Materials for Supercritical CO₂ Applications **Pre-Conference** Tutorial







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8th International sCO₂ Power Cycles Symposium February 26-29, 2024 San Antonio, Texas







ELECTRIC POWER **RESEARCH INSTITUTE**

Pre-Conference Tutorial 8th International sCO₂ PCS February 26-29, 2024 San Antonio, Texas





- - Cost

 - Lifetime

• Processing (availability, fabricability, weldability, repairability) • Tutorial outline

- development)

Materials for sCO₂ Applications - Outline

Heat engines (steam turbines, gas turbines, sCO₂ Brayton cycles) – higher temperatures lead to higher efficiencies e.g. gas turbine blades (increased strength, cooling, coatings) Materials selection based on • Properties for performance (strength – design codes, creep, toughness)

Compatibility with service environment

Power cycles and materials options (existing materials, AUSC)

• sCO₂ materials challenges

Corrosion and other testing

• sCO₂ materials selection







Power Cycle Comparisons

[GE Power Systems]

sCO₂ Turbomachinery (550 MW_e Plant)

Power Cycle Comparisons

Cycle	Component	Inlet		Outlet	
		T, °C	P, bar	<i>T</i> , °C	P, bar
Indirect	Heater Turbine HX Heater	450–535 650–750 550–650 750	10-100 200-300 80-100 200-300	650–750 550–650 100–200 1150	10-100 80-100 80-100 200-300
Direct	Turbine	1150 800	200–300 200–300 30–80	800 100	200–300 30–80 30–80

Operating conditions in indirect- and direct-cycle sCO₂ power systems [Holcomb, 2016]

Materials performance and degradation identified as barrier to commercialization.

	COMPONENT	M
nger	HEX, <1,000F	Αι
Excha B Kcha	HEX, >1,000F	Ni str
	Compressor Housing	St
	Compressor Impeller	(tr
	Compressor Rotor	
	Seals, Dry Gas	
	Turbine Housing	
Lod Lod Lod	Turbine Disk	Ni
Ę	Turbine Blade	Hi Ni
	Ducts, <1,000F	Αι
	Ducts, >1,000F	Hi
ce-of-	High Temp. Valves	Αι
Baland	High Temp. Piping	Αι

sCO₂ Brayton Cycle Materials Options

ATERIAL TYPE	SUPPORTING TECH BASE	RISK / RATIONA
ustenitic Stainless Steel	Various	High Technology lower pressures
i-Cr, solid solution rengthened alloys	USC/A-USC Steam	sCO ₂ oxidation, strength
eel casting	UK Nuclear Magnox / AGR	High TRL for low
rades incomplete)	Cryo Propellant Turbopump	High TRL, Low te
loy Steel	Various	High TRL, Low te
eramics / Cermet coating	Process Industries	Accelerated wea
i-based casting alloys	A-USC Steam	Low TRL (in deve
i-base superalloy	Gas / Power Turbine	Tech Base is sho Creep
igh Cr, Ni-base superalloy i-base superalloy + Pt-Al	Gas / Power Turbine	Tech base is sho Creep, HCF
ustenitic Stainless Steel	Various	High TRL for low
igh Cr, Ni-base superalloy	USC Steam	Low TRL. Oxidat weldability.
ustenitic/Nickel-based	A-USC Steam	Intermediate TR supply chain que
ustenitic/Nickel-based	USC/A-USC Steam	High TRL, but su for many needed

- v Readiness Level (TRL) for
- Weldable, moderate
- ver pressures
- emp limits oxidation
- emp limits oxidation
- ar in sCO₂
- elopment)
- orter life, sCO₂ Oxidation,
- orter life, sCO₂ Oxidation,
- ver pressures
- tion, Creep., Demonstrated
- RL, not tested in service, estionable
- apply chain is not established defined and components

Fabrication demonstrations:

set 20 Years of DOE-funded AUSC Materials Research, \$90M

Higher efficiency for new and existing fossil fuel plants +25% HHV efficiency improvement over the average U.S. power plants • 760°C/1400°F steam conditions drives the need to use nickel-based superalloys Minimizing risk for utilities to build A-USC power plants Close technology gaps leading to commercial scale demonstration Development of fabrication, welding, corrosion/materials database, ... Validation of technology applicable to multiple fossil, nuclear, sCO₂, and renewable power generation options Focusing on development of U.S. supply chain that can produce boiler and turbine components out of AUSC materials Industry partnership under DOE demonstrating supplier readiness for fabricating 760°C/1400°F-capable components **ASME code case approval for two new alloys:** Inconel ® 740H, CC-2702 (2012) Haynes ® 282, CC-3024 (2021) Welding: DMWs, thick section, overlay Forming: bending, extruding, swaging Casting: flowability, modeling

Component	Method	Primary Alloy(s)	Key Dimensions
/Jain Steam Pipe/Header + Bends	Extrusion	Inconel ® 740H	25,000 lbs 22 x 3.7 inch
lot Reheat Pipe/Header + Bends	Extrusion	Inconel ® 740H	25,000 lbs 28 x 1.5 inch
leader Assembly	Welding	740H, H282, stainless	About 14' x 16' 16 tube lengths
Aembrane Panel	Welding	P92	10' x 4'
Vye (VIM-ESR-VAR)	Forging	Inconel ® 740H	25,000 lbs
Jozzle Carrier / Shell	Casting	Haynes [®] 282	21,000 lbs
Rotor (VIM-ESR-VAR)	Forging	Haynes [®] 282	22,000 lbs
			-

Tubing Fabrication Demonstration

Header Welding Demonstration

AUSC – Major Achievements in Material Research and Fabrication

In-Plant Corrosion Demonstration

First 740H Pipe Extrusion

Final Header Assembly

H282 Valve Body Casting (after PT)

H282 Rotor Forging (Mid-processing)

H282 Nozzle Carrier Casting

Existing materials/mechanical properties OK for main components (piping, valves, turbomachinery, etc.) **Environmental considerations – high temperature sCO**₂

Availability

Environmental considerations – high temperature sCO₂

- Reliability of data

Performance of actual components/material forms

- Cost-effective alloys
- Higher temperature applications
- Coatings

Materials are available...but there are knowledge gaps...efforts are required to complete the picture.

sCO₂ Materials Technical Challenges

Current Knowledge

Corrosion testing – short-term, coupons, representative temps/pressures, mass gain (vs. depth) **Gaps Remaining/Technical Challenges**

Code compliant materials/qualified alloys; design codes Supply chain – in required forms and sizes

Longer-term (short-term testing inadequate – breakaway corrosion, intergranular corrosion) Testing under "real" conditions (flow, stress, impurities – H_2O , O_2 , others; indirect vs. direct cycles)

Mechanical property degradation

Thin sections – property differences, effect of geometry Diffusion bonded, brazed, welded joints – corrosion resistance Erosion, fouling of microchannel heat exchangers

Thermal fatigue, creep-fatigue interactions, high blade bending loads/temperature + pressure combinations Other challenges and considerations

Non-metallics – degradation of seals via swelling, rapid gas depressurization Leverage previous work – comparison to steam and SCW corrosion, pressure effects

• Effects of contaminants

Materials in CO₂ Environments – Before ~2008

Previous Knowledge • CO, gas-cooled Magnox reactors

Many reactor years of operation Structural material behaviour well-characterized • High temperature (650 °C) but low pressure ($< P_c$) • Corrosion rates higher during operation Oil and gas industry CO₂ experience • Enhanced oil recovery (EOR), CO₂ transport pipelines • High pressure (<21 MPa) but low temperature (< 200 °C) • Pure, dry – virtually inert < 500 °C

• Significant corrosion of steels and nickel alloys with ppm H₂O, > 600 °C Austenitic alloys better than ferritic-martensitic steel High levels of Cr and Ni increase corrosion resistance

- Breakaway corrosion caused by exfoliation and nucleation of oxides mainly influenced by exposure time and/or CO₂ gas pressure

Fundamentals (oxidation and carburization) Mechanisms and scales

- **Testing results**
 - Weight gain, oxidation scales
 - Factors affecting corrosion
 - Temperature, pressure, impurities
 - Materials (alloying, structure, thickness, etc.)
 - Effects of stress
- - Strength and corrosion vs. temperature
- - Progress in Materials Science 136 (2023)
 - State of the art overview material degradation in high-temperature supercritical CO₂ environments

Materials in CO₂ Environments – 2008-2023

Recent Corrosion and Other Testing

Materials selection strategy

Recent review paper (Li et al. 2023)

CO₂ Corrosion at Elevated Temperatures Oxidation $xM + yCO_2 = M_xO_y + yCO$ $xM + (y/2)CO_2 = M_xO_y + (y/2)C$ Oxygen partial pressure high enough to induce oxidation • Formation of oxide layer Carburization $2CO_{2}$ $2CO_2 \leftrightarrow CO_2 + C$ Penetration of C through the oxide layer Reaction with metallic elements

$$\leftrightarrow 2CO + O_2$$

[Li et al., 2023]

General Weight Gain Trends FM > austenitic > nickel-base X (>16% Cr)

sCO₂ Corrosion Testing – Weight Gain

be like those in steam

3000

- CO_2 and recent sCO_2 Recent sCO₂ and steam
- Mass gain plotted
- Larson-Miller plots for comparison

 $P = T(^{\circ}K)[20 + logt(hr)] \times 10^{-3}$

Ferritic-Martensitic Steels

Ferritic steels in CO₂ (left) and recent data vs. steam $P = T(^{\circ}K)[20+logt(hr)]x10^{-3}$ (right) [Kung, 2018]

Mass gains for sCO₂ and steam are approx. similar...the scale morphologies in sCO₂ would be like those in steam

(a) 12Cr-steel, 20MPa 600°C 1000h

[Furukawa et al., JPES, 2010]

- 12Cr Martensitic steel
- Two successive layers, no breakaway corrosion
 - Outer: Fe oxide, Fe_3O_4
- Thin internal oxide zone (IOZ) between base metal and inner layer
- Carburizing observed near surface in base metal
 - Factor in breakaway corrosion, degradation of ductility

Inner: Fe+Cr oxide, Fe(Fe_{1-x}, Cr_x)₂O₄

Ferritic-Martensitic Steels

sCO₂ Corrosion Testing – Corrosion Scales

CO., 550°C/250 BARS : CROSS SECTION - FESEM

[Rouillard, sCO₂ PCS 2011]

T91 9Cr F-M steel Duplex oxide layer Outer: magnetite, Fe_3O_4 Inner: spinel, Fe_{3-x}Cr_xO₄ Internal oxidation also Extrapolation to 20 years Corrosion layer thickness = $500 \ \mu m$ Static tests at 50 °C @ 10 MPa No corrosion observed Flowing CO₂ Similar oxide scale at 550 °C @ 1 bar (+ outer Fe_2O_3 haematite)

Schematics of Corrosion Scales for Various Alloy Systems

/		· - · - · - · -	Oxide layer str
Eo Cr allour	Low Cr	substrate	Fe-Cr-O spinel
	High Cr (>17%)	substrate	
	Low Cr	substrate	Cr ₂ O ₃ and/or NiO
Ni-Cr alloys	High Cr (>14%)	substrate	Cr ₂ O ₃

[Li et al., 2023]

sCO₂ Corrosion Testing – Corrosion Scales

ide layer structure

Cr-O spinel Fe-O

Fe-Cr-O spinel

Ni-Cr-O spinel

Cr-depletion zone. Internal oxidation zone

Matrix of ferrite-martensite steel (Fe20Cr alloy) (650°C/1bar,120h) (b)

Fe-Al spinel oxide-

Al-depletion zone/

Grain boundary

Matrix of alumina-forming alloy (Fe-Cr-Al alloy) (650°C/20MPa,3000h)

(a) FM steel (b) austenitic steel (c) AFA (d) nickel-base alloy [Yang et al., 2022]

austenitic steels [Strakey, 2014]

nickel-base alloys in sCO₂ [Strakey, 2014]

Temperature

- corrosion
- breakaway corrosion

Pressure

- No "real" consensus

Some results show increased weight gain • Going from 0.1 to 20 MPa has minimal effect Some results show more protective scale Infiltration of C becomes easier – more carburization?

• However, are there any "surprises" at low temperatures • Very high temperatures?

Most important factor – increasing temperature leads to increasing

sCO₂ Corrosion Testing – Factors

- Parabolic growth breaks down, scale becomes non-protective,

 - Coatings, very high temperature materials, cooling schemes
- As pressure increases, oxidation and carburization increase?

Impurities

- Indirect cycles: ppm levels
- Direct cycles: % levels
- kinetics change
- Corrosion behaviour expected to change as thermodynamics and • Various results

Alloying

- Cr determines corrosion resistance of alloys chromia stable oxide, good protection

 - corrosion
- Ni Ni-base alloys show better carburization resistance than steels Facilitates stable chromia layer • Higher solubility of carbides – higher tolerance to C • Al – improves corrosion resistance by forming protective alumina layer

- - Higher stability, lower oxidation kinetics, higher resistance to C

sCO₂ Corrosion Testing – Factors

• High Cr favours Cr_2O_3

• Low Cr fails to form continuous scale – less protective, prone to internal

Component thickness Strength considerations • Thicker sections have better corrosion resistance • Alloy reservoir • Also consider component geometry Welding and joining • Heat affected zone, microstructural changes, segregation of alloying elements joints Coatings alloyed materials

sCO₂ Corrosion Testing – Factors

- Due to oxidation and carburization
- Application of coatings to protect metal substrate

Must consider tolerance of corrosion-induced thickness loss

• Leads to weak points, can affect corrosion and mechanical behaviour of the

• Alloys with poor corrosion resistance will be worse after welding • Fe- and Ni-base alloys with high alloying levels may not be so affected

Reduce oxidation and carburization – may allow application of low-

Stress-assisted corrosion

- Combined effects of mechanical loading/stresses and corrosion will accelerate material degradation leading to early failure
- Tensile behaviour
 - Increased strength and reduced ductility (carbide precipitation)
 - Fe-alloys more prone to degradation vs. Ni-alloys
- Creep
 - \bullet
- Stress corrosion cracking
 - Combined effects of chemical and mechanical loading
 - Some work here
 - C-rings
 - Pressurized tubes
- - Limited work

sCO₂ Corrosion Testing – Factors

Not much work done here

• Fatigue and thermal cycling

21

Code compliant materials/qualified alloys; design codes IN740H code case approved • Haynes 282 code case approved Design codes for valves, heat exchangers? Supply chain – in required forms and sizes Market pull to enable capability IN740H for tube, pipe, fittings [deBarbadillo, 2018 and 2022] Tube and pipe available Smallest tube made at Greenville Tube Fittings, etc. not available from stock Largest pipe made at Wyman-Gordon • Various TRL levels Manufacturing mill products (TRL 8, full plant required for TRL 9) \bullet • Manufacturing components, fabricating systems (TRL 6, moving to 8)

• Systems (limited experience, TRL 4)

sCO₂ Materials Challenges – Availability

sCO₂ Materials Challenges – Environmental Considerations

Currently max test duration in

Carburization and internal oxidation leading to breakaway

Issue with ferritic steels

• Seen in austenitic steels (initial stages, with duplex scales, after

Ni-based alloys with high Cr

Estimate via micro hardness measurements [Kung, 2018] or quantify via Glow Discharge **Optical Emission Spectroscopy** (GDOES) [Lance, 2018] and

Results in Grade 92 Steel

[Brittan, 2020]

 Testing under "real" conditions (flow, stress, impurities – H₂O, O₂, others; indirect vs. direct cycles) GTI/Oak Ridge National Laboratory 1000 hours) CSIRO impurities [Pint, 2019]

sCO₂ Materials Challenges – Environmental Considerations

• C-ring testing (stressed material) in sCO₂ (750 °C, 20 MPa, 500-• Various materials (housing, disk, blade) [Keiser, 2016+2017] No SCC seen

Pressure vessel as test specimen (stressed material) Various labs examining effects of

 CO₂ composition (RG vs IG) Not much difference Open cycle conditions [Shingledecker, 2016], [Kung, 2018], [Lolla, 2018], [Pint, 2018], [Tylczak, 2018], [Walker, 2018],

[Keiser, sCO₂ PCS 2016]

External tube surface temperatures

[CSIRO]

[Pint, 2018]

SCO, flow

sCO₂ Materials Challenges – Environmental Considerations Round Robin Testing and Fundamental Modeling (US DOE Nuclear Energy University Programs) Various test facilities, but previously no formal test program to validate data consistency Comparable and reproducible results desired Mass Gain of Alloy 625 Exposed to CO₂ at 550°C and 20 MPa Lead: OSU, Collaborators: UofW-Madison, 0.25 ORNL, NETL, Carleton University, KAIST, - - - KAIST - - - KAIST - - 🔶 - NETL 🕂 🕂 – NETI - - 🕂 - UW – – 💆 – UW EPRI - - - - OSU Initial results [Tucker, 2018] +/- consistent 2 0.15 2 0.15 Recent results [Zanganeh, 2022] O Compact tension specimens exposed, 0.05 0.05 study subsequent fatigue crack growth [Holcomb, 2016] 1400 200 Time [h] Time [h] Evaluate effects of sCO₂ exposure on [Tucker, 2018] tensile properties [Pint, 2016] and [Jang, 2014] Ex-situ fatigue response after sCO₂ 90 As-received exposure [Rozman, 2018] 80 S-CO, at 550°C (1000h) 625 1200 S-CO₂ at 600°C (1000h) 70 - 310S In-situ environmentally induced cracking % S-CO, at 650°C (1000h) Teeter, 2018] 1000 atio **G91** Effect of sCO₂ on steel ductility [Pint, 2021] MP Bu 800 -TS,

Reliability of data Mechanical property degradation

- Degradation of steels in CO₂ [Rozman, 2022]

D

ganization	Maximum Temperature	Maximum Pressure	Chamber Volume	Flow rate (mL/min)	Autoclave Material
OSU	800°C	26 MPa	1235 cm ³	0-24	Haynes 230
UW	750°C	25 MPa	900 cm ³	0-24	Inconel 625
2 systems)	760°C	38 MPa	(combined)	0-24	Haynes 282
ORNL	850°C	30 MPa	1400 cm ³	0-24	Haynes 282
NETL	800°C	28 MPa	1040 cm ³	0-24	Haynes 230
Carleton	750°C	25 MPa	1150 cm ³	0-250	Inconel 625
KAIST	700°C	25 MPa	1077 cm ³	0-24	Inconel 625

Thin sections – property differences, effect of geometry

- [Pint, 2016]
- [Kung 2016]

Diffusion bonded, brazed, welded joints – corrosion resistance

- 2020]
- [Shingledecker, 2022]

exchangers

- [Sabau, 2016]
- Thermal fatigue, creep-fatigue interactions, high blade bending loads/temperature + pressure combinations

sCO₂ Materials Challenges – Performance of Actual **Components/Material Forms**

Creep debit for thin sections [Pint, 2016] Microstructure and creep of 740H sheet [Shingledecker, 2022]

Oxide thickness not extent of damage

Heat flux, stress from complex geometries

Performance of welded 740H and 282 [Brittan, 2018] and Grade 92 [Brittan,

Weldment cracking of sCO₂ heater **Erosion, fouling of microchannel heat**

> Is erosion a real problem – fluid or debris, exfoliation? [Fleming, 2014], [He, 2018] Oxide scale itself may cause blockage

[Sabau, sCO₂ PCS 2016]

Cos	st-effective a
	Steels for dire
Hig	her-tempera
	SiC piping [Ne
	[Barringer, 202
	Material option
Coa	atings
	Allow use of Ic
	n-metailics –
vid dor	Swennig, rap
	Tunnison 200
	mnatihility of
	[Nanon 2022]
	INCION, ZUZZ
LC V Cor	nnarison to a
CO	rosion. pres
in ((0, 0, 0)
	Extend ORNL
	steam to sCO
	2018 Exfeliction
	 Exionation Predicts so
	evolution c
	Little pressure
	pressure CO ₂

sCO₂ Materials Challenges – Other Considerations

lloys ect-fired [Oleksak, 2022] **Ature applications** eiderer, 2022] and 22] ns [Pint, 2022]

ower-cost alloys or push of material

degradation of seals pid gas

09] and oil and gas studies polymers

ous work – steam and SCW sure effects (testing

/EPRI exfoliation model for ₂ [Sabau, 2016], [Kung,

of oxide scales on boiler tubes cale failure and loss based on of oxide

effect seen in sCO₂ – low testing OK?

[Tunnison, 2015]

Polymers – Rapid Gas Depressurization (RGD)

Rapid depressurization after a polymer seal is diffused with S-CO₂ can damage the seal as the S-CO₂ quickly expands to escape the seal

RGD is increased by:

- High pressure
- High gas concentration

[sCO2 Fundamentals Tutorial, 2013]

[Sabau, sCO₂ PCS 2016]

High decompression rate

 High temperature Poor seal constraint

Alloys with high Cr and Ni, and Ti and Al more corrosion resistant

Build a stable, tight oxide layer that resists corrosion

Alloys with low Cr levels less corrosion resistant

Build a duplex layer with that does not resist corrosion as well

In general, decreasing corrosion resistance:

Nickel Ni-Cr-X Cr>16%	Austenitic Fe-Cr-Ni Cr>16%	FM Fe-Cr Cr<12%
IN625	800H	12Cr
IN617	AL6-XN	HCM12A
Haynes 230	316SS	NF616
IN718	310SS	T91
IN738		

Increased corrosion with temperature Not much (if any) pressure effect

Summary of Main Findings

[[]Pint and Brese, 2017]

Key step in design **Poor choices?**

- Failure, increased cost
- **Best material?**
 - Properties to provide necessary service performance
 - Processing of material into finished components
 - Selection process
 - conditions into required material properties)
 - Analysis of material requirements (translate service and environmental Screen candidate materials (compare needed properties with databases)
 - Select candidate material(s) (analyze candidates trade-offs, value analysis, cost-benefit, etc.)
 - Develop design data (testing, pilots, etc.)

 \bullet

Piping/casing/heat exchangers

- Governed by ASME B&PV Code, Piping Codes, Material Standards • Code approved materials, allowable stresses
- Compatibility corrosion allowance

Turbines/compressors/shafts

- More flexibility
- Lots of materials available
- OEM designs
- Compatibility

Other components

- Seals
- Bearings
- Electrical components
- Etc.

Materials Selection

Materials Selection – Strength and Corrosion

RY TECHNOLOGICAL RECOMMANDATIONS FOR STEELS

Corrosion behaviour os« Mild steel » with %Si > or < 0,4% under SC-CO₂ at 250 bars at 400°C?

[Rouillard, sCO₂ PCS 2011]

- From past studies carried out in the 60-70's for MAGNOX reactors and AGR :
- $CO_2 + 1\%$ vol CO + 300 vpm $H_2O + 300$ vpm $H_2 + 350$ vpm CH_4 at 20-40 bars

		Auster
teel	Austenitic steels	(31
	(316L type)	Nim
		Ρ
	<660°C	>6

nitic steels 0 type, onic 80, PE16) 660°C

Existing data from other high-temperature systems, and sCO₂ work are valuable

ASME codes – temperature limits based on allowable mechanical strength • 100,000 hr creep rupture at 100 MPa

Corrosion effects?

Recommended maximum working temperature of Fe- and Ni-based alloys as candidate materials for different components in S-CO₂ environments.

Alloy type

Fe-based

Low Cr ferritic s High Cr ferritic

AFA

Austenitic stainl steels (Cr < 20%Austenitic stainl steels (Cr > 20%Cr > 14%

Ni-based

Materials Selection – Strength and Corrosion

• Steam-sCO₂ – results generally applicable to sCO₂

• SCW-sCO₂ – SCW usually more corrosive

	Typical alloys	Recommended temp. (°C)	Notes
steels	T22	<450	
steels	T/P91, T/P92, T/P122, HCM12A	<550	
	OC6, OC7, OC10, MA957	<550	Up to 650 > 20%.
less %)	TP347HFG, Super SS304H, SS316,	<620	
less %)	Alloy 800, SS310	<650	
_	Alloys 230, C-276, 282, 740, 617, 600, 690, 625	<750 °C or other severe environments (high contents of impurities)	For crucia recomme

Air-sCO₂ – corrosion generally same for corrosion resistant alloys, corrosion worse in sCO₂ for less corrosion resistant alloys

ial parts like turbine, ending Cr > 22%; Alloy 625 could be welding fillers.

[Li et al., 2023]

0 °C for some AFAs with Cr

(MPa)

Stress

Materials Selection – Strength and Corrosion

Alternate valve from <u>316SS</u>

10 MW_e STEP Facility and Materials

[Marion, 2021] and [Lariviere, 2019]

Previous loops Sandia National Laboratories • Piping, erosion Naval Nuclear Laboratory Erosion • Others? STEP

• Materials availability, processing, fabrication Compatibility with service environment • Operation under "real" conditions – flow, stress, etc. • Failures – failures modes, analysis

Thank You!

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