AMM Advanced Methods for Manufacturing

DEC 2 – 3, 2020

Development of Low Temperature Spray Process for Manufacturing Fuel Cladding and Surface Modification of Reactor Components

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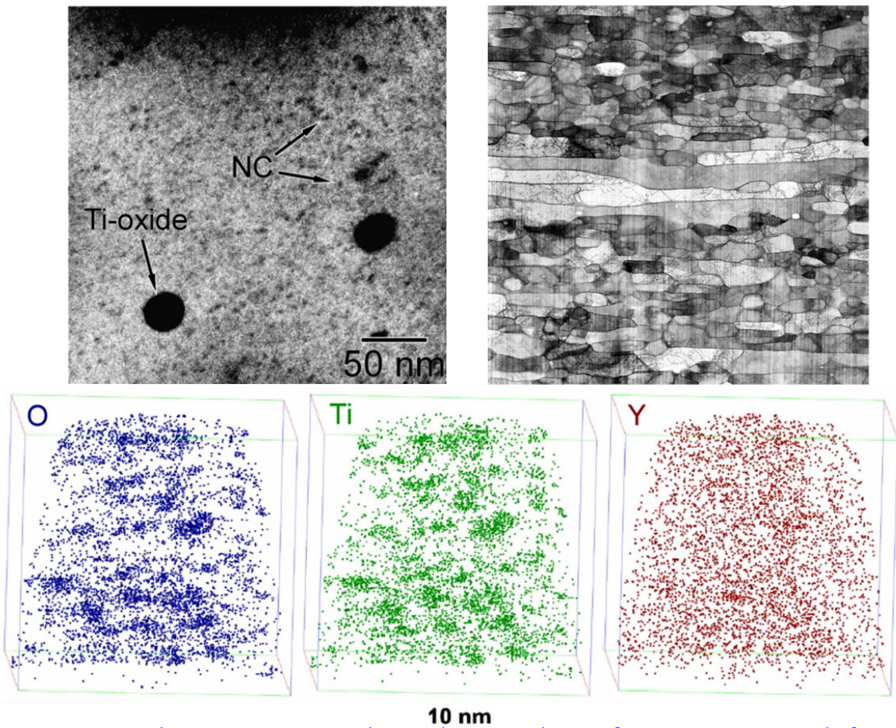
Project Objectives

Develop low temperature, solid-state (no-melting involved) powder spray deposition process (cold spray process):

- Rapid, near-net shape manufacturing of oxide-dispersion strengthened (ODS) steel cladding tubes
- High deposition rate coating technology for corrosion and wear protection, and repair of nuclear reactor components

Oxide Dispersion Strengthened (ODS) Steels



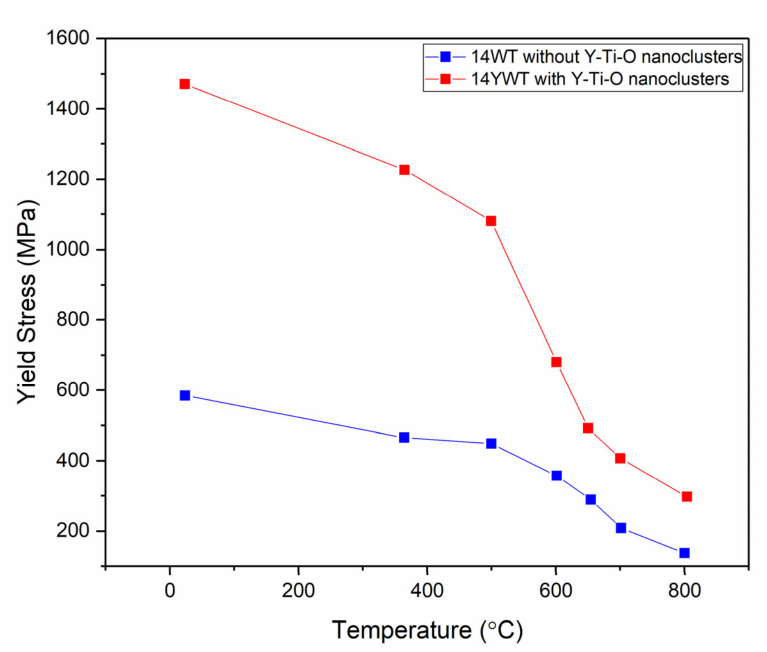
TEM images showing nano-scale oxide particles in ferritic matrix with fine grains and atomic probe tomography (APT) image of nanoclusters [Hoelzer et al, JNM 2007]

ODS steels are ferritic steel with uniform dispersion of nanometer-sized oxide particles and nanoclusters (e.g., MA957, 9CrODS, 12YWT, 14YWT, etc)

- Has the low radiation-induced swelling of conventional ferritic steels
- High temperature strength superior to conventional ferritic steels
- Regarded as a cross-cutting material for multiple advanced reactor concepts (e.g., SFR, LFR, fusion reactors)

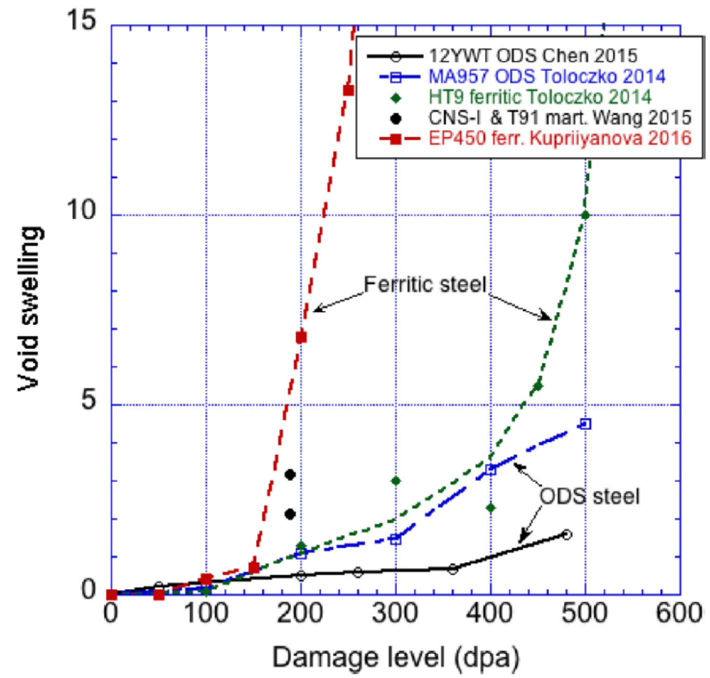
Role of Oxide Nanoparticles/Nanoclusters in Ferritic Steels

Increased High Temperature Strength



Yield strength as a function of temperature for 14WT and 14YWT [Hoelzer et al, JNM 2007]

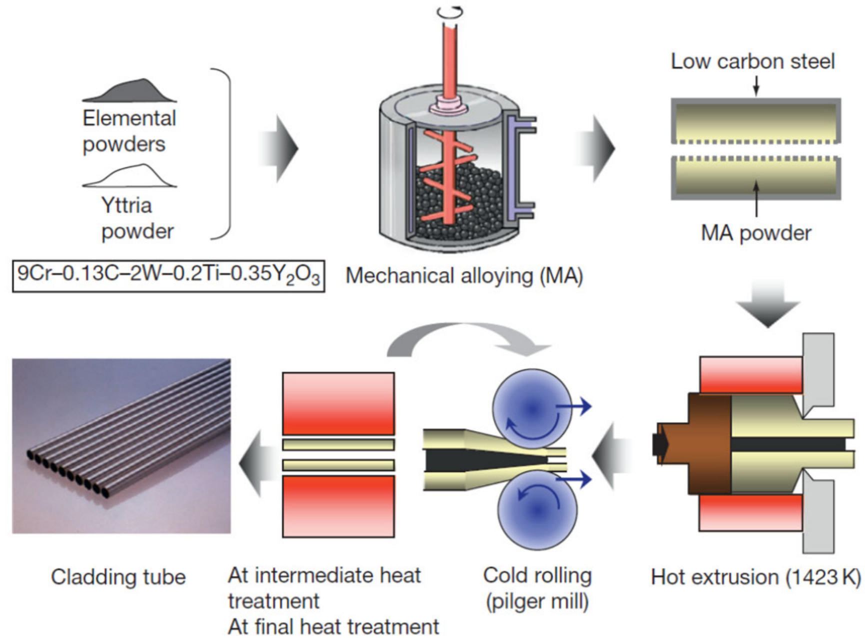
Enhanced Radiation Damage Tolerance



Comparison of void swelling due to single ion irradiation at 450 – 480 ºC [Zinkle et al, Nucl Fusion 2017]

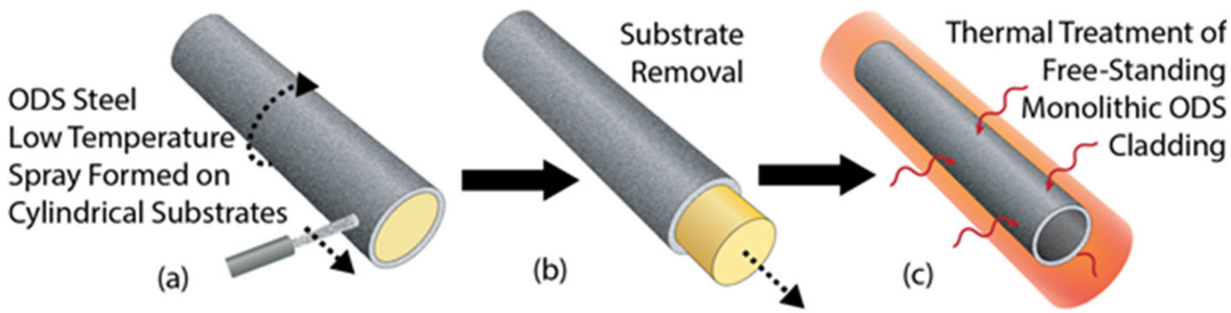
Conventional Manufacturing of ODS Steel Tubes – Slow and Expensive Process

- Milled powders are canned and degassed at 400 ˚C and subjected to multiple hot/ warm extrusion steps (8 -10 steps) at temperatures > 1000 ˚C and annealing.
- Low strain rate extrusion processes not conducive to large-scale manufacturing
- May lead to grain anisotropy, and anisotropy in mechanical properties
- Melting processes cannot be used as they lead to upward stratification of oxide nanoparticles (heterogeneous dispersion)



Conventional fabrication of ODS steel tubes requires mechanical alloying and multiple extrusion steps [G. Odette et al, Ann. Rev. Mater Res., 2008]

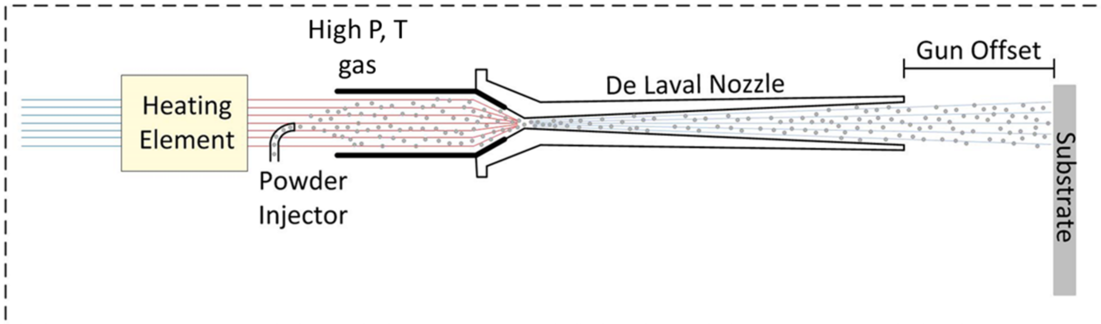
Concept of Manufacturing ODS tube via Cold Spray Process – Three Major Steps



Potential Benefits:

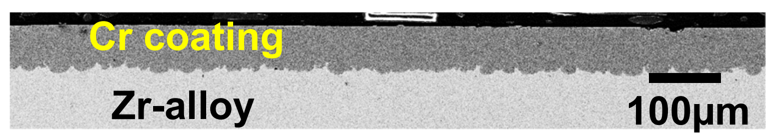
- **Eliminates multiple extrusion steps**
- **Eliminate ball milling step**
- **Faster and cheaper manufacturing process**

Cold Spray Additive Manufacturing at University of Wisconsin, Madison



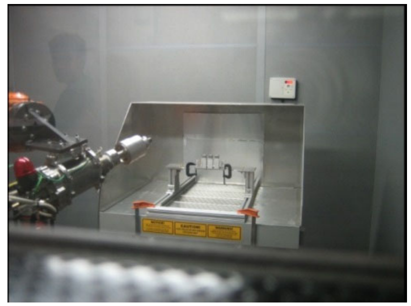
- High velocity powder particles propelled by a gas onto the surface of a substrate/part to form a coating or a deposit
- Particle temperature is much lower than the melting point – deposition occurs in solid state
- Atmospheric pressure operation - high deposition efficiency (solid-state additive manufacturing)
- Supports factory fabrication and expeditious deployment

Cr coating on Zr-alloy cladding for ATF (with Westinghouse)



Cold Spray Laboratory at the University of Wisconsin (est. 2012)

Robot for pre-programmed movement of spray gun



Sample stage and dust collector (below that)

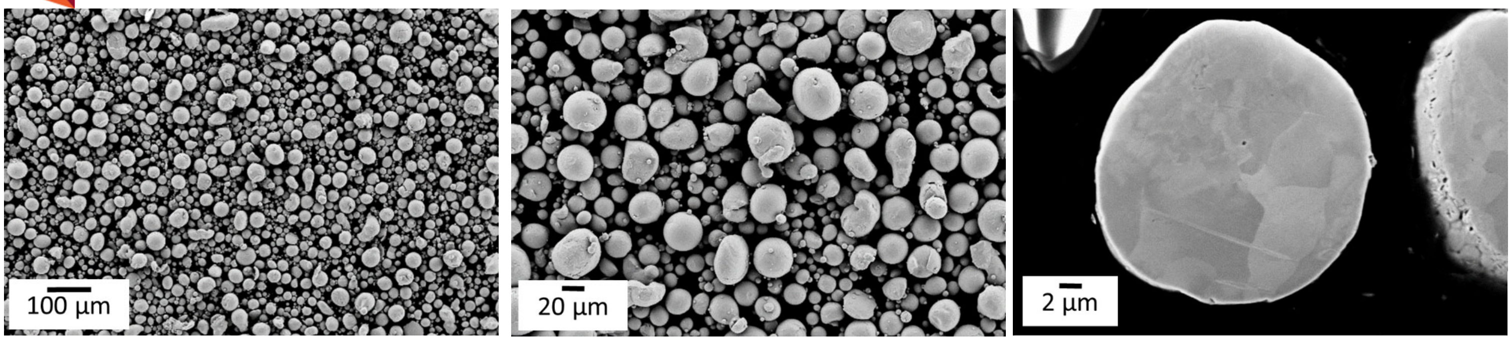
Sound-proof spray booth

- **4000-34 KINETIK System, from ASB Industries/CGT-GmBH**
- **Spray booth from Noise Barriers**
- **Robot controlled (Nachi system, from Antennen)**

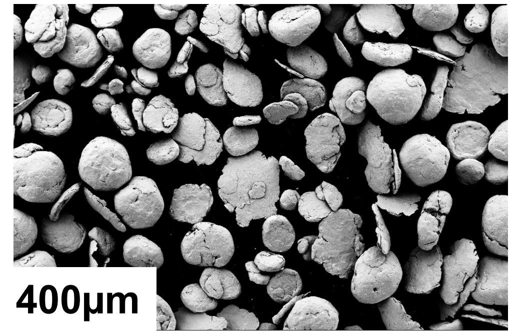
Nitrogen/helium gas cylinders

Robot controls (left) and spray gun control (right)

Feedstock Powder for Cold Spray Process



Gas atomized powder (ORNL)



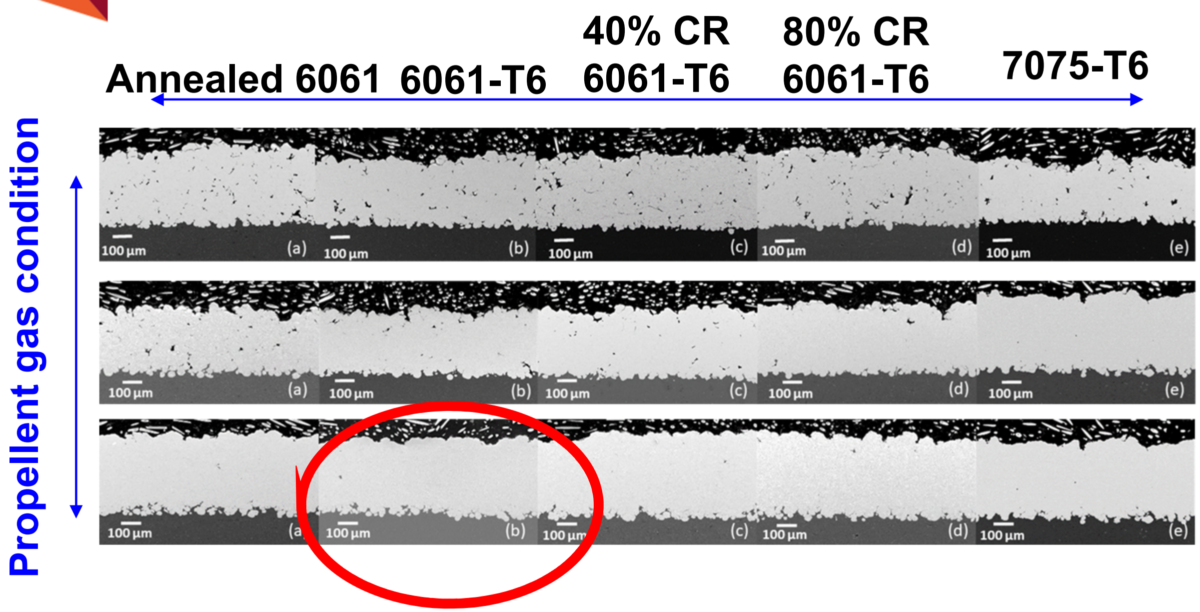
Ball milled powder (LANL)

- Received from collaborator at Oak Ridge National Laboratory (project collaborator)
- Gas atomized powder (-44 µm) L2308
- Gas atomized powder composition (wt%):
  - Fe-14Cr-3W-0.32Ti-0.11Y-0.12O
  - Fe-14Cr-3W-0.39Ti-0.23Y-0.14O
- Ball-milled powders from Los Alamos National Lab (project collaborator)

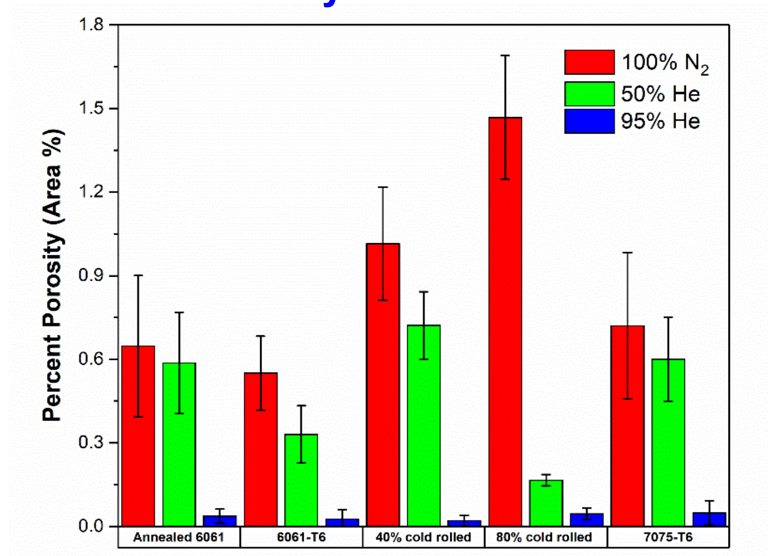
Parameters Investigated for Cold Spray Manufacture of ODS Steel Cladding

- Propelling gas composition (nitrogen, helium, and mixtures thereof)
- Gas preheat temperature
- Disposable Al-alloy substrates (different hardness substrates)
- Powder size and size distribution
- Two compositions of ODS

Parametric Investigation of Cold Spray Process (powder size effect)



Porosity measurement

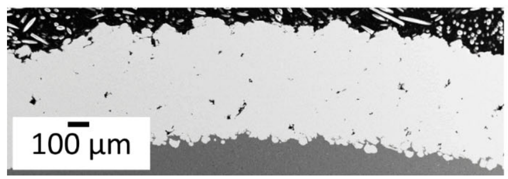


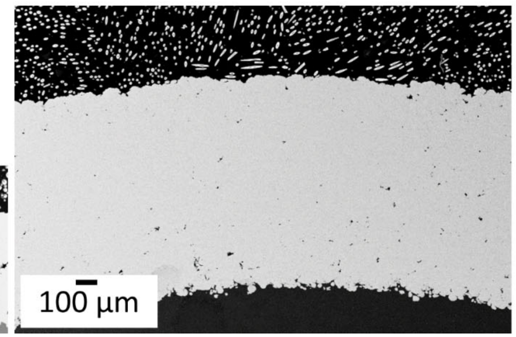
A deposit with 0.03% porosity and 1.2 mm in thickness is achievable by single pass process

Parametric Investigation of Cold Spray Process (powder size effect)

ODS tube < 44 µm, including < 25 µm powder

ODS tube with 25-44 µm powder

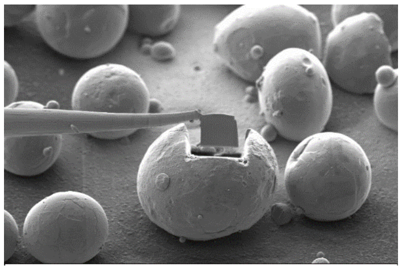




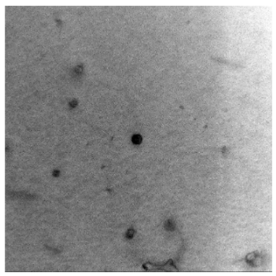
- Two different powder size distributions investigated:
  - 25-44 µm size powder
  - -44 um size powder that included powder sizes less than 25 um
- The -44 um sized powder increased both the deposit density and thickness due to interstitial gap filling and peening effect of larger particles

TEM Imaging of Powders and Cold Sprayed Deposit

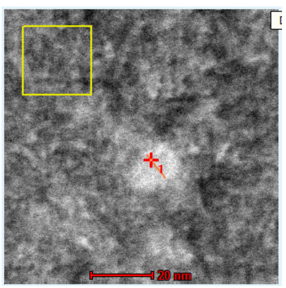
Powder



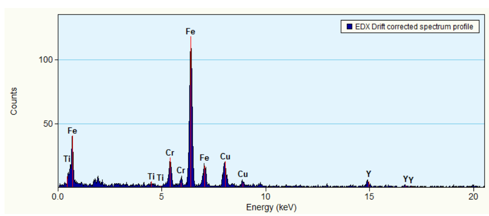
Transmission Electron Microscopy (TEM) lamellae prepared from a particle



TEM images showing fine particles in the matrix

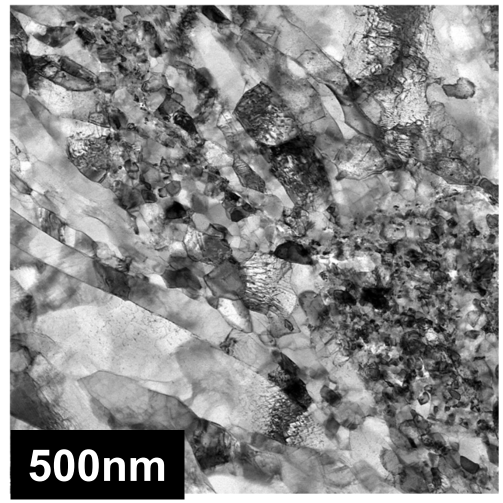


TEM images showing fine particles in the matrix

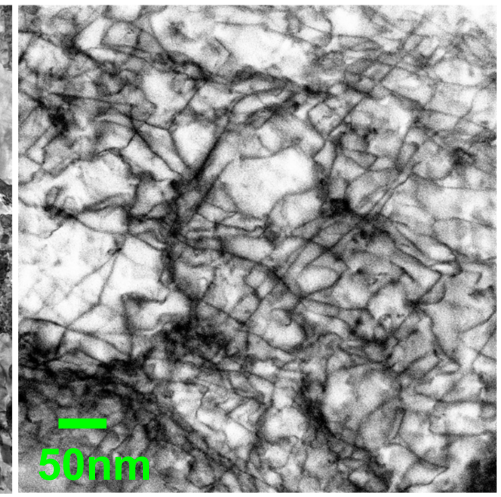


EDS spectra showing Ti/Y enriched particles

Cold Spray Deposited Cladding

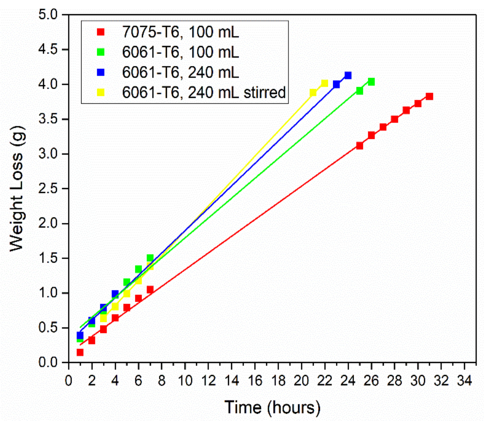


Grain refinement and inhomogeneous grain size distribution

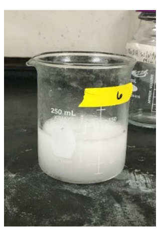


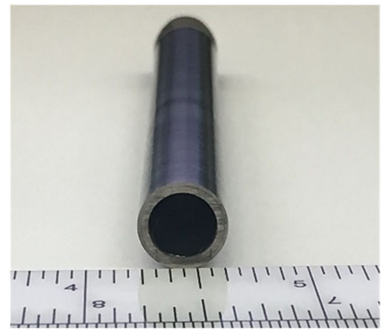
Dislocation forest and disappearance of the nanoparticles

Dissolution of Al-alloy Mandrel resulting in a Free-Standing Tube

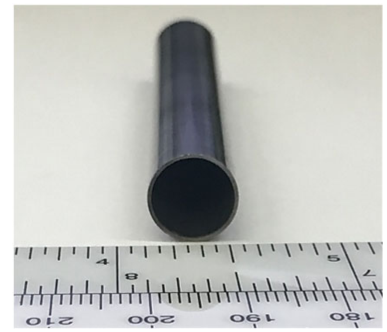


Dissolution kinetics of various Al-alloys in NaOH solution





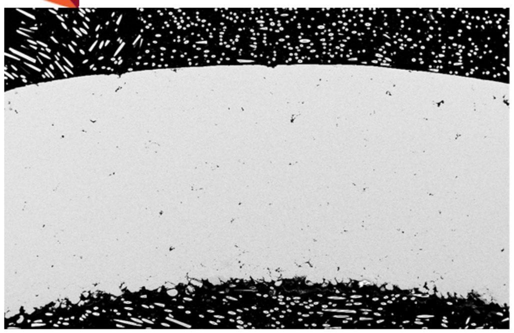
ODS coated Al-alloy mandrel

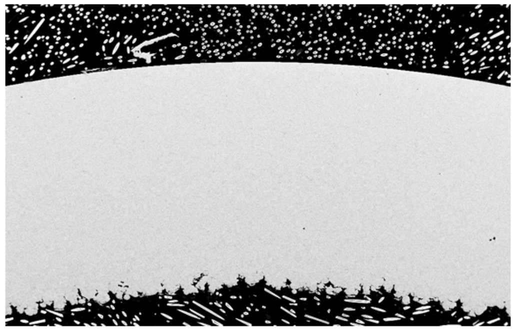


Removal of Al-alloy mandrel

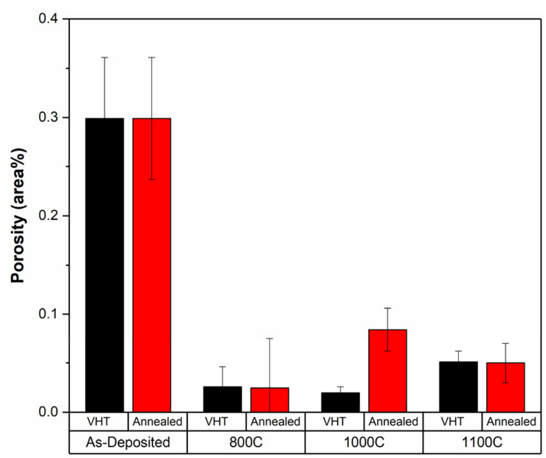
- Polishing of the as-deposited tube to decrease surface roughness and wall thickness
- Dissolution of the aluminum tube mandrel in 20% NaOH solution

Post Vacuum Heat Treatment As-fabricated Tube led to further densification

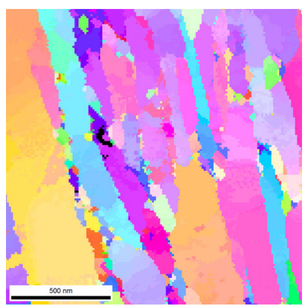




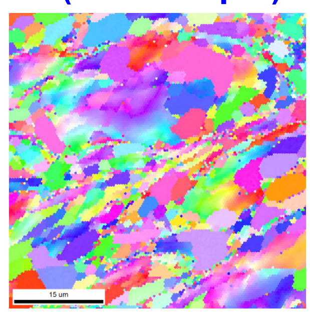
- Heat treatment at 800 ºC to 1100 ºC for 1 hour in inert gas or partial vacuum
- Improved density of the deposit but grain growth was observed
- No specific grain texture



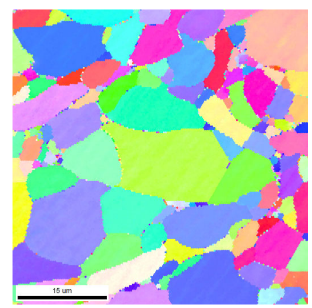
As-deposited (dynamic recrystallization)



800 ºC, 1hr (4.9 ± 3.8 μm)

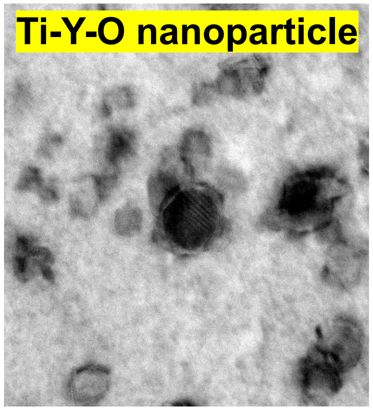


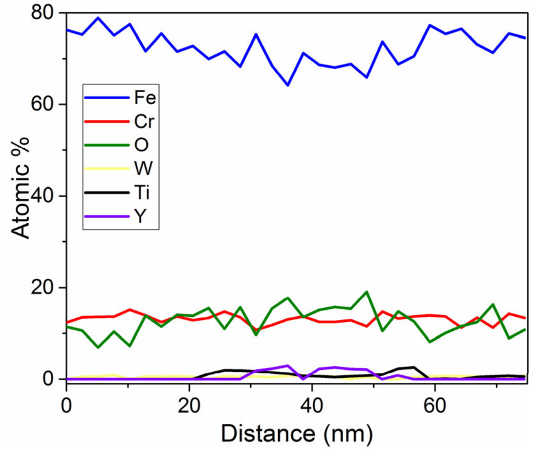
1100 ºC, 1hr (8.0 ± 4.0 μm)



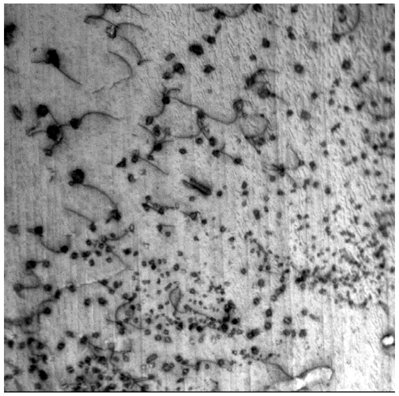
Post Vacuum Heat Treatment As-fabricated Tube led to Oxide Nanoparticle Precipitation

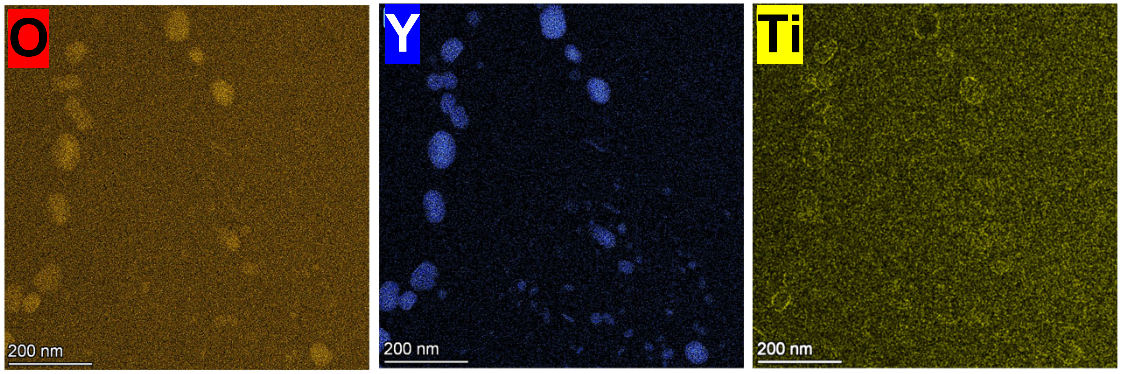
- High number density oxide nanoparticles were precipitated in grains and GBs during post-heat treatment
- TiO_2 particles (50 to 150 nm) and Ti-Y-O particles (20 to 80 nm)





STEM BF/DF images for ODS after 1100 ºC 1hr

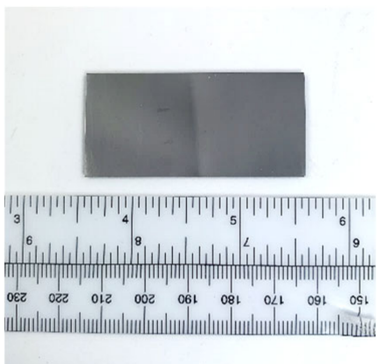




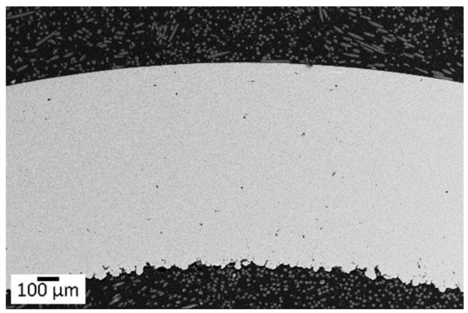
EDS maps showing Ti-Y-O nanoparticles

Free-Standing ODS Cladding Tube Manufactured Using Gas Atomized Powder using Cold Spray

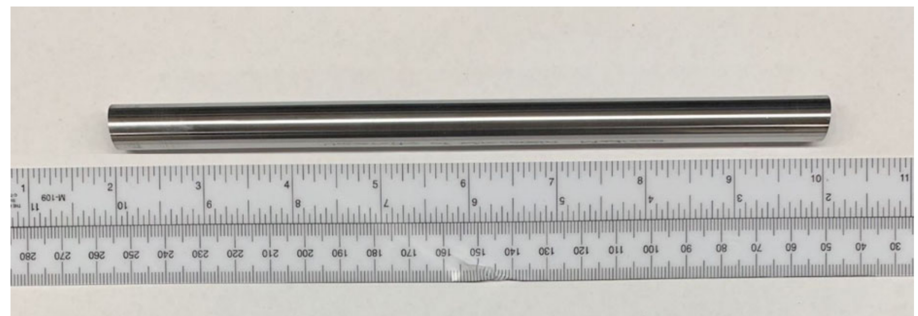
ODS Steel Flat



Cross-section of ODS cladding tube



ODS cladding tube

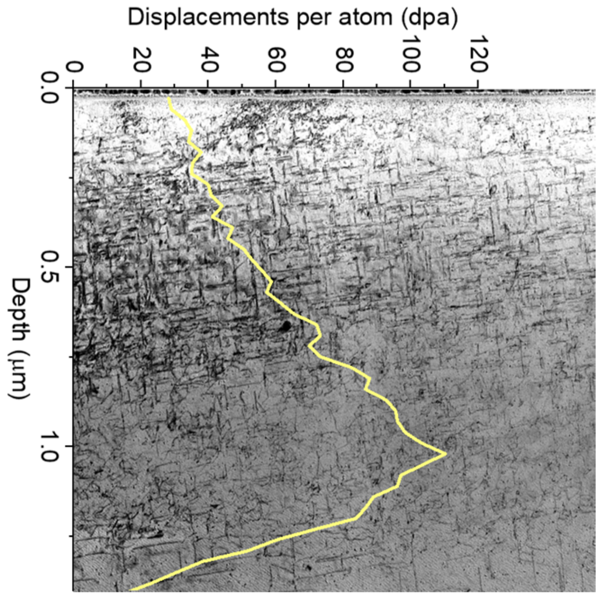


Length: 204 mm (8”) cladding tube
O.D.: 11.5 mm
Wall Thickness: ~ 1 mm

Heavy Ion-irradiation Response of ODS Steel Materials

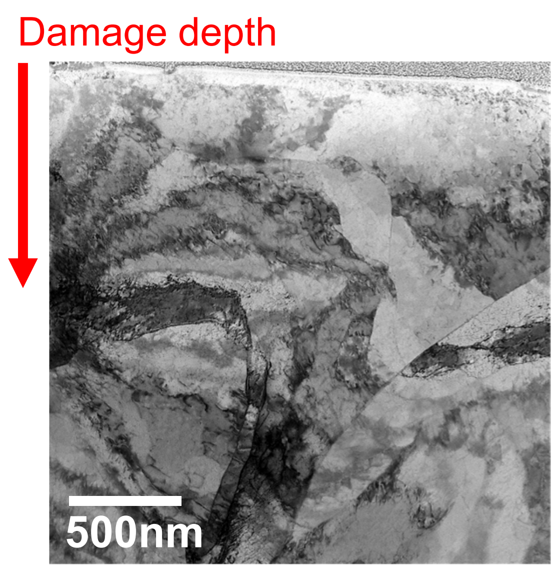
3.7 MeV Fe^{2+} at 500 ºC, 6.5 × 10^{-3} dpa/s, Max. 110 dpa

Heat Treated CS ODS steel

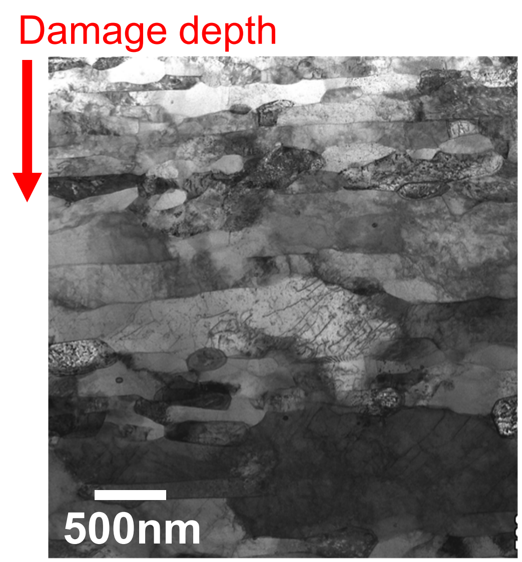


Formation of radiation defects (dislocation loops - mostly <100> type loops and line dislocations)

As-deposited CS ODS steel



Conventional ODS steel



No significant microstructural changes (fine grains and pre-existing dislocations structure)

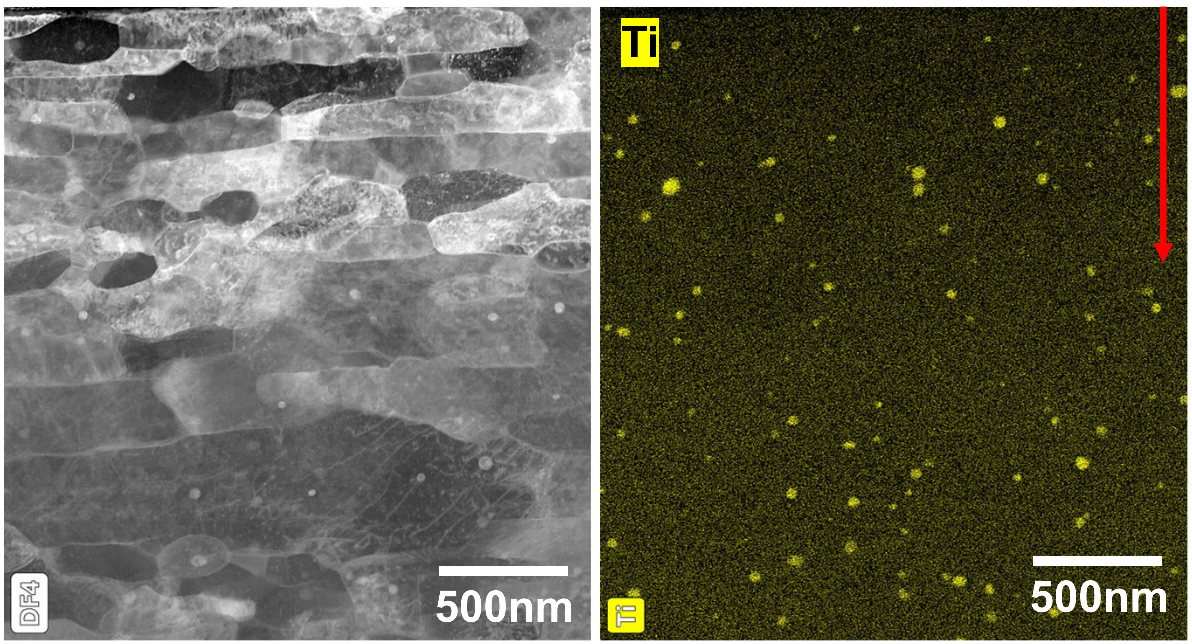
Stability of Oxide Nanoparticles in ODS Steel under Ion Irradiation

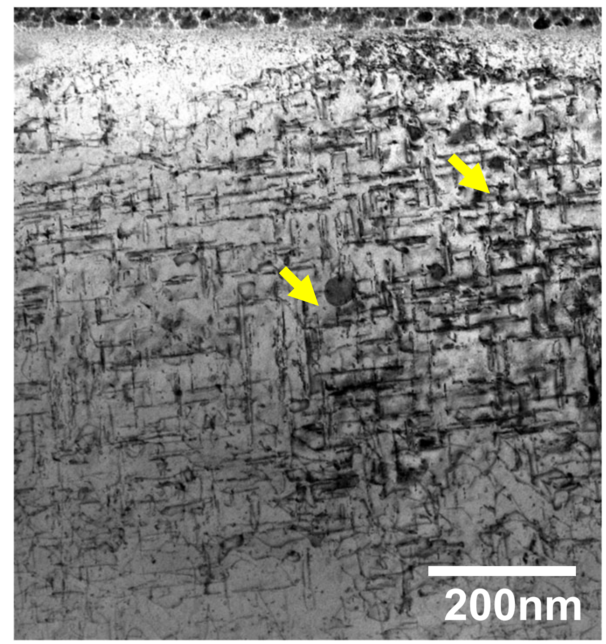
Coarse oxide particles are stable under the radiation damage

Conventional ODS steel

Damage depth

Heat Treated CS ODS steel



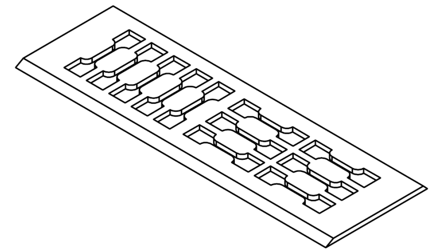


APT work is being performed to evaluate stability fine oxide particles under the ion irradiation

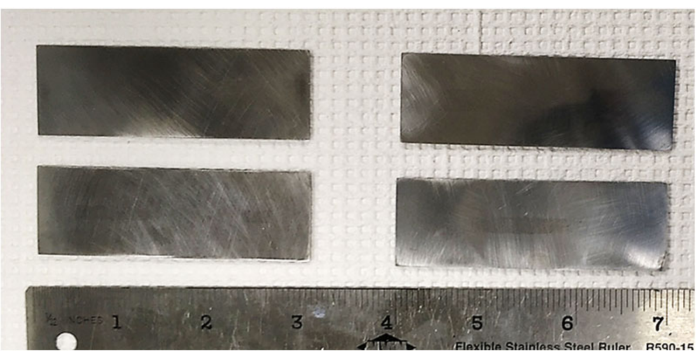
Ongoing Work for Tensile Testing of Cold Spray ODS Steel

Tensile tests for following cold spray ODS steel manufacturing conditions underway:

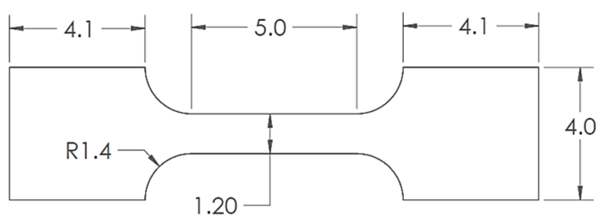
1. As-deposited
2. Heat treated at 900 °C at 1 hour
3. Heat treated at 1000 °C at 1 hour
4. Heat treated at 1100 °C at 1 hour

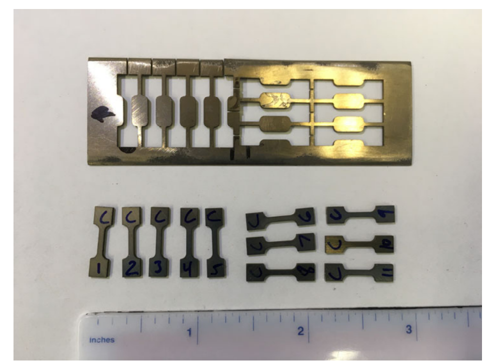


Samples have been EDM cut – tests to be performed at Cal Nano



Free standing ODS steel materials



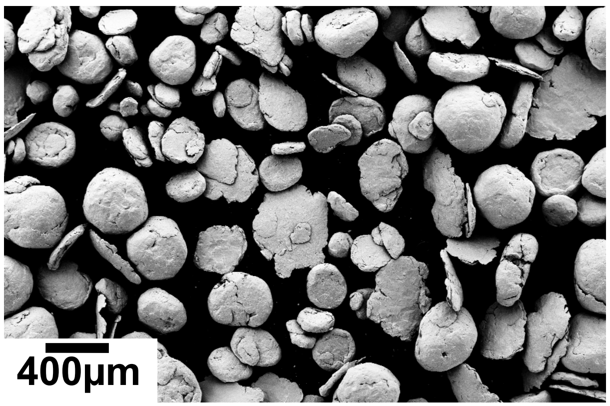


SSJ3 type tensile specimens

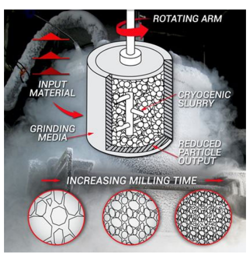
Advanced Processing Strategy

- Work shown so far has been performed using atomized powders
- The (atomized + ball-milled) powders are best because ball-milling further puts the Y, Ti, and O in solid solution which is good for subsequent precipitation of nanoscale oxide precipitates (these ball-milled powders have been supplied to us by collaborator LANL)
- But ball milled powders are too large – few hundred microns and also work hardened for cold spray
- Cryomilling to reduce ball-milled particle size was successful, but still had high work-hardening
- We have conducted annealing experiments to soften the cryomilled particles to make them sprayable (working with company Solar Therm in PA)
- This would lead to fine powders, with low hardness, and with microstructure identical to LANL's ball-milled powder

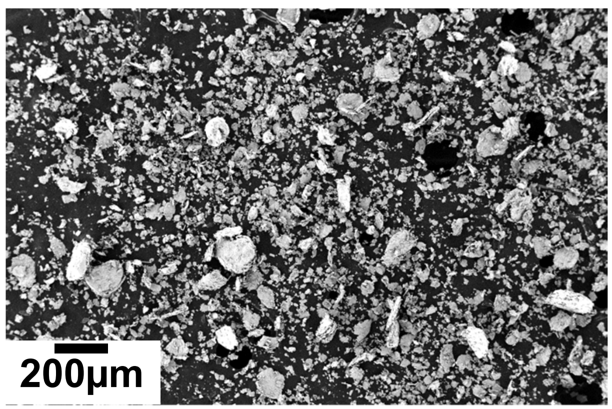
Use of LANL Ball-Milled Powders for Manufacturing ODS Cladding



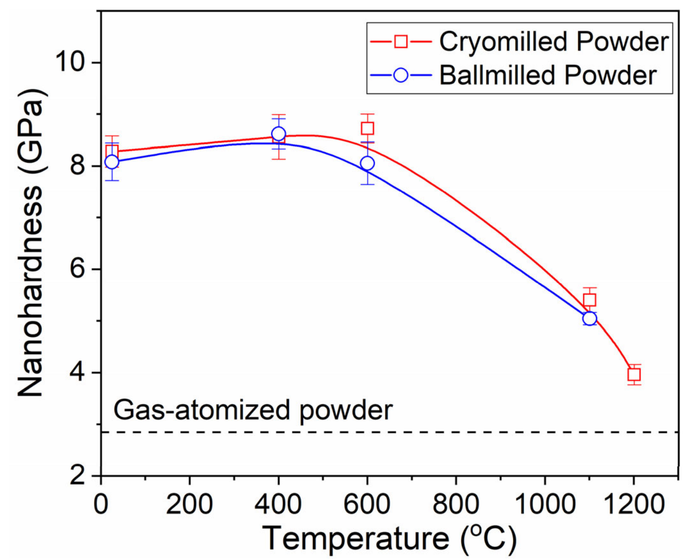
- **Powder size reduction (Cryogenic milling)**
- **Hardness reduction (Heat treatment)**



Cryo-milling in liquid N_2



Hardness reduction by heat treatment



Challenges:

- ***Powder oxidation***
- ***Powder agglomeration***

Alternative Route Involving Ball-Milled ODS Powders for Manufacturing ODS Cladding Tubes

Ball-Milled Powders from Partner, LANL
- Desired solutionized microstructure
- Particle size too large for cold spray
- Particle hardness too high for cold spray

→

Cryomilling of ball-milled powders
- Particle size reduced to suit cold spray
- Particle hardness same as ball-milled

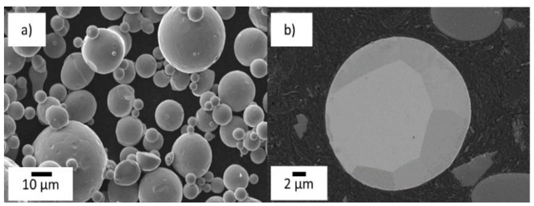
Annealing of cryomilled powders
- Particle hardness lowered and suited for cold spray
- Particle size suited for cold spray

→

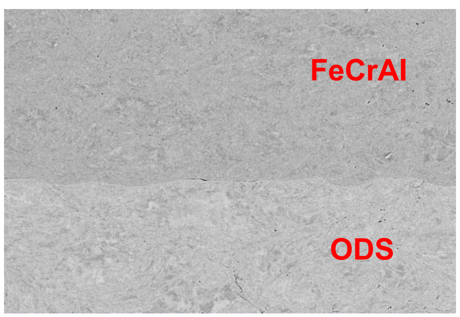
Cold Spray to form ODS tubes
- Best microstructure
- Best properties

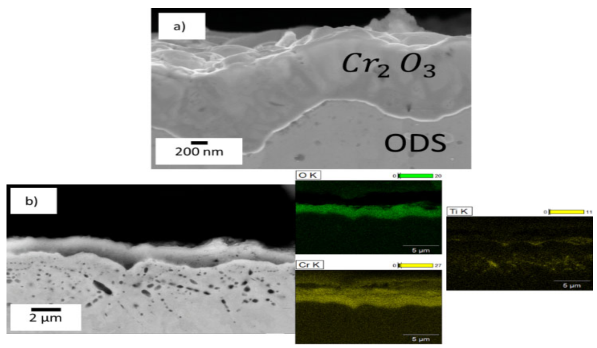
Cold Spray Coatings for Nuclear Energy Applications (FeCrAl Oxidation-Resistant Coatings)

The cold spray additive manufacturing process has a capability of produce compositionally (hence functionally) graded ODS steel across thickness direction

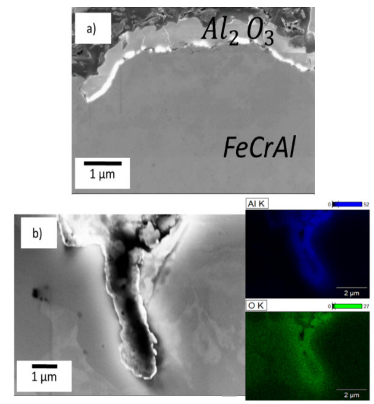


SEM images of FeCrAl powder





ODS:
~ 1 µm thick Cr oxide layer

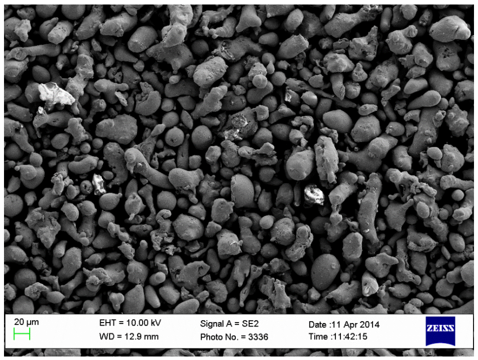


FeCrAl Coated ODS:
~ 150 nm thick Al oxide layer

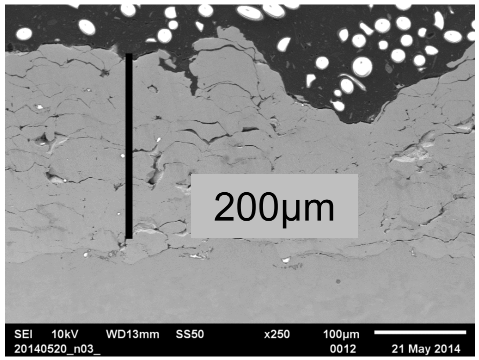
1100 °C for 1 hour oxidation tests

- Protective oxide layers developed on ODS and FeCrAl coated ODS
- No spallation of the FeCrAl coating from the ODS steel

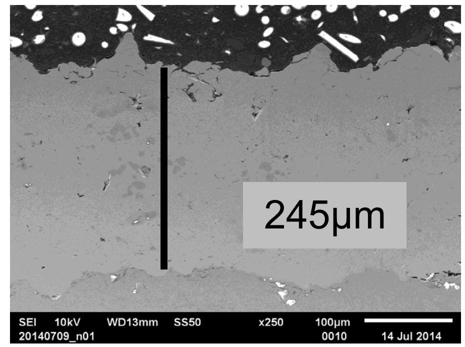
Inconel 600 (~72%Ni-15%Cr-13%Fe) Cold Spray Coatings



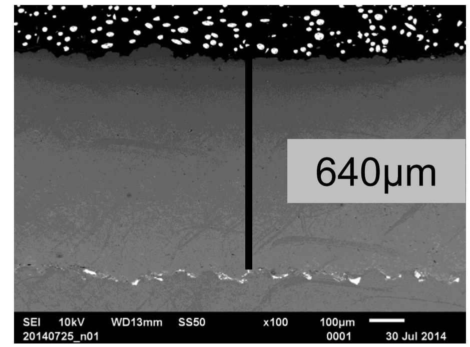
Powders (< 44um)



20um - 44um powder size (nitrogen)



< 20um powder size (nitrogen)



< 20um
<1% Porosity
(helium)

Smaller particles sizes lead superior and thicker coatings

Significantly superior coatings with helium

Ongoing Research

- Further investigation of cold spray process using annealed cryogenic milled ODS powder
- Examine mechanical properties of ODS steel materials produced by gas-atomized ODS powders
- Expand the solid-state additive manufacturing capabilities of cold spray process to investigate other coating materials for multiple applications

Issues and Concerns

- Delays encountered due to COVID-19 – University completely shut-down from mid-March to mid-June 2020.
- Students and researchers allowed into campus on a rotational basis under strict protocols
- Training of new students has been a challenge because of minimum 6 feet social distancing
- DOE-NE granted an automatic 1-year no-cost extension (very helpful)
- Some delays encountered for ‘small tensile sample’ testing, now resolved and working with Cal Nano for these tests

Project Impacts

- "Improving Deposition Efficiency in Cold Spraying Chromium Coatings by Powder Annealing", H. Yeom, T. Dabney, G. Johnson, B. Maier, M. Lenling, and K. Sridharan, **The International Journal of Advanced Manufacturing Technology**, 100(5), 2019, p. 1373.
- "A Novel Approach for Manufacturing Oxide Dispersion Strengthened (ODS) Steel Cladding Tubes using Cold Spray Technology", B. Maier, M.Lenling, H. Yeom, G. Johnson, S. Maloy, and Kumar Sridharan, **Nuclear Engineering and Technology**, 51, 4, 2019, p. 1069.
- "Manufacturing Oxide Dispersion Strengthened (ODS) Steel Fuel Cladding Tubes using the Cold Spray Process", M. Lenling, H. Yeom, B. Maier, G. Johnson, T. Dabney, J. Graham, P. Hosemann, D. Hoelzer, S. Maloy, and K. Sridharan, **Journal of Minerals, Metals, and Materials Society (JOM)**, 171, 8, 2019, p. 2868 (invited).
- "Development of Cold Spray Process for Manufacturing of Oxide Dispersion Strengthened (ODS) Steel Cladding Tubes", M. Lenling, H. Yeom, B. Maier, G. Johnson, T. Dabney, J. Graham, P. Hosemann, D. Hoelzer, S. Maloy, and K. Sridharan, **proc. Trans. 2019 American Nuclear Society Annual Conf., Minneapolis, MN**, 2019.
- "Investigation of Manufacturing Oxide Dispersion Strengthened (ODS) Steel Fuel Cladding Tubes using Cold Spray Technology", M. Lenling, H. Yeom, B. Maier, G. Johnson, K. Sridharan, P. Hosemann, D. Hoelzer, S. Maloy, and J. Graham, **The Metallurgical Society (TMS) Annual Conf., Symp. on Additive Manufacturing for Energy Applications, San Antonio, TX**, March 2019.
- "Novel Fabrication Route for Oxide Dispersion Strengthened (ODS) Steel Cladding Tubes using Cold Spray Technology", H. Yeom, M. Lenling, J. Graham, P. Hosemann, D. Hoelzer, S. Maloy, P. Grant, and K. Sridharan, **Materials in Nuclear Energy Systems (MiNES) Conference, Baltimore, MD**, October 2019.
- "Manufacturing Oxide Dispersion Strengthened (ODS) Steel Fuel Cladding Tubes by Cold Spray Technology", H. Yeom, M. Lenling, T. Dabney, J. Graham, P. Hosemann, D. Hoelzer, S. Maloy, and K. Sridharan, **The Metallurgical Society (TMS) Annual Conf., Symposium on Additive Manufacturing for Energy Applications II,** San Diego, CA, February 2020.
- Methods for Manufacturing Nanostructured and Compositionally-Tailored Tubes and Components by Low Temperature, Solid State Cold Spray Powder Deposition", patent filed 2020
- "Recent advances of cold spray technology in nuclear energy applications" H. Yeom and K. Sridharan, **Advanced Materials and Processes** (invited), July 2020.
- "Cold Spray Technology in Nuclear Energy Applications: A Review of Recent Advances", H. Yeom and K. Sridharan, **Annals of Nuclear Energy**, 150, 2021, 107835.
- "Evaluation of Oxide Dispersion Strengthened (ODS) Steel Fuel Cladding Tubes Manufactured by Cold Spray Technology", H. Yeom, V. Ramasawmy, M. Lenling, P. Hosemann, D. Hoelzer, S. Maloy, and K. Sridharan, **Symp. Additive Manufacturing for Energy Applications III, The Metallurgical Society (TMS) Annual Conf., Orlando, FL**, March, 2021.

Milestones and Deliverables for FY-20 and FY-21

- **Period December 2020 through September 2021**
  - Mechanical property testing data: July 2021
  - Cryomilling approach for ball-milled powders: May 2021
  - Development of coating treatments for cross-cutting applications in nuclear: September 2021
  - At least two more publications: September 2021
  - Final report: September 2021 + 3 months

Contact Information and Questions

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