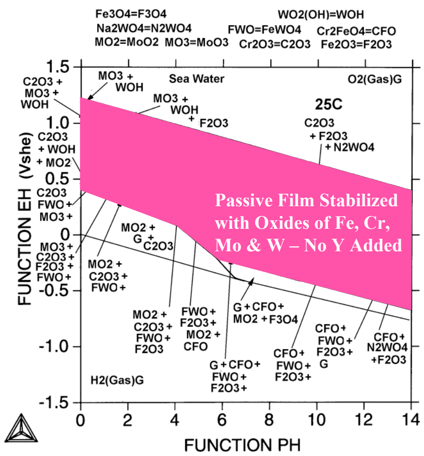
The operating power is 1.5 MW. The usable space is 17 centimeters (7 inches) in diameter and 22 centimeters (9 inches) in length.

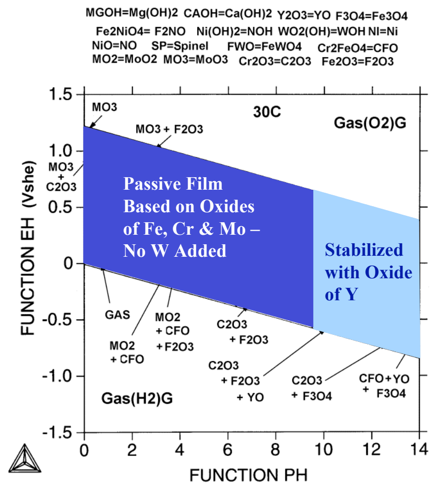
$\varphi_{1\text{-MeV eq.}} = 2.3 \times 10^{10}$ n/cm^2·sec $D_{\text{fast neutrons} > 0.1\text{ MeV}}$ (Si) $\cong$ 60 Gy/hr
$\varphi_{\text{thermal}} = 1\%$ of $\varphi_{1\text{-MeV eq.}}$ $D_{\text{gamma rays}}$ (Si) $\cong$ 200 Gy/hr

Pulsing

Typical Pulse Reactivity $\cong$ \$1.6 (or \$0.60 prompt reactivity)
Peak Power $\approx$ 400 MW
FWHM $\approx$ 30 milliseconds
Total Energy Release = 14-15 MW·sec

Pourbaix Diagrams for SAM40X3 & SAM1651

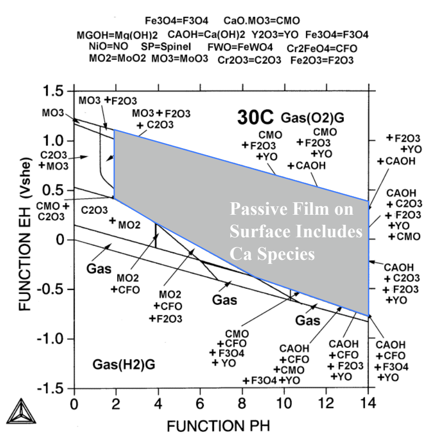


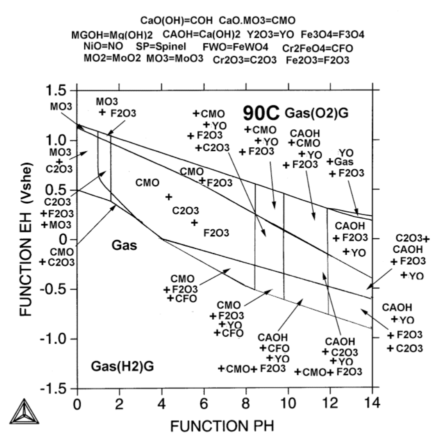


Prediction for early variant of SAM40X3 (Fe – 3Mo – 21Cr – 2W – 17B – atomic percent) in seawater (40 g NaCl + 1000 g H_2O) at 25°C

Prediction for SAM1651 (Fe – 14Mo – 15Cr – 2Y – 15C – 6B – atomic percent) in seawater (40 g NaCl + 1000 g H_2O) at 30°C

Calculated Pourbaix Diagram for SAM1651 in Concentrated Calcium Chloride





Fe-14Mo-15Cr-2Y-15C-6B 30°C 5M $CaCl_2$

Fe-14Mo-15Cr-2Y-15C-6B 90°C 5M $CaCl_2$

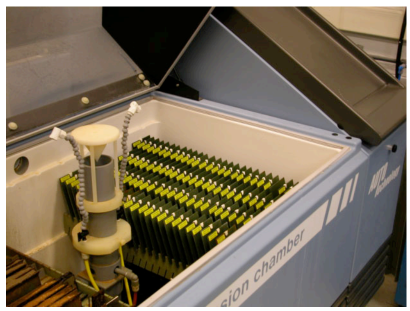
High-Performance Corrosion-Resistant Materials: Salt Fog Testing

Dr. Louis F. Aprigliano (NSWC Retired)
Naval Surface Warfare Center
Carderock Division

Introduction

- Original Goals
  - Use salt fog test to screen alloys for Naval application
  - Provide means to produce "bulk" samples of amorphous alloys
- Approach
  - Use GM salt fog test for evaluating materials resistance to sea water spray
  - Use Spray Metal Forming to produce bulk samples

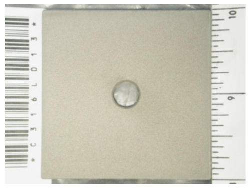
Salt Fog Chamber at NSWC

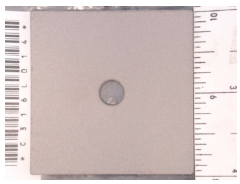


24 Hour Test Cycle for GM9540P Accelerated Corrosion Test (Salt Fog Test)

Shift	***Elapsed Time (hrs)***	***Event***
Ambient Soak	0	*Salt solution mist for 30 seconds, followed by ambient exposure (13-28 ºC (55-82 ºF))
	1.5	*Salt solution mist for 30 seconds, followed by ambient exposure (13-28 ºC (55-82 ºF))
	3	*Salt solution mist for 30 seconds, followed by ambient exposure (13-28 ºC (55-82 ºF))
	4.5	*Salt solution mist for 30 seconds, followed by ambient exposure (13-28 ºC (55-82 ºF))
Wet Soak	8-16	8 hour high humidity exposure (49 ± 0.5 ºC (120 ± 1 ºF), 100% RH) including 55 minute ramp to wet conditions
Dry Soak	16-24	8 hour elevated dry exposure (60 ± 0.5 ºC (140 ± 1 ºF), <30% RH) including 175 minute ramp to dry conditions
*Salt solution mist consists of 1.25% solution containing 0.9% sodium chloride, 0.1% calcium chloride, and 0.25% sodium bicarbonate.		

Macrographs of Typical SAM Coating (SAM40) First Salt Fog Test



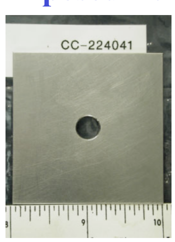


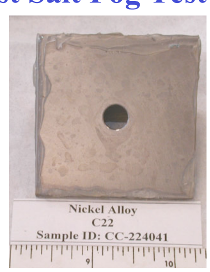
As-received

After 13 cycles (days)

Macrographs of Wrought & HVOF Samples of Alloy C-22 Exposed During First Salt Fog Test

Nickel-Based Alloy C-22 (Bulk Plate)



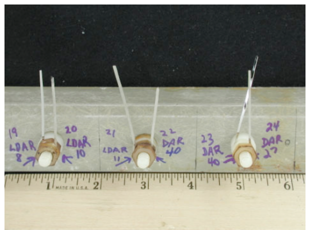


Nickel-Based Alloy C-22 (HVOF on 316L)

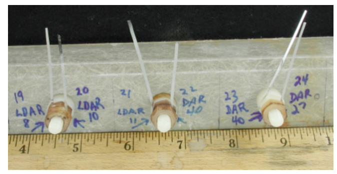
As-received (zero cycles)

After 13 cycles (days)

Salt Fog Test of Melt Spun Ribbons (Third Salt Fog Test)



As-received (Zero Cycles)



24 Cycles (days)

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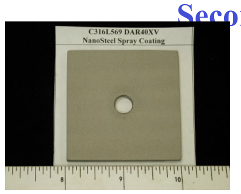
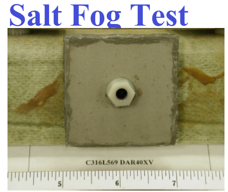
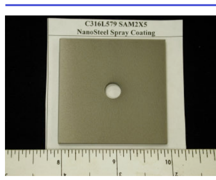
Macrographs of SAM Coatings from Second Salt Fog Test

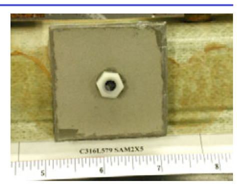
SAM40XV

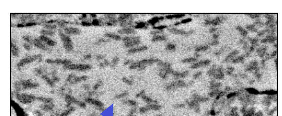




SAM2X5

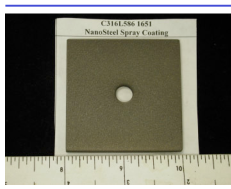


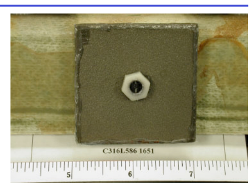


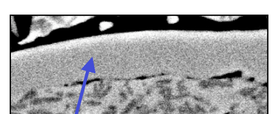


Amorphous matrix with Cr_2B precipitate

SAM7 (SAM1651)







Amorphous

Zero Cycles | 28 Cycles (days)

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Chemical Composition of Coatings (Atomic Percent)

SAM40	Cr – 19, Mn – 2, Mo – 2.5, W – 1.7, B – 16, C – 4, Si – 2.5, Fe – balance
SAM2X5	Cr – 18.1, Mn – 1.9, Mo – 7.4, W – 1.6, B – 15.2, C – 3.8, Si – 2.4, Fe – balance
SAM7 (SAM1651)	Cr – 15.0, Mo – 14.0, B – 6.0, C – 15.0, Y - 2, Fe - balance

Conclusions from Salt Fog Testing

- Early SAM coatings rusted during first test
  - Rusting initiated at de-vitrified areas in coatings
- Fully amorphous material (melt spun ribbons) did not rust
- Improved coatings in third test were rust free
  - Believed to be fully dense and amorphous or at ferrite free
  - At end of exposure test, samples will be subjected to detailed analysis at Sandia

Accomplishments – Salt Fog Testing

- Latest coatings are resistant to salt spray corrosion and are probably fully dense and amorphous or at least ferrite free
- Latest coatings are as resistant to salt spray corrosion as Type 316 stainless steel or nickel-based Alloy C-22
- De-vitrified areas in prior coatings rusted within a day or two
- Developed rapid screening test for coating quality
- Variation of the test could be used to verify the quality of coatings on the actual waste packages

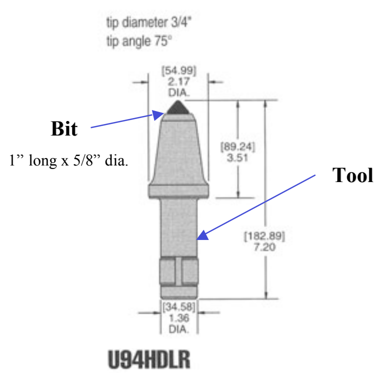
High-Performance Corrosion-Resistant Materials: Material for Tunnel Boring Application

Dr. Louis F. Aprigliano (NSWC Retired)
Naval Surface Warfare Center
Carderock Division

Near-Term Feasibility Activities: SAM-Tipped Alpine Picks

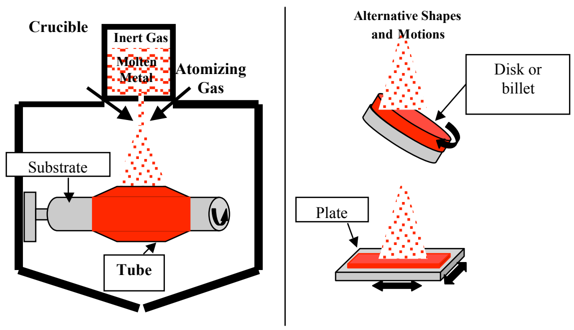
- NSWC: Tungsten carbide “plug tip” replaced by a spray-formed, EDM’d DAR35 “plug tip”
- NSWC: Spray-formed DAR35 “plug tip” brazed to hold it in place and heat treated (700 C for 96 hrs) to significantly increase hardness
- Colorado School of Mines: Will test the SAM-tipped alpine picks (2) in the Linear Cutting Machine with an hard, abrasive sandstone (standard alpine pick wear test) and assess SAM-tipped alpine pick performance

Bits for Mining Tool (Alpine Picks)



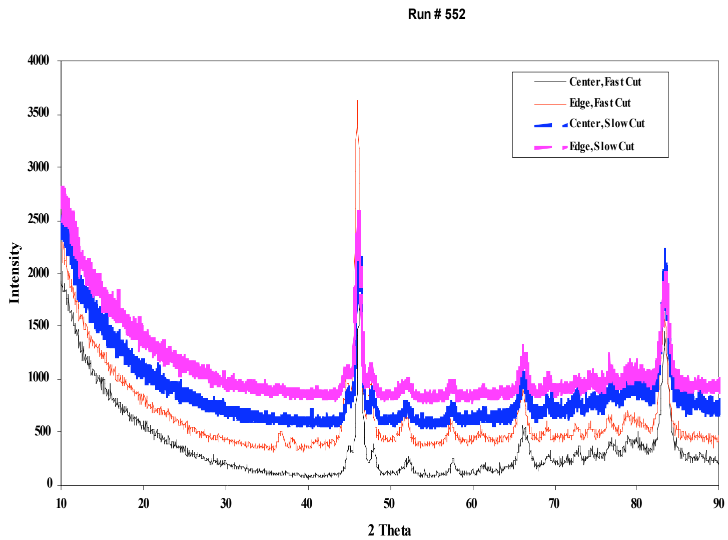
Goal - Fabricate bits for mining tool from bulk iron-based SAM

Schematic of Spray Metal Forming Process



Macrograph of As-Sprayed Low-Carbon SAM40 (SHS727)

X-ray Diffraction of Material From Spray Metal Forming Run 552, Low-Carbon SAM40 (SHS727)



Bulk Amorphous Metal Plates Cut from NSWC Spray Form

Microhardness (Vickers) Measurements

Material Designation	SAM 40 SHS727	Low C Run 552		C22			316L SS	
Indenter Load	100 g	1000 g	100 g	300 g	1000 g	100 g	300 g	1000 g
Readings	796 821 833 886 886	660 746 710 750 726	242 230 238 227 240	191 202 201 187 208	177 177 179 187 208	282 305 329 314 305	245 238 245 259 246	231 225 217 227 227
Average	844	718	235	198	181	307	246	225

Macrograph of Machined Bits (Tips)

Tensile Data
Low-Carbon SAM40 (SHS727)

Sample #	Tensile Strength	Yield Strength	Elongation	Reduction of Area
1	120 ksi	*	1.5%	1.5%
2	95ksi	*	2.5%	2.0%
3	128 ksi	*	1.5%	3.0%

*Broke before yielding @ 0.2%

Composition of
Low-Carbon SAM40 (SHS727)

Alloy / ID	Fe[1]	Cr	Mo	C	B	Si	W	Mn	Al
Low Carbon SAM40	60.2	20.3	4.9	0.1	3.6	1.4	6.4	2.3	-
	60.0	19.8	5.15	0.16	2.9	1.65	6.31	2.23	-

Zr	Co	Y	N	O
0.8	-	-	-	-
0.017			0.053	0.014

Notes:
1. Fe value by difference.
2. Ingot purchased from Special Metals, Incorporated
3. Powders supplied by NanoSteel, Inc.

Accomplishments – Bulk Amorphous Metals

- Fabrication of Slabs with Thickness > ¾ Inches
- Bits Made for Mining Tool

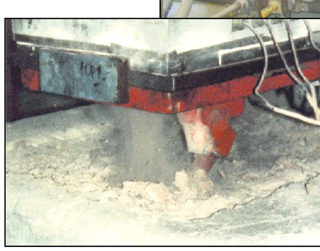
Backup Slides

Feasibility Studies: SAM-Coated Disc Cutters

- Herrenknecht (USA) provided gratis two state-of-the-art 17” disc cutters for SAM coating trials; Caterpillar forged 5 dummy 17” discs, based on the Herrenknecht geometry for spray/coating and HDIF parameters optimization
- 17” disc cutters (Herrenknecht & dummies) to be SAM coated by Caterpillar
- All SAM coated 17” disc cutters will be High Density Infrared Fused (HDIF) by ORNL (to prevent spalling of coating during impact with rock)
- The best dummy HDIF 17” disc cutters will be tested with Topopah Spring Welded Tuff at the Colorado School of Mines (e.g. to initially determine if coating spalls upon rock impact)
- If a few of the 17” dummy disc cutters show good performance, Herrenknecht (USA) will test the Herrenknecht-supplied disc cutters on a TBM operating on a hard rock job in the US (gratis). Only way to assess disc cutter performance as it is not possible to obtain the required linear travel distances to assess wear resistance in a laboratory-scale environment.

Linear Cutting Machine Tests

Linear Cutting Machine Tests will be used to assess SAM-tipped alpine pick performance



Courtesy of the Colorado School of Mines

High-Performance Corrosion-Resistant Materials: Microstructure Characterization

Nancy Yang
Sandia National Laboratory
Livermore, California

Collaborations

- Sandia National Laboratory
  - T. Headley
  - G. Lucadamo
  - J. L Yio
  - J. Chames,
    A. Gardea
  - M. Clift
- University of California, Davis
  - E. Lavernia
  - L. Ajdelsztajn
- Lawrence Livermore National Laboratory
  - J. Farmer
  - J. Haslam
  - D. Day

Outline

- Objective
- Work Scope
- Scientific Tasks
- Accomplishments for FY06
- Experimental Findings
  - Materials
  - Experimental Approaches
  - Results
- Summary

Objective and Work Scope

- Grand Challenge (HPCRM Team)
  - Develop high strength corrosion resistant Fe-based amorphous metal coatings for long term storage of spent nuclear fuel and naval applications

- Work scope
  - Establish correlations between material properties, process variables and corrosion resistance of amorphous metals coatings
  - Collaborate with UC Davis and UNR to optimize the thermal spray and atomization for producing new SAM2X5 and SAM1651 coatings
  - Orchestrate transfer of new technical insights to industrial partners for implementation
  - Provide material characterization support- for HPCRM team activities including witness samples from industrial partners and Tunnel Boring Machine (TBM) application

Scientific Tasks

- Task I: Effect of Metallurgy on Corrosion Performance (SNL/LLNL)
  - Devitrification & thermal stabilities of 2x5 and 1651 amorphous ribbons
  - Effect of devitrification on corrosion performance
- Task II: Process Optimization (SNL / UCD / UNR)
  - Physical properties of feedstock powders
  - Effect of powder property on coating morphology
  - Implication of coating morphology on corrosion performance
  - Process refinement of HVOF spray and atomization at UCD
  - New nanocrystalline SAM Y2O3 composite at UCD/UNR
- Task III: Physical Metallurgy of Drop-cast SAM 2x5 & 1651 for TBM Application (SNL/ORNL)

Accomplishments for FY06

- The studies show that a combination of high density and amorphous structure are the key ingredients for corrosion resistant SAM2X5 and SAM1651 ribbons
- We have gained good insights into devitrification and thermal stability of SAM40, 1651 and 2x5 ribbons and their influence on corrosion performance
- We have established a correlation between HVOF coating morphology, i.e., microstructure & integrity, and corrosion performance.
- We have identified the following key elements of coating morphology affecting corrosion performance of SAM2x5&1651 coatings
  - Cr-Mo-depleted α-ferrite in devitrified SAM40, SAM2X5 and SAM1651 ribbons is susceptible to corrosion in sea water and $CaCl_2$ environment
  - - *Interconnected* devitrified powders, pores and open inter-particle interfaces create- an adversarial pathway for corrosion advancement
  - Optimizing powder size and shape are effective in mitigating devitrification and porosity, and therefore reducing interconnected α-ferrite, pores and open interfaces
  - Thermal management of HVOF spray and atomization are also effective in decreasing devitrification and improving powder consolidation

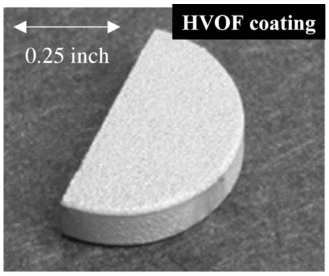
Accomplishments for FY06

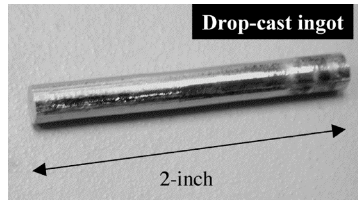
- The above scientific findings have provided the necessary scientific bases and
- guidance to on-going process optimization activities at UCD and industry
- We have successfully produced corrosion resistant coatings using optimized
- HVOF spray parameters in PlasmaTech (PTI)

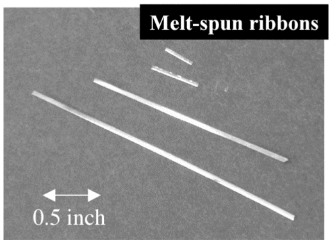
Material Characterization Approaches

- Microstructure
  - Optical Metallographic Imaging (OMI)
  - Scanning Electron Microscopy (SEM) Imaging
    - Backscattered Electron Imaging (BEI)
    - Secondary Electron Imaging (SEI)
  - Transmission Electron Microscopy (TEM)
    - Bright Field (BF)
    - Dark Field (DF)
- Porosity
  - Metallographic / SEM Image Analysis
- Crystalline Structure
  - X-Ray Diffraction (XRD)
  - TEM /Selected Area Electron Diffraction (SAED).
- Alloy Composition, Chemical Variation and Phase Transformation
  - Electron Microprobe
    - Wavelength (WDS)
    - Energy Dispersive X-Ray (EMPA/EDS)
- Mechanical Properties
  - Hardness (VHN): Vickers Micro-indentation at 100 grams load
  - Damage Tolerance (DT): Based on fracture toughness measurements at 1000 grams
- Thermal Stability
  - Thermal annealing temperatures are: 150°C, 300 °C, 500 °C, 600 °C, 800 °C and 1000 °C

Typical geometry and dimensions and the nominal composition of the three SAM formulations being evaluated







	SAM 40		SAM 1651		SAM 2x5	
	wt%	at%	wt%	at%	wt%	at%
Fe	59.79	52.00	51.34	48.00	54.37	49.70
Cr	20.29	19.00	14.90	15.00	18.49	18.20
Mn	2.26	2.00			2.04	1.90
Mo	4.93	2.50	25.67	14.00	13.88	7.40
W	7.56	2.00			5.75	1.60
B	3.61	16.00	1.26	6.00	3.27	15.20
C	0.99	4.00	3.44	15.00	0.89	3.80
Si	0.57	1.00			1.31	2.40
Y			3.40	2.00		
total	100.00	98.50	100.00	100.00	100.00	100.20

The three specimens were designed for R&D purposes

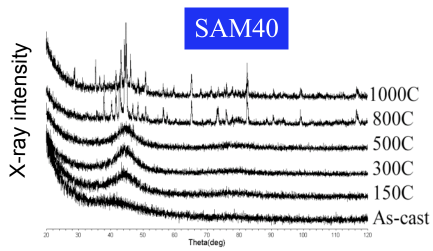
Devitrification of SAM 40, SAM2X5 & SAM1651 Melt Spun Ribbons

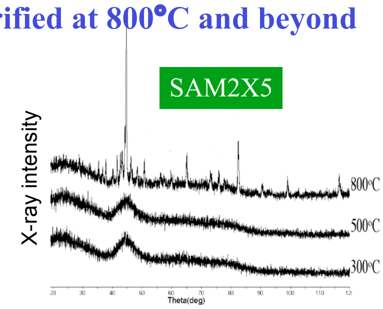
Identify undesirable elements lowering corrosion performance

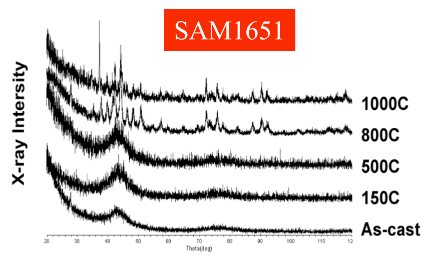
↓

Determine scientific strategy for mitigating the undesirable elements

Amorphous structure of SAM40, SAM1651 & SAM2X5 ribbons is stable up to 500°C and devitrified at 800°C and beyond

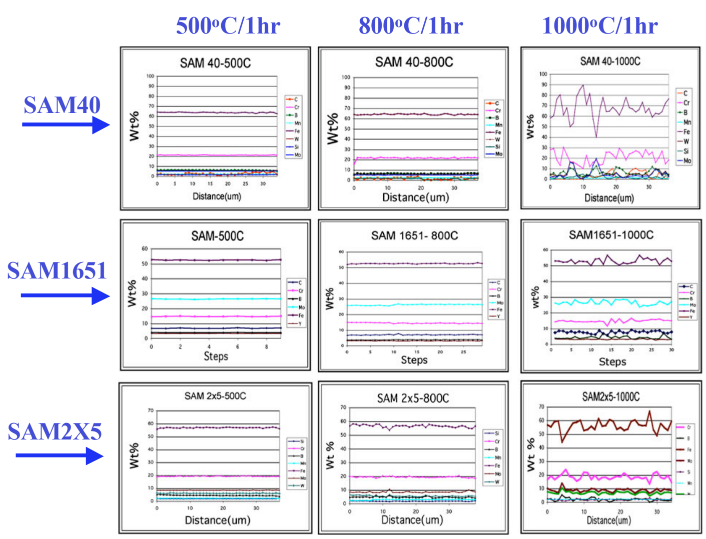






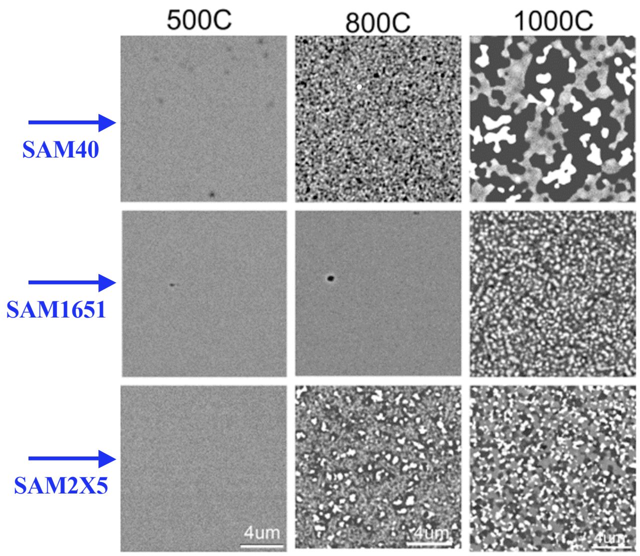
α-ferrite and $M_{23}C_6$ observed in all three devitrification ribbons

WDS profiles show uniform chemical composition through thickness of all three ribbons up to 500°C/1hr



Chemical variations seen in SAM40, SAM2X5 & SAM1651 at 800°C and 1000°C are indicative of phase separation

BEI images of SAM 40, SAM1651 and SAM2X5 ribbons show amorphous structure up to 500°C/1hr



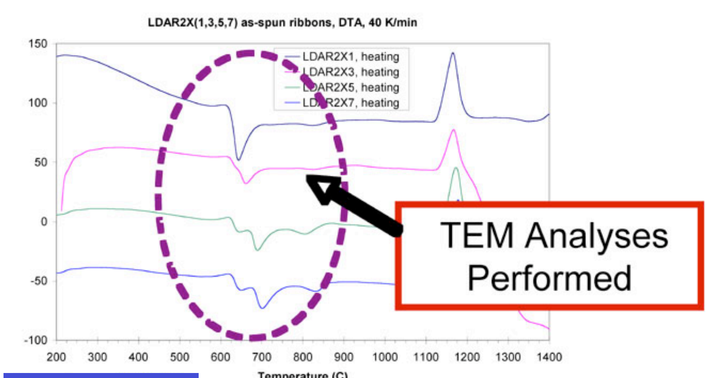
Submicron microstructure feature was visible at 800°C. The feature coarsened substantially at 1000°C

Submicron feature became visible at higher annealing temperature, 1000°C

Submicron microstructure feature also was visible at 800°C and beyond

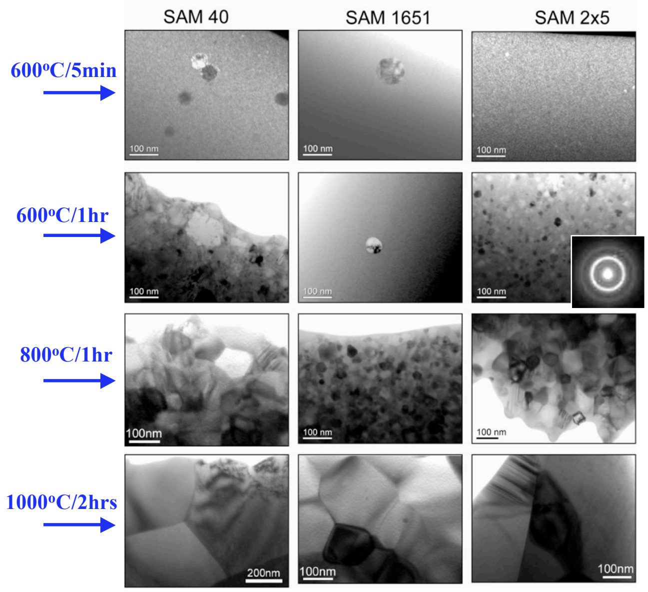
- Submicron features were being examined using TEM analyses below

TEM analyses were focused on ribbons annealed between 600-1000°C where microstructure & phase separation are evolving



Courtesy of UWM

TEM/BF shows SAM1651 devitrified at 800°C, instead of 600°C/1hr seen in SAM40 & SAM2X5 formulations



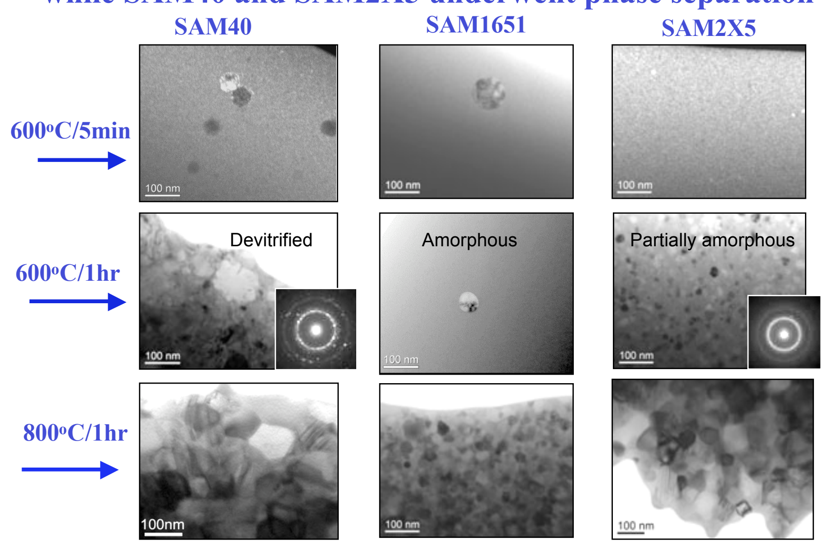
Amorphous structure with exception of few crystalline particles initiated in

SAM1651 ribbon devitrified at higher temperature, SAED of SAM2X5 exhibit mixture of amorphous halos background with crystalline rings

Nanocrystalline composite observed in all three ribbons

At 1000°C, all three ribbons were fully devitrified and the phases were coarsened

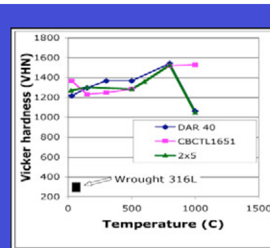
SAM1651 remained amorphous after annealing at 600°C for 1hr, while SAM40 and SAM2X5 underwent phase separation



- TEM/SAED show all ribbons retain amorphous structure up to 600°C/5min
- SAM1651 devitrified at higher temperature (800°C/1hr) than SAM40 and SAM2X5

Hardness changes with annealing temperature in all three SAM formulations is related to microstructure evolution

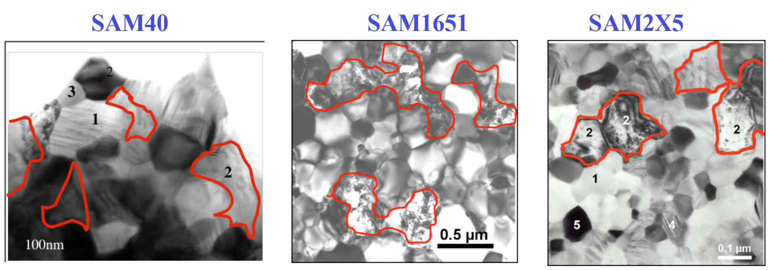
- Hardness rises slowly with annealing temperature up to 500°C. Is this due to short range ordering?
- Drastic increases in hardness between 500-800°C attributed to formation of nanocrystalline composite.
- Hardness drop at 800°C for SAM40 and SAM2X5 attributed to microstructure coarsening.

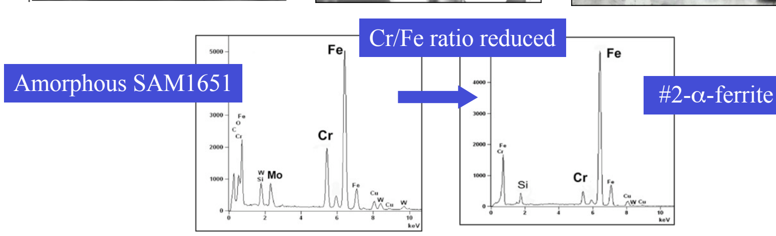


Crystalline phases dectected in the devitrified ribbon							
Sample ID	bcc Fe	M23(CB)6	M6C	Fe3B	M3B2	Cr2B	MB2
SAM40 ribbon 1000°C	x	x				x	
SAM1651rod/ribbon 800°C	x	x	x				
SAM1651 ribbon 1000oC	x	x	x		x		
SAM 2x5 ribbon 800°C	x	x	x	x	x		x

•α-ferrite and $M_{23}(CB)_6$ type phases were detected in all devitrified SAM40, SAM2X5 and SAM1651 ribbons.

TEM/BF images show typical microstructure of Cr-Mo-depleted α-ferrite in the devitrified SAM1651 and SAM2X5 ribbons



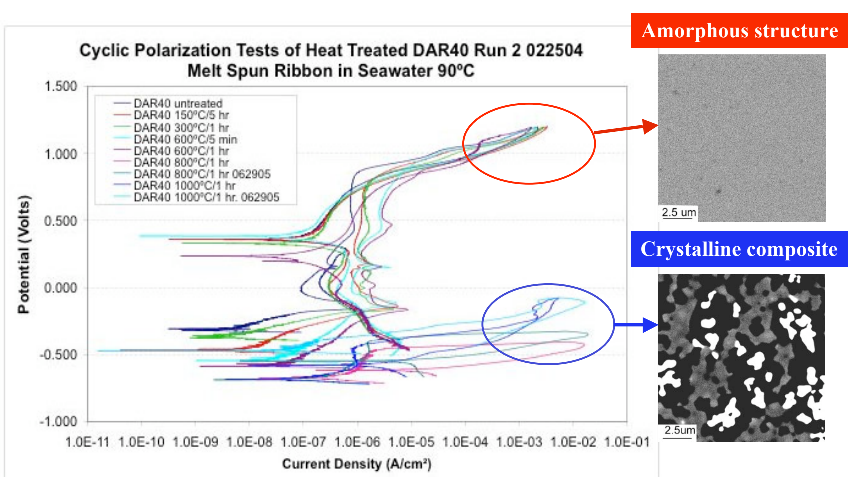


EDS α-ferrite contain < 10 wt. % Cr and < 1 wt. % Mo, which is much reduced from their starting amorphous matrix

Effect of Devitrification on Corrosion Performance of SAM40, SAM1651 and SAM2X5 Ribbons

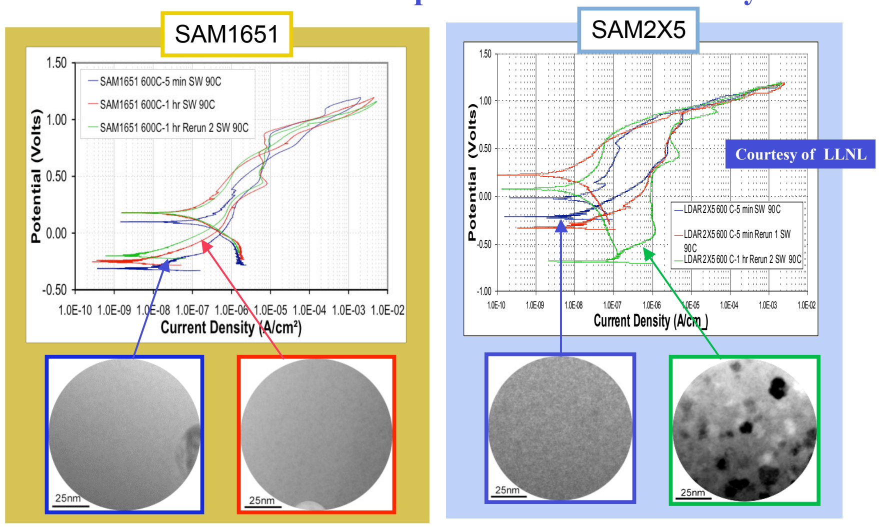
Work Supporting
Corrosion Studies at
Lawrence Livermore National Laboratory

As-received amorphous SAM40 ribbons show higher corrosion resistance relative to devitrified ribbons



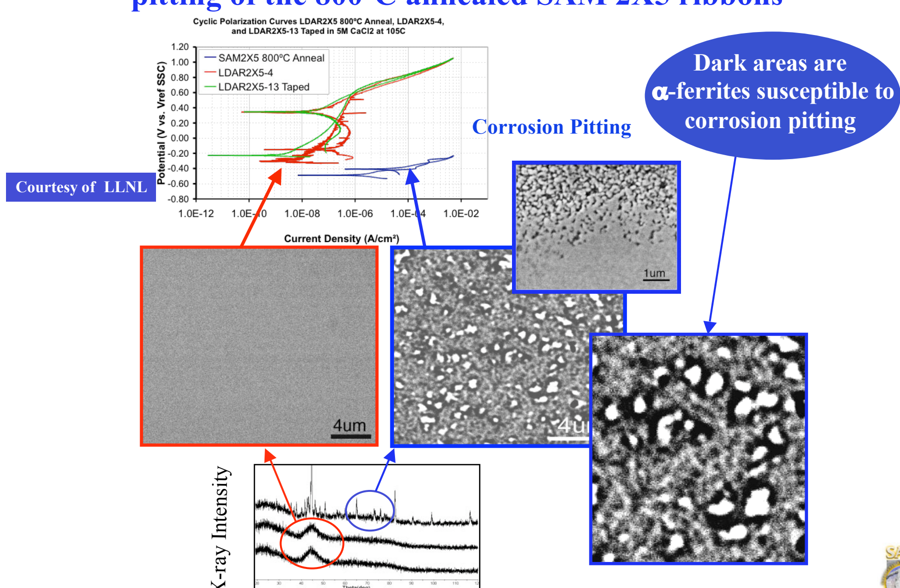
Courtesy of LLNL

Amorphous structure of SAM2X5 and SAM1651 ribbons are corrosion resistant despite their thermal history



Nanocrystalline α-ferrites may be responsible for the changes in cyclic polarization behavior of SAM2X5 ribbon annealed at 600°C/1hr.

Presence of nanocrystalline α-ferrites was responsible for corrosion pitting of the 800°C annealed SAM 2X5 ribbons



Relationship of Devitrified Microstructure to Corrosion Performance Observed by LLNL

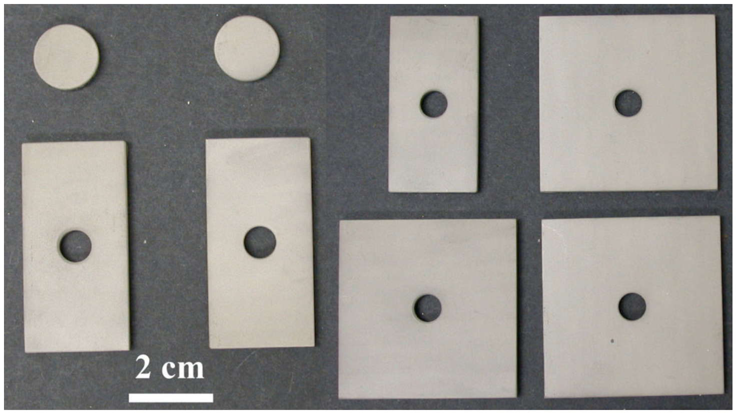
- SAM2X5 and SAM1651 melt-spun ribbons are fully dense and retain amorphous structure up to 600°C/5min.
- Vickers hardness of all ribbons stayed above 1200 VHN up to 800°C. Drastic increase in harness near 800°C related to formation of nanocrystalline composites. Hardness drop in SAM40 and SAM2X5 ribbons beyond 800°C attributed to microstructure coarsening
- SAM1651 devitrified at relatively higher temperature, 800°C. The ribbons retain a strong nanocrystalline composite structure up to 1000°C/2hrs.
- All the fully devitrified ribbons contained Cr-Mo-depleted α-ferrite network that are susceptible to corrosion pitting.

Process Optimization Studies by UCD & SNL

Physical property of feedstock powder and its effect on HVOF coating morphology

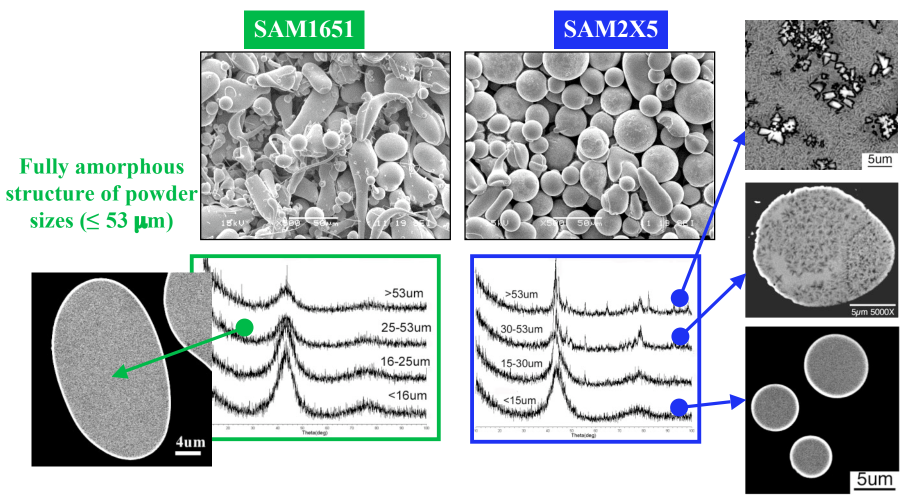
HVOF coatings presented here were produced by Dr. Leo Ajdelsztajn of UCD at facilities at UCD and Plasma Tech Incorporated (PTI)

HVOF coatings produced for HPCRM are sprayed from atomized powders of SAM formulations



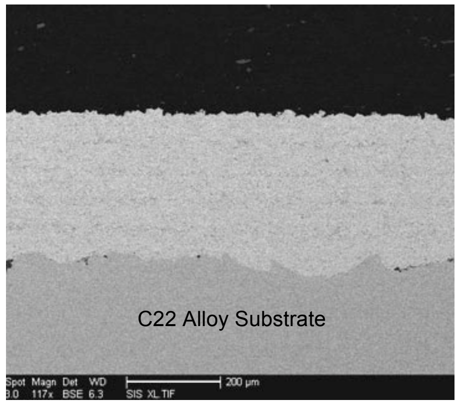
Courtesy of UCD & PTI

Distinct difference in powder morphology and state of amorphous structure between SAM1651 and SAM2X5 powders



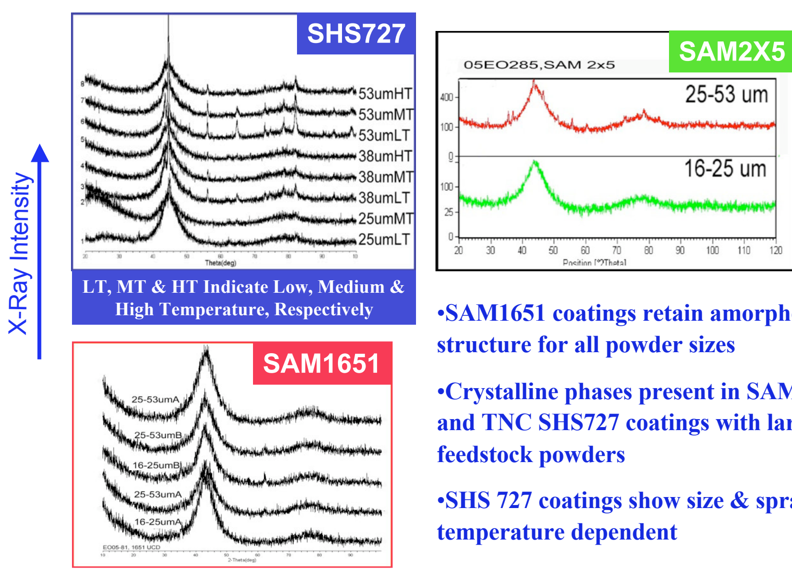
Microstructure of SAM2X5 powders is size dependent. Large powders tend to devitrify and small powders tend to remain amorphous structure.

Typical Morphology of HVOF Coatings Produced by the University of California at Davis for HPCRM



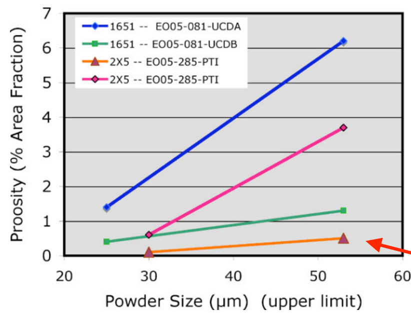
SEM/BSE image showing typical morphology of 200 µm-thick SAM1651 coatings (metallographic polished cross section).

Without process optimization, amorphous structure of SAM2X5 coatings is powder size dependent



- SAM1651 coatings retain amorphous structure for all powder sizes
- Crystalline phases present in SAM2X5 and TNC SHS727 coatings with large feedstock powders
- SHS 727 coatings show size & spray temperature dependent

Porosity of SAM1651 & SAM2X5 coatings is dependent on powder size & HVOF spray parameters



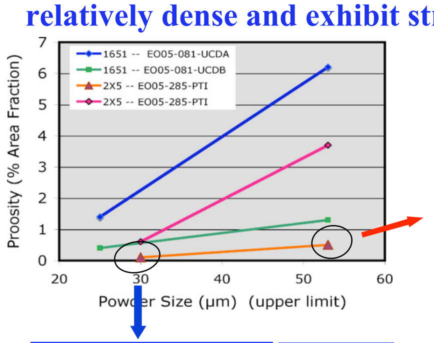
- All four plots show a trend of porosity increase with feedstock powder size
- Porosity level appeared to vary with H_2/O_2 fuel mix ratio

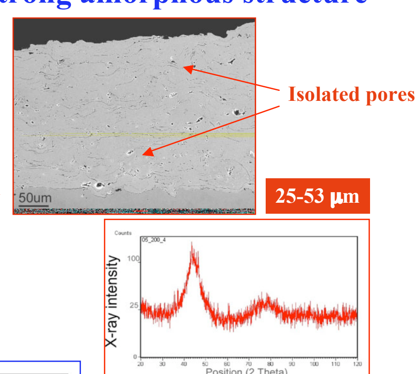
Focus of discussion

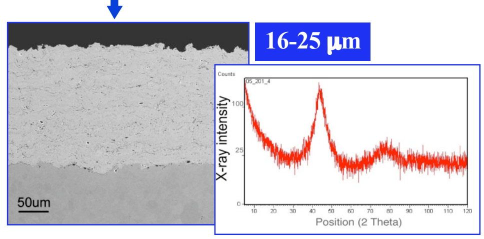
Spary parameter	HVOF spray condition							
	O_2		H_2		Air		Distance	Gun type
	FMR**	psi	FRM	psi	FMR	psi		
PTI-SAM 2x5	86	120	40	120			9.5"	JK2000
UCD-SAM 2x5	32	170	62	140	44	100	9.5"	DJ 2700
UCD-SAM1651-A	32	170	62	140	44	100	9.5"	DJ 2701
UCD-SAM1651 B	32	170	68	140	44	100	9.5"	DJ 2702

** Flow meter reading

SAM2X5 coatings sprayed with high O_2 / H_2 ratio fuel at PTI are relatively dense and exhibit strong amorphous structure



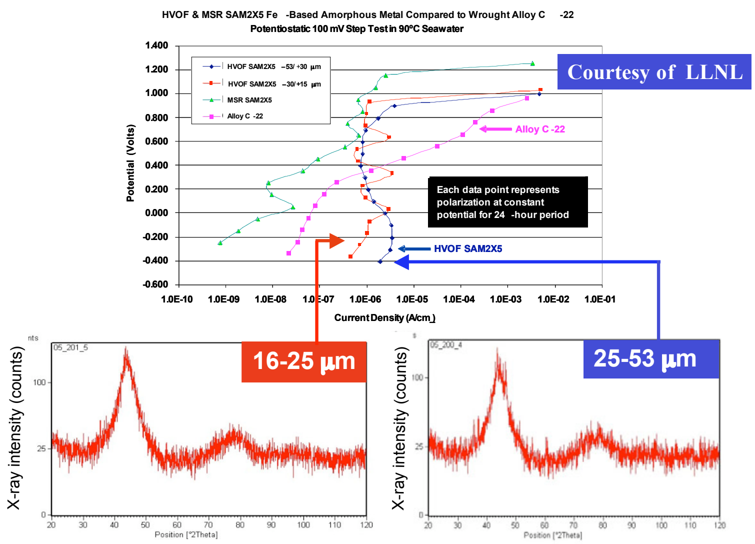




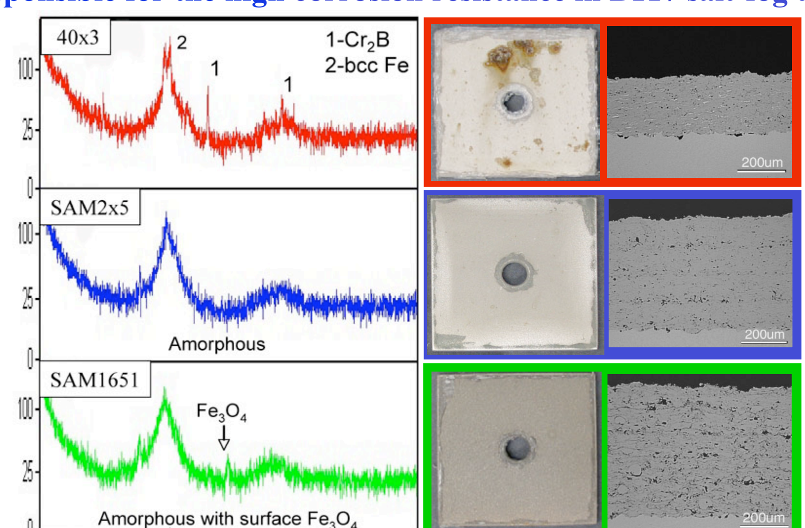
- Most pores seen in the coatings were not interconnected
- Minimal crystalline peak presence in the XRD

Implication to Corrosion Performance ?

Highly amorphous SAM2X5 coatings without interconnected pores are very resistant to corrosion in 90°C Seawater

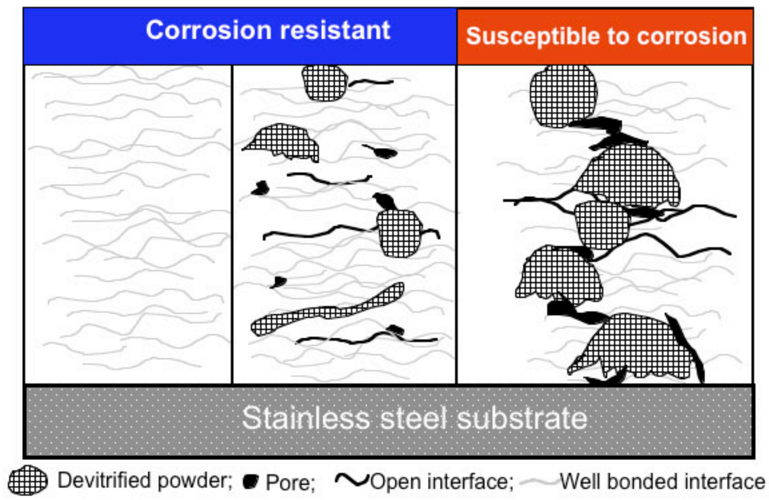


Amorphous structure of SAM2X5 and SAM 1651 HVOF coatings is responsible for the high corrosion resistance in B117 salt-fog test

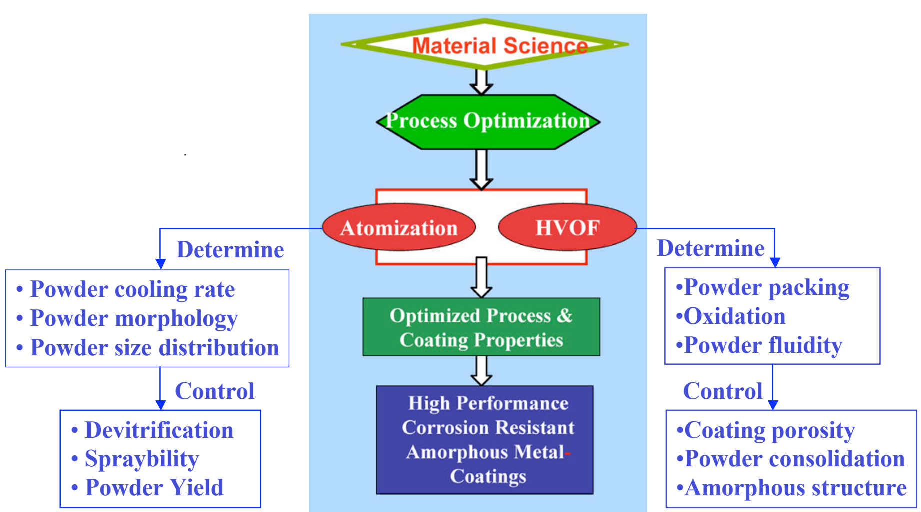


Corrosion pitting of SAM40X3 coating attributed to interconnected devitrified powders and pores

Schematic for Various Scenarios of Corrosion Susceptibility



What do we gain from material science study and process optimization?



Summary

- SAM1651 powders retain amorphous structure for all size ranges (< 16 μm to ≤ 53 μm). However, the powders contains irregular shapes with ligaments
- SAM2X5 powders are spherical and state of amorphous structure is size dependent. Large unmelted powders (>30 μm) tend to devitrify during atomization.
- HVOF coating morphology is impacted greatly by feedstock powder properties including microstructure, powder size and shape.
- During HVOF spray, large powders of SAM2X5 stay devitrified and rigid which results in undesirable crystalline composites with high porosity.
- Presence of Cr-Mo-depleted α-ferrites in the devitrified SAM composite is a likely source of corrosion in sea water and $CaCl_2$ environments.
- Interconnected devitrified powders, pores and open powder interfaces creates an effective pathway for corrosion advancement.
- The above findings provided the scientific bases for the on-going process optimization activities.

Summary (cont'd.)

- Dense amorphous melt spun ribbons (MSR) of SAM2X5 are stable up to 600°C and at 600°C, the degree of devitrification is time dependent. SAM1651 ribbon on the other hand, devitrified at higher temperature (> 600°C)
- Hardness increases beyond the T_g are attributed to formation of a nanocrystalline composite which contains α-ferrite susceptible to corrosion
- Size distribution of feedstock powders due to the strong effect on powder microstructure, a crucial factor determining coating morphology, impact corrosion performance
- Thermal management during HVOF is also essential for controlling coating morphology and porosity in particular
- Process optimization of atomization and HVOF spray are necessary for improving coating morphology

Accomplishments for FY06

- The studies show that a combination of high density and amorphous structure are the key ingredients for corrosion resistant SAM2X5 and SAM1651 ribbons
- We have gained good insights into devitrification and thermal stability of SAM40, 1651 and 2x5 ribbons and their influence on corrosion performance
- We have established a correlation between HVOF coating morphology, i.e., microstructure & integrity, and corrosion performance.
- We have identified the following key elements of coating morphology affecting corrosion performance of SAM2x5&1651 coatings
  - Cr-Mo-depleted α-ferrite in devitrified SAM40, SAM2X5 and SAM1651 ribbons is susceptible to corrosion in sea water and $CaCl_2$ environment
  - - *Interconnected* devitrified powders, pores and open inter-particle interfaces create- an adversarial pathway for corrosion advancement
  - Optimizing powder size and shape are effective in mitigating devitrification and porosity, and therefore reducing interconnected α-ferrite, pores and open interfaces
  - Thermal management of HVOF spray and atomization are also effective in decreasing devitrification and improving powder consolidation

Accomplishments for FY06

- The above scientific findings have provided the necessary scientific bases and
- guidance to on-going process optimization activities at UCD and industry
- We have successfully produced corrosion resistant coatings using optimized
- HVOF spray parameters in PlasmaTech (PTI)

Future Work

- **Continue to focus on process optimization**
  - <u>Atomization</u>
    Optimize powder size distribution and morphology for mitigating devitrified microstructure and improve powder yield
  - <u>HVOF spray</u>
    Improve powder consolidation and coating integrity for minimizing undesirable interconnected pores and open interfaces

- **Continue to provide material characterization support for HPCRM & TB programs:**
  - Quality verification for witness samples from industrial partners
  - Materials relevant to the HPCRM & TB team activities

Backup Slides

High-Performance Corrosion-Resistant Materials: Thermal Stability

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University of Wisconsin – Madison
Madison, Wisconsin

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Preview

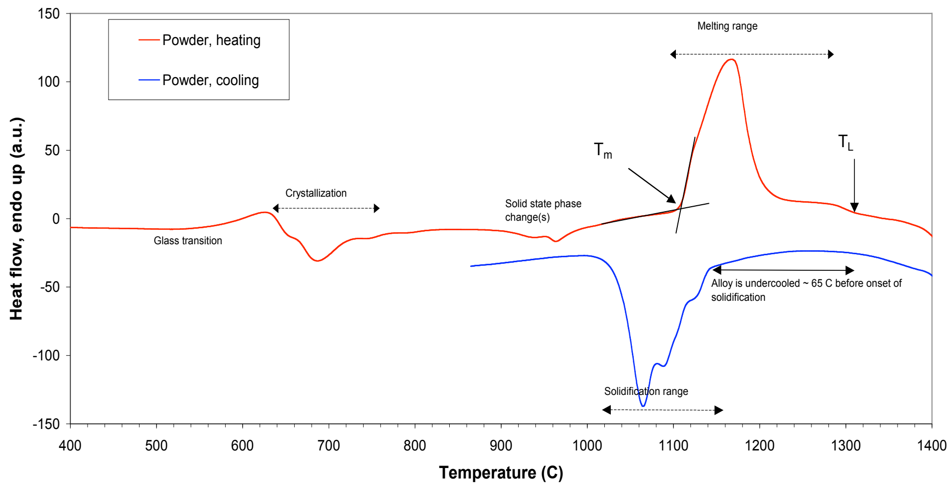
- University of Wisconsin objectives
- Accomplishments
- UWM objectives
- Kinetic analysis – approach and examples (SAM40)
- Wedge cast experiments
  - SAM2X5
  - SAM7
- Kinetics
  - Preliminary results and model analysis (melt-spun ribbons)
    - SAM2X5
    - SAM7
- Powder analysis – XRD, DTA and SEM
  - SAM2X5
  - SAM7
- Summary
- Future work

Introduction/objectives

- Thermal analysis (DTA, DSC) of amorphous melt-spun ribbons:
  - glass transition temperature T_g, crystallization temperature T_x, melting temperature T_m, liquidus temperature T_l
  - first estimate of glass forming ability (GFA) based on reduced glass transition temperature T_{rg} (T_g/T_l) of respective alloys
- Microstructure characterization (TEM, SEM, XRD) of powder and sprayed bulk samples.
  - determination of crystalline phases limiting GFA ⇒ modification of composition to avoid primary crystallization on cooling
- Wedge casting of selected alloys
  - determination of critical cooling rates for glass formation
  - analysis of microstructure transitions
- Kinetic analysis of amorphous phase synthesis and stability
  - isothermal and continuous heating crystallization kinetics
  - model analysis for crystallization
  - time-temperature transformation curves
  - test analysis predicitions

Accomplishments – FY05

- Pearl Harbor review:
  - Thermal analysis complete for 27 alloys
  - CCR measured for SAM35, SAM40, SAM2X7 and SAM7 (SAM1651)
  - Detailed devitrification studies have identified Fe-borides as the primary phases
  - Improved accuracy in kinetics analysis
  - Preliminary TTT-curves for SAM35 and SAM40
  - Powder processing analysis in progress.
- Key West review:
  - CCR measured for SAM2X5
  - More complete microstructure analysis of wedge cast SAM7 (SAM1651)
    - TEM & SEM
  - Low-temperature annealing of SAM2X5 and SAM7 (SAM1651)
  - Completed kinetic evaluation of SAM40
  - Preliminary TTT-diagrams assessed for SAM2X5 and SAM7 (SAM1651)
  - Powder analysis of SAM2X5 and SAM7 (SAM1651)
    - SEM: Morphology of powder as function of powder size
    - DSC: Devitrification kinetics as function of powder size.
    - DTA: Thermal analysis for various powder sizes.



Differential thermal analysis (DTA) of atomized SAM1651 powder, illustrating typical features of an amorphous sample undergoing continuous heating (40 K/min).

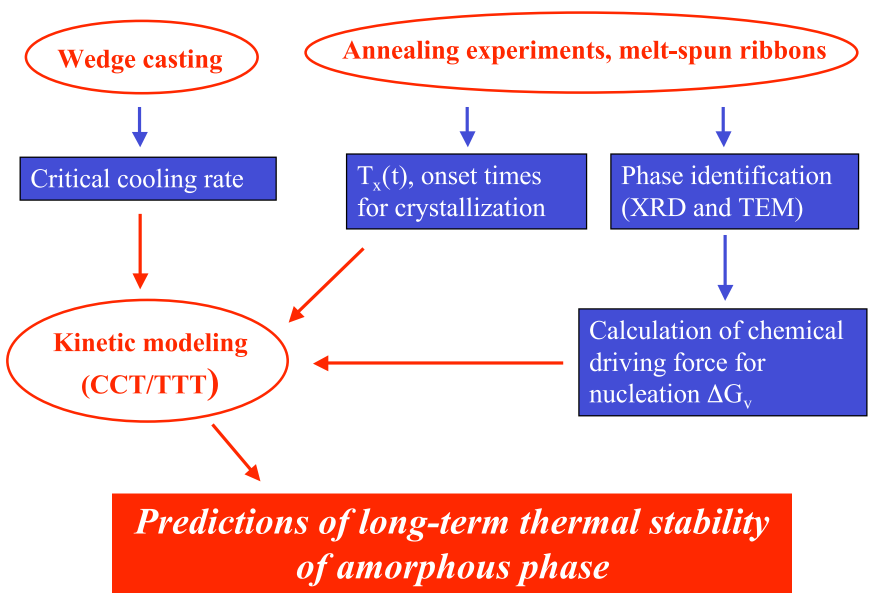
Thermal Analyses: Summary of Data for HPCRM Materials

Alloy	T_g (°C)	T_x (°C)	T_m (°C)	T_L (°C)	T_{rg} (°C)	CCR (K/s)
SAM2X1	575	620	1124	1190-1210	0.57	
SAM2X3	578	626	1131	1190-1210	0.57	
SAM2X5	579	628	1133	1190-1210	0.57	
SAM2X7	573	630	1137	1190-1210	0.57	610
SAM6	580	623	995	1238-1250	0.56	
SAM9	572	677	1146	1223	0.56	
SAM40X3	561-567	630	1130	1260	0.55	
SAM1X1	570	612	1121	min. 1270	0.55	
SAM4X5	580	623	1194	1290	0.55	
SAM7 (SAM1651)	584	653	1121	1290	0.55	85
SAM4X1	573	621	1135	min. 1300	0.54	
SAM4X7	558	616	1198	1255	0.54	
SAM40	568-574	623	1110	1338	0.53	550
SAM1X3	560	589	1119	min. 1300	0.53	
SAM4X3	568	623	1146	min. 1320	0.53	
SAM1X5	540	572	1115	min. 1300	0.52	
SAM3X1	560	614	1108	min. 1320	0.52	
SAM3X5	590	677	1143	min. 1400	0.52	
SAM3X7	605	697	1164	min. 1420	0.52	
SAM5X1	570	622	1134	min. 1360	0.52	
SAM8	565	637	1137	1350-1370	0.52	
SAM35	545-565	613	1074	1350	0.51	450
SAM3X3	573	659	1138	min. 1380	0.51	
SAM5X5	596	659	1193	min. 1420	0.51	
SAM1X7	510	545	1112	min. 1300	0.5	
SAM5X3	575	641	1147	min. 1410	0.5	
SAM10	535	568	1210	1350-1370	0.5	

Kinetic Transformation Diagrams - Introduction

- **Improved corrosion properties of amorphous Fe-based compared to conventional alloys arise mostly from the lack of grain boundaries.**
- **The amorphous structure must be maintained over long times and elevated temperatures in order to keep beneficial corrosion properties.**
- **The stability of amorphous Fe-alloys must be evaluated**
  - **Assessment of Time-Temperature-Transformation diagrams**
- **Combinatorial approach utilizing isothermal annealing, wedge-casting and thermodynamic modeling.**

Procedure for Kinetic Transformation Diagram Assessment



Steady state nucleation rate (heterogeneous):

$$J = \Omega \exp\left[-\frac{\Delta G^{*} f(\theta)}{kT}\right], \quad \Omega = \rho_S \nu = \rho_S D_L / a^2$$

$$\Delta G^{*} = \frac{A\sigma_{LS}^{3}}{\Delta G_v^{2}}$$

$$JVt = 1, \quad D_L = \frac{kT}{3\pi a \eta}, \quad \eta = \Gamma \exp\left[\frac{B}{T - T_0}\right]$$

$$\ln(t) = \ln\left[\frac{3\pi a^3 \Gamma}{\rho_s V k}\right] + \frac{B}{T - T_0} - \ln T + \frac{C}{T \Delta G_v^2}$$

ρ_s = Available site density, ν = Attachment frequency, D_L = Liquid diffusivity, a = Jump distance, A = Geometrical factor, σ_{LS} = L/S interfacial energy, G_v = Driving free energy for nucleation, F(θ) = Catalysis factor (0 < f(θ) $\leq$ 1), V = nucleation volume, t = time

Continuous Cooling Transformation (CCT) diagrams

- **Isokinetic behavior (Christian, 1975): the reaction rate depends solely on the state of the assembly.**
- **Additivity rule (Scheil, 1935):**
  - **For non-isothermal treatment the reaction starts when the sum of all fractional transformation times equals unity:**

$$\sum_{i=1}^{n} \frac{t_i}{\tau_i} = 1$$

- **Generalization of this equation:**

$$\int_{t_0}^{t_{CCT}} \frac{dt}{\tau_{TTT}(T)} = 1$$

$T=T_L$ at $t=t_{018}$

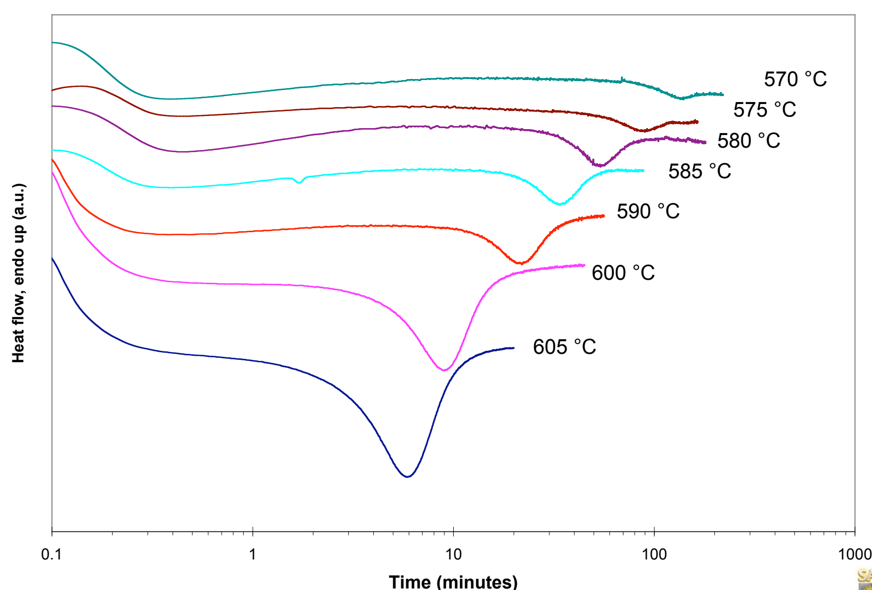
$$\int_{T_{CCT}}^{T_L} \frac{dT}{\tau_{TTT}(T) R(T)} = 1$$

Changing integration variables and their corresponding limits (Pham, 1995); R = (-dT/dt)

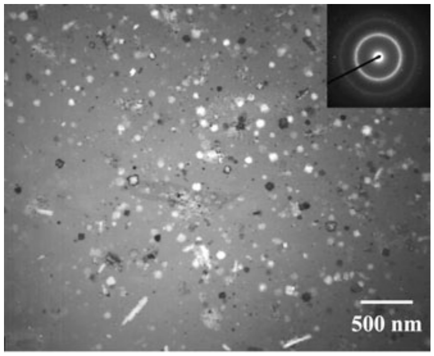
Example: SAM40

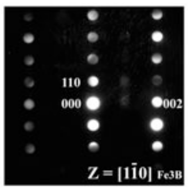
- Thermal analysis of melt-spun ribbons (DTA/DSC)
- Wedge casting
- Thermodynamic modeling
- Transformation diagrams

Isothermal Annealing of SAM40 Melt Spun Ribbons (MSRs)



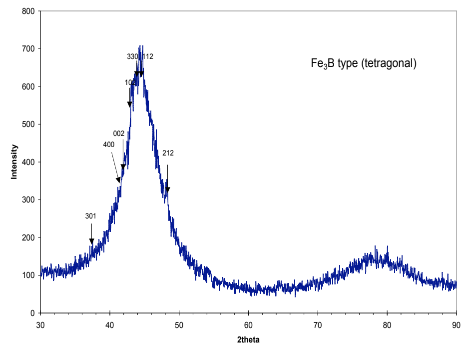
SAM40: Devitrification Analysis



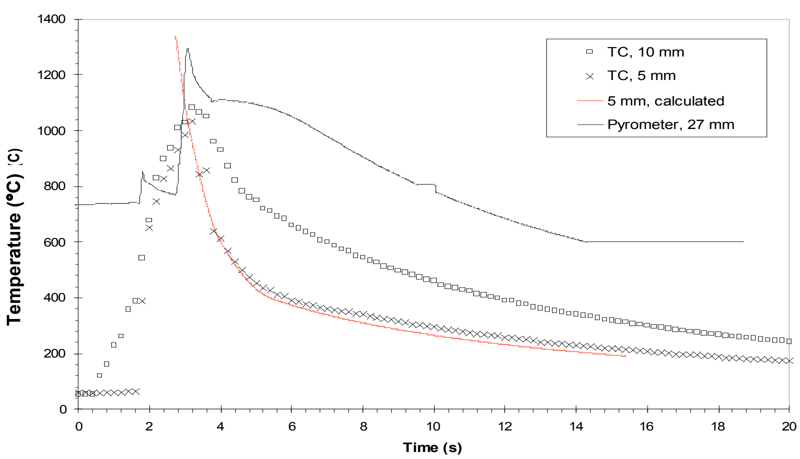


Micro-diffraction from a small particle

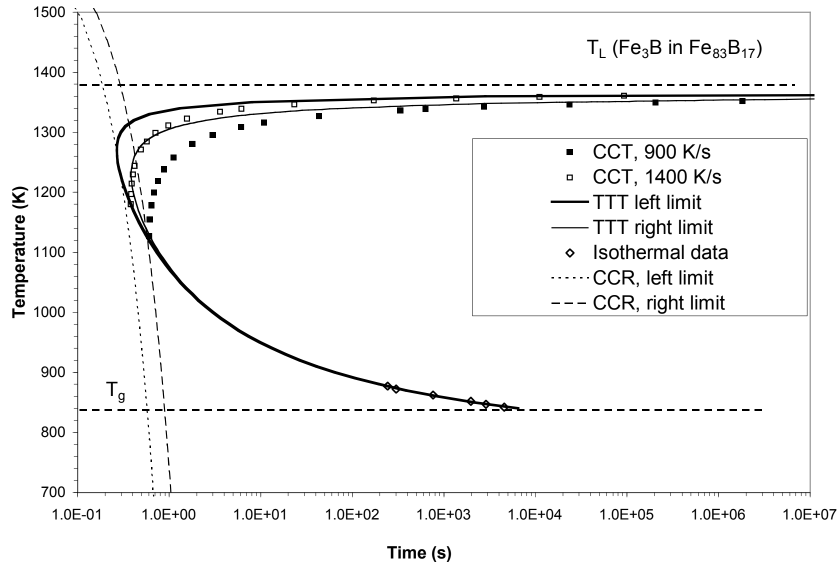
- SAM40 ribbons, 15 min at 595 °C,
- Both rod-like and spherical particles appear to be Fe_3B.
- TEM analysis consistent with XRD.



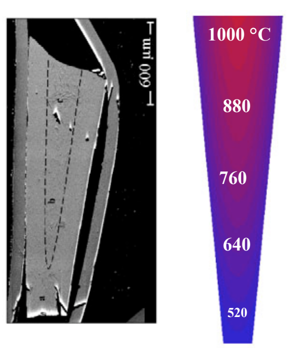
Measured and Calculated Cooling Curves for Wedge Cast SAM40



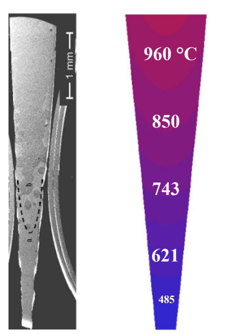
Kinetic Transformation Diagrams for SAM40



A conservative extrapolation of the TTT-curve yields intersect of 100 000 years at 370 °C.

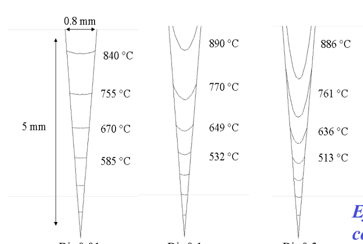


SAM40 (Bi = 0.46)



SAM35 (Bi=0.03)

Microstructure and calculated temperature distribution in two wedge cast alloys after 0.4 seconds. Morphology features are linked to Biot numbers (Bi = hL/k).



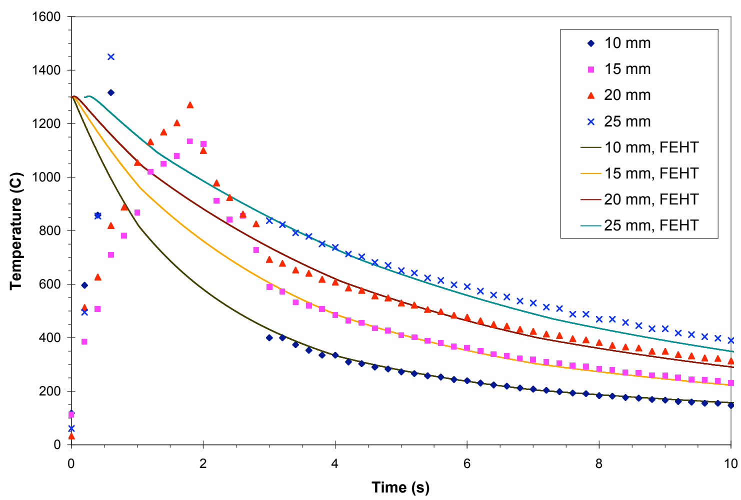
Effect of Biot number on temperature contours during cooling

- **Wedge casting**
- **SEM analysis**
- **Heat transfer analysis using initial parameters**
- **Adjusting heat transfer parameters (Biot number) to achieve agreement between thermal gradient distribution, microstructure morhpology at crystal/glass transition and measured cooling curves**
- **Final assesment of Biot number yields heat transfer coefficient, h and thermal conductivity,k**
- **Thermal property determination can be used in analysis of HVOF spray processes.**

SAM1651 (SAM7)

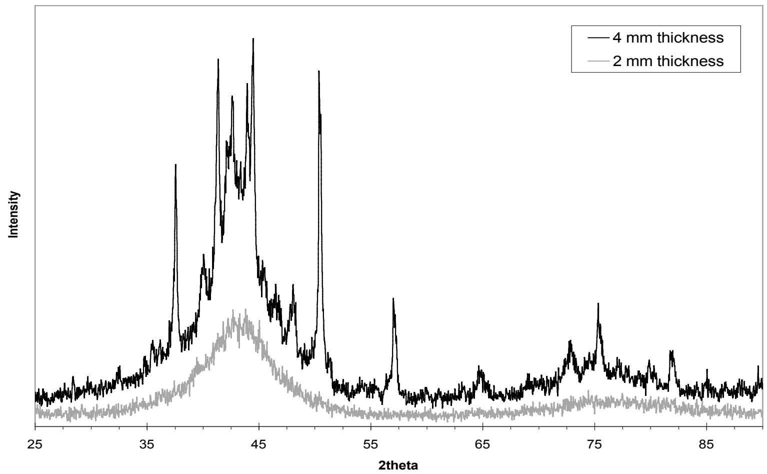
- Wedge cast
- Thermal analysis
- Microstructure characterization
- Preliminary transformation diagram

Cooling Rates Acquired During Wedge Cast of SAM1651 (SAM7)



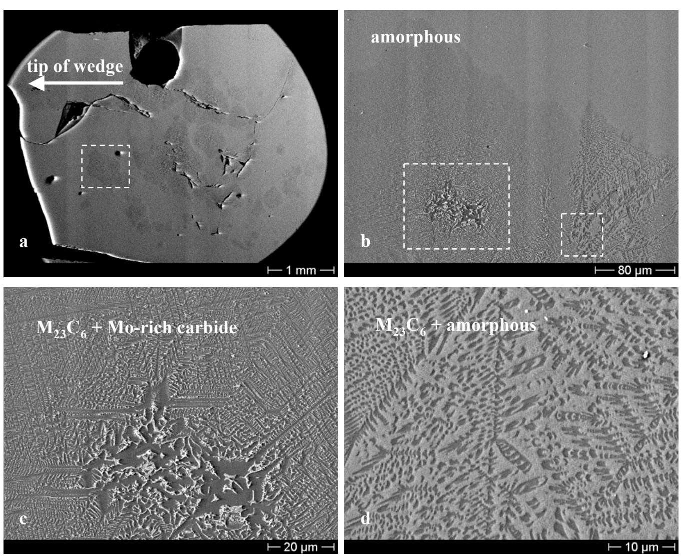
The solid lines are calculated using the heat transfer coefficient h(t) as a fitting parameter.

X-Ray Diffraction of Wedge Cast SAM1651 (SAM7).



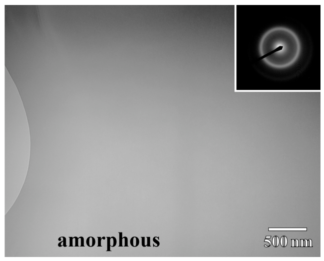
- No peaks for wedge thickness < 2 mm (fully amorphous)
- $M_{23}C_6$ peaks at 4 mm, one unidentified phase and retained glass (see next slide)

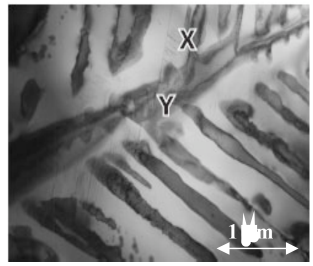
Back-Scattered Electron Images from Wedge Cast SAM1651



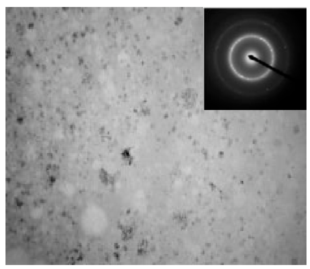
Back-scattered electron images from wedge cast SAM7, featuring the transition zone (glassy → crystalline microstructure).

TEM Images from Wedge Cast SAM1651 (SAM7) Transition Zone

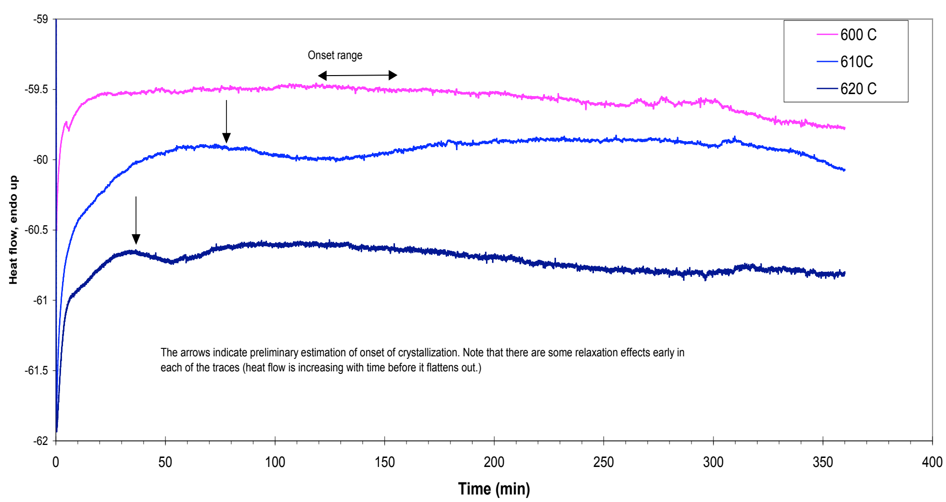




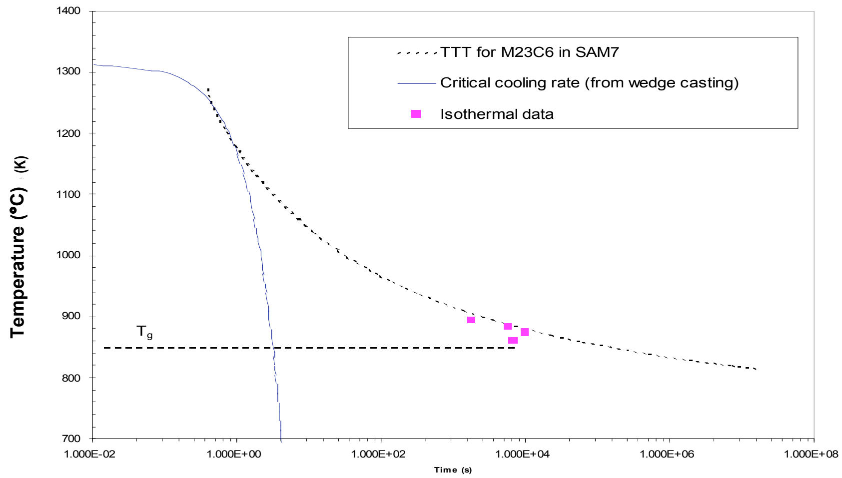
$M_{23}C_6$ phase of two different compositions (X & Y has the same structure)



← **Amorphous + nano-sized precipitates**



Preliminary TTT-Curve for SAM1651 (SAM7)

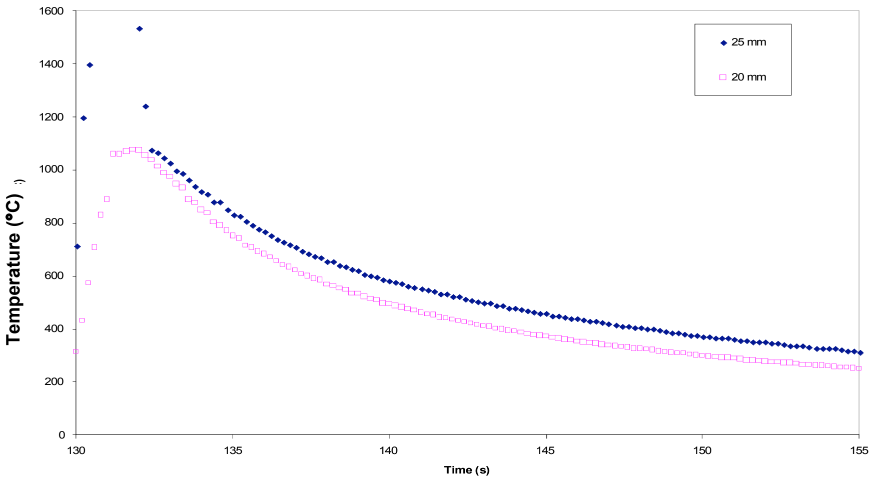


Time (s)

SAM2X5

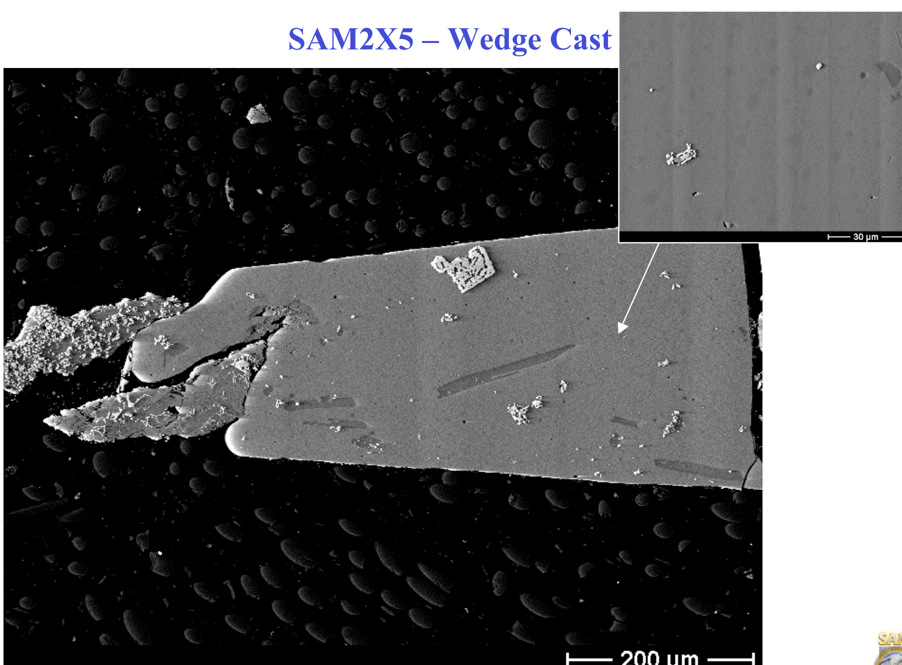
- Wedge cast
- Thermal analysis
- Microstructure characterization
- Powder analysis

SAM2X5 – Cooling curves

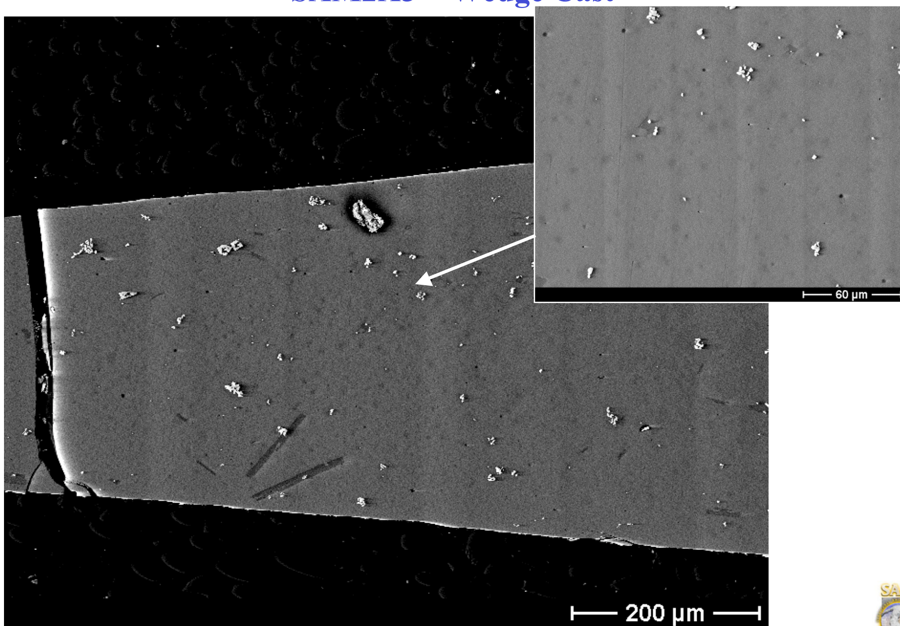


Time (s)

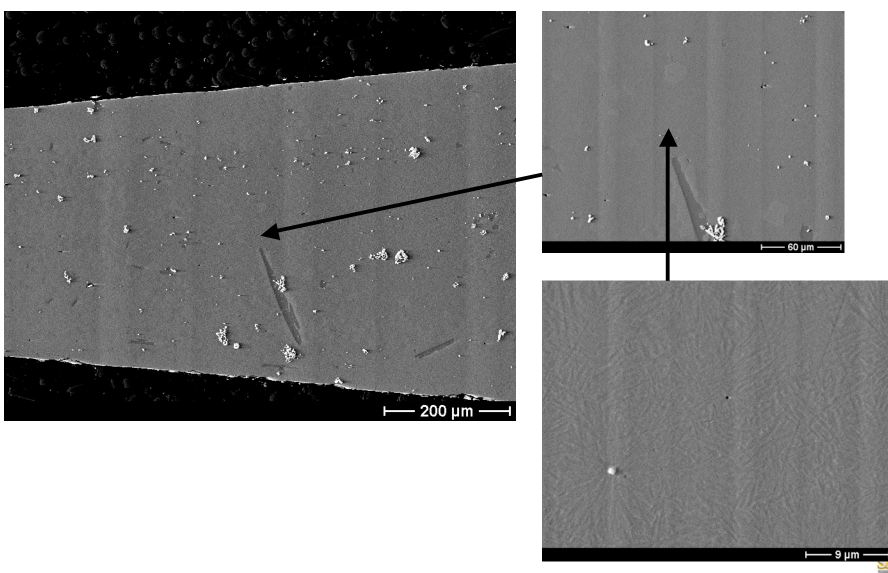
SAM2X5 – Wedge Cast



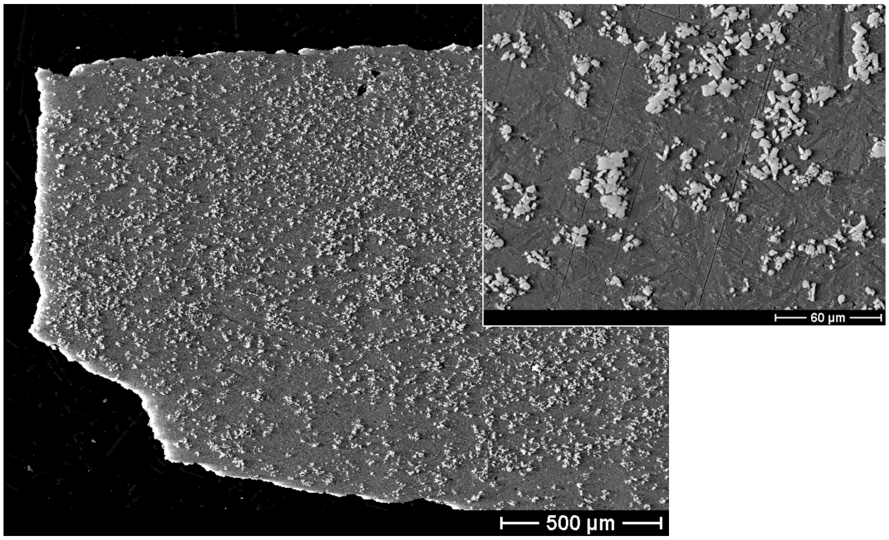
SAM2X5 – Wedge Cast



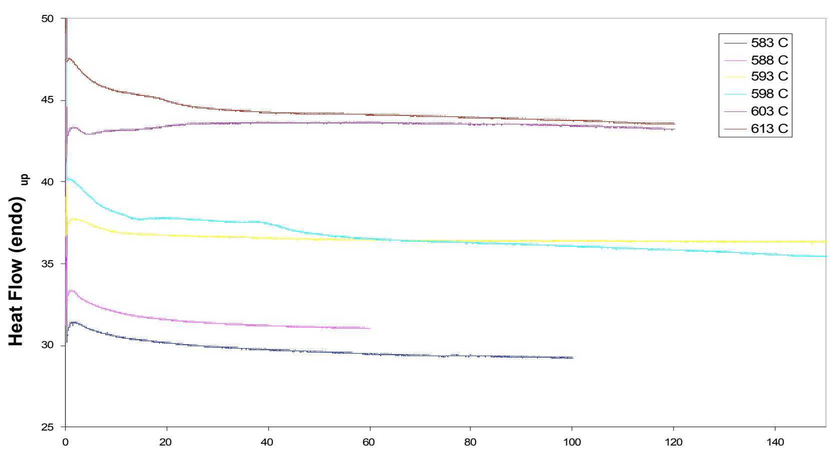
SAM2X5 – Wedge Cast



SAM2X5 – Wedge Cast

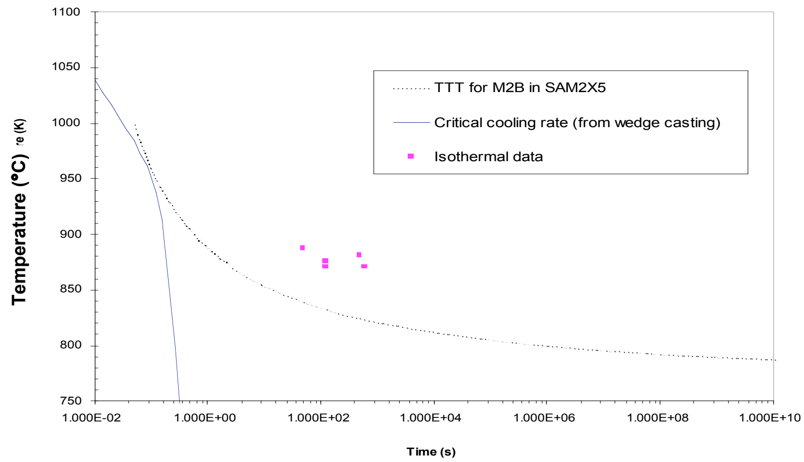


SAM2X5 – Isothermal Annealing



Time (min)

Preliminary TTT-curve for SAM2X5

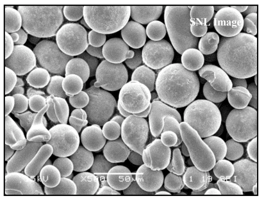


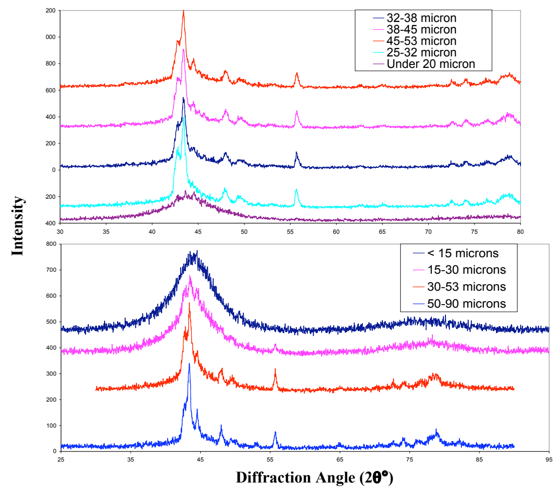
Time (s)

Powder analysis

- SAM2X5 and SAM7
- Carpenter/Caterpillar
- XRD, SEM, DTA

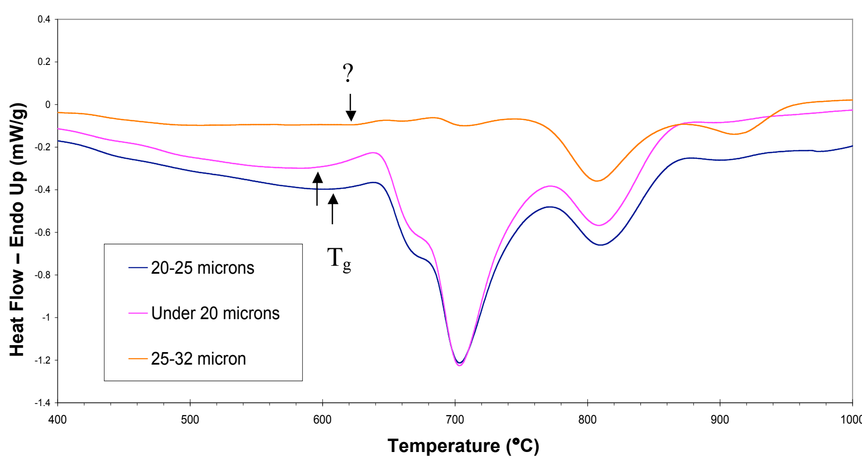
X-Ray Diffraction of Various Sizes of SAM2X5 Powder





- Crystalline phases:
- An unidentified carbide phase appears for powder sizes ~ 20 μm.
- Preliminary TEM results suggest Mo_3B_2, however, XRD/TEM analysis is ongoing.
- The bcc-Fe phase appears for powders ~ 25 μm.

Heat flow vs. Temperature Measured During Continuous Heating of SAM2X5 Powders



- T_g depends on cooling rate (powder size)
- The amorphous fraction of the droplets depend on size

Powder Processing – Nucleation Kinetics

- **During atomization an effective nucleant isolation is developed**
- **For independent powder particles the nucleant-free fraction is given by a Poisson analysis as**

$$X = \exp\left[-N_V V\right]$$

- **with N_V = number of nucleants per cm^3, V = volume**
- **For volume dispersed nucleants in powders of diameter d, the fraction that form glass, X, is**

$$X = \exp\left[-\left(d/d_0\right)^3\right]$$

- **with $N_V = 6/\pi d_0^3$**
- **For surface catalyzed nucleation, with $N_S = 1/\pi d_0^2$ (nuclei/cm^3)**

$$X = \exp\left[-\left(d/d_0\right)^2\right]$$

Powder Processing – Nucleation Kinetics

- **For homogeneous nucleation, the number of nuclei is**

$$\int_{T_g}^{T_m} J(T)dT \cong J_{max}\Delta T$$

- **where ΔT~50 K for T_{rg}=0.6. Then the fraction of amorphous powder during the time t to cool ΔT at a rate R is**

$$X \approx \exp\left[-J_{max}Vt\right] = \exp\left[-J_{max}V\Delta T / R\right]$$

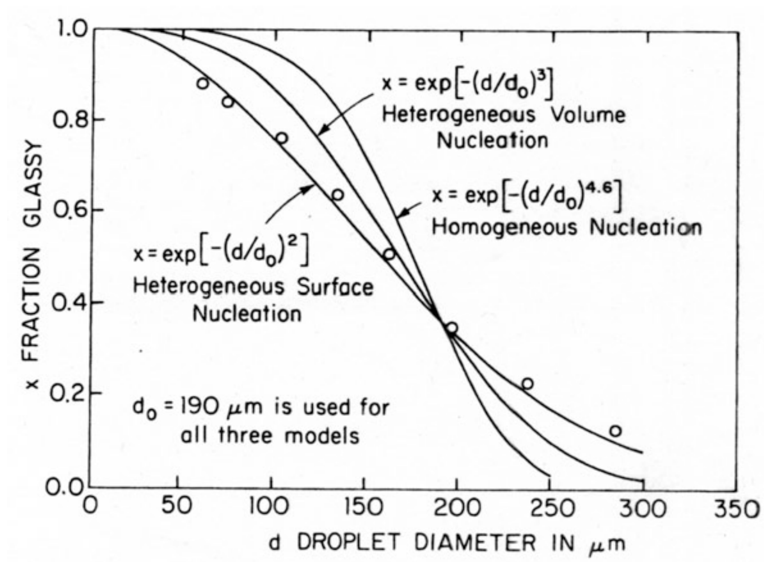
- **The cooling rate for powders can be estimated by**

$$R \approx A / d^{1.6}$$

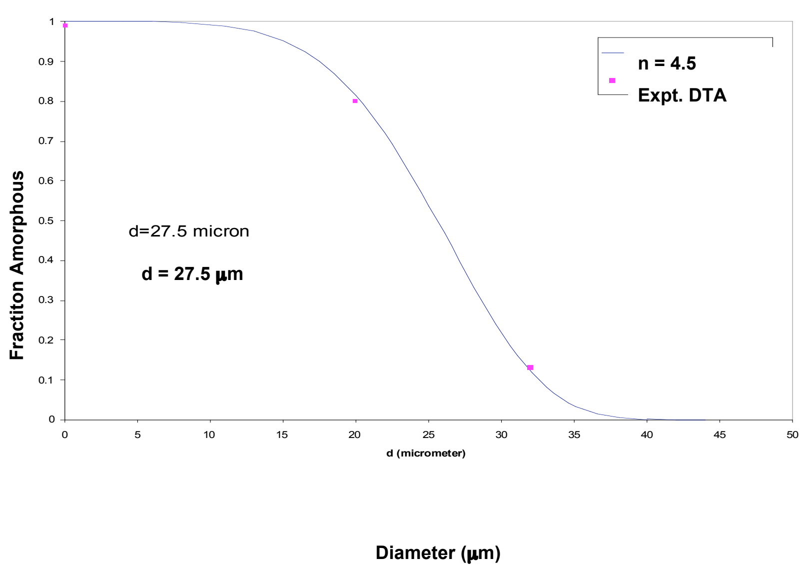
- **Then**

$$X \approx \exp\left[-(d/d_0)^{4.6}\right]$$

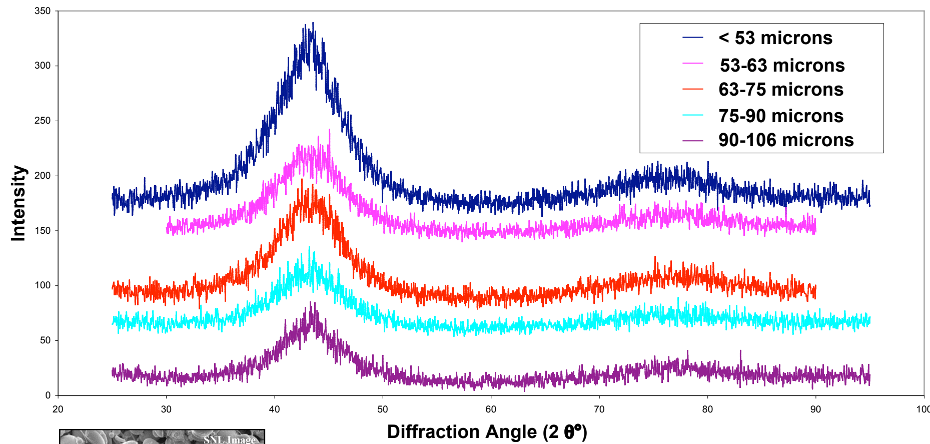
- **An analysis of the dependence of X on d can be used to evaluate the nucleation mechanism.**

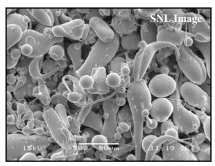


SAM2X5 – Fraction Amorphous vs. Powder Size



XRD of SAM1651 Powder for Various Powder Sizes





No crystalline peaks are observed.

Summary

- Thermal analysis measurements for 27 glass forming alloys
- Wedge casting (CCR) and kinetics measurements (meltspun ribbons) for SAM35, SAM40, SAM7, SAM2X5 and SAM2X7
- Initial model analysis improved and applied to SAM35 and SAM40, ongoing for SAM7 and SAM2X5
- Analysis of powder solidification
- Several excellent candidate alloys have been identified with good glass forming ability and corrosion resistance.
  - Detailed kinetics analysis is necessary
  - Critical guidance for processing
  - Basis for lifetime assessment

Accomplishments – FY05

- **Pearl Harbor review:**
  - Thermal analysis complete for 27 alloys
  - CCR measured for SAM35, SAM40, SAM2X7 and SAM7 (SAM1651)
  - Detailed devitrification studies have identified Fe-borides as the primary phases
  - Improved accuracy in kinetics analysis
  - Preliminary TTT-curves for SAM35 and SAM40
  - Powder processing analysis in progress.
- **Key West review:**
  - CCR measured for SAM2X5
  - More complete microstructure analysis of wedge cast SAM7 (SAM1651)
    - TEM & SEM
  - Low-temperature annealing of SAM2X5 and SAM7 (SAM1651)
  - Completed kinetic evaluation of SAM40
  - Preliminary TTT-diagrams assessed for SAM2X5 and SAM7 (SAM1651)
  - Powder analysis of SAM2X5 and SAM7 (SAM1651)
    - SEM: Morphology of powder as function of powder size
    - DSC: Devitrification kinetics as function of powder size.
    - DTA: Thermal analysis for various powder sizes.

Future Work – FY06 & Beyond

- Measurement of ΔC_P for more accurate ΔG_V.
- Improvements in diffusivity analysis.
- Growth kinetics analysis of primary crystallization.
- Heat transfer modeling
  - Establish confidence intervals for long-term predictions
  - Validate assumptions
    - boundary conditions
    - mold temperature
    - materials properties

Acknowledgements

- This work was done for LLNL in direct support of the High Performance Corrosion Resistant Materials (HPCRM) Project under sub-contract number B529197. Lawrence Livermore National Laboratory (LLNL) is gratefully acknowledged.
- The HPCRM Project at LLNL is co-sponsored by the Defense Advanced Projects Agency (DARPA) Defense Sciences Office (DSO) and the Department of Energy (DOE) Office of Science & Technology International (OSTI).

Backup Slides

High-Performance Corrosion-Resistant Materials: Corrosion Testing

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Introduction

- **The development of HPCRM compositions that are damage tolerant, environmentally friendly and applicable as a coating to a wide variety of substrates under a range of conditions is a remarkable achievement that has been accomplished in just over two years by the dedicated efforts of a team of experienced scientists and engineers who made judicious choices in charting the direction of their work. The reduction to practice of a process based on amorphous metal alloys in a few short years has left some substantial gaps in knowledge that will limit effective application of this development to DOD/DOE requirements and prevent rapid solutions to problems which may arise in effective use of this promising new technology.**

- **This report describes important progress that has been made in development of new methods for predicting the temperature dependence of the stability of the SAM series of alloys under equilibrium and glass forming conditions. This progress has important implications for prediction of the kinetics of devitrification and processing of coating/substrate systems and composites based on this new class of alloys.**

Accomplishments

1.0 During the past year Pourbaix Diagrams have been for SAM2X5, SAM1651 (SAM7) and C-22 in 5M $CaCl_2$ at 105°C and in seawater at 90°C. In addition, voltage verses phase fraction relations have been calculated for these alloys at pH=4, pH=7 and pH=10 for these alloys in both these environments for comparison with observations.

2.0 The TCFE3.TDB database has been adapted by utilizing the description of the metastable Fe_3B described in the literature to characterize the nucleation source for devitrification of the SAM alloys in keeping with the suggestion of Perepezko and Hidal made last year after measuring the devitrification of more than one dozen SAM alloys. This provided the tool for calculating the phase equilibria and glass forming characteristics in the SAM alloy in agreements with measurements performed in the current study.

3.0 This analysis provides a path for calculating the kinetics of devitrification and has already been used in suggesting that the current values of the liquidus temperature used to characterize the SAM alloys in HVOF processing is too low by 100-200°C. The present description of the glass forming characteristics of SAM2X5 and SAM1651 (SAM7) provides a very evident explanation for the experimental observation by Perepezko and Hidal that the critical cooling rate for glass formation in SAM2X5 is and order of magnitude faster than that for SAM1651 (SAM7).

Significance of the Current Accomplishments

Limitations of Current Methods

1. Current Descriptions of the relations between Phase Equilibria and Glass Formation are qualitative and do not permit application to quantitative design activities.

2. Current Descriptions of the Kinetics of devitrification of SAM alloys would very long time experimental data to estimates which subject to considerable uncertainty.

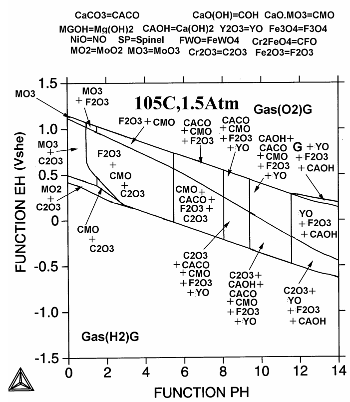
Advantages of New Methods

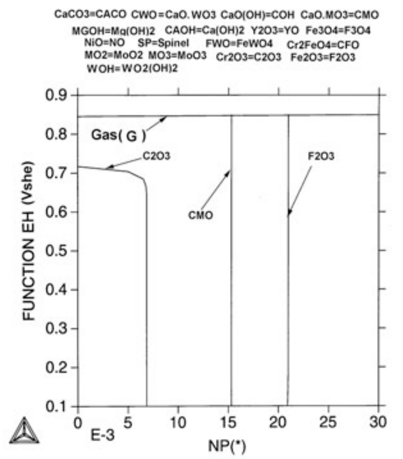
1. Explicit description of phase equilibria as a function of temperature and composition can be used to detail predictions of interactions between HPCRM coatings and substrates and can be used to design optimum coating compositions and conditions.

2. The present method for describing the phase equilibria and glass forming characteristics can be used as a basis for making explicit predictions of the kinetics of devitrification which could be applied to calculating the expected kinetics of devitrification from radioactive decay heat.

Calculation of Pourbaix and Voltage vs. Phase Fraction Relationships for SAM1651 in 5M $CaCl_2$ at 105°C

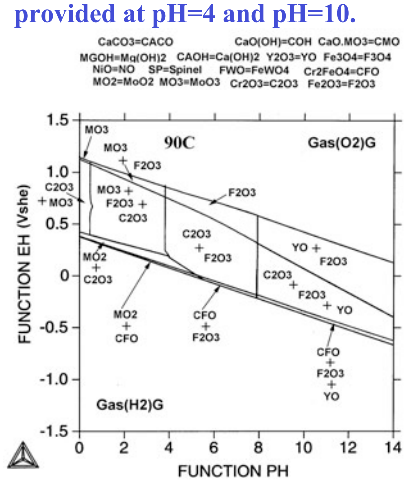
Calculated Pourbaix diagrams for SAM1651 (SAM7) at 105°C in 5M $CaCl_2$ and voltage verses phase fraction diagram shown at pH=4 for comparison with measurements (calculations also provided at pH=7 and 10).

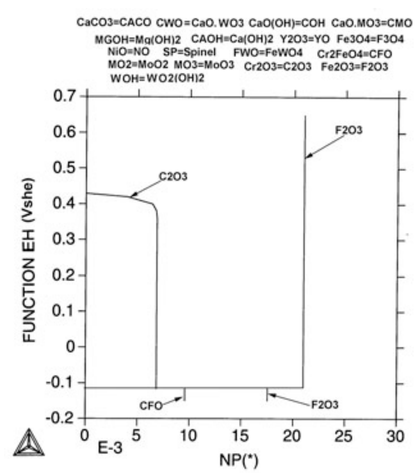




Calculation of Pourbaix and Voltage vs. Phase Fraction Relationships for SAM1651 in Seawater at 90°C

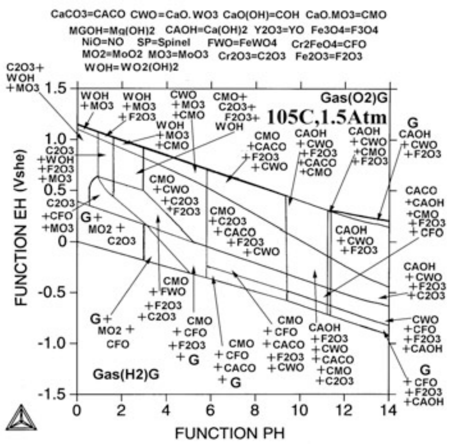
Calculated Pourbaix diagrams for SAM1651 (SAM7) at 90°C in seawater and voltage verses phase fraction relations at pH=7. Calculations are also provided at pH=4 and pH=10.

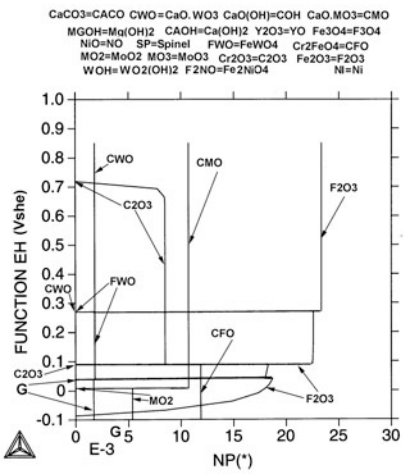




Calculation of Pourbaix and Voltage vs. Phase Fraction Relationships for SAM2X5 in 5M $CaCl_2$ at 105°C

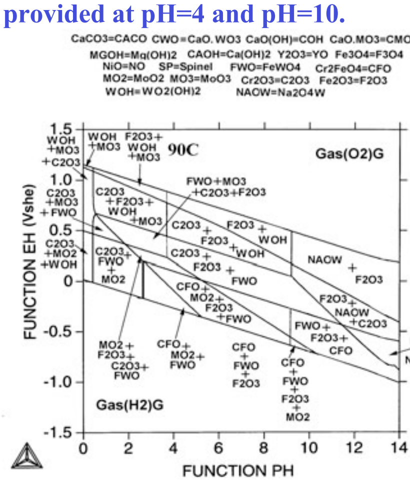
Calculated Pourbaix diagrams for SAM2X5 at 105°C in 5M $CaCl_2$ and voltage verses phase fraction relations at pH=4. Calculations are also provided at pH=4 and pH=10.

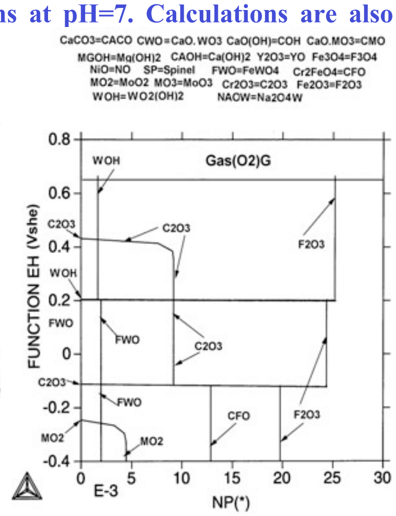




Calculation of Pourbaix and Voltage vs. Phase Fraction Relationships for SAM2X5 in Seawater at 90°C

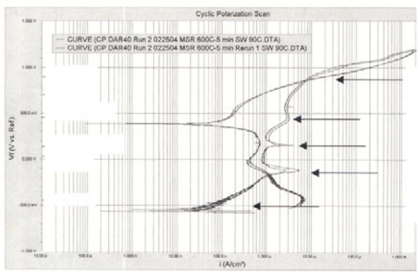
Calculated Pourbaix diagrams for SAM2X5 at 90°C in seawater and voltage verses phase fraction relations at pH=7. Calculations are also provided at pH=4 and pH=10.



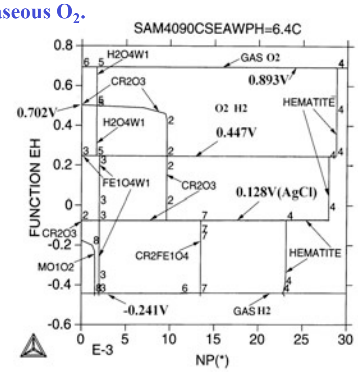


Calculation of Pourbaix and Voltage vs. Phase Fraction Relationships for SAM40 in Seawater at 90°C

Cyclic polarization data from LLNL and calculated phase fraction verses voltage diagrams for SAM40 at pH=6.4 and 90°C in seawater. Voltages are measured relative to a Ag/AgCl reference electrode in 4M KCl. Observed anodic oxidation peaks were observed at approximately –0.50, +0.15, +0.2, +0.45 and +0.90 volts. These coincide with calculated transitions in the surface oxide. These peaks correspond to the predicted formation of gaseous H_2, the dissolution of molybdenum dioxide, the conversion of iron chromate to chromium dioxide, the conversion of iron tungstate to tungsten hydroxide, the dissolution of chromium dioxide, and finally the formation of gaseous O_2.

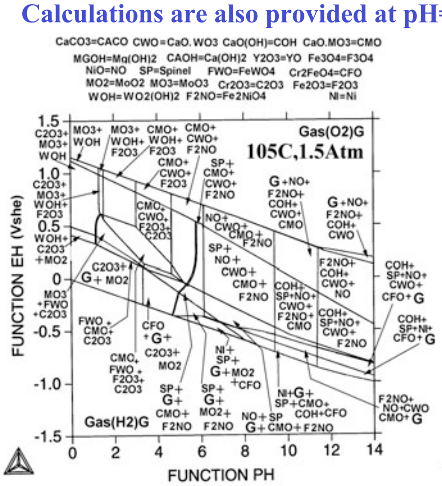


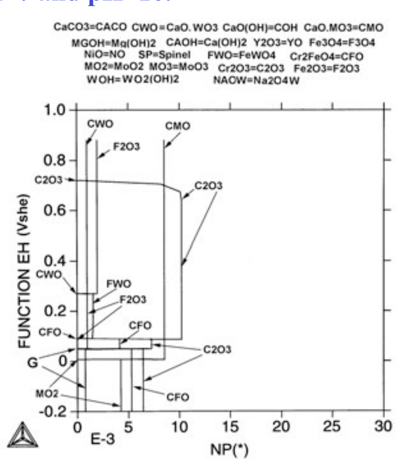
SAM40, Tested in 90C, Seawater, after devitrification at 600C for 5 minutes



Calculation of Pourbaix and Voltage vs. Phase Fraction Relationships for Alloy C-22 in 5M $CaCl_2$ at 105°C

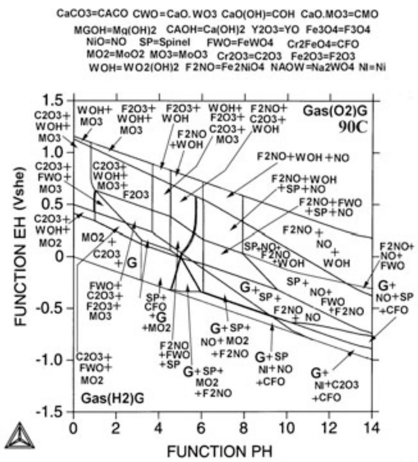
Calculated Pourbaix diagrams for nickel-based Alloy C-22 at 105°C in 5M $CaCl_2$ and voltage verses phase fraction relations at pH=4. Calculations are also provided at pH=7 and pH=10.

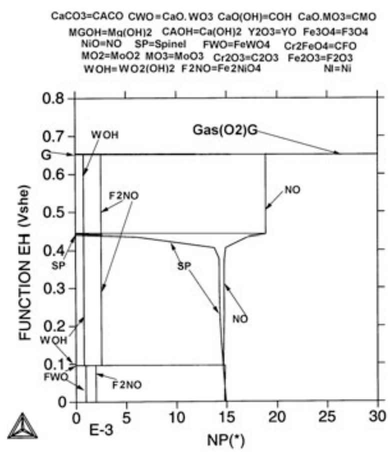




Calculation of Pourbaix and Voltage vs. Phase Fraction Relationships for Alloy C-22 in Seawater at 90°C

Calculated Pourbaix diagrams for Alloy C-22 at 90°C in seawater and voltage verses phase fraction relations at pH=7. Calculations are also provided at pH=4 and pH=10.

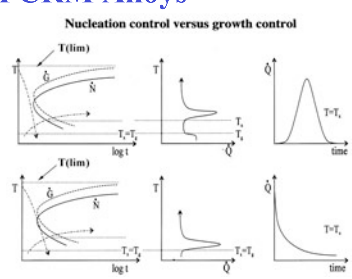


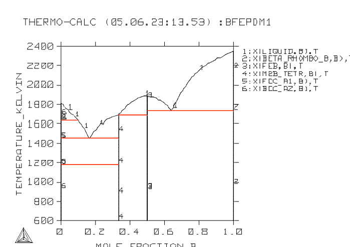


Thermodynamic and Kinetic Description of Glass Formation in HPCRM Alloys

Nucleation verses growth control was discussed by Perepezko and Hildal at HPCRM 2005 in Pearl Harbor. When devitrification results from the formation of a single crystalline phase $T_x = T_g$.

Palumbo et.al. at CALPHAD 26 have discussed the metastable Fe_3B phase in the Fe-B system as the nucleation site for devitrification and have characterized it. This description was used in adapting the 'TCFE3.TDB' database in order to calculate the stable Fe-B database at the right and the metastable Fe-B phase diagrams shown on the next slide. The new adapted database was used to calculate phase equilibria and glass formation in the SAM-type alloys in good agreement with the experimental results of Perepezko, Hidal and Yang.

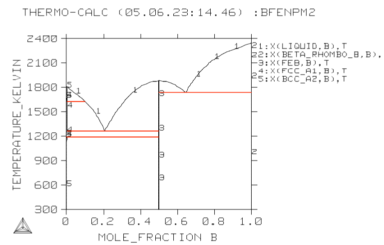


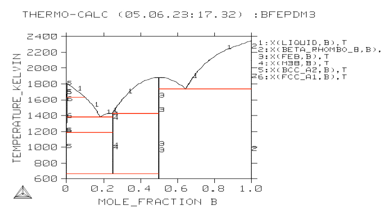


Metastable Phase Equilibrium in the Fe-B System

The stable Fe-B phase diagram shown on the previous slide displays the liquid, bcc, fcc, B, M_2B, and FeB phases. The Fe_3B phase is not stable. In the figure on the right the M_2B phase is suspended without including the Fe_3B phase. In the figure below the phase diagram is calculated including both the Fe_3M and FeB phases but suspending M_2B.

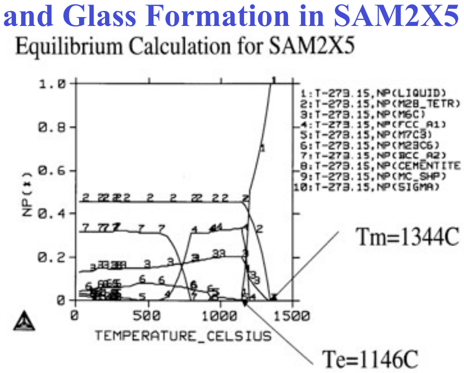
The studies carried out by Perepezko and Hildal last year suggested that phase that was responsible for initiating the devitrification of the SAM alloys was M_3B. The current set of calculations applies this idea by adapting the the 'TCFE3.TDB' database and adding the Fe_3B description due to Palumbo et. al. In order to provide a thermochemical rational for the HPCRM experimental studies. The value of these results is that it provides the basis for a new theoretical tool for understanding and predicting the behavior of the current and future SAM-type alloys.

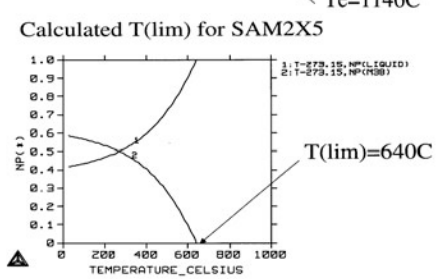




Calculation of Phase Equilibria and Glass Formation in SAM2X5

The results for SAM2X5 are show in this figure, and those for SAM1651 (SAM7) are shown on the next figure. The following table (Table 1) lists most of the compositions studied by Perepezko and Hidal along with a comparison of all the measured and calculated results listed in Tables 2 and 3. Detailed calculations like those shown for SAM1651 and SAM2X5 were made for all of the alloys. The equilibrium calculations shown for SAM1651 define the liquidus, T_m, at 1344°C and the quasi-eutectic, T_e, where the liquid disappears and is converted to the solid phases as 1146°C. The ordinate, NP(*), is the fraction of phases that must equal 1.0. At high temperatures the fraction of liquid is 1.0. The calculation of T(lim) is performed by suspending all phases except liquid and M_3B which is approximated by Fe_3B and not the highest temperature where this phase can form from the liquid. For SAM2X5 this comes at 640°C. Slide 11 illustrates the relation between T(lim), Tg and Tx. This relation will be discussed further in the slide entitle future work.

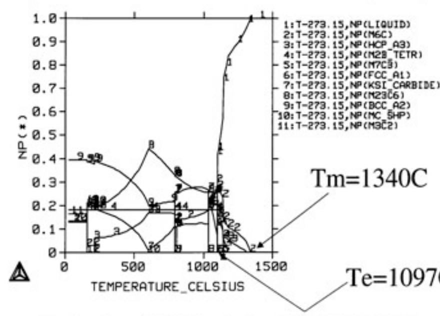




Calculation of Phase Equilibria and Glass Formation in SAM1651

The phase equilibria and glass forming relations shown for SAM2X5 are provided here for SAM1651 with T_m = 1340°C, T_e = 1097°C and T(lim) = 586°C. Examination of Table 2 shows that the comparison between the experimental and calculated results is is very good for the quasi-eutectic where the mean deviation is 16°C or less than 2%. The difference in the calculated and measured liquidus is nearly an order of magnitude higher. Part of the reason for this is due to the fact that the transitions at T_e are abrupt while the changes at T_m are gradual. Finally Table 3 compares the values calculated for T(lim) with the observed values of T_g and T_x. Although this comparison cannot be made definitively at present at present the results are promising. Finally the T(lim) results for SAM2X5 and SAM1651 are consistent with the critical cooling rates required for amorphous SAM2X5 are ten times those required for SAM1651 (SAM7).

Equilibrium Calculation for SAM7(1651)



Calculated T(lim) for SAM7(1651)

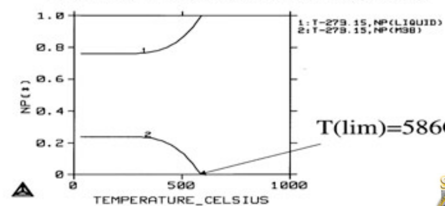


Table 1. Summary of SAM Alloy Compositions

(Atomic Percent)

Alloy	Fe	Cr	Mn	Mo	W	B	C	Si	Ni	Ti
SAM 35	54.2	15.0	2.0	2.0	1.5	16.0	4.0	5.0	0	0
SAM 40	52.3	19.0	2.0	2.5	1.7	16.0	4.0	2.5	0	0
SAM 40X3	50.7	18.4	1.9	5.4	1.6	15.5	3.9	2.4	0	0
SAM 1X1	51.8	18.8	2.0	2.5	1.7	15.8	4.0	2.5	1	0
SAM 1X7	48.6	17.7	1.9	2.3	1.6	14.9	3.9	2.3	7	0
SAM 2X1	51.8	18.8	2.0	3.5	1.7	15.8	4.0	2.5	0	0
SAM 2X3	50.7	18.4	1.9	5.4	1.6	15.5	3.9	2.4	0	0
SAM 2X5	49.7	18.1	1.9	7.4	1.6	15.2	3.8	2.4	0	0
SAM 2X7	48.6	17.7	1.9	9.3	1.6	14.9	3.7	2.3	0	0
SAM 4X1	51.8	18.8	2.0	2.5	1.7	15.8	4.0	2.5	0	1
SAM 4X7	48.6	17.7	1.9	2.3	1.7	14.9	3.7	2.3	0	7
SAM 7(1651)	48.0	15.0	0	14.0	0	6.0	15.0	0	0	0
SAM 8	46.6	14.6	0	13.6	3.0	5.8	14.6	0	0	0
SAM 9	47.1	17.1	1.8	9.3	1.5	14.4	3.6	2.3	0	0
SAM10	57.3	21.4	0	2.6	1.8	16.9	0	0	0	0

Table 2. Summary of Calculated Results T_L^* and T_e^* for SAM-Type Alloys with Experimental Results T_l and T_m

(Temperature, OC)

Alloy	T_m	T_e^*	$\{T_e^* - T_m\}$	T_L	T_L^*	$\{T_L^* - T_L\}$
SAM35	1074	1133	59	1350	1384	34
SAM40	1110	1156	46	1338	1357	19
SAM40X3	1130	1150	20	1260	1350	90
SAM1X1	1121	1157	36	1270 min	1357	87 max
SAM1X7	1112	1121	19	1300 min	1354	54 max
SAM2X1	1124	1152	28	1200	1355	155
SAM2X3	1131	1147	16	1200	1348	148
SAM2X5	1133	1146	13	1200	1344	144
SAM2X7	1137	1166	29	1200	1349	149
SAM4X1	1135	1142	7	1300 min	1562	262
SAM4X7	1198	1114	16	1255	1785	530
SAM7(1651)	1121	1097	-24	1290	1340	50
SAM 8	1137	1130	-7	1360	1498	138
SAM 9	1146	1170	24	1223	1340	117
SAM10	1210	1138	-72	1360	1408	48
			-----			-----
Mean			16			134 max

Table 3. Comparison of Calculated and Experimental Glass Forming Temperatures

Alloy	T_g	T_x	$\{T_x - T_g\}$	T^*(lim)	$\{T^*(lim) - T_g\}$
SAM35	555	613	58	710	155
SAM40	571	623	62	658	87
SAM40X3	564	630	66	649	85
SAM1X1	570	612	42	645	75
SAM1X7	510	545	35	608	98
SAM2X1	575	620	45	654	79
SAM2X3	578	626	48	648	70
SAM2X5	579	628	49	640	61
SAM2X7	573	630	57	633	60
SAM4X1	573	621	48	639	66
SAM4X7	568	616	48	502	-66
SAM7(1651)	584	653	69	586	2
SAM8	565	637	72	586	21
SAM9	572	677	105	640	68
SAM10	535	568	33	634	99
			-----		-----
Mean			55		64

Note: T_g, T_x were measured by Perepezko and Hildal while T^*(lim) has been calculated. The temperature T^*(lim) lies above T_g and T_x.

Table 4. Experimental Observations of Phase Equilibrium in SAM-Type Alloys by Sandia

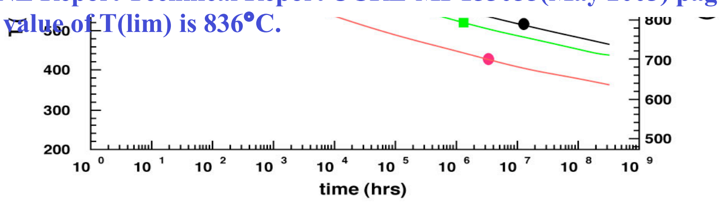
Summary of Phases Present

		M23(CB)6	M6C	Fe2B	Fe3B	M3B2	Cr2B	MB	bcc Fe	Y-Mo(CB)
DAR 35	HDIF fusing	XRD/WDS				XRD/WDS	XRD/WDS		XRD/WDS	
DAR40	ribbon 1000°C	XRD			XRD				XRD/WDS	
SAM1651	800°C	XRD/TEM	XRD/TEM			XRD			XRD	
SAM1651	1000°C	XRD/TEM	XRD/TEM			XRD/TEM		XRD	XRD/TEM	TEM
SAM1651	plate 1150C	XRD	XRD						XRD	
2x5	ORNL rod	XRD				XRD			XRD	
2x1	ribbon 800°C	XRD	XRD(W)		XRD(s)	XRD			XRD	
2x5	ribbon 800°C	XRD	XRD(M)		XRD(M)	XRD			XRD	
2x7	ribbon 800°C	XRD	XRD(s)		XRD(w)	XRD			XRD	
SHS727	HVOF coating						XRD		XRD	

Backup Slides

Suggestions for Future Work

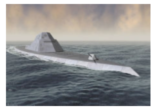
The figure shown below is a calculated isothermal TTT diagram for a FCC-base matrix of a ternary Ni-Cr-13.5Mo (wt. %) alloy transforming into the P-phase. This figure is taken from an LLNL report by Turchi, Kaufman and Zi-Kui Liu entitled "Modeling of Stability and Aging of Candidate Ni-Cr-Mo Based Alloys for the Yucca Mountain Project [LLNL Report Technical Report UCRL-MI-153055(May 2003) page 27]. The value of T(lim) is 836°C.



Suggestions for Future Work

The current database that has been applied to calculate the equilibrium and glass forming relations for the SAM-type alloys is adequate for calculating TTT and CTT diagrams similar to that shown for the FCC/P-Phase case shown above using the DICTRA software developed by TCS.

In order to perform the required calculation a Mobility Database for the amorphous phase must be developed and applied. Palumbo and his colleagues as well as the TCS Group in Stockholm have made some progress along this Path. It is suggested that future work be directed toward completing this task.



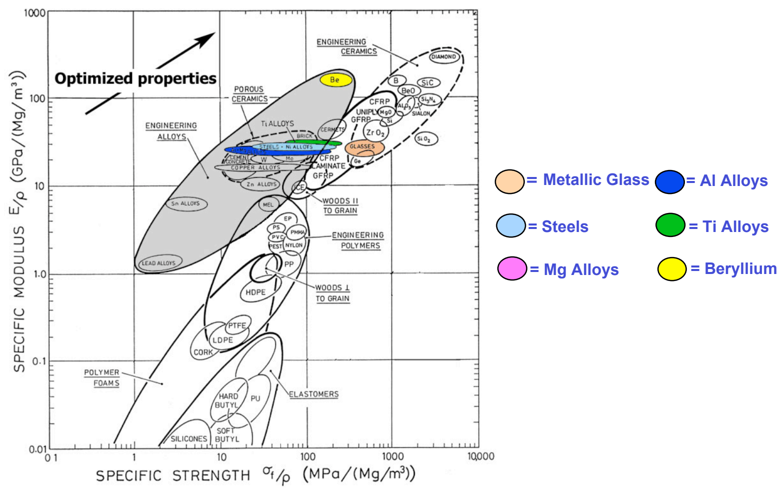
High-Performance Corrosion-Resistant Materials: Mechanical Properties

John J. Lewandowski
Case Western Reserve University
Cleveland, Ohio

Outline

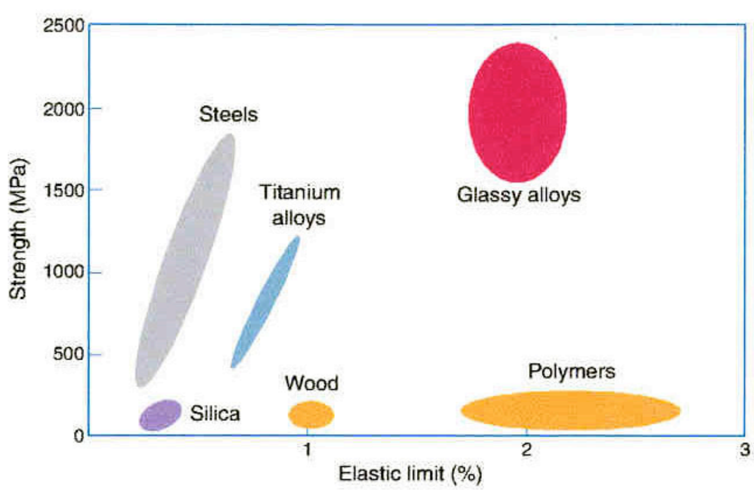
- Strength of Amorphous Metals
  - Comparison to Other Materials
- Processing Background
  - Critical Cooling Rate
  - Effects on Critical Casting Thickness
    - Bulk Metallic Glasses
    - Amorphous Powders/Ribbons
- Hardness/Strength as f(Temperature)
  - Deformation Mechanism Map
  - Examples of Various Metallic Glass Systems
    - Hardness vs Temperature
    - Hardness Evolution vs Time at Temperature
      - Relevance to Transformation Studies
      - Relevance to Processing
  - Comparison to 316L Stainless Steel
- Toughness
  - Examples of Various Metallic Glasses
    - Effects of Notch Radius
    - Fracture Energy (G)
- Loading Rate Effects
  - High Strain Rate Testing
    - Relevance to Impact Loading
    - Examples of Metallic Glasses
      - Use of High Speed Video
      - Effects of Length/Diameter

Summary of Engineering Materials and Unique Location of Metallic Glass



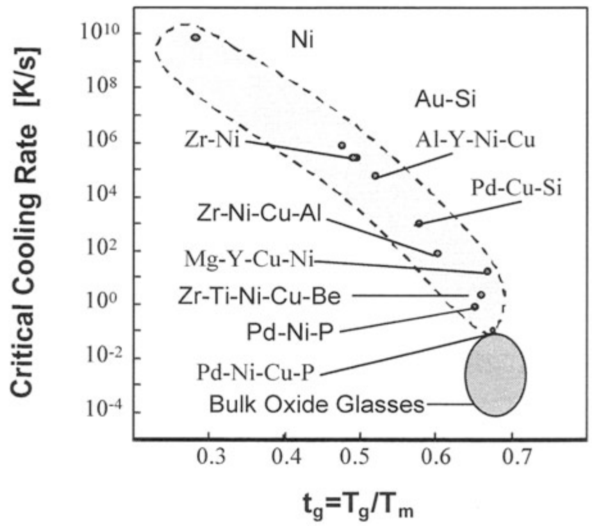
Adapted from: Ashby, M. F. *Materials Selection in Mechanical Design*, Pergamon Press, 1992, pp. 28-36

Glassy Metal Alloys Possess High Strength and Elastic Limit



Reference: Telford, M. The Case for Bulk Metallic Glass, Materials Today, March 2004, pp.36-43.

Increase in t_g (Reduced Glass Transition Temperature) Lowers the Critical Cooling Rate to Produce Bulk Metallic Glasses



Reference: Peker, A. and Johnson, W.L. Appl. Phys. Lett., vol. 63, 1993, p. 2342.

Reduced Glass Transition Temperature Indicative of Glass Forming Ability

SAM2X5 → (SAM2X5)

SAM1651 → (SAM7)

Alloy	T_g (°C)	T_x (°C)	T_m (°C)	T_L (°C)	T_{rg}
SAM35	545-565	613	1074	1350	0.51
SAM40	568-574	623	1110	1338	0.53
SAM40X3	561-567	630	1130	1260	0.55
SAM1X1	not clear	612	1121	min. 1270	N.A.
SAM1X3	560	589	1119	min. 1300	0.53
SAM1X5	540	572	1115	min. 1300	0.52
SAM1X7	510	545	1112	min. 1300	0.50
SAM2X1	575	620	1124	1190-1210	0.57
SAM2X3	578	626	1131	1190-1210	0.57
SAM2X5	579	628	1133	1190-1210	0.57
SAM2X7	573	630	1137	1190-1210	0.57
SAM3X1	560	614	1108	min. 1320	0.52
SAM3X3	573	659	1138	min. 1380	0.51
SAM3X5	590	677	1143	min. 1400	0.52
SAM3X7	not clear	697	1164	min. 1420	
SAM4X1	573	621	1135	min. 1300	0.54
SAM4X3	568	623	1146	min. 1320	0.53
SAM4X5	580	623	1194	1290	0.55
SAM4X7	558	616	1198	1255	0.54
SAM5X1	570	622	1134	min. 1360	0.52
SAM5X3	575	641	1147	min. 1410	0.50
SAM5X5	596	659	1193	min. 1420	0.51
SAM6	580	623 [2)]	995	1238-1250	0.56
SAM7	584	653 [2)]	1121	1290	0.55
SAM8	565	637 [2)]	1137	1350-1370	0.52
SAM9	572	677 [2)]	1146	1223	0.56
SAM10	535	568 [1)]	1210	1350-1370	0.50
SAM11	535	572 [1)]	1202	1365-1395	0.49

Effects of Temperature on Flow Behavior of Amorphous Metals

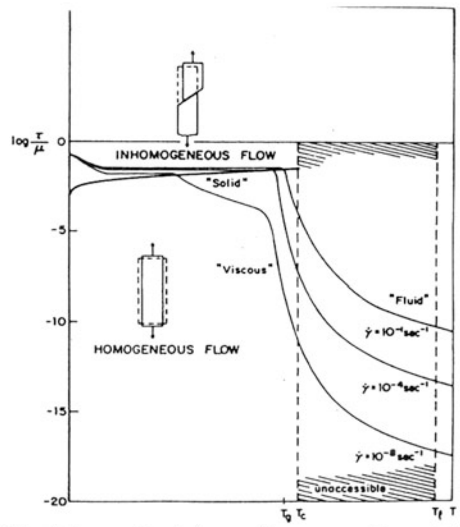
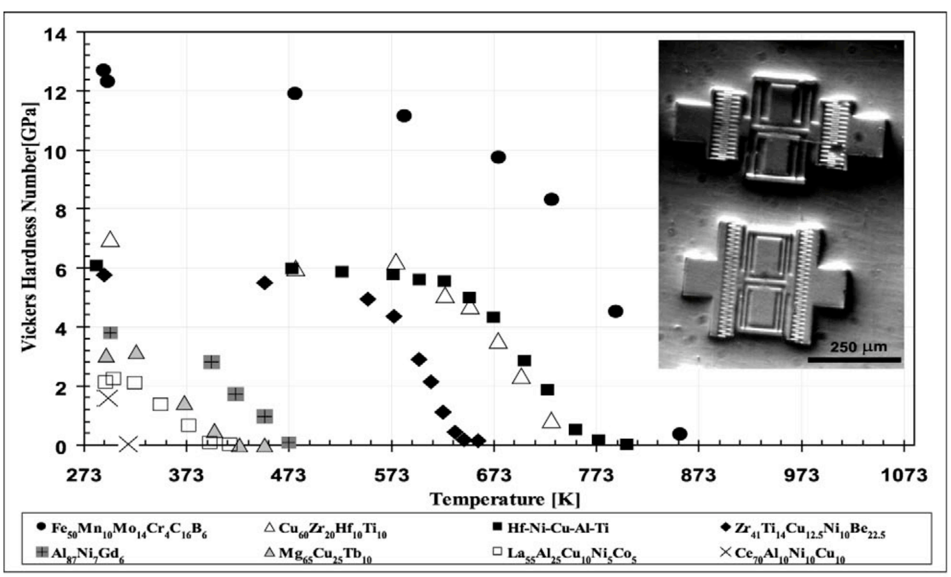


Figure 2.1: Schematic deformation map of a metallic glass [43]

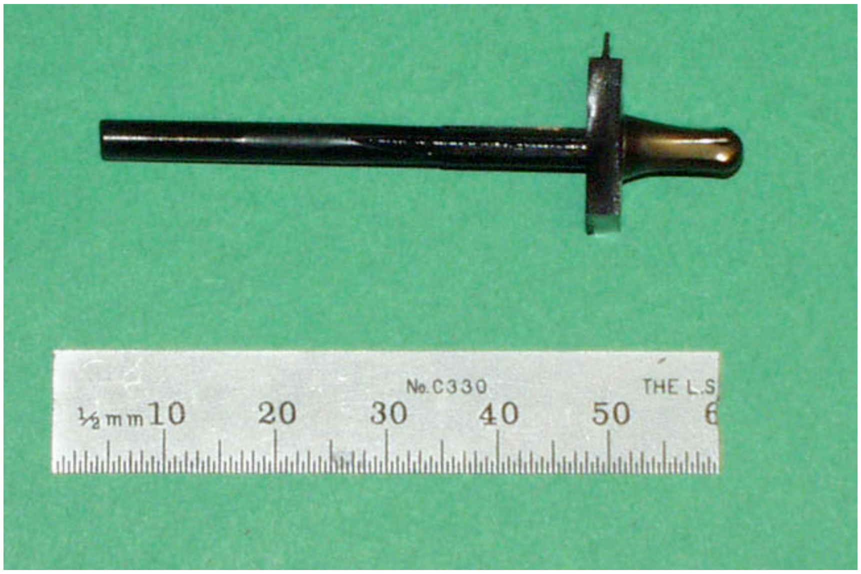
Reference: Spaepen, F. Acta Metall., 1977, 25, 407.

Hardness Reduction Near Tg - Unique Processing Possibilities

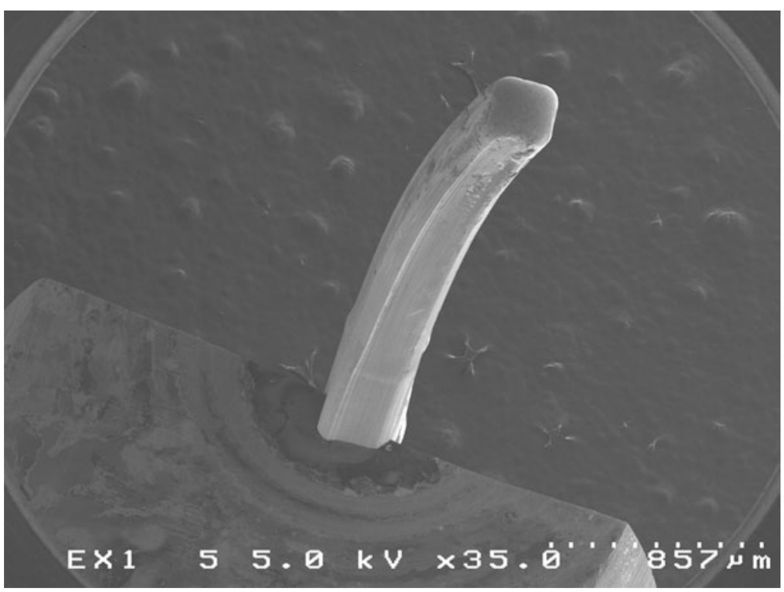


Reference: Lewandowski, J., Shazly, M., Shamimi Nouri, A. Scripta Mater., 2006, 54(3), pp. 337-342.

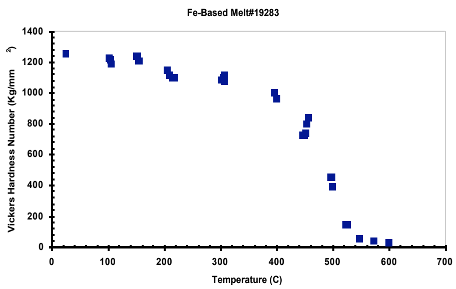
Example of Unique Forming Using Zr-based Metallic Glass

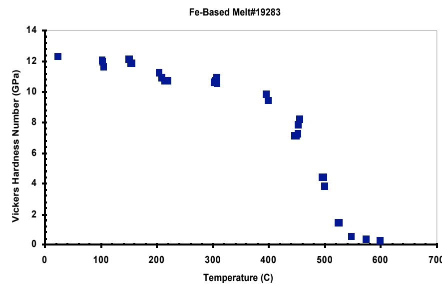


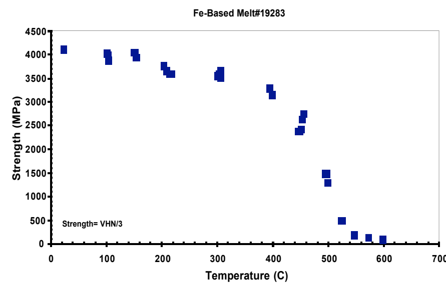
Extrusion of Metallic Glass - Possible Wire Production Process



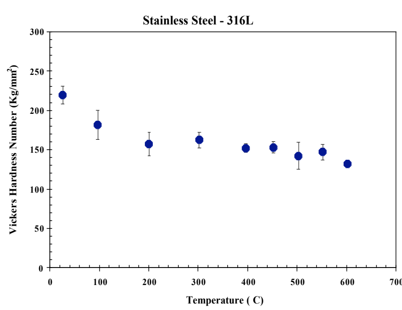
Hot Micro-hardness Measurements of SAM1651 Ingots (Tg = 584C)

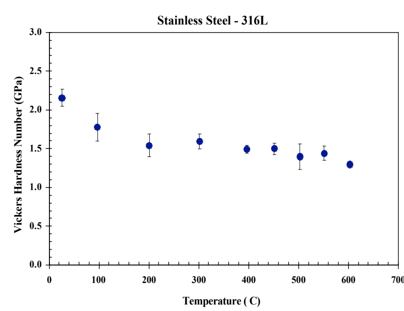


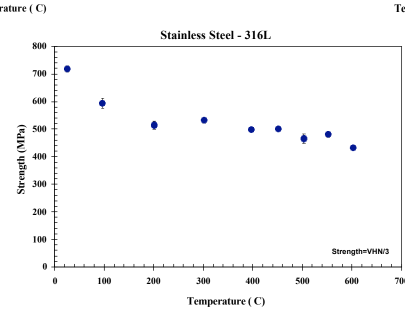




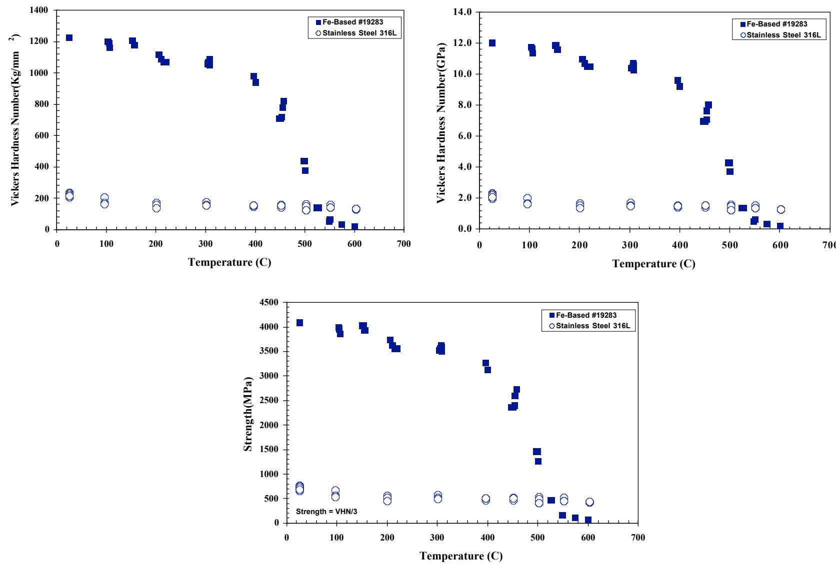
Hot Micro-hardness Measurements of 316L



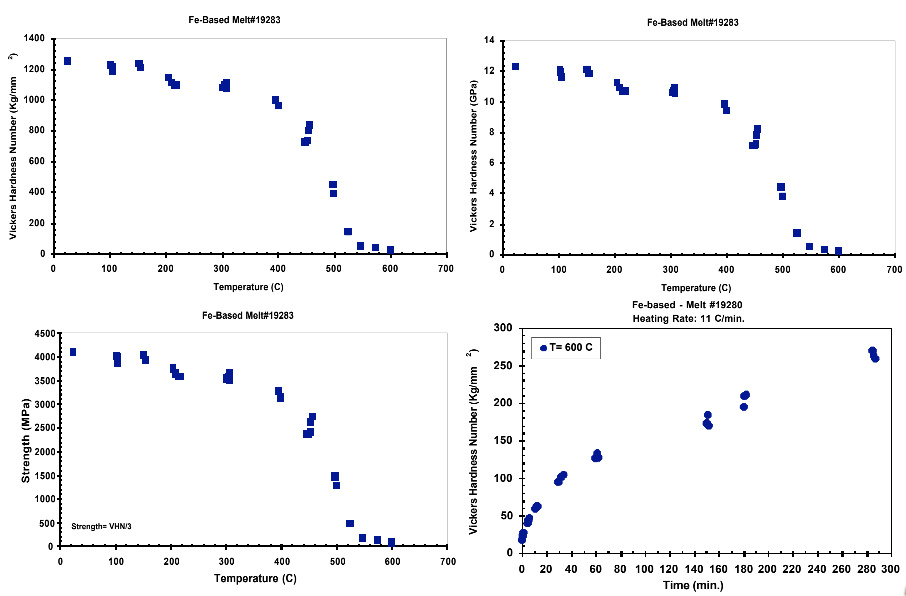




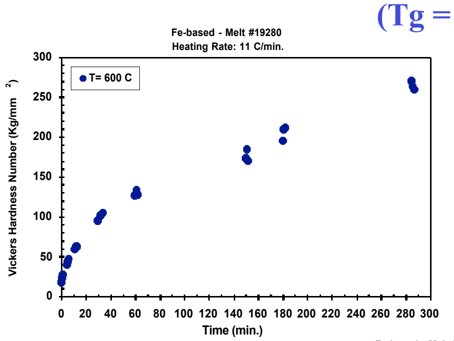
Hot Micro-hardness Measurements of SAM1651 vs 316L

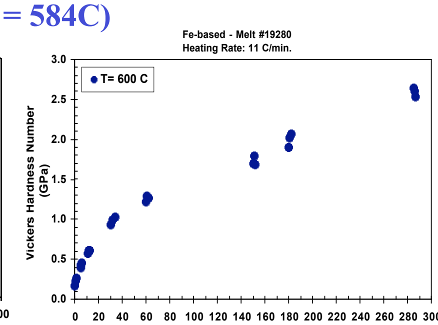


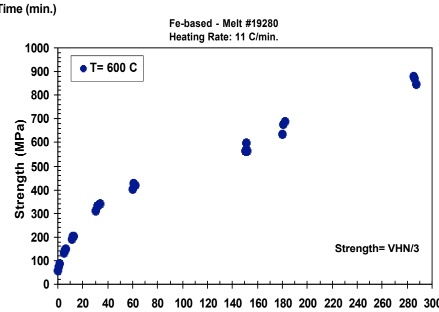
Hot Micro-hardness Measurements of SAM1651 Ingots (Tg = 584C)



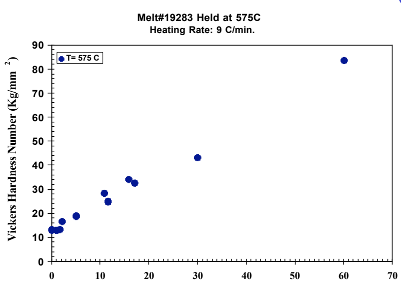
Micro-hardness Evolution vs Time at 600C - SAM1651 Ingots (Tg = 584C)

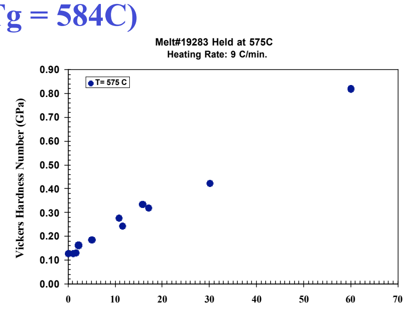


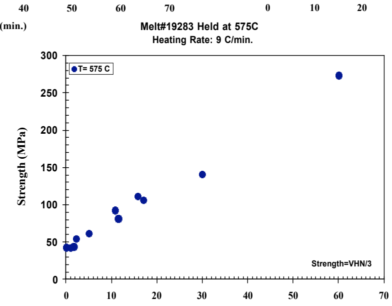




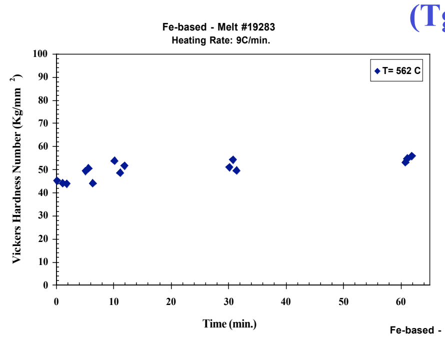
Micro-hardness Evolution vs Time at 575C - SAM1651 Ingots (Tg = 584C)

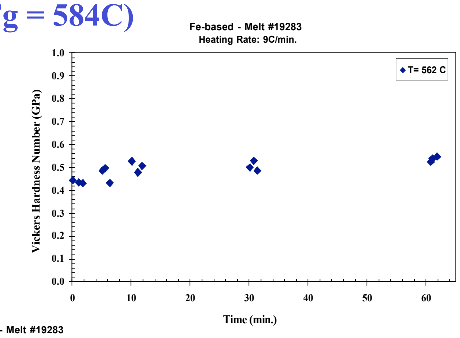


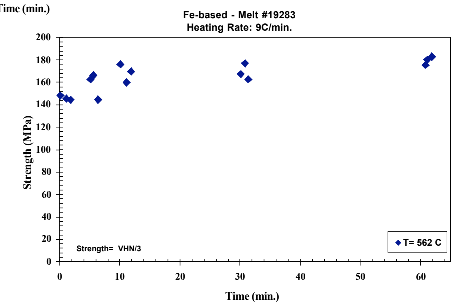




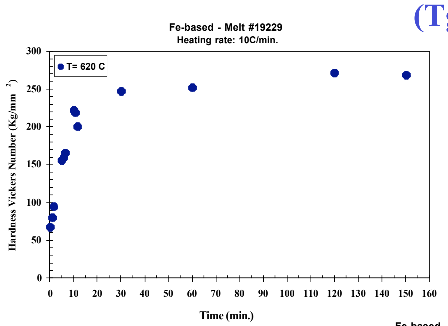
Micro-hardness Evolution vs Time at 562C - SAM1651 Ingots (Tg = 584C)

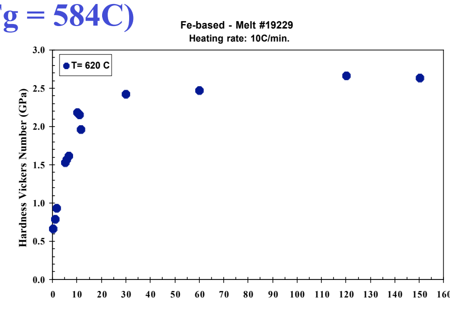


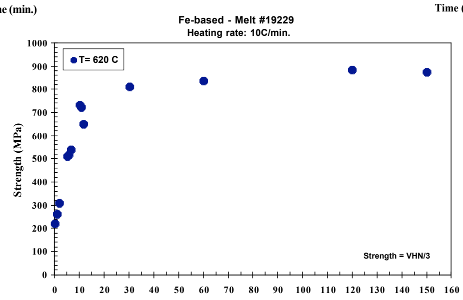




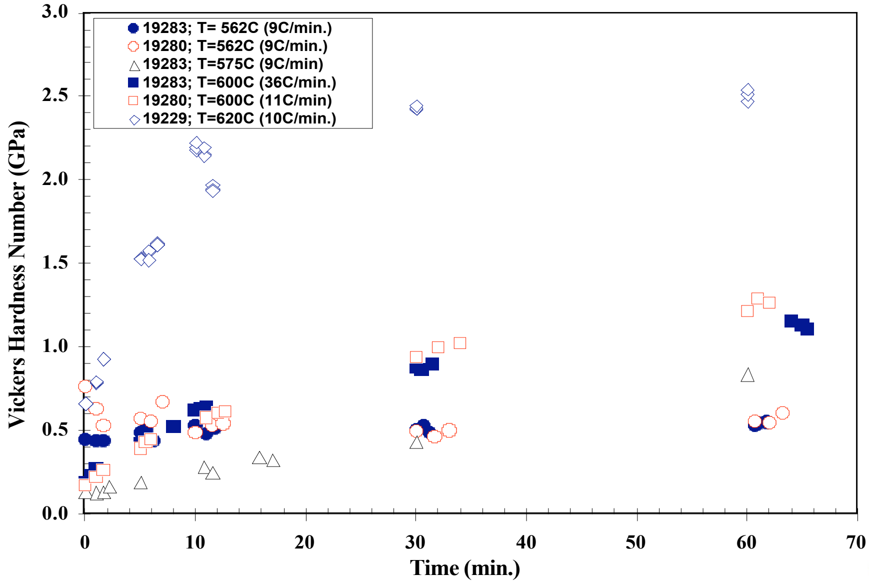
Micro-hardness Evolution vs Time at 620C- SAM1651 Ingots (Tg = 584C)



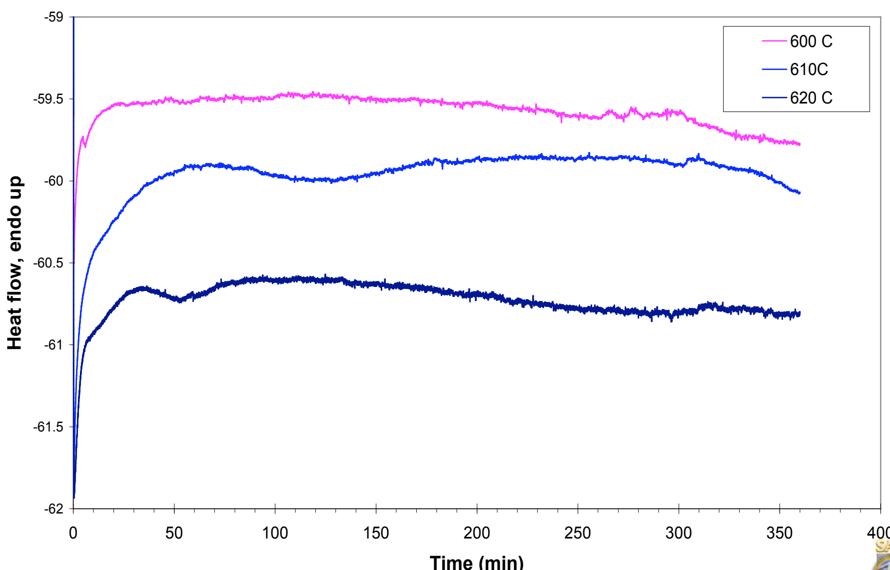




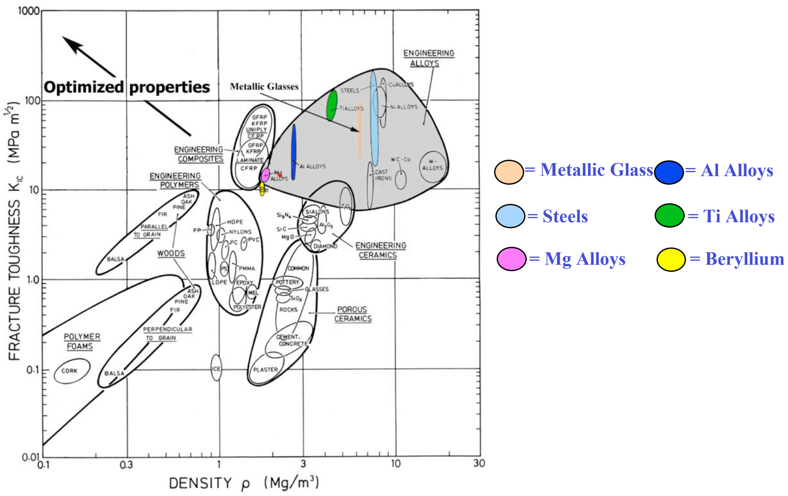
Summary: Hot Micro-hardness vs Time at Temp - SAM1651 Ingots (Tg = 584C)



Isothermal Annealing of SAM1651 (SAM7) Melt Spun Ribbons



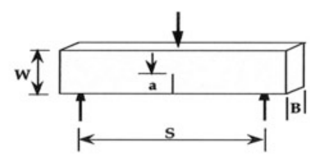
Summary of Toughness for Engineering Materials and Location of Bulk Metallic Glasses



Adapted from: Ashby, M. F. Materials Selection in Mechanical Design, Pergamon Press, 1992, pp. 28-36

Fracture Toughness Evaluation Methods for Bulk Metallic Glasses

Experimental Methods



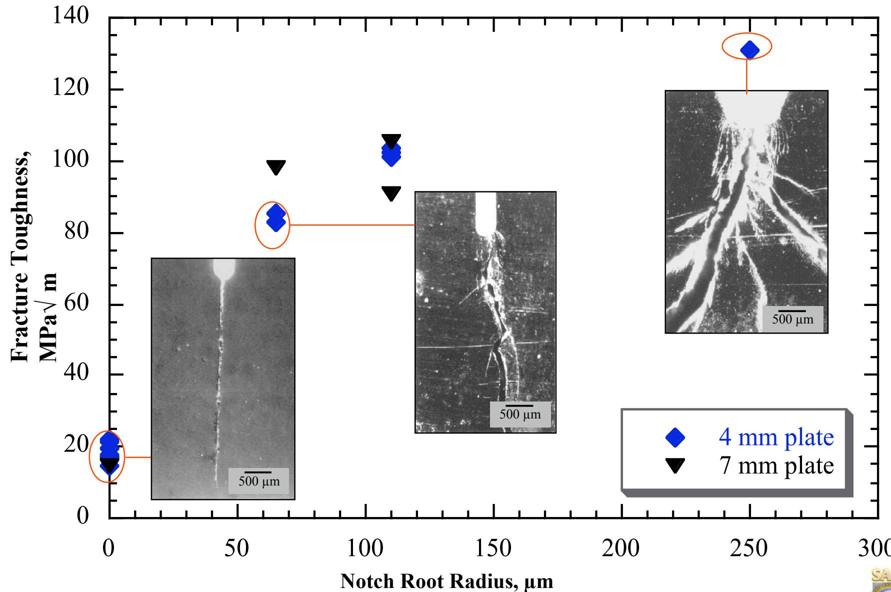
Fracture Toughness/Notched Toughness Tests
- ASTM E399-90
- fatigue precracked at 20 Hz
- U-shape notch (ρ = 65 and 110 μm)
- V-shape notch (ρ = 250 μm)
- Const. Displacement rate of 100 μm/min

a/W = 0.3-0.5
W = 8 or 12 mm
B = 4 mm
S = 24 to 48 mm

$$K_Q = \frac{P_Q S}{B W^{3/2}} \cdot f\left(\frac{a}{W}\right)$$

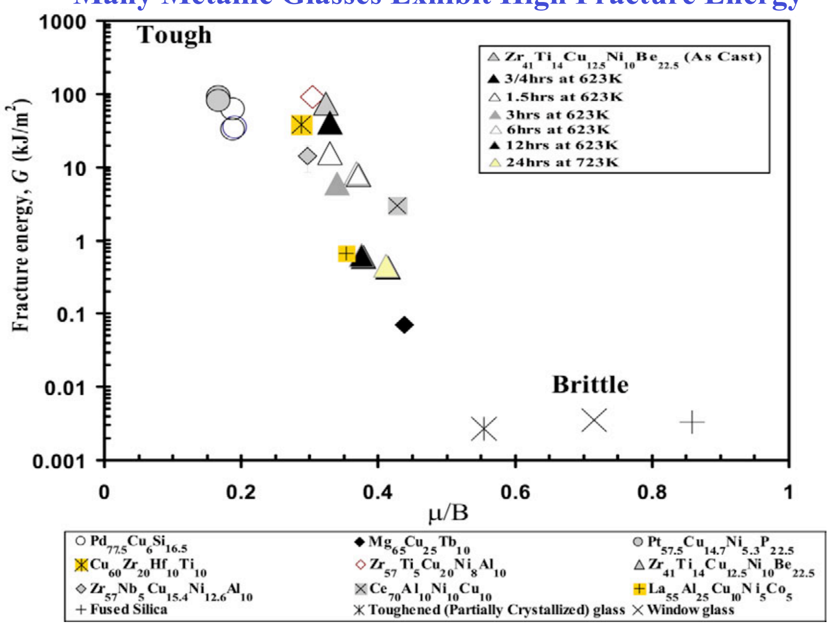
$$f\left(\frac{a}{W}\right) = \frac{3(a/W)^{1/2}\left[1.99 - (a/W)(1 - a/W)(2.15 - 3.93a/W + 2.7a^2/W^2)\right]}{2(1 + 2a/W)(1 - a/W)^{3/2}}$$

High Toughness Exhibited by Zr-based Bulk Metallic Glass



Reference: Lowhaphandu, P. and Lewandowski, J. Scripta Mater., 1998, 38, pp. 1811-1816.

Many Metallic Glasses Exhibit High Fracture Energy



Reference: Lewandowski, J., Shazly, M., Shamimi Nouri, A., Scripta Mater., 2006, 54(3), pp. 337-342.

Dynamic Yield, Flow and Fracture
- Relevant to Impact/Ballistic Loading

- Servo-hydraulic Machines– 10^{-3} s^{-1} to 10 s^{-1}
- Deformation Simulator Machine- 10^{-1} s^{-1} to 10^{2} s^{-1}
- Split Hopkinson Pressure Bars– 500 s^{-1} to 10^{4} s^{-1}
- High Speed Particle Impact
- Single Stage Gas Gun (plate impact experiments)

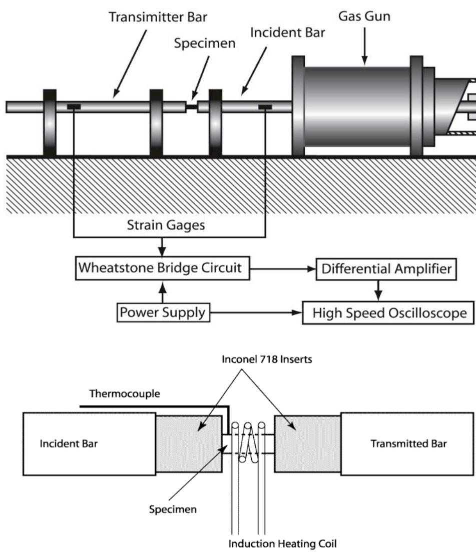
  strain rate >10^{4} s^{-1}

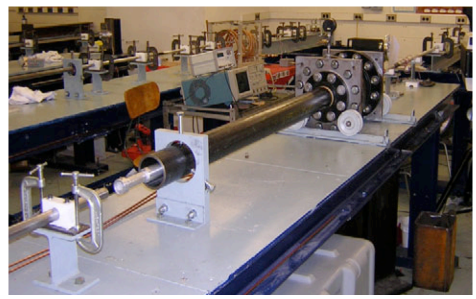
Test Temperature Range: -100°C to 1000°C

Advanced Deformation Simulator

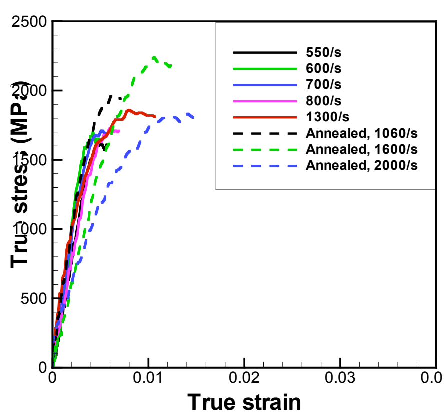
- High load capacity (110 kip, 220 kip)
- Ability to test large specimens (e.g 5” diam.)
- Intermediate Loading rates (0.1 - 120”/sec)
  - > Servo-hydraulic rates
  - < SHPB
- Strain Rates (0.1 - 500/sec)
- Single and Multiple-hit capability

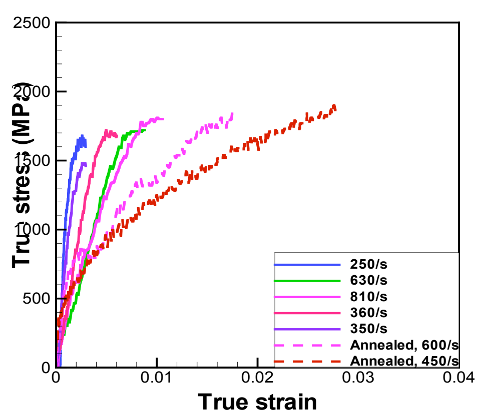
Split Hopkinson Tension Bar for High Strain Rate Testing





Effects of L/D and Annealing on Zr-Glass Tested in Compression



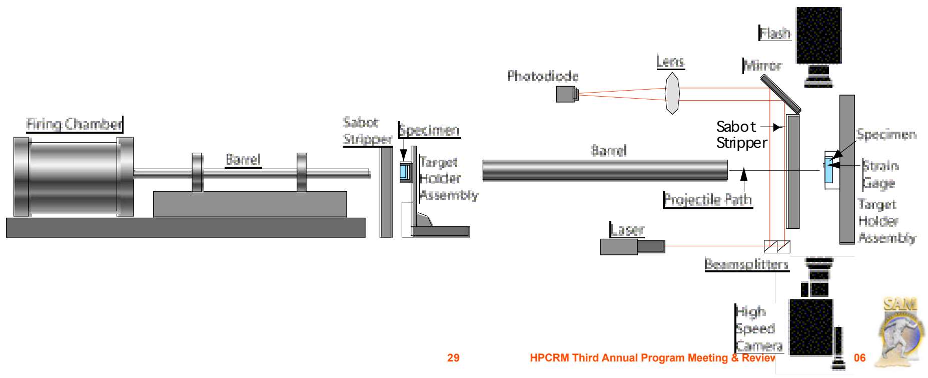


- L/D = 1.0, As-Cast
  - Peak stress: 1600-1800 MPa
  - Failure strain: 0.5-1.0%
- L/D = 1.0, annealed
  - Peak stress: 1800-2200 MPa
  - Failure strain: 1.0-2.0%
- Strain rates: 500-2000/s

- L/D = 2.0, As-Cast
  - Peak stress: 1500-1800 MPa
  - Failure strain: 0.2-1.0%
- L/D = 2.0, annealed
  - Peak stress: 1800-1900 MPa
  - Failure strain: 1.5-3.0%
- Strain rates: 200-1000/s

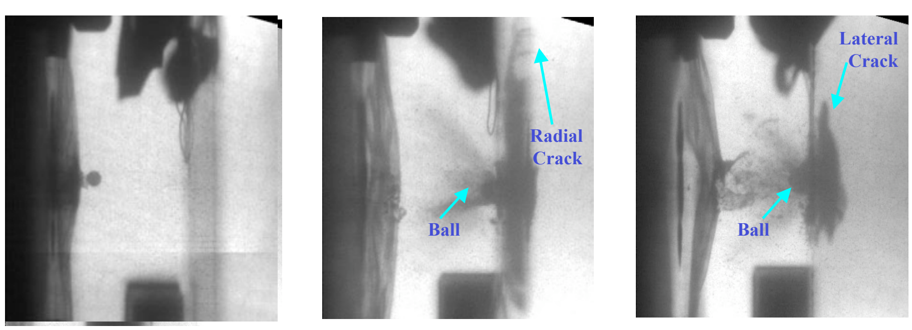
High Speed Particle Impact

- Compressed air gas gun with 0.75”Ø by 4.5’ long barrel
- Hardened chrome steel ball bearing 1/16”Ø on 1.5” long nylon sabot
- Impact measured by:
  - DRS Hadland IMACON 200 high speed camera
    - 2.5-6 μs interframe time
  - Vishay Micromeasurements group stacked rosette strain gages



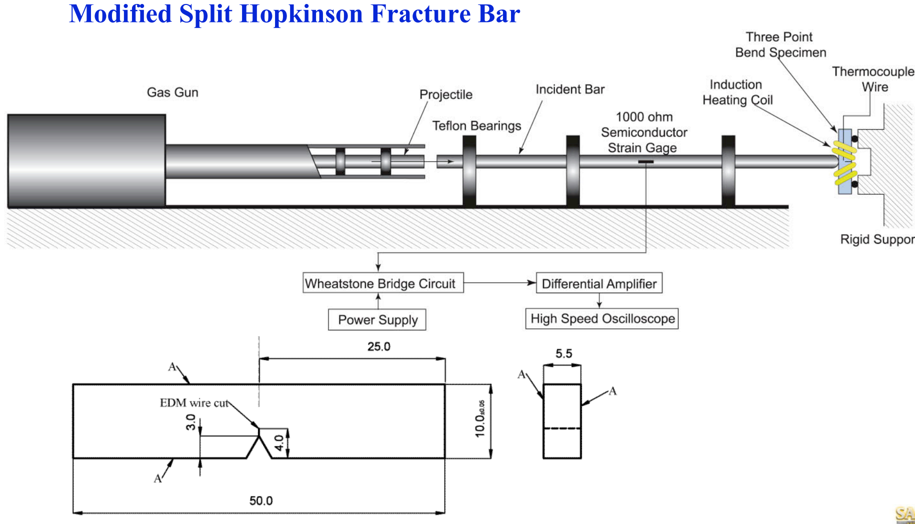
Cracking Patterns in Ceramic During High Speed Particle Impact

- Crushed zones seen beneath impact site in all cases
- Surface chipping seen on impact face
- Four types of internal cracking observed:
  - Conical, radial, lateral, splinter



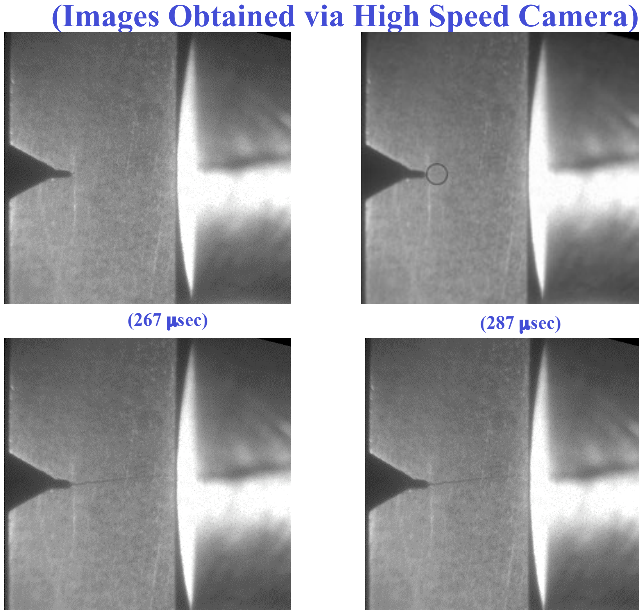
Elevated Temperature Dynamic Fracture Toughness Test

Modified Split Hopkinson Fracture Bar



All Dimensions in mm
"A" surfaces should be perpendicular and parallel as appicable within 0.01 mm TIR

High Strain Rate Fracture Toughness Testing of Metals (Images Obtained via High Speed Camera)



(267 μsec) (287 μsec)

(307 μsec) (327 μsec)

General Mechanical Behavior Issues for Coated Samples

- 'Free-film' vs bulk behavior of glass
  - Coating properties likely different than bulk metallic glass

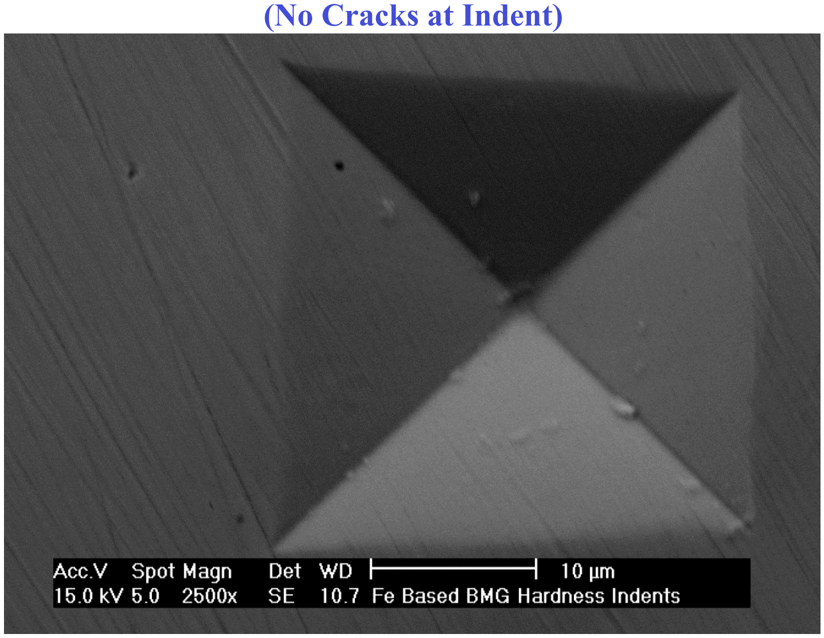
- Deformation/fracture of coating on substrate
  - Substrate may affect coating behavior

- Hardness/Micro-hardness
  - Crack vs No Crack, Crack Length Anisotropy
  - Calculation of Indentation Toughness
  - Shear Bands vs No Shear Bands
  - Indent Shape Effects
  - Diamond Pyramid vs Knoop
  - Temperature Effects (Hot Hardness)

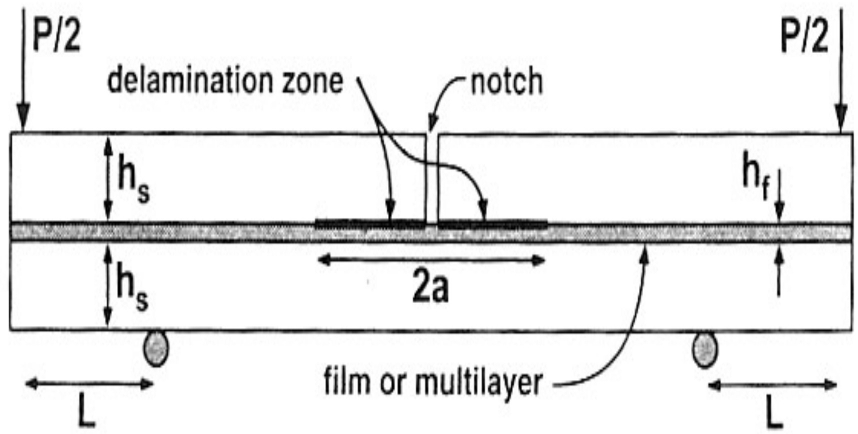
- Effects of edges/interfaces on fracture
  - Bond strength/interface toughness

- Residual stress effects
  - Effects of Processing Conditions
  - Effects of Coating Thickness

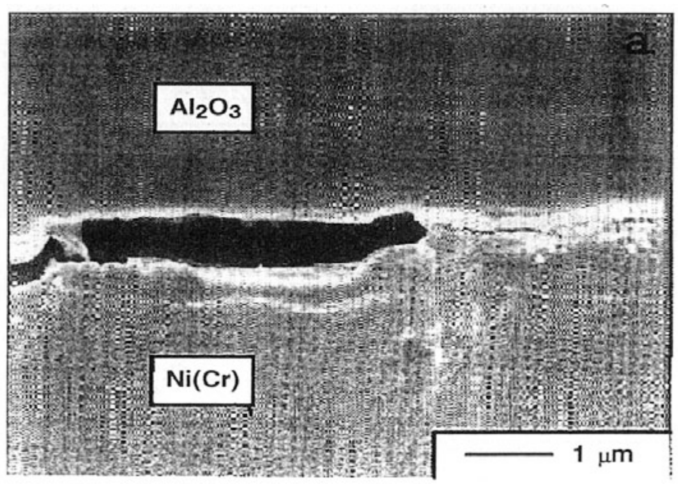
SAM1651 Micro-hardness Indentation (No Cracks at Indent)



Four-point Flexure Specimen for Interface Toughness Tests



Plasticity at Interface Increases Interface Toughness



Blunting of a crack along an interface between Ni and Al_2O_3 which is toughened by an order of magnitude by the addition of 20 at.% Cr. Plastic flow in the metal causes the interface crack to blunt. Reproduced with permission from Gaudette et al. (1997)

Effects of Edges/interfaces on Fracture

- Hardness Indentation at Interface
  - Crack vs No Crack Along Interface
  - Length of Crack
  - Indentation Shape (Diamond vs Knoop)
- Industry Screening Tests (TBC's, other)
- Interface Fracture Toughness
  - Bend Test
  - In-Situ Fracture test in SEM

Summary

- Strength of Amorphous Metals
  - Comparison to Other Materials
- Processing Background
  - Critical Cooling Rate
  - Effects on Critical Casting Thickness
    - Bulk Metallic Glasses
    - Amorphous Powders/Ribbons
- Hardness/Strength as f(Temperature)
  - Deformation Mechanism Map
  - Examples of Various Metallic Glass Systems
    - Hardness vs Temperature
    - Hardness Evolution vs Time at Temperature
      - Relevance to Transformation Studies
      - Relevance to Processing
  - Comparison to 316L Stainless Steel
- Toughness
  - Examples of Metallic Glasses
    - Effects of Notch Radius
    - Fracture Energy (G)
- Loading Rate Effects
  - High Strain Rate Testing
    - Relevance to Impact Loading
    - Examples of Metallic Glasses
      - Use of High Speed Video
      - Effects of Length/Diameter

Future Work – Bulk SAM2X5 vs. SAM1651 Amorphous Metal

- Hardness vs. Temperature
  - Comparison to 316L and C-22
- Hardness Evolution vs Time At Temperature
- Link To Microstructure Evolution Studies
- Link To Processing Studies
  - Structure Evolution During Spray Deposition
    - Wire Extrusion for Spray Deposition
- Toughness
  - Notch Toughness and Fatigue Precracked Fracture Toughness
  - Comparison to Other Coating Materials
  - Effects of Annealing on Toughness
  - Effects of Changes in Test Temperature Relevant to Government Applications
- Effects Of Changes In Loading Rate
  - High Strain Rate Testing (e.g. > 1000/sec)
  - High Speed Video (200,000 frames/sec) of Deformation/Fracture Events
  - Effects of Annealing on Behavior

Future Work – Bulk SAM2X5 vs. SAM1651 Amorphous Metal

- Environmental Effects
  - Environmental Effects on Cracking
  - Relevant Environments

Future Work – HVOF SAM2X5 & SAM1651 Coatings

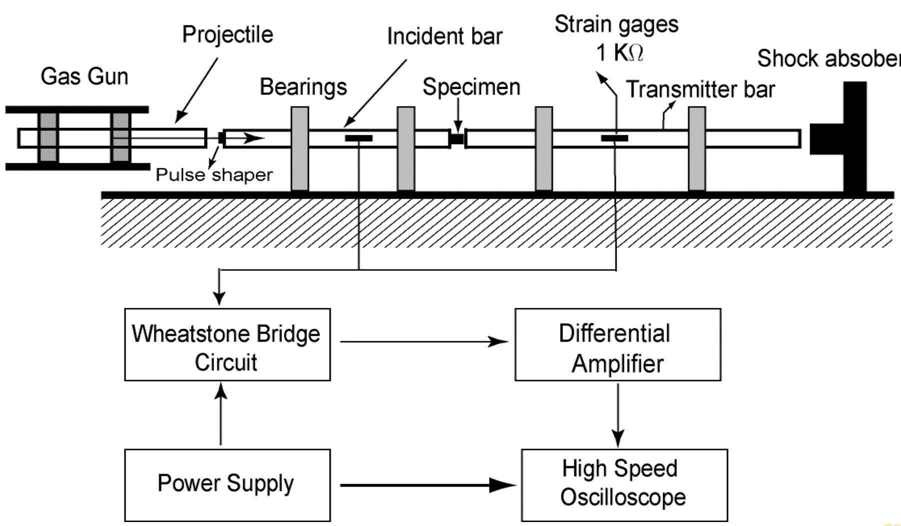
- Effects of Changes In Loading Rate
  - High Strain Rate Testing (e.g. > 1000/sec)
  - High Speed Video (200,000 frames/sec) of Deformation/Fracture Events
  - Effects of Annealing on Behavior
- Coated Samples (SAM 2X5, SAM 1651)
  - Interface Characterization
  - Effects of Surface Preparation
  - Reaction Products
  - SEM/EDS/TEM
- Evaluation of Coating Materials (SAM2X5 & SAM 1651)
  - 'Free film' properties may be different than bulk metallic glass
- Interface Strength/Toughness Tests
  - Effects of Surface Preparation
  - Effects of Reaction Products
  - Locus of Cracking
    - In-situ Testing
    - SEM/EDS/AES (Auger Electron Spectroscopy) of Fracture Path

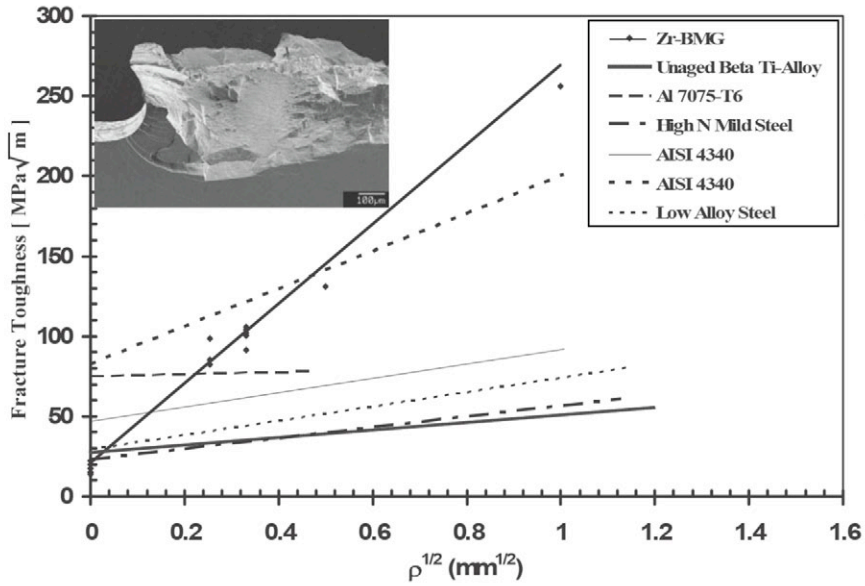
Future Work – HVOF SAM2X5 & SAM1651 Coatings

- Effects of Loading Rate
- Effects of Annealing
- Environmental Effects

Backup Slides

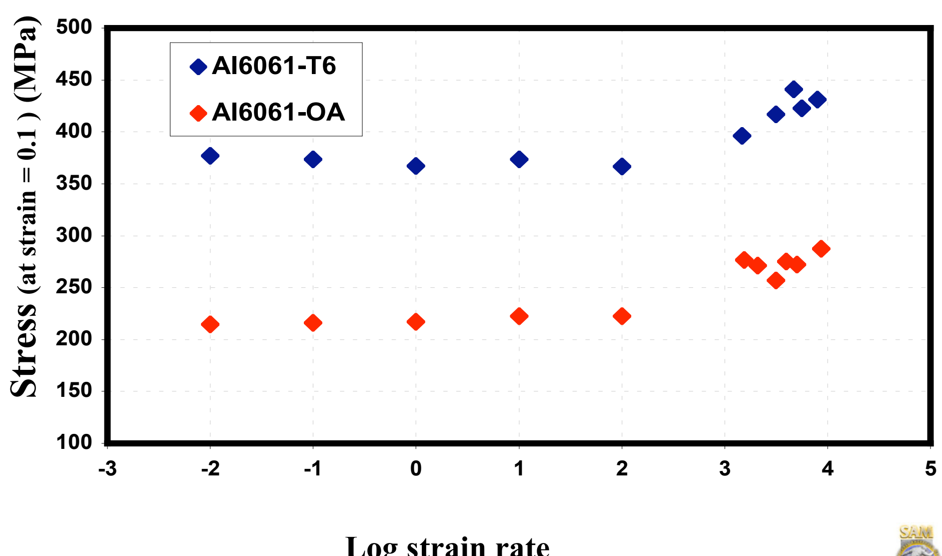
Split Hopkinson Compression Bar for Testing at High Strain Rate



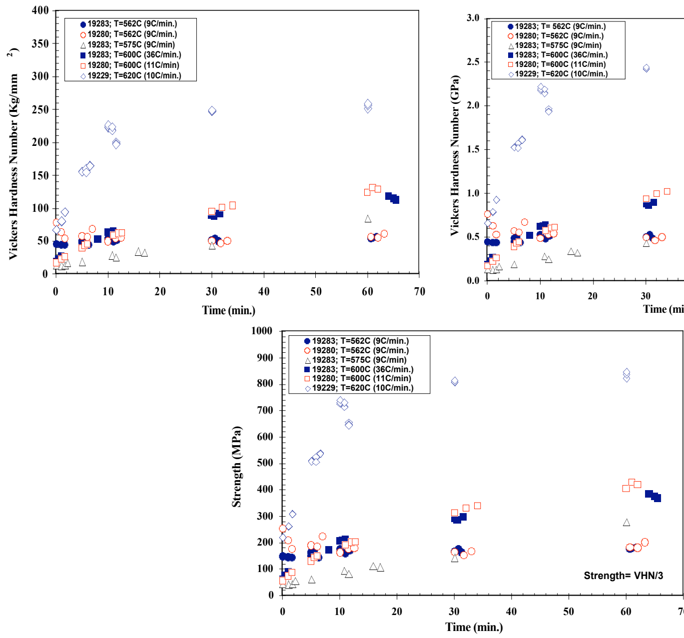


Reference: Lewandowski, J., Shazly, M., Shamimi Nouri, A., Scripta Mater., 2006, 54(3), pp. 337-342.

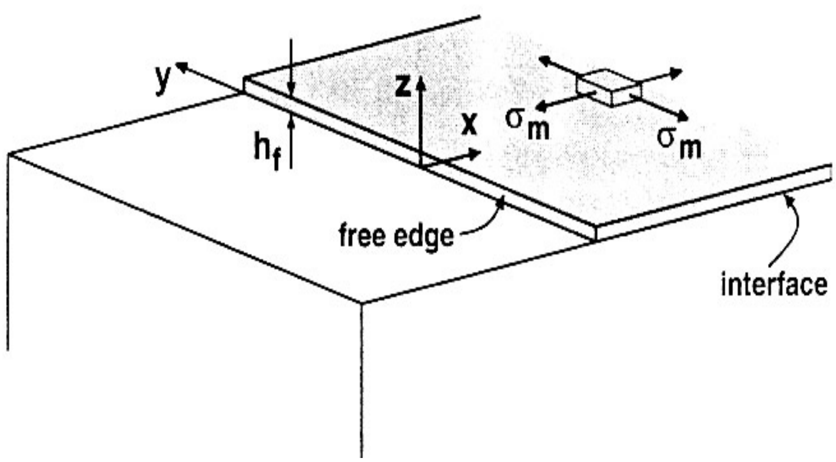
Effects of Changes in Strain Rate on Strength of 6061 Al Alloy



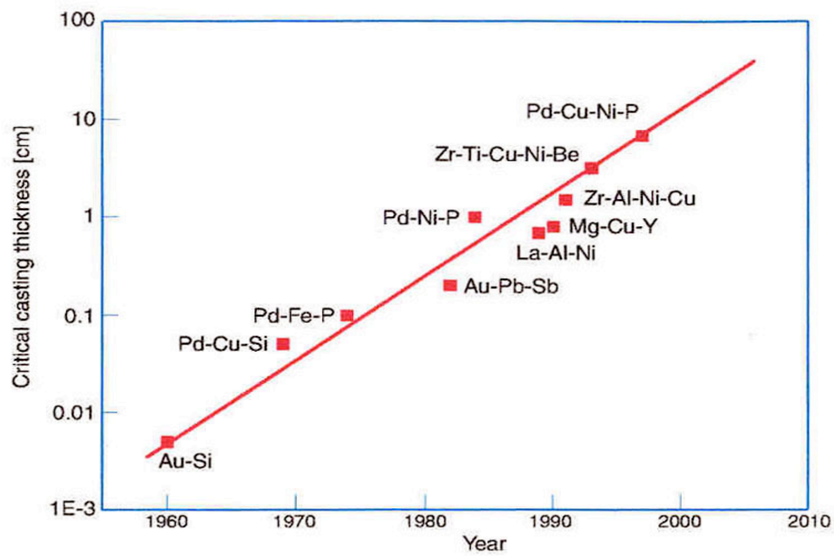
Hot Micro-hardness vs Time at Temp - SAM1651 Ingots



Effects of Edges/interfaces on Fracture

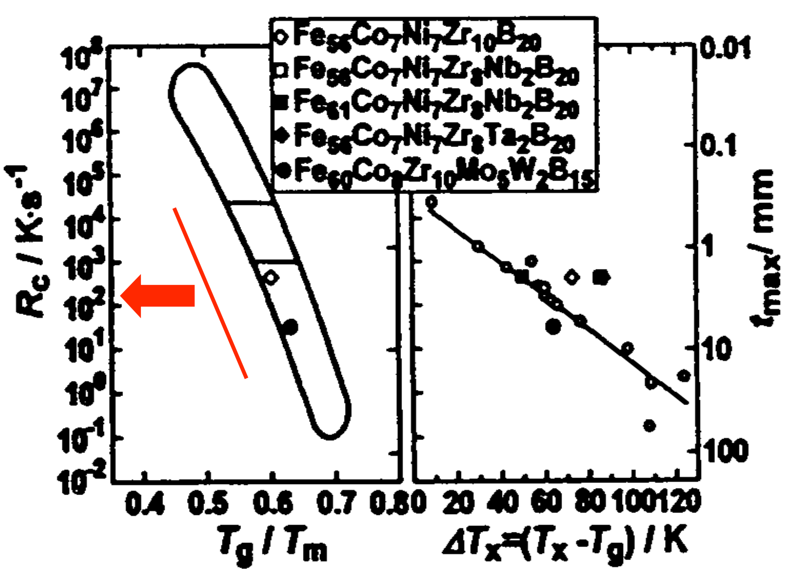


Schematic diagram of a thin film with a free edge bonded to a thick substrate. The equi-biaxial stress in the film is σ_m at points far from the film edge compared to h_f. The planar edge of the film x = 0 is traction-free.

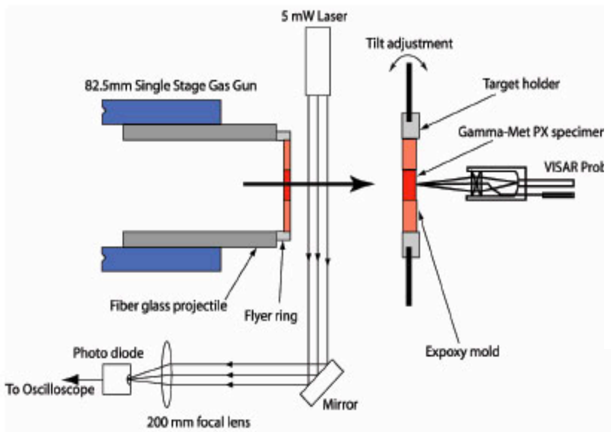


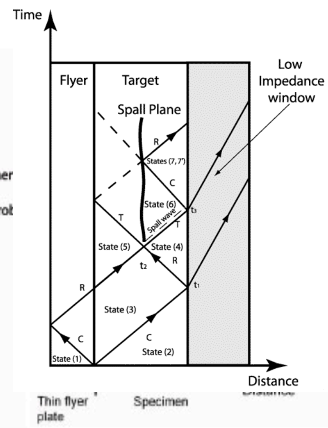
Reference: Telford, M., The Case for Bulk Metallic Glass, Materials Today, March 2004, 36-43.

Critical Cooling Rate vs. Casting Thickness and Reduced Glass Transition Temperature for Fe-based Glasses

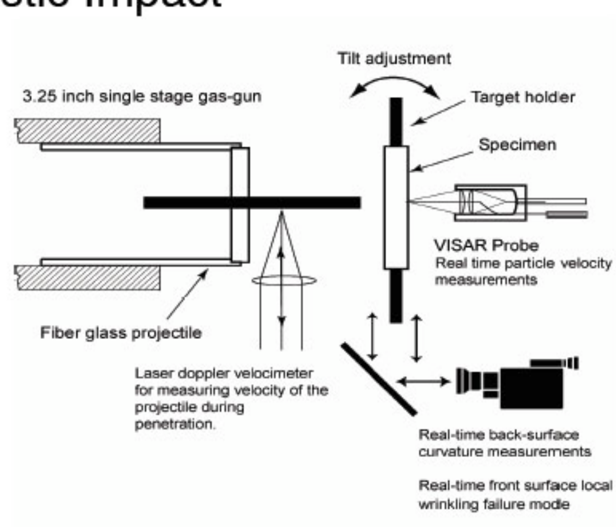


Dynamic Spall Strength





Ballistic Impact



High-Performance Corrosion-Resistant Materials: Overview of Tasks & Deliverables for FY06 – FY08

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Jay Boudreau
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John Rivard
Lou Aprigliano

TNC
Dan Branagan
SAM2X5 Powder
SAM2X5 Coatings

ORNL
Craig Blue
SAM1651 Powder
Drop Cast Ingots

CAT
Brad Beardsley
SAM2X5 Coatings
SAM1651 Coatings

NRL
Bob Bayles
Surface Ship
Applications

NRL
Bob Brown
Submarine
Applications

SNL CA
Nancy Yang
Microstructure
Characterization

UCD
Enrique Lavernia
HVOF Optimization
Atomization

UNR
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Nanometer -Scale
Ceramic Additives

BAH
Jon Kirkwood
DOE Applications
Economic Analysis

LLNL/ORNL/CSM
Frank Wong et al.
TBM & Alpine Pick
Applications

WISCONSIN
John Perepezko
Thermal Analysis
Phase Stability

CALPHAD
Larry Kaufman
Thermochemical
Modeling

CASE -WESTERN
John Lewandowski
Mechanical
Properties

LLNL
Joe Farmer
Corrosion Testing
Gamma Effects

NRL
Bob Bayles
Corrosion Testing
Microbial Effects

CASE-WESTERN
Joe Payer
Corrosion Thrust
Area Leader

BERKELEY
Tom Devine
Passive Film Study
SERS & SAW

LLNL
Jor-Shan Choi
Criticality Control
Neutron Effects

MNRC/UCD
Robert Flocchini
Moe Boussoufi
Ben Liu
Irradiation
Attenuation
Radiography

Program Management & Completion of Non-Q Testing

Activity		Responsibility
1	Program Management	Program Management
1.1	Technical Direction	DOE Walker / DARPA Christo / LLNL Farmer / CASE Payer
1.3	Administration	LLNL Farmer & Christman
1.4	Data Analysis & Interpretation	LLNL Farmer et al.
1.5	Optimization Studies - Materials & Structures	LLNL Farmer et al.
1.6	Formal Documentation	LLNL Farmer et al.
1.7	Formal Program Reviews	LLNL Farmer et al.
1.8	Quality Assurance	LLNL Farmer & Barish
1.9	Budget & Schedule	LLNL Farmer & Tippens
1.10	Proposal, Negotiaion, & Receipt of FY06 Funding & Opening Account Numbers	DOE Walker / DARPA Christo / LLNL Farmer / CASE Payer
1.11	Proposal, Negotiation, & Receipt of FY07 Funding & Opening Account Numbers	DOE Walker / DARPA Christo / LLNL Farmer / CASE Payer
1.12	Proposal, Negotiaion, & Receipt of FY08Funding & Opening Account Numbers	DOE Walker / DARPA Christo / LLNL Farmer / CASE Payer
1.13	Award FY06 Contracts	DOE Walker / DARPA Christo / LLNL Farmer / CASE Payer
1.14	Award FY07 Contracts	DOE Walker / DARPA Christo / LLNL Farmer / CASE Payer
1.15	Award FY08 Contracts	DOE Walker / DARPA Christo / LLNL Farmer / CASE Payer
	(Non-Q) Data Generation for Scientific Understanding & Materials Selection	*Complete On-Going Non-Q Testing*
2	(Non-Q) Corrosion Resistance - General & Localized	(Non-Q) Corrosion Resistance - General & Localized
2.1	Specify Relevant Environments - Seawater, Mixed Salts/Brines & Boric Acid	LLNL Carroll, Woolery & Gdowski
2.2	Linear Polarization (LP) - After 24-Hour Hold - Oxygenated Solution	LLNL Farmer, Rebak, Lian & Day
2.3	Cyclic Polarization (CP) - After 24-Hour Hold - Oxygenated Solution	LLNL Farmer, Rebak, Lian & Day
2.4	Long-Term Open Circuit Corrosion Potential (OCP)	LLNL Farmer, Rebak, Lian & Day
2.5	Linear Polarization (LP) - After Long-Term OCP Measurements	LLNL Farmer, Rebak, Lian & Day
2.6	Cyclic Polarization (CP) - After Long-Term OCP Measurements	LLNL Farmer, Rebak, Lian & Day
2.7	Long-Term Weight Loss Samples (WL)	LLNL Farmer, Rebak, Lian & Day
2.8	Long-Term Crevice Corrosion Samples (CC)	LLNL Farmer, Rebak, Lian & Day
2.9	Long-Term Drip-Testing of Hot Inclined Plates (DT)	LLNL Farmer, Rebak, Lian & Day
2.10	Extended-Duration Potential Step (PS) - Breakdown Potential	LLNL Farmer, Rebak, Lian & Day
2.11	Extended-Duration Potential Step (PS) - Repassivation Potential	LLNL Farmer, Rebak, Lian & Day
2.12	Stifling of Active Crevice Corrosion	LLNL Farmer, Rebak, Lian & Day / CASE Payer
2.13	Galvanic Coupling with Other Engineering Alloys	LLNL Farmer, Rebak, Lian & Day / CASE Payer
2.14	Cathodic Hydrogen Charging in Sulfuric Acid with Sodium Arsenate	LLNL Farmer, Rebak, Lian & Day / CASE Payer
2.15	Depth Profiling with Time of Flight Secondary Ion Mass Spectrometry (TOF/SIMS)	LLNL Farmer, Rebak, Lian & Day / CASE Payer
2.16	Thermal Gravimetric Analysis (TGA) - Dust Deliquescence	LLNL Gdowski, Woolery & Carroll
2.17	Documentation & Publication of Results from Team	LLNL Farmer / CASE Payer

Program Management & Completion of Non-Q Testing

3	(Non-Q) Corrosion Resistance - Passive Film Stability & Oxygen Reduction Kinetics	(Non-Q) Corrosion Resistance - Passive Film Stability
3.1	Pourbaix Diagram Calculation	CALPHAD Kaufman / LLNL Turchi
3.2	Quartz Microbalance Studies with Potential-Controlled Samples	UCB Tom Devine
3.3	Effects of Extreme Environments - In Situ Optical Studies in Autoclave	UCB Tom Devine
3.4	In Situ Surface Enhanced Raman Spectroscopy (SERS)	UCB Tom Devine
3.5	In Situ Ordinary & Resonant Raman Spectroscopy	UCB Tom Devine
3.6	Oxygen Reduction Kinetics Study with CP - Exchange Current Density & Tafel Slopes	CASE Payer / LLNL Farmer et al.
3.7	Electrochemical Impeadance Spectroscopy (EIS) - Capacitance vs. Potential	LLNL Farmer et al. / CASE Payer
3.8	X-Ray Fluorescence Spectroscopy (XPS) - Oxidation State Changes	LLNL Farmer et al. / CASE Payer
3.9	Auger Electron Spectroscopy (AES) - Composition Profile	LLNL Farmer et al. / CASE Payer
3.10	Time of Flight Secondary Ion Mass Spectrometry (TOF/SIMS) - Composition Profile	LLNL Farmer et al. / CASE Payer
3.11	Transmission Electron Microscopy (TEM) - Cross Section	CASE Payer / SNL Yang / LLNL Farmer et al.
3.12	Atomic Force Microscopy with Electrochemical Stage (AFM/ES) - Plan View	CASE Payer / LLNL Farmer et al. / SNL Yang
4	(Non-Q) Environmental Cracking	(Non-Q) Environmental Cracking
4.1	Slow Strain Rate Testing of Cylindrical Dog Bones (SCC)	CASE Lewandowski & Payer / LLNL Rebak & Lian / NRL Bayles
4.2	Constant Load Testing & Time-To-Failure (SCC)	CASE Lewandowski & Payer / LLNL Rebak & Lian / NRL Bayles
4.3	Determination of Threshold Stress & Stress Intensity Factor (SCC)	CASE Lewandowski & Payer / LLNL Rebak & Lian / NRL Bayles
4.4	Slow Strain Rate Testing of Cylindrical Dog Bone Test	CASE Lewandowski & Payer / LLNL Rebak & Lian / NRL Bayles
4.5	Constant Load Testing & Time-To-Failure	CASE Lewandowski & Payer / LLNL Rebak & Lian / NRL Bayles
4.6	Determination of Threshold Stress & Stress Intensity Factor	CASE Lewandowski & Payer / LLNL Rebak & Lian / NRL Bayles
5	(Non-Q) Microbial Influenced Corrosion	(Non-Q) Microbial Influenced Corrosion
5.1	Microbe Identification & Formulation of Environment	NRL Bayles & Little / LLNL Lian & Horn
5.2	Long-Term Immersion Testing in Identified Environment	NRL Bayles & Little / LLNL Lian & Horn
6	(Non-Q) Radiation Effects	(Non-Q) Radiation Effects
6.1	Gamma Radiolysis Effects on Electrolyte Chemistry	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
6.2	Gamma Radiolysis Effects on Mixed Potential	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
6.3	Gamma Radiolysis Effects on General Corrosion	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
6.4	Gamma Radiolysis Effects on Localized Corrosion	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
6.5	Gamma Radiolysis Effects on Stress Corrosion Cracking	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
6.6	Gamma Radiolysis Effects on Hydrogen Induced Cracking	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
6.7	Neutron Radiation Effects on Void Swelling	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
6.8	Neutron Radiation Effects on Mechanical Property Degradation	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
6.9	Neutron Radiation Effects on Mixed Potential	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
6.10	Neutron Radiation Effects on General Corrosion	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
6.11	Neutron Radiation Effects on Localized Corrosion	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
6.12	Neutron Radiation Effects on Stress Corrosion Cracking	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
6.13	Neutron Radiation Effects on Hydrogen Induced Cracking	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
6.14	Neutron Capture Cross Section Determination	LLNL Farmer, Choi & Upadhye - McClellon Reactor

Program Management & Completion of Non-Q Testing

7	(Non-Q) Differential Thermal Analysis (DSC, DTA)	(Non-Q) Thermal Stability
7.1	Differential Thermal Analysis (DSC, DTA)	UWM Perepezko & Hildal
7.2	Wedge Casting	UWM Perepezko & Hildal
7.3	Effects on Corrosion	LLNL Farmer, Rebak, Lian & Day; SNL Yang
7.4	Effects on Mechanical Properties	UWM Perepezko & Hildal; SNL Yang; Case Lewandowski
7.5	Data Analysis & Interpretation	UWM Perepezko & Hildal; Others
8	(Non-Q) Initial Mechanical Testing	(Non-Q) Mechanical Testing
8.1	Hot Hardness Testing - In Vacuum	CASE Lewandowski
8.2	Slow Strain Rate Testing of Cylindrical Dog Bone Samples in Air	Case Lewandowski
8.3	Creep Test Under Constant Stress - In Air	Case Lewandowski
8.4	Split Hopkinson Bar Test	Case Lewandowski
8.5	Impact Testing with Drop Tower	LLNL Farmer et al.
8.6	Data Analysis & Interpretation	CASE Lewandowski
8.7	Technical Project Review & Report Lab Scale Test of SAM Coatings	CASE Lewandoski
	Go / No-Go Material Performance, Economic Analysis, and Proposed Application: Data Approved by RW-1	

Further Optimization of Industrial Atomization & Thermal Spray

9	(Non-Q) Optimized Process for Powder Production	Powder Optimization
9.1	Optimization of SAM2X5 Atomization Process	TNC Branagan
9.2	Produce Lots of SAM2X5 for CAT / INL / UCD	TNC Branagan
9.3	Fractionation of SAM2X5 Powder / Standard Cut (-53+20 mm / mean 35 mm)	TNC Branagan
9.4	Fractionation of SAM2X5 Powder / Kerosene Torches (-45+15 mm / mean 30 mm)	TNC Branagan
9.5	Fractionation of SAM2X5 Powder / DJ Torches (-45+10 mm / mean 30 mm)	TNC Branagan
9.6	Fractionation of SAM2X5 Powder / Both Processes (-36+15 mm / mean 25 mm)	TNC Branagan
9.7	Yield, Morphology, Composition & Crystallinity for Various Size Fractions of SAM2X5 Powder	TNC Branagan / SNL Yang
9.8	Pourabilty Index - Ease of Pneumatic Conveyance	INL Swank / UCD Lavernia / CAT Beardsley
9.9	Demonstrate Recycle Unacceptable SAM2X5 Powder	TNC Branagan
9.10	Develop Viable Atomization Process for SAM1651 Powder - Production of Spherical Powder	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
9.11	Produce Lots of SAM1651 for CAT / INL / UCD	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
9.12	Fractionation of SAM1651 Powder / Standard Cut (-53+20 mm / mean 35 mm)	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
9.13	Fractionation of SAM1651 Powder / Kerosene Torches (-45+15 mm / mean 30 mm)	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
9.14	Fractionation of SAM1651 Powder / DJ Torches (-45+10 mm / mean 30 mm)	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
9.15	Fractionation of SAM1651 Powder / Both Processes (-36+15 mm / mean 25 mm)	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
9.16	Yield, Morphology, Composition, Crystallinity - Various Size Fractions of SAM1651 Powder	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
9.17	Pourabilty Index - Ease of Pneumatic Conveyance	INL Swank / UCD Lavernia / CAT Beardsley
9.18	Demonstrate Recycle Unacceptable SAM1651 Powder	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
10	(Non-Q) Optimized Process for Substrate Coating - Robust Process	Coating Optimization - Robust Process
10.1	Coating Process Effects on Substrates	CAT Beardsley
10.2	Acquire/Manufacture Required Substrates	CAT Beardsley
10.3	Develop Robust Surface Preparation Process - Grit Blasting	CAT Beardsley
10.4	Develop Robust Surface Preparation Process - PROTAL™ Processing	CAT Beardsley
10.5	Develop Robust Thermal Spray (TS) Process for SAM2X5 - Tafa JP	CAT Beardsley
10.6	Develop Robust Thermal Spray (TS) Process for SAM2X5 - Sulzer Metco DJ	CAT Beardsley
10.7	Develop Robust Thermal Spray (TS) Process for SAM2X5 - GTV K2	CAT Beardsley
10.8	Develop Robust Thermal Spray (TS) Process for SAM2X5 - SM WOKA Star 600	CAT Beardsley
10.9	Develop Robust Thermal Spray (TS) Process for SAM 2X5 - JetKote ™	CAT Beardsley
10.10	Develop Robust Thermal Spray (TS) Process for SAM1651 - Specific Technology TBD	CAT Beardsley / SNL Yang
10.11	Develop Robust Thermal Spray (TS) Process for SAM2X5/1651 - Process Interruptions	CAT Beardsley / SNL Yang
10.12	Develop Robust Thermal Spray (TS) Process for SAM2X5/1651 - Coating Properties	CATBeardsley/SNLYang/LLNLFarmer/CASELewandowski
10.14	Reporting by Caterpillar	CAT Beardsley
10.15	Equipment Acquisitions - Thermal Spray Systems	CAT Beardsley
10.16	Equipment Acquisitions - Laser for Surface Preparation	CAT Beardsley

Industrial Partners Attain ‘QSL’ Status & Produce ‘Q’ Materials

11	TNC on Qualified Supplier List (QSL)	Qualification of Powder Supplier
11.1	Telecon Briefing	LLNL Farmer & Barish
11.2	Preparation of Written Materials	LLNL Farmer & Barish
11.3	Site Visit & Briefing	LLNL Farmer & Barish
11.4	Personnel Training	LLNL Farmer & Barish
11.5	Preparation of Procedures	LLNL Farmer & Barish
11.6	Implementation of Procedures	LLNL Farmer & Barish
11.7	Traceable Calibrations	LLNL Farmer & Barish
11.8	OQA Audit	LLNL Farmer & Barish
11.9	Follow-up Audit	LLNL Farmer & Barish
11.10	Certification	LLNL Farmer & Barish
11.11	Initial Proof Production of Qualified Materials	LLNL Farmer & Barish
12	Caterpillar on Qualified Supplier List (QSL)	Qualification of Supplier
12.1	Telecon Briefing	LLNL Farmer & Barish
12.2	Preparation of Written Materials	LLNL Farmer & Barish
12.3	Site Visit & Briefing	LLNL Farmer & Barish
12.4	Personnel Training	LLNL Farmer & Barish
12.5	Preparation of Procedures	LLNL Farmer & Barish
12.6	Implementation of Procedures	LLNL Farmer & Barish
12.7	Traceable Calibrations	LLNL Farmer & Barish
12.8	OQA Audit	LLNL Farmer & Barish
12.9	Follow-up Audit	LLNL Farmer & Barish
12.10	Certification	LLNL Farmer & Barish
12.11	Initial Proof Production of Qualified Materials	LLNL Farmer & Barish
13	ORNL/Carpenter on Qualified Supplier List (QSL)	Qualification of Supplier
13.1	Telecon Briefing	LLNL Farmer & Barish
13.2	Preparation of Written Materials	LLNL Farmer & Barish
13.3	Site Visit & Briefing	LLNL Farmer & Barish
13.4	Personnel Training	LLNL Farmer & Barish
13.5	Preparation of Procedures	LLNL Farmer & Barish
13.6	Implementation of Procedures	LLNL Farmer & Barish
13.7	Traceable Calibrations	LLNL Farmer & Barish
13.8	OQA Audit	LLNL Farmer & Barish
13.9	Follow-up Audit	LLNL Farmer & Barish
13.10	Certification	LLNL Farmer & Barish
13.11	Initial Proof Production of Qualified Materials	LLNL Farmer & Barish
14	(Q) Powder from Vendor on QSL	Produce Qualified (Q) Powder
14.1	SAM2X5 Powder at TNC	TNC Branagan
14.2	SAM1651 Powder at TNC/UCD/ORNL/Carpenter	TNC Branagan / UCD Lavernia / ORNL Peters / SNL Yang
15	(Q) Coated Samples from Vendor on QSL	Produce Qualified (Q) Samples
15.1	SAM2X5 Samples from CAT / INL / UCD	CAT Beardsley / INL Swank / UCD Lavernia
15.2	SAM1651 Samples from CAT / INL / UCD	CAT Beardsley / INL Swank / UCD Lavernia
15.3	Electron Microscopy & X-Ray Diffraction	SNL Yang

Obtain Fully Qualified Data with ‘Q’ Materials & Samples

	(Q) Data Generation for Total System Performance Assessment	Testing of Qualified (Q) Samples
16	(Q) Corrosion Resistance - General & Localized	(Q) Corrosion Resistance - General & Localized
16.1	Specify Relevant Environments - Seawater, Mixed Salts/Brines & Boric Acid	LLNL Carroll, Woolery & Gdowski
16.2	Linear Polarization (LP) - After 24-Hour Hold - Oxygenated Solution	LLNL Farmer, Rebak, Lian & Day
16.3	Cyclic Polarization (CP) - After 24-Hour Hold - Oxygenated Solution	LLNL Farmer, Rebak, Lian & Day
16.4	Long-Term Open Circuit Corrosion Potential (OCP)	LLNL Farmer, Rebak, Lian & Day
16.5	Linear Polarization (LP) - After Long-Term OCP Measurements	LLNL Farmer, Rebak, Lian & Day
16.6	Cyclic Polarization (CP) - After Long-Term OCP Measurements	LLNL Farmer, Rebak, Lian & Day
16.7	Long-Term Weight Loss Samples (WL)	LLNL Farmer, Rebak, Lian & Day
16.8	Long-Term Crevice Corrosion Samples (CC)	LLNL Farmer, Rebak, Lian & Day
16.9	Long-Term Drip-Testing of Hot Inclined Plates (DT)	LLNL Farmer, Rebak, Lian & Day
16.10	Extended-Duration Potential Step (PS) - Breakdown Potential	LLNL Farmer, Rebak, Lian & Day
16.11	Extended-Duration Potential Step (PS) - Repassivation Potential	LLNL Farmer, Rebak, Lian & Day
16.12	Stifling of Active Crevice Corrosion	LLNL Farmer, Rebak, Lian & Day / CASE Payer
16.13	Galvanic Coupling with Other Engineering Alloys	LLNL Farmer, Rebak, Lian & Day / CASE Payer
16.14	Cathodic Hydrogen Charging in Sulfuric Acid with Sodium Arsenate	LLNL Farmer, Rebak, Lian & Day / CASE Payer
16.15	Depth Profiling with Time of Flight Secondary Ion Mass Spectrometry (TOF/SIMS)	LLNL Farmer, Rebak, Lian & Day / CASE Payer
16.16	Thermal Gravimetric Analysis (TGA) - Dust Deliquescence	LLNL Gdowski, Woolery & Carroll
16.17	Documentation & Publication of Results from Team	LLNL Farmer / CASE Payer
17	(Q) Corrosion Resistance - Passive Film Stability & Oxygen Reduction Kinetics	(Q) Corrosion Resistance - Passive Film Stability
17.1	Pourbaix Diagram Calculation	CALPHAD Kaufman / LLNL Turchi
17.2	Quartz Microbalance Studies with Potential-Controlled Samples	UCB Tom Devine
17.3	Effects of Extreme Environments - In Situ Optical Studies in Autoclave	UCB Tom Devine
17.4	In Situ Surface Enhanced Raman Spectroscopy (SERS)	UCB Tom Devine
17.5	In Situ Ordinary & Resonant Raman Spectroscopy	UCB Tom Devine
17.6	Oxygen Reduction Kinetics Study with CP - Exchange Current Density & Tafel Slopes	CASE Payer / LLNL Farmer et al.
17.7	Electrochemical Impeadance Spectroscopy (EIS) - Capacitance vs. Potential	LLNL Farmer et al. / CASE Payer
17.8	X-Ray Fluorescence Spectroscopy (XPS) - Oxidation State Changes	LLNL Farmer et al. / CASE Payer
17.9	Auger Electron Spectroscopy (AES) - Composition Profile	LLNL Farmer et al. / CASE Payer
17.10	Time of Flight Secondary Ion Mass Spectrometry (TOF/SIMS) - Composition Profile	LLNL Farmer et al. / CASE Payer
17.11	Transmission Electron Microscopy (TEM) - Cross Section	LLNL Farmer et al. / SNL Yang / CASE Payer
17.12	Atomic Force Microscopy with Electrochemical Stage (AFM/ES) - Plan View	LLNL Farmer et al. / SNL Yang / CASE Payer
18	(Q) Environmental Cracking	(Q) Environmental Cracking
18.1	Slow Strain Rate Testing of Cylindrical Dog Bones (SCC)	CASE Lewandowski / LLNL Rebak & Lian / NRL Bayles
18.2	Constant Load Testing & Time-To-Failure (SCC)	CASE Lewandowski / LLNL Rebak & Lian / NRL Bayles
18.3	Determination of Threshold Stress & Stress Intensity Factor (SCC)	CASE Lewandowski / LLNL Rebak & Lian / NRL Bayles
18.4	Slow Strain Rate Testing of Cylindrical Dog Bone Test	CASE Lewandowski / LLNL Rebak & Lian / NRL Bayles
18.5	Constant Load Testing & Time-To-Failure	CASE Lewandowski / LLNL Rebak & Lian / NRL Bayles
18.6	Determination of Threshold Stress & Stress Intensity Factor	CASE Lewandowski / LLNL Rebak & Lian / NRL Bayles
19	(Q) Microbial Influenced Corrosion	(Q) Microbial Influenced Corrosion
19.1	Microbe Identification & Formulation of Environment	NRL Bayles & Little / LLNL Lian & Horn
19.2	Long-Term Immersion Testing in Identified Environment	NRL Bayles & Little / LLNL Lian & Horn

Obtain Fully Qualified Data with 'Q' Materials & Samples

20	(Q) Radiation Effects	(Q) Radiation Effects
20.1	Gamma Radiolysis Effects on Electrolyte Chemistry	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
20.2	Gamma Radiolysis Effects on Mixed Potential	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
20.3	Gamma Radiolysis Effects on General Corrosion	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
20.4	Gamma Radiolysis Effects on Localized Corrosion	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
20.5	Gamma Radiolysis Effects on Stress Corrosion Cracking	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
20.6	Gamma Radiolysis Effects on Hydrogen Induced Cracking	LLNL Farmer, Upadhye, Glass & Day - LLNL Gamma Pit
20.7	Neutron Radiation Effects on Void Swelling	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
20.8	Neutron Radiation Effects on Mechanical Property Degradation	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
20.9	Neutron Radiation Effects on Mixed Potential	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
20.10	Neutron Radiation Effects on General Corrosion	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
20.11	Neutron Radiation Effects on Localized Corrosion	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
20.12	Neutron Radiation Effects on Stress Corrosion Cracking	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
20.13	Neutron Radiation Effects on Hydrogen Induced Cracking	LLNL Farmer, Upadhye, Glass & Day - McClellon Reactor
20.14	Neutron Capture Cross Section Determination	LLNL Farmer, Choi & Upadhye - McClellon Reactor
21	(Q) Differential Thermal Analysis (DSC, DTA)	(Q) Thermal Stability
21.1	Differential Thermal Analysis (DSC, DTA)	UWM Perepezko & Hildal
21.2	Wedge Casting	UWM Perepezko & Hildal
21.3	Effects on Corrosion	LLNL Farmer, Rebak, Lian & Day; SNL Yang
21.4	Effects on Mechanical Properties	UWM Perepezko & Hildal; SNL Yang; Case Lewandowski
21.5	Data Analysis & Interpretation	UWM Perepezko & Hildal; Others
22	(Q) Final Mechanical Testing	(Q) Mechanical Testing
22.1	Hot Hardness Testing - In Vacuum	CASE Lewandowski
22.2	Slow Strain Rate Testing of Cylindrical Dog Bone Samples in Air	Case Lewandowski
22.3	Creep Test Under Constant Stress - In Air	Case Lewandowski
22.4	Split Hopkinson Bar Test	Case Lewandowski
22.5	Impact Testing with Drop Tower	LLNL Farmer & Haslam
22.6	Data Analysis & Interpretation	CASE Lewandowski
22.7	Technical Project Review & Report Lab Scale Test of SAM Coatings	CASE Lewandoski
23	(Non-Q) Economics/Application Analysis	Economics/Application Analysis
23.1	Small Packages in Large Package	LLNL Farmer / BOOZ-ALLEN Kirkwood / CAT Beardsley
23.2	Multipurpose Container	LLNL Farmer / BOOZ-ALLEN Kirkwood / CAT Beardsley
23.3	Coated Criticality Control Structure (Basket)	LLNL Farmer / BOOZ-ALLEN Kirkwood / CAT Beardsley
	Go / No-Go Decision: Material Performance, Economic Analysis, and Proposed Application Data Approved by RW-1	

Demonstrate Viability of Process for Sub-Scale Container/Basket

24	(Non-Q) Optimized Designs Enabled by Coating	Optimized Designs Enabled by Coating
24.1	Small Packages in Large Package	DOE Walker / BOOZ-ALLEN Kirkwood / LLNL Farmer & Choi
24.2	Coated Multipurpose Container (MPC)	DOE Walker / BOOZ-ALLEN Kirkwood / LLNL Farmer & Choi
24.3	Coated Criticality Control Structure (Basket)	DOE Walker / BOOZ-ALLEN Kirkwood / LLNL Farmer & Choi
25	(Non-Q) Large Scale Model Containers (2-Ton Limit of Existing Crane)	(Non-Q) Large-Scale Model Containers
25.1	Procurement of Single-Wall 316L Stainless Steel Container & Basket	CAT Beardsley
25.2	Closure Lid	CAT Beardsley
25.3	Fixturing at Existing Spray Facility	CAT Beardsley
25.4	Powder Production	CAT Beardsley
25.5	Coating Container & Lid	CAT Beardsley
25.6	Coating Basket Materials	CAT Beardsley
25.7	Closure Weld	CAT Beardsley
25.8	Coating Closure Weld	CAT Beardsley
	Go / No-Go Decision: Fabricability, Durability and Corrosion of Coated Scale-Model Container, Basket & Closure	
26	(Non-Q) Testing Large Scale Model Containers (2-Ton Limit of Existing Crane)	(Non-Q) Testing of Large-Scale Model Containers
26.1	Lid Weldability	LLNL Rebak / SNL Yang / INL Swank / ORNL Blue
26.2	Electron Microscopy & X-Ray Diffraction of Witness & Core Samples	LLNL Rebak / SNL Yang / INL Swank / ORNL Blue
26.3	Consistency Based on Core Samples Data	LLNL Rebak / SNL Yang / INL Swank / ORNL Blue
26.4	NDE Method Development - Continuity Mapping with Eddy Current	LLNL Farmer / SNL Yang
26.5	NDE Method Development - Continuity Mapping with Gamma Sources	LLNL Farmer / SNL Yang
26.6	NDE Method Development - Continuity Mapping with X-Ray Methods	LLNL Farmer / SNL Yang
26.7	NDE Method Development - Continuity Mapping with Ultrasonic Methods	LLNL Farmer / SONALYSTS Halter
26.8	NDE Method Development - Continuity Mapping with Portable SEM Surface Probe	LLNL Farmer / NRL Bayles / SNL Yang
26.9	NDE Method Development - Continuity Mapping with Portable Electrochemical Surface Probe	LLNL Farmer / CASE Payer
26.10	Heat Transfer	LLNL Farmer & Choi
26.11	Coefficient of Thermal Expansion	LLNL Farmer & Choi
26.12	Handling	LLNL Farmer & Choi
26.13	Drop Test	LLNL Farmer & Choi
26.14	Impact Analysis	LLNL Farmer & Choi
26.15	Corrosion at Damaged Sites	LLNL Farmer / CASE Payer
26.16	Patching and Repair	CAT Beardsley
26.17	Deliquescence & Drip Testing	LLNL Farmer & Choi
26.18	Fabrication Process Evaluation & Modification	DOE Walker / LLNL Farmer
26.19	Draft Design & Specification Documents for Full-Scale Multipurpose Container (MPC)	LLNL Farmer & Choi
26.20	Design of Seismic Shaker Table Test for Full-Scale Demo MPC	LLNL Farmer et al.

Demonstrate Viability of Process for Full-Scale MPC

27	(Q) Full-Scale Demo Multipurpose Containers (MPCs)	(Q) Full-Scale Demo Multipurpose Containers (MPCs)
27.1	Powder Production	CAT Beardsley
27.2	Procurement of Three (3) Multipurpose Containers	LLNL Farmer
27.3	Spray Facility Preparation	CAT Beardsley
27.4	Coating Container & Lid Coating	CAT Beardsley
27.5	Coating Basket Materials	CAT Beardsley
27.6	Closure Weld	CAT Beardsley
27.7	Coating Closure Weld	CAT Beardsley
28	(Q) Testing Full-Scale Demonstration MPCs	(Q) Testing Full-Scale Demonstration MPCs
28.1	Lid Weldability	LLNL Rebak / SNL Yang / INL Swank / ORNL Blue
28.2	Electron Microscopy & X-Ray Diffraction of Witness & Core Samples	LLNL Rebak / SNL Yang / INL Swank / ORNL Blue
28.3	Consistency Based on Core Samples Data	LLNL Rebak / SNL Yang / INL Swank / ORNL Blue
28.4	NDE Method Development - Continuity Mapping with Eddy Current	LLNL Farmer / SNL Yang
28.5	NDE Method Development - Continuity Mapping with Gamma Sources	LLNL Farmer / SNL Yang
28.6	NDE Method Development - Continuity Mapping with X-Ray Methods	LLNL Farmer / SNL Yang
28.7	NDE Method Development - Continuity Mapping with Ultrasonic Methods	LLNL Farmer / SONALYSTS Halter
28.8	NDE Method Development - Continuity Mapping with Portable SEM Surface Probe	LLNL Farmer / NRL Bayles / SNL Yang
28.9	NDE Method Development - Continuity Mapping with Portable Electrochemical Surface Probe	LLNL Farmer / CASE Payer
28.10	Heat Transfer	LLNL Farmer et al.
28.11	Coefficient of Thermal Expansion	LLNL Farmer et al.
28.12	Handling	LLNL Farmer et al.
28.13	Drop Test	LLNL Farmer et al.
28.14	Impact Analysis	LLNL Farmer et al.
28.15	Seismic Shaker Test	LLNL Farmer et al.
28.16	Corrosion at Damaged Sites	LLNL Farmer / CASE Payer
28.17	Patching and Repair	CAT Beardsley
28.18	Deliquescence & Drip Testing	LLNL Farmer et al.
28.19	Fabrication Process Evaluation & Modification	DOE Walker / LLNL Farmer
28.20	Design & Specification Documents for Full-Scale Multipurpose Container	LLNL Farmer et al.
29	(Q) Initial Qualified Data Through Peer Review	Qualification of Any Existing Non-Q Samples & Data
29.1	Peer Review	LLNL Farmer & Barish
29.2	Publication in Peer Reviewed Journal	LLNL Farmer et al.
30	(Q) Regulatory/License/Baseline Ammendment	Regulatory/License/Baseline Ammendment
30.1	Material Selection Report	DOE Walker /B-A Kirkwood / M&O Stahl / LLNL Farmer
30.2	Waste Package Design Review	DOE Walker /B-A Kirkwood / M&O Stahl / LLNL Farmer
30.3	Modify TSPA Process Model	DOE Walker /B-A Kirkwood / M&O Stahl / LLNL Farmer
30.4	Prepare AMR	DOE Walker /B-A Kirkwood / M&O Stahl / LLNL Farmer
30.5	Update TSPA	DOE Walker /B-A Kirkwood / M&O Stahl / LLNL Farmer
31	(Q) Regulatory/License/Baseline Ammendment	Intellectual Property & Licensing Issues
31.1	Filing Additional Provisionals & Patents	DOE Daubenspeck / LLNL Thompson / TNC Buffa
31.2	Review of Complete Intellectual Property Portfolio Developed by HPCRM Program	DOE Daubenspeck / LLNL Thompson / TNC Buffa
31.3	Review of Relevant Intellectual Prorperty Outside HPCRM Program	DOE Daubenspeck / LLNL Thompson / TNC Buffa
31.4	Establish Appropriate Legal Vehicles for Commercialization & Enforcement	DOE Daubenspeck / LLNL Thompson / TNC Buffa
31.5	Prepare Licensing Strategy to Enable Commercializaiton of Processes for Containers	DOE Daubenspeck / LLNL Thompson / TNC Buffa

Overview of Accomplishments

- Fe-based amorphous metal formulations have been identified with corrosion resistance comparable to that of Ni-based Alloy C-22
  - Cr & Mo provide corrosion resistance
  - B enables glass formation
  - Y lowers critical cooling rate
    - SAM1651 = 80 K/s (yttrium added)
    - SAM2X7 = 610 K/s (no yttrium) … similar to SAM2X5
- These materials are also extremely hard provide enhanced resistance to abrasion and gouges (stress risers) from backfill operation
  - Type 316L Stainless Steel = 150 VHN
  - Alloy C-22 = 250 VHN
  - HVOF SAM2X5 = 1100-1300 VHN
- Optimization has been used to produce high-quality coatings in laboratory
  - Full-density pore-free completely amorphous coatings can be achieved by limiting the powder's size distribution so that the critical cooling rate can be maintained within particles during thermal spray (PSO)

Technology Provides Several Potential Benefits

- These new materials provide a viable coating option for repository engineers
  - SAM2X5 & SAM1651 coatings can be applied with thermal spray processes without any significant loss of corrosion resistance
  - Both Alloy C-22 and Type 316L stainless lose their resistance to corrosion during thermal spraying
- SNF/HLW containers with corrosion resistant coatings are envisioned
  - Enhanced multi-purpose container (MPC) … leverage existing capability
  - Protected closure weld … eliminate need for stress mitigation
  - Integral drip shield … elimination of titanium drip shield
  - Thicken areas where greater corrosion is expected (crevices)
- Both SAM2X5 & SAM1651 have high boron content which enable them to absorb neutrons and therefore be used for criticality control in baskets
  - Alloy C-22 and 316L have no neutron absorber
  - Problems encountered with borated stainless and Gd-doped Ni-Cr-Mo
  - Variable thickness absorber

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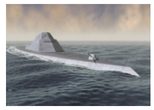
Backup Slides

High-Performance Corrosion-Resistant Materials: Virtual Research Group Coordination Activities

Jay Boudreau
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Milestones

- Chair or Co-Chair weekly HPCRM teleconferences
- Chair or Co-Chair all "Side-bar" teleconferences

Deliverables

- Management (Co-Chair) of HPCRM Teleconference & Videoconference Meetings
- Written Documentation of All Teleconferences
- Concise Streamlined Requirements Document for DOE Applications
- Documentation of all technology associated with FY04 successful "Salt Fog" Samples
- Formal Document Review with Written Comments at Project Leader's request
- Comment Resolution Satisfactory to Reviewer & Authors

Contribution to the HPCRM Project

- Helped the Project Leader maintain continuity and frequent interaction amongst Team members
- Identified "Action Items" for each weekly teleconference and followed up with Team members to ensure closure
- Provided thorough, clear, and accurate documentation of each meeting and distributed minutes to all appropriate Team members to enable all participants to focus on evolving project goals
- Provided formal documentation for "Salt Fog" specimens and for DOE Requirements

Backup Slides