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Advanced Sensors and Instrumentation

PNNL-SA-205622

Project: D9H60 Cobalt Magnetostrictive ElectroMagnetic Acoustic Transducer

Advanced Sensors and Instrumentation (ASI) Annual Program Webinar

November 4, 6-7, 2024

[Advisor/Project Manager: Bill Glass; Contributors; Morris Good, Nicholas Conway, Tianhao Wang

[Pacific Northwest National Laboratory

Project Overview

Purpose: Advanced reactors will be difficult or impossible to inspect or monitor with existing sensors and techniques. This project goal is to demonstrate feasibility of a high temperature permanently installed sensor that can monitor a significant pipe or vessel material volume for structural integrity.

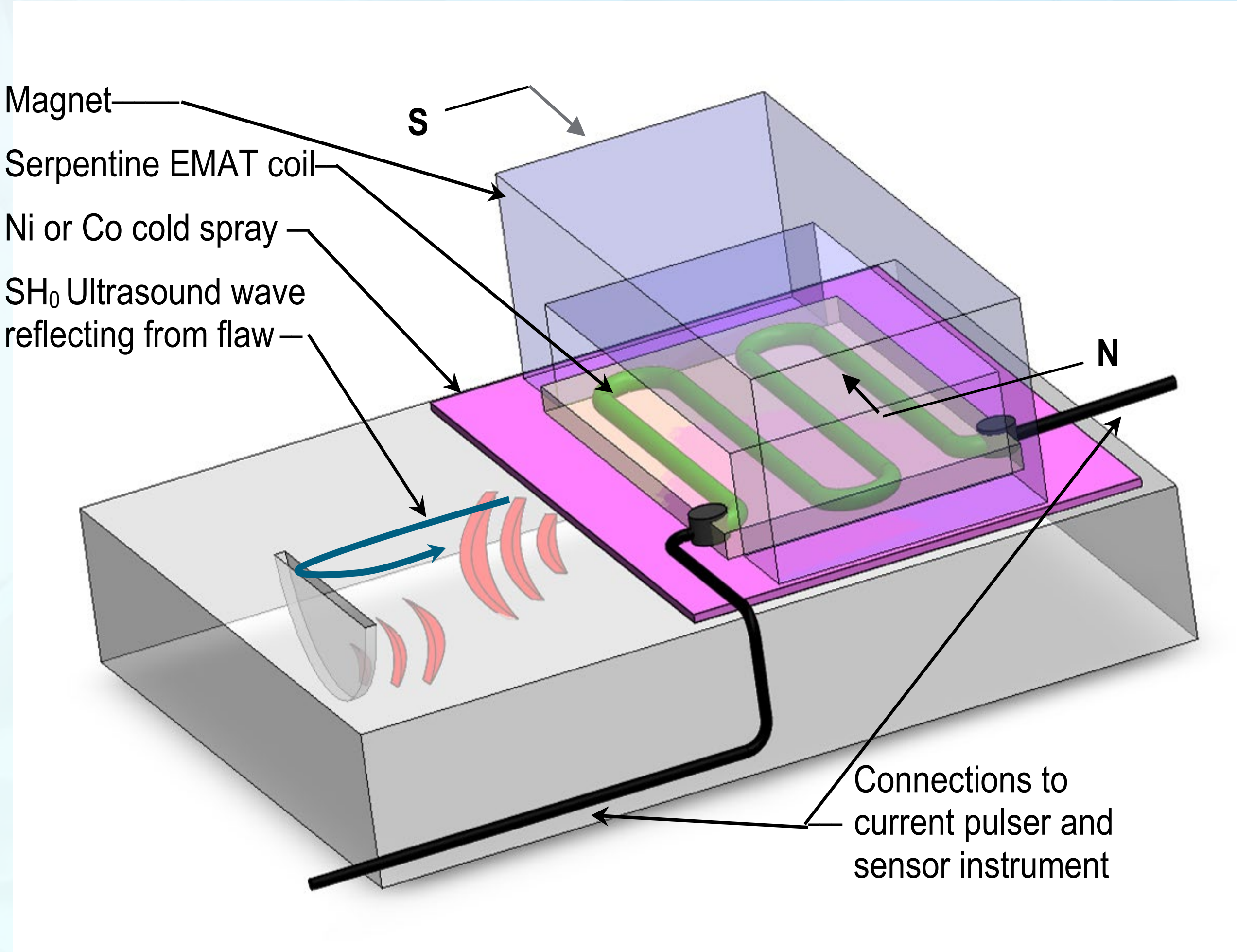
Objectives: Ni Magnetostrictive Electromagnetic Acoustic Transducers (MSEMAT) are efficient at generating guided wave ultrasound up to ~300 ^{o}C. This project is to design, build, and test a high temperature cobalt MSEMAT for higher temperature advanced reactors – first to 350 ^{o}C with a permanent magnet and then hopefully up to 800^{o}C with an electromagnet. The test program will show:

- Performance (S/N response to reference flaw) as temperatures ramp from 20^{o}C to 350^{o}C using a permanent AlNiCo magnet (no magnet wires). Note: Due to Contracts delays, purchased hardware was delayed, and due to surprise behavior of Co, in FY24, only permanent AlNiCo magnet EMAT was tested.
- Performance of electromagnet using Magnetostrictive Co. Target test will be to 500, 600, 700, and 800^{o}C. This part of the program delayed until FY25.

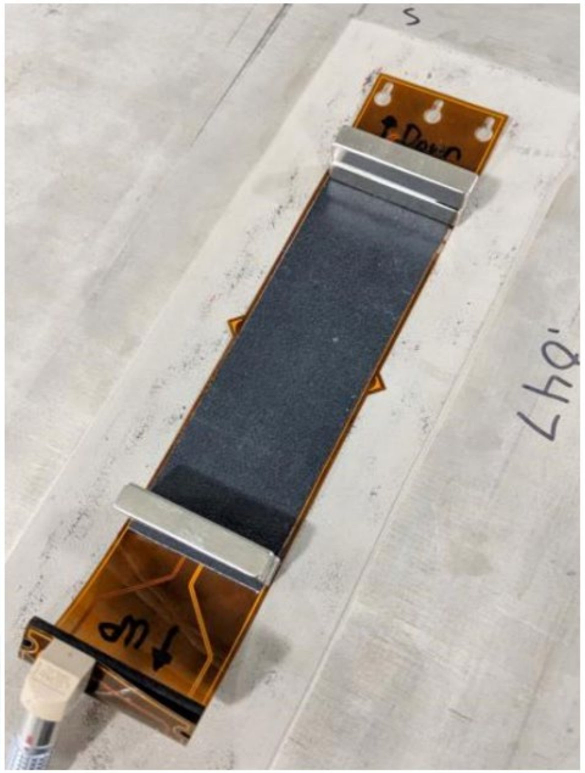
:

MS EMAT principle of opperation

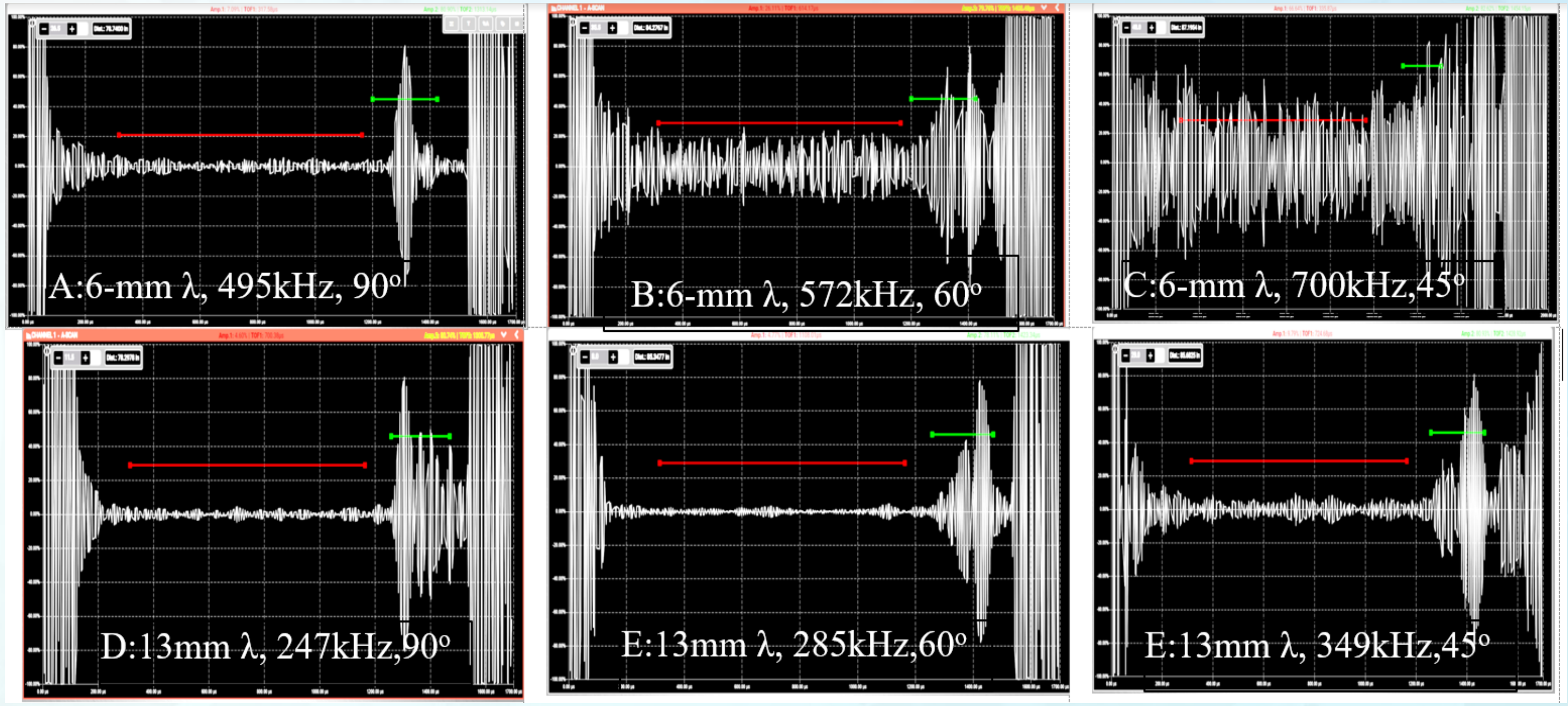
Magnetostrictive materials change shape in response to a change in the magnetic field. A magnetostrictive cold spray sensor configured as shown preferentially sends Shear Horizontal Waves perpendicular to coil and magnet N-S (Joule). These waves will reflect from anomalies (like cracks or welds or plate ends) and as they pass through the magnetic field, they produce a signal in the coil that may be sensed. (Villari).



Laboratory EMAT on Cold Spray Ni detected 25% TW 6mm dia. Flat Bottom Hole at a distance of 2-m



Laboratory EMAT with permanent magnet on Ni Cold Spray

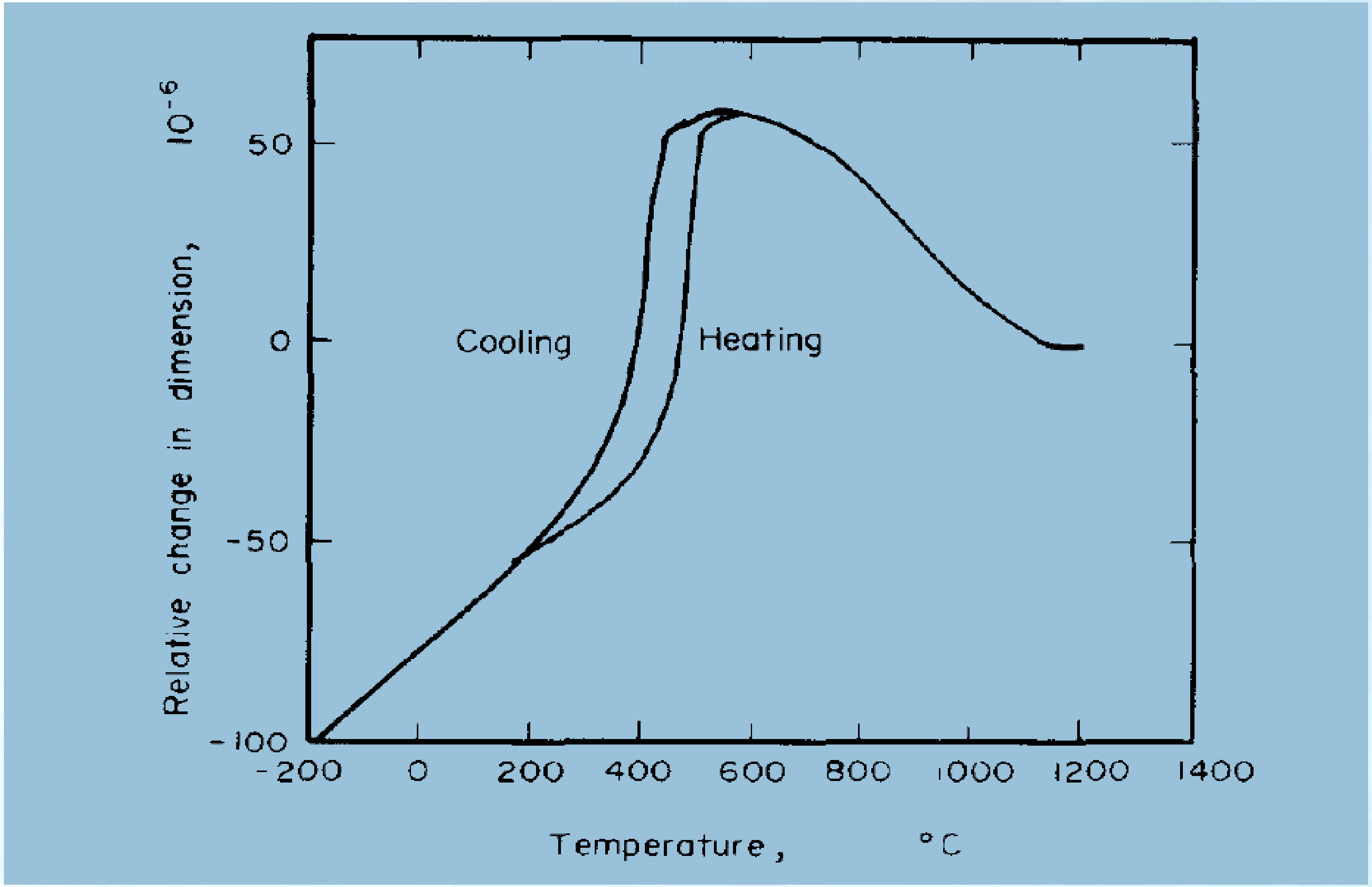


Response to 25% TW 6mm dia. Flat Bottom Hole at a distance of 2-m from sensor [S.W. Glass et. al. published in Materials Evaluation, October, 2024]

Literature references show Cobalt magnetostriction to be ~ 2x that of Ni

Material	Magnetostrictive Coefficient (ppm)	Curie Temperature °C	Reference
Nickel (Ni)	-34	354-360	(Legendre and Sghaier 2011)
Cobalt (Co) HCP@20°C	~-50 to -62	1121	(Kim and Yang 2018) (Britanica 2016) (Betteridge 1980)
Cobalt (Co) FCC @ 547°C	+50	1121	(Betteridge 1980)
Ferrous Cobalt (FeCo)	70-90	977-1227	(MacLaren et al. 1999); (Nolte, Severin, and Hoffmann 2000)
Galfenol	250	700	(Britanica 2016)
Iron	20-30	770	(Britanica 2016)
Steel	2-10	750-770	(Britanica 2016)

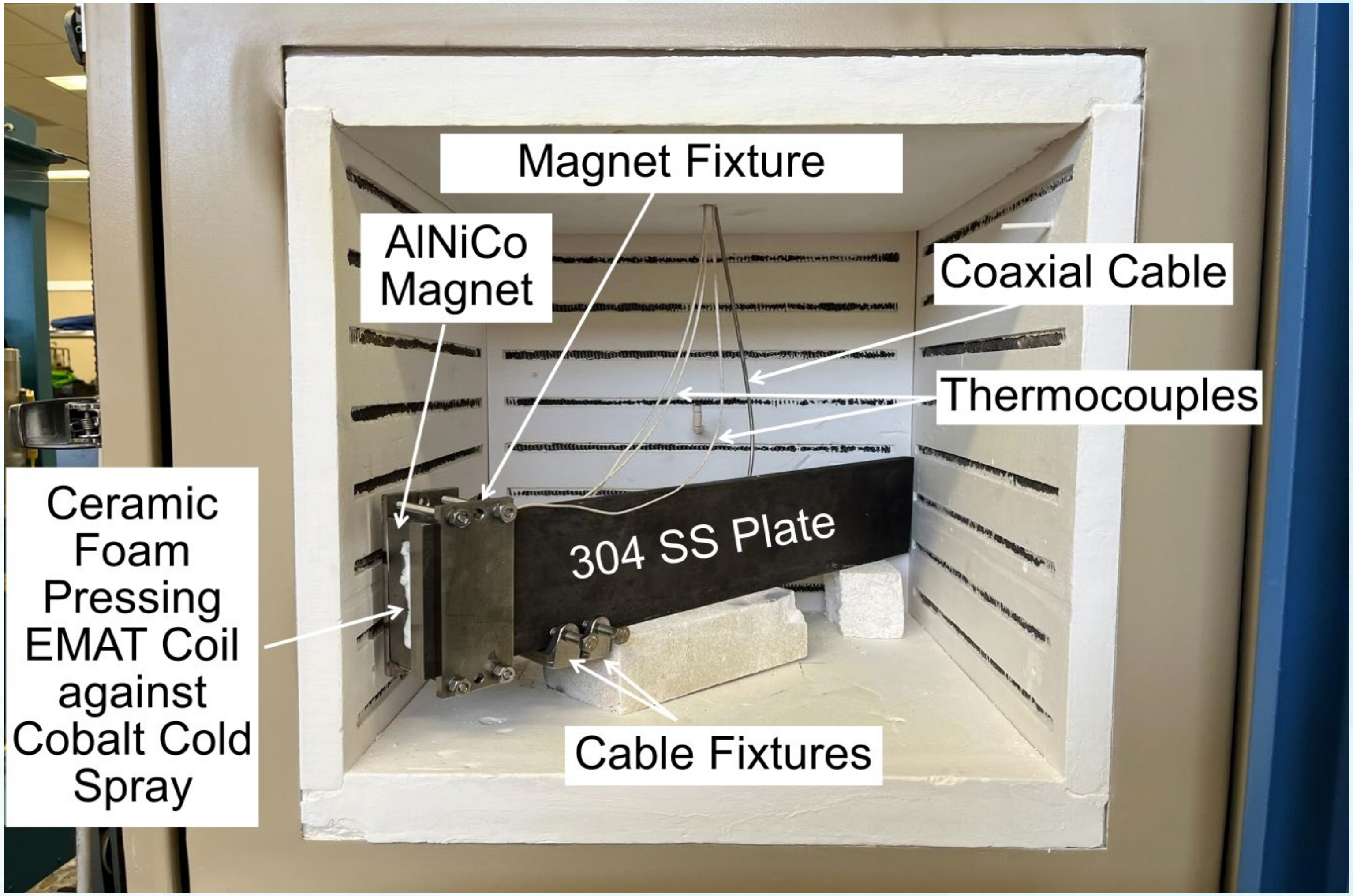
One reference shows magnetostriction of Co range from -100 to +55 as a function of temperature



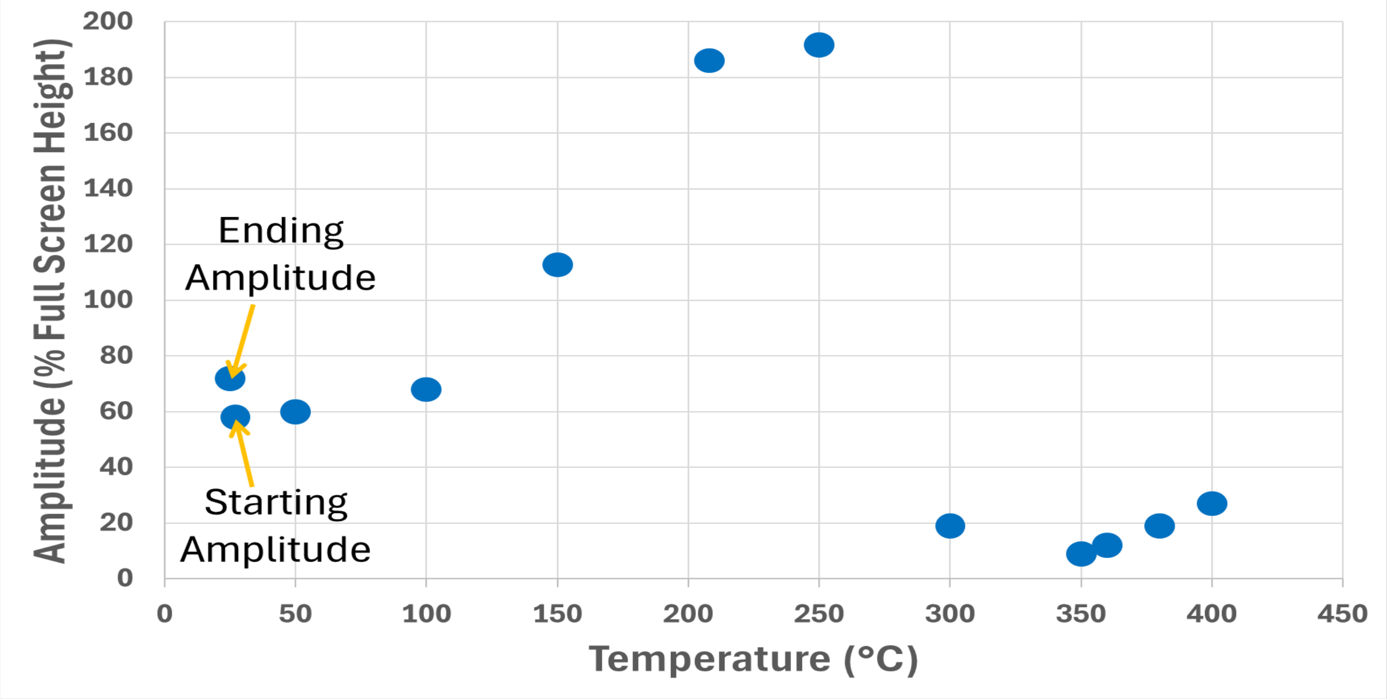
The Cobalt Suprise

- As-sprayed Co had little or no magnetostrictive response
- After thermal annealing (tested anneals to 500^{o}C and 600^{o}C), Co showed magnetostrictive response that was ~ 32dB less than Ni. (literature values expected +6dB greater than Ni. This would add a step to the cold spray and sensor process but this kind of thermal annealing is possible and regularly performed for weld stress relief.
- Co has two allotropic forms; a hexagonal-close-packed form (HCP), which is stable at temperatures below about 400° C (673 K), and a face-centered cubic form (FCC) that is stable at higher temperatures up to its melting point. At atmospheric pressure, Co undergoes its phase transformation from HCP to FCC at ~ 450-470^{o}C.
- Co seems to saturate more readily than Ni. SuCo magnets required liftoff of several inches. AlNiCo magnets (<20% field strength) had superiorEMAT performance.

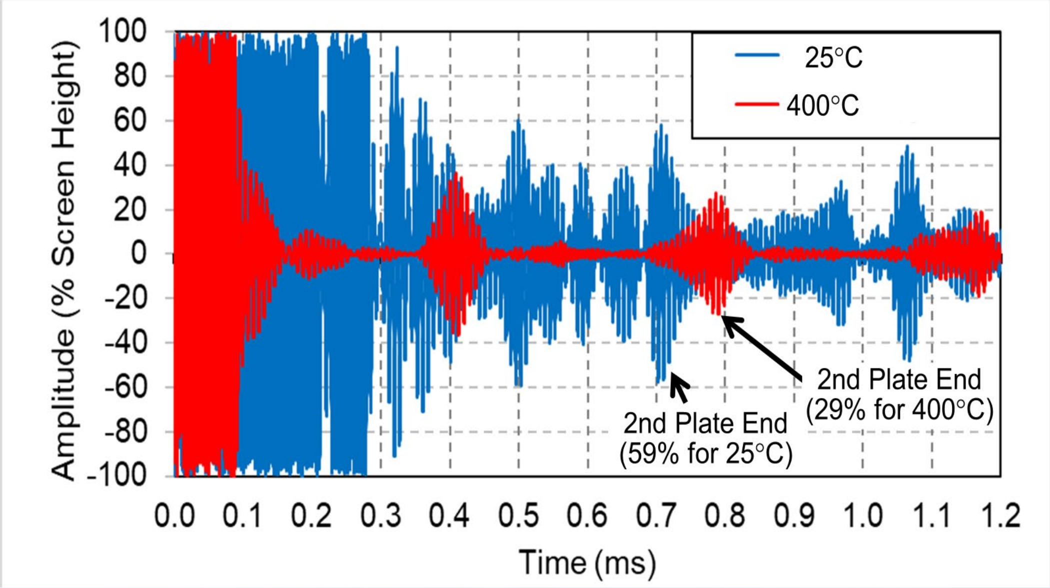
High Temperature Tests (to 400°C) were performed on Annealed Co Cold Spray with AlNiCo permanent magnet



MS EMAT response increased to 250^{o}C, dropped at 300-350^{o}C than continued to increase. Although the non-monotonic temperature response was not ideal, the plate end response at 400oC was 29% of room temperature response. (Yea!)



Second Plate-End response versus temperature (originating at 25^{o}C, increasing to 400^{o}C and returning back to 25^{o}C). The drop in amplitude from 250-300^{o}C is unexplained however the high temperature signal was clear with a similar signal/noise ratio at both 25 and 400^{o}C.



Room Temperature and Elevated Temperature He Cold sprayed Co Annealed to 650^{o}C and with an AlNiCo magnet EMAT previously heated to 400^{o}C

Continuing Work

- Electromagnet test to higher temperatures. (EM shown to be noisier with a response 25% of the room temperature AlNiCo magnet). Hopes are still high for high temperature(>500^{o}C) sensor response.
- Repeat the high temperature test of the AlNiCo magnet EMAT with silver wire. (the difficulty of inconel sheath high temperature wire and unexpected drop then continued rise of the EMAT response is unexplained, unexpected, and should be confirmed.
- Budget Permitting.
- EBSD micrograph to estimate phase distribution of annealed and unannealed Co.
- Make a revision-2 differential EMAT for lower noise sensor response (per INNERSPEC recommendation)?
- Would like to publish at ASNT research symposium (December Abstract, June Conference in Indianapolis).
- Submit entry to ASI sensor website.

Concluding Remarks/Questions/Comments

- The 400^{o}C response with the AlNiCo magnet showed a clear EMAT backwall response confirming feasibility of using cold spray annealed Co for an EMAT sensor.
- The electromagnet tests to date were noisier than the permanent magnet and the field strength was less but backwall responses are clear at room temperature and we are encouraged that the sensor should be able to go to 500^{o}C and higher. This will be verified soon.
- Direct all Questions/ Comments to:

Bill Glass; PNNL Advisor
Phone: 509 372 6190
Email: Bill.Glass@pnnl.gov
ORCID ID: 0000-0001-6723-4803

D9H60 Cobalt Magnetostrictive ElectroMagnetic Acoustic Transducer

OVERVIEW

Purpose: Advanced reactors will be difficult or impossible to inspect or monitor with existing sensors and techniques. This project goal is to demonstrate feasibility of a high temperature permanently installed sensor that can monitor a significant pipe or vessel material volume for structural integrity.

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- Performance (S/N response to reference flaw) as temperatures ramp from 20°C to 350°C using a permanent SmCo magnet (no magnet wires). #
- Performance of electromagnet to 500°C. #

IMPACT

Logical Path:

- Design the sensor based on Ni experience at room temperature but using materials that can withstand higher temperatures and that can be tested in the available oven.
- Identify magnetic material and high temperature wire vendors to fabricate critical components (magnet, coil, connecting wires) and order components. (Long leads)
- Assemble the EMAT – first with a permanent magnet (PM) good to 350°C.
- Test the PM EMAT - ramp up to temperature and a long-term soak @ 350°C.
- Assemble the Electromagnet (EM)EMAT that hopefully will function to 500°C.
- Test the EM EMAT - ramp up to temperature and a long-term soak @ 500°C.

Expected outcomes are:

- The sensor response will degrade with temperature but still should be sufficient to see the test flaw.
- The PM EMAT will weaken due to well-understood material behavior as the magnet temperature exceeds 350°C. The test must stay below that temperature.
- The EM EMAT will fail above ~800°C due to well-understood material behavior. The test must stay below that temperature.

DETAILS

Principal Investigator: Bill Glass; Team: Andy Casella, Morris Good, Kevin Gervais, Yanming Guo.

Institution: Pacific Northwest National Laboratory

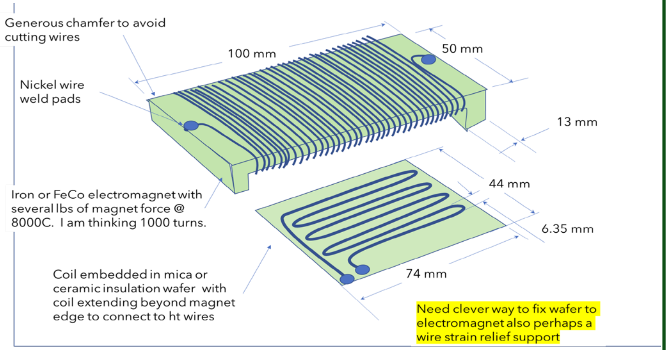
Duration: 2 years **Total Funding Level: $4**00,000

TPOC: Chris Petrie

Federal Manager: Dan Nichols

Workscope: 3.01 Crosscutting Technology Development (CT)

PICS:NE Workpackage #:
CT-24PN070208



RESULTS

Results to date:

- As sprayed Co shows suprisingly weak MS response. Heat annealing significantly improves the MS response. Suprisingly, the response is weaker than Ni. AlNiCo permanent magnet on the annealed Co sample plate showed good backwall to 400oC.

Accomplishments:

- AlNiCo magnet EMAT on annealed Co sample tested successfully to 400oC.
- Electromagnet tested at room temperature.
- XRD tests showed a distribution of 2 Co phases after annealing but results were inconclusive for unannealed.

Upcoming Tasks:

- Perform high temperature tests with permanent magnets and with electromagnets.

Cost Variance:

NA

Thank You

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Clean. Reliable. Nuclear.

Embedding Fiber Optic Sensors in SS316 Wrought Products Through Confined Rolling

Period: FY24

PI: Vineet V. Joshi

Rajib Kalsar, Lei Li, Shivakant Shukla,

Mark Rhodes, Ayoub Soulami, and Ali Zbib

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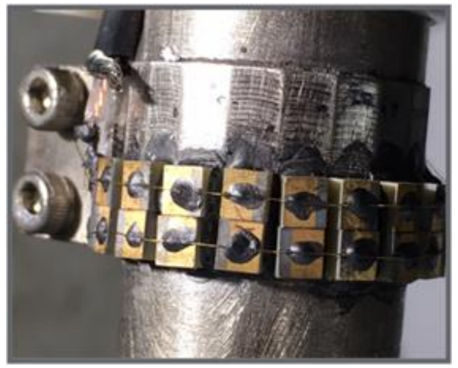
Outline

- Current State of the Art
  - Why Wrought Products?
- Our Approaches: Concept of Confined Rolling
- Approach 1:
  - Analytical Equations
  - Modeling
  - Experiments
  - Summary
- Approach 2:
  - Analytical Equations
  - Modeling
  - Experiments
  - Summary
- Post Fabrication Testing
- Next steps

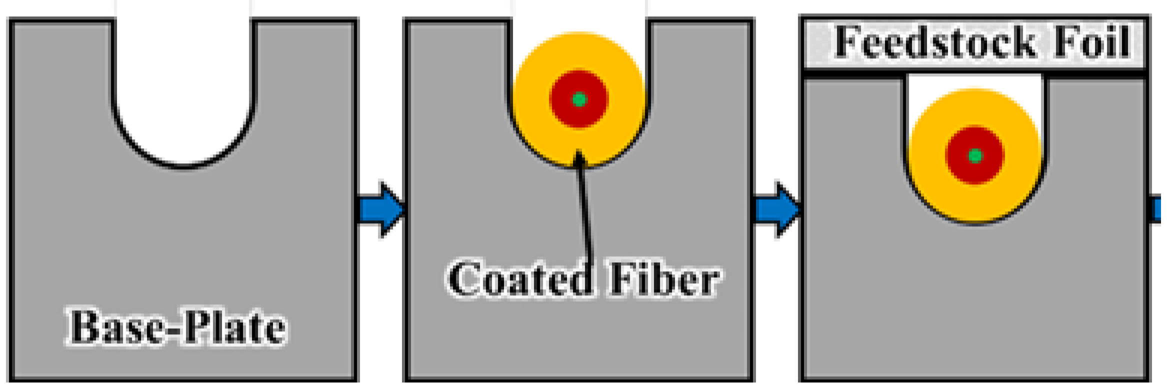
Current State of the Art and Concepts

Embedding sensors in wrought products has always been desired for a wide range of applications, especially, in harsh conditions to detect off normal events.

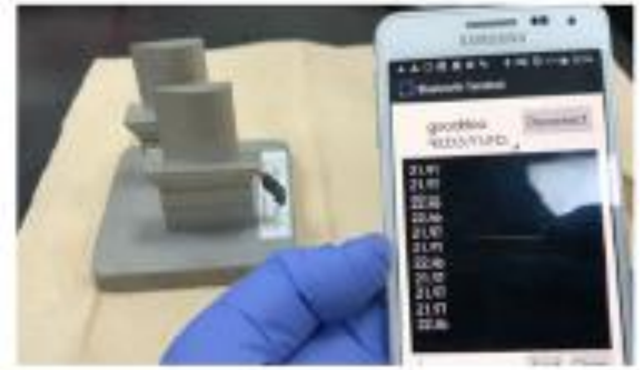
External mount



Welding



Additive Manufacturing

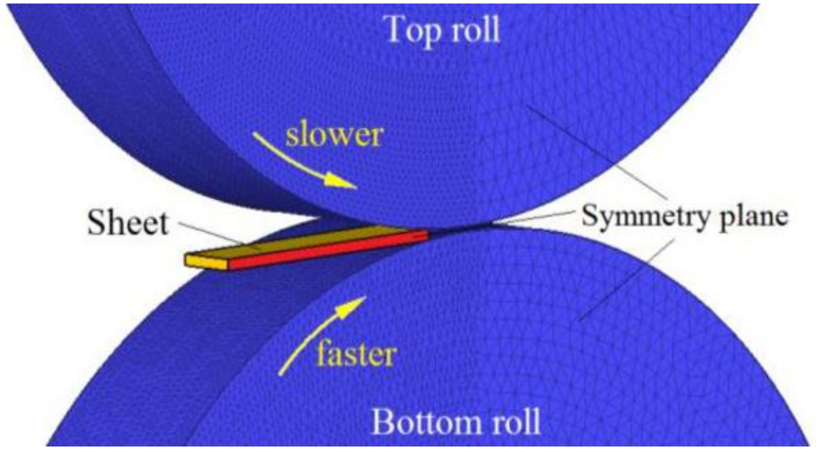


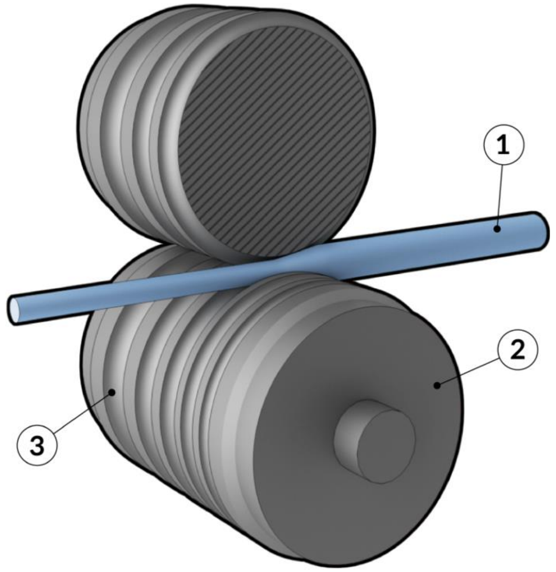
- Sensors are glued/joined on external surfaces
  - ➢ Limited applications in harsh environments, looses integrity
- Drilling holes and embedding sensors and welding plates
  - ➢ Component looses structural integrity, and welded structure is not suitable in harsh environments
- Embedding sensors in metal additive components
  - ➢ Component has as-cast microstructure

Wrought products are heavily used in nuclear and harsh environments

References
1. Functional fiber-optic sensors embedded in stainless steel components using ultrasonic additive manufacturing for distributed temperature and strain measurements, Additive Manufacturing 52 (2022) 102681.
2. Embedding sensors using selective laser melting for self-cognitive metal Parts, Additive Manufacturing 33 (2020) 101151.

Challenges with Embedding Sensors in Wrought Products





- Conventional processes such as rolling, forging, extrusion and pilgering introduce large plastic deformation and stresses
- These products are cheaper and faster to make
- Known technologies and products properties are well understood

Preserving the structural integrity of the sensor

Systematically understanding the nature of stress and manipulating the deformation mechanism we can place/ embedded sensors in wrought products

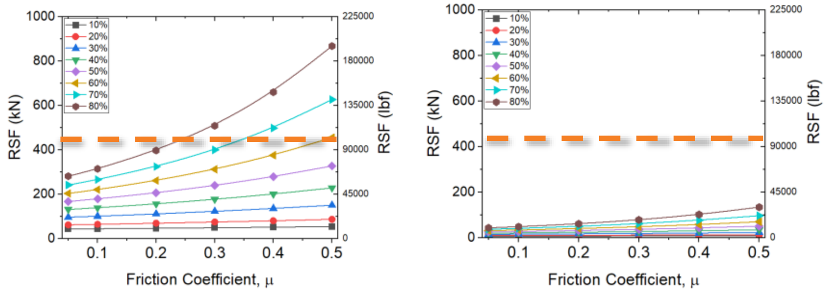
Approach: *Minimize the Number of Experiments*

Analytical Calculation

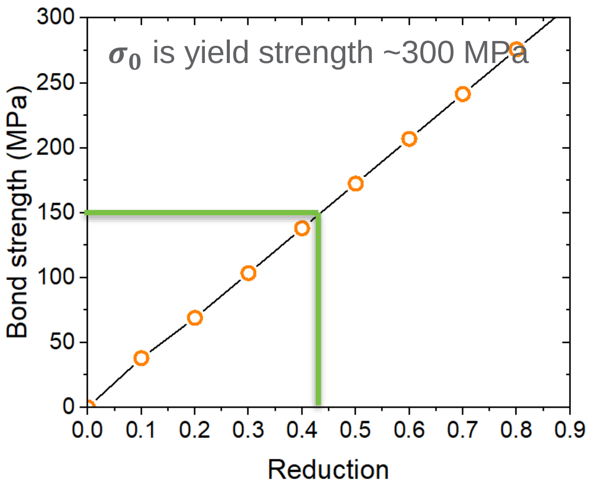
- Roll separation force

RSF @RT

RSF@1000°C



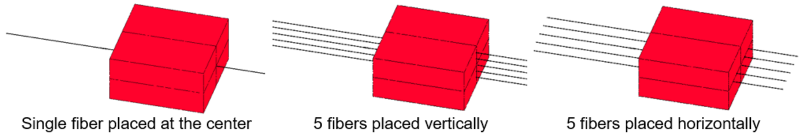
- Roll bonding criteria



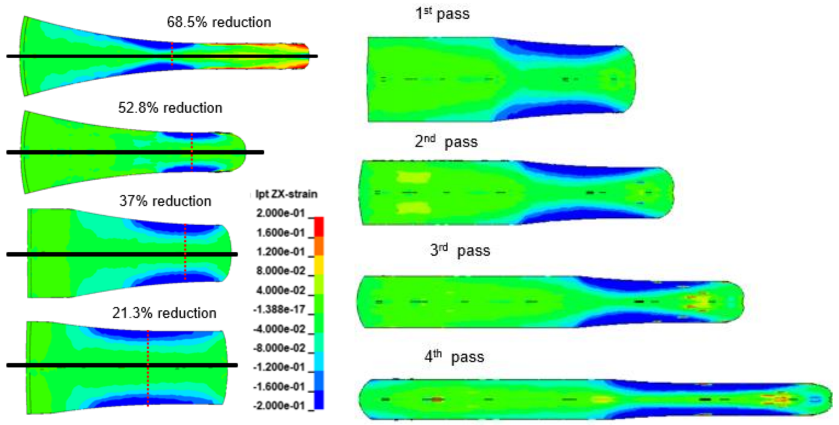
~43% reduction needed to have bond strength of ~ $\frac{1}{2}\sigma_0$

FEM Modeling

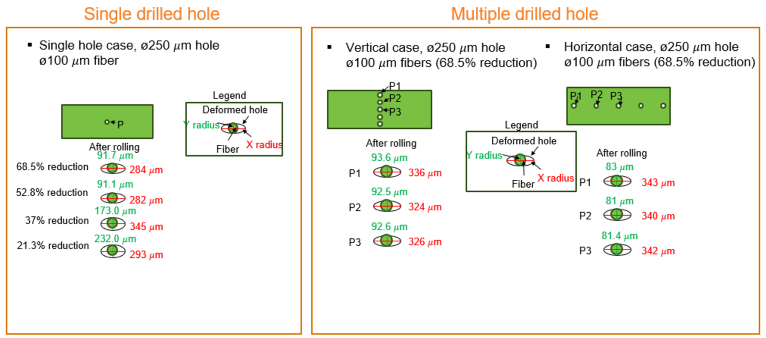
- Fiber location



- Single vs Multi pass strain

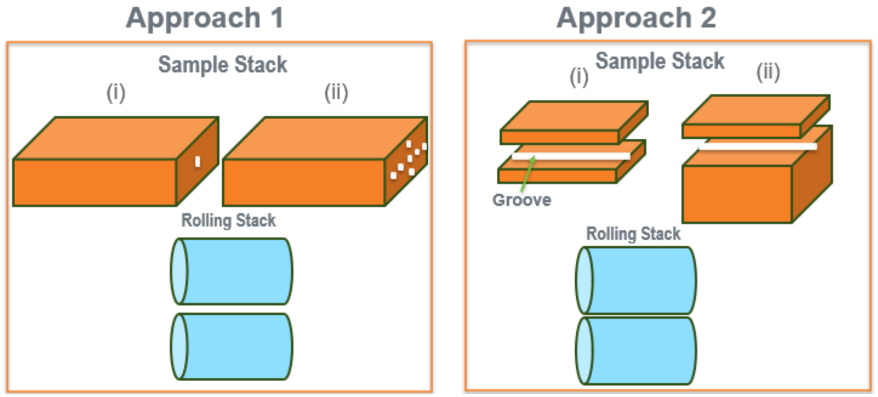


- Channel collapse



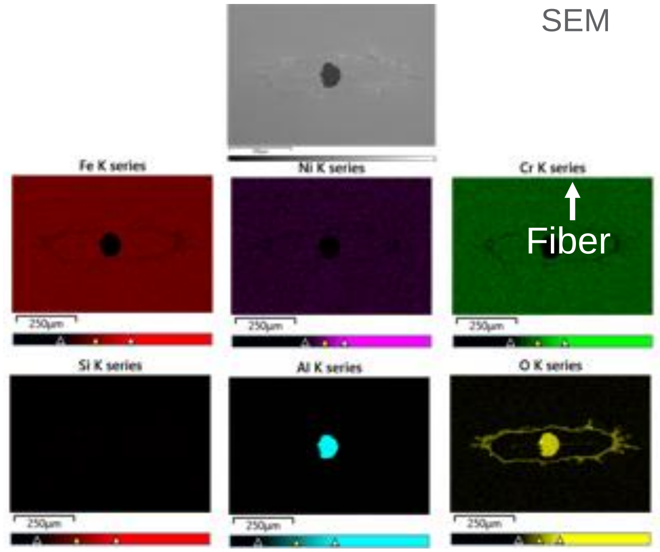
Experimental

- Rolling configuration



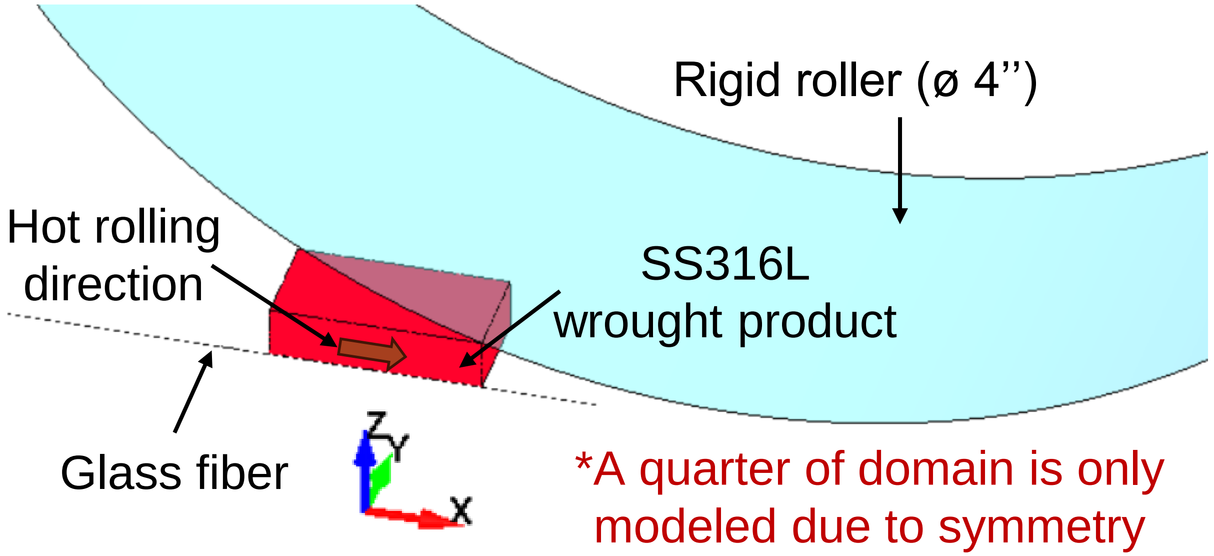
- Microstructure (Approach 1)

SEM



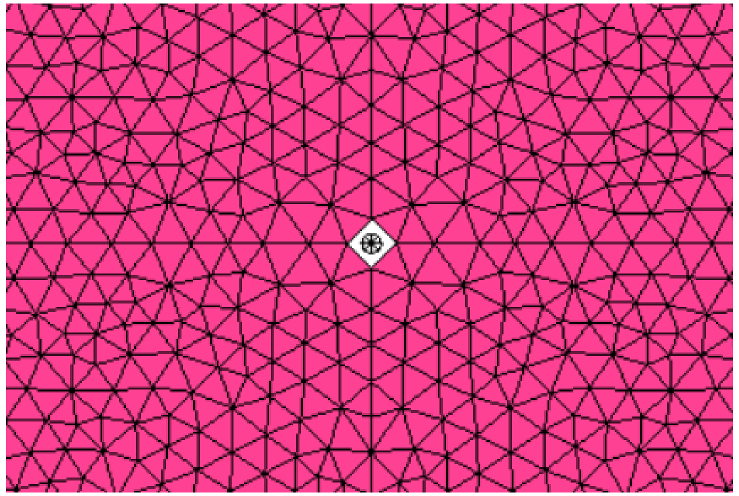
Finite Element Model Setup for Hot Rolling of Wrought Product with Fiber Embedded

- Finite element method (FEM) model setup

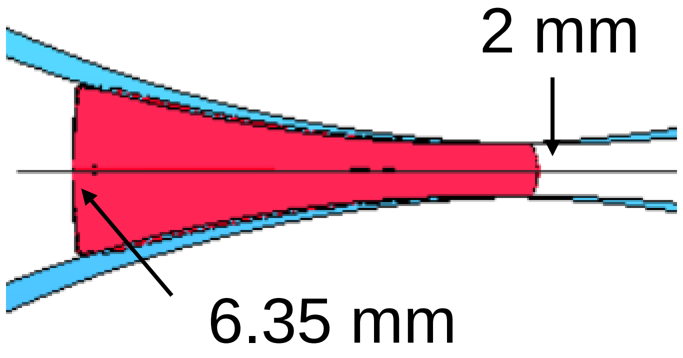


*A quarter of domain is only modeled due to symmetry

- ø250 μm drilled hole to hold a ø100 μm fiber

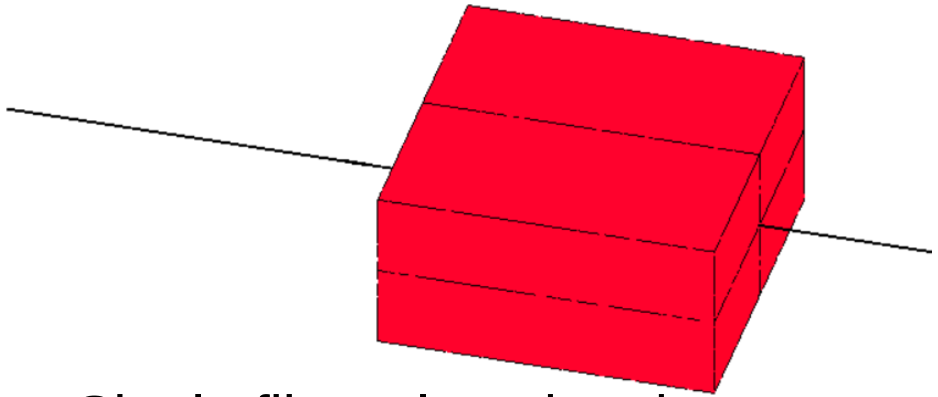


- Baseline reduction rate: 68.5%

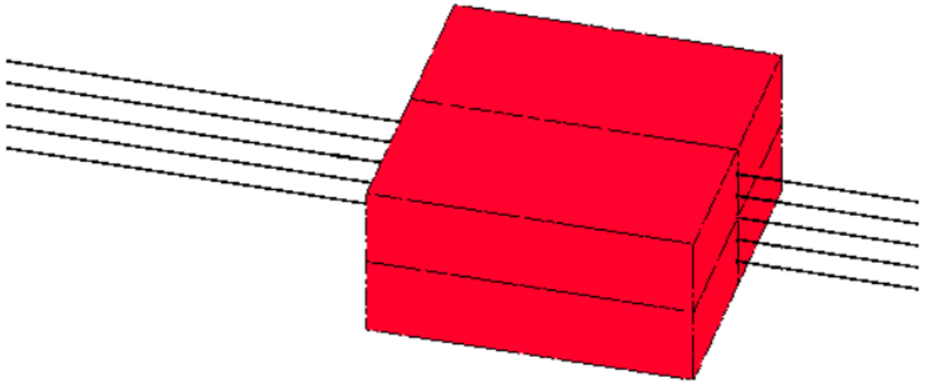


- Fiber embedding cases with wrought product sized in 0.5'' x 0.5'' x 6.35 mm

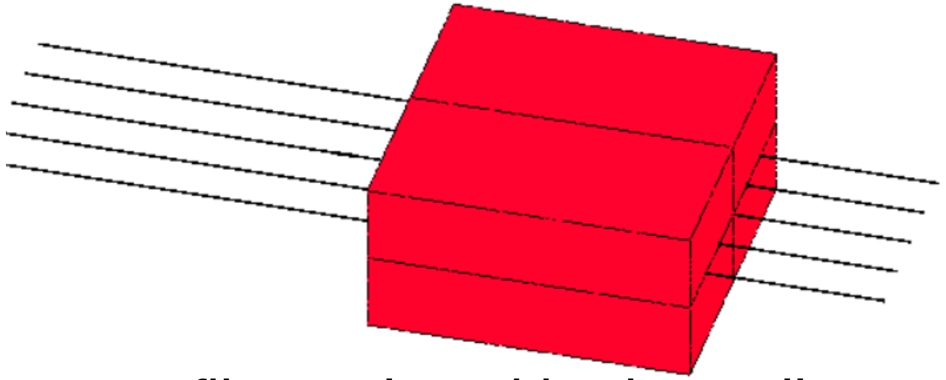
Al_2O_3 fiber: 100 µm



Single fiber placed at the center



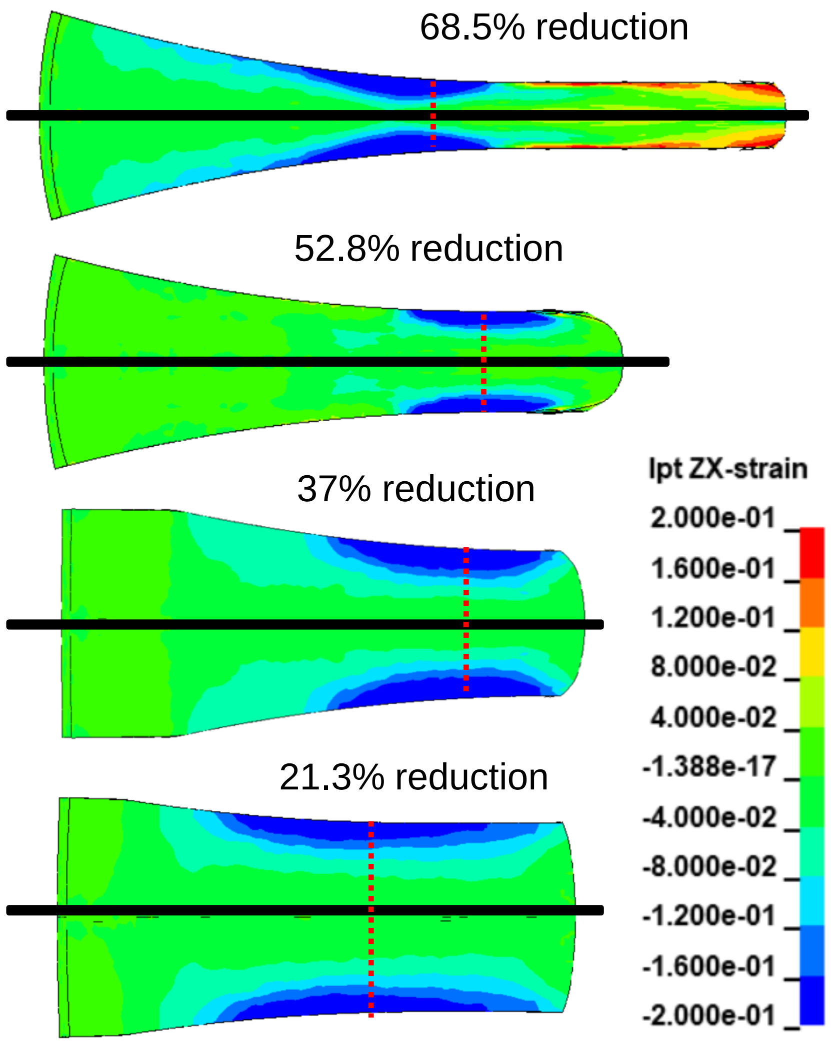
5 fibers placed vertically



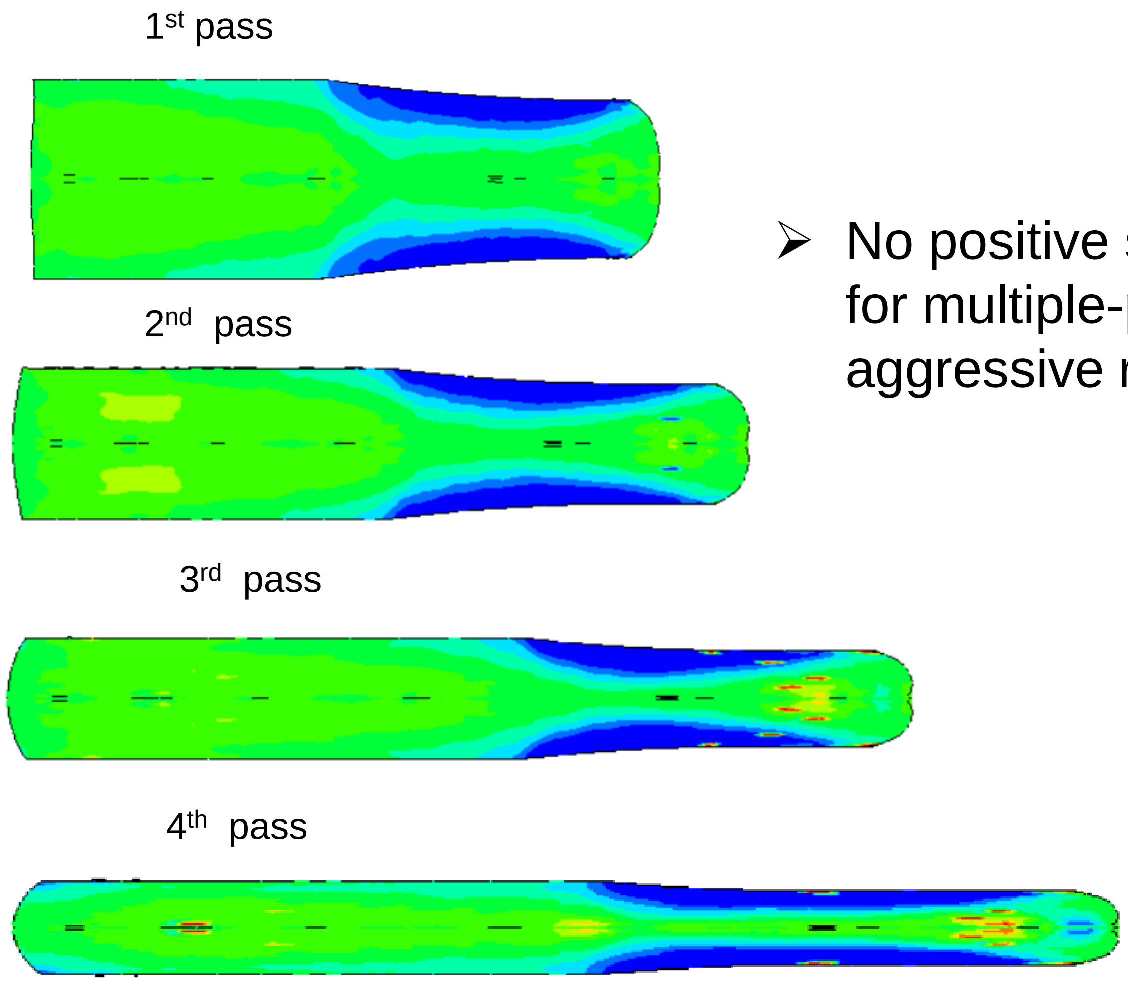
5 fibers placed horizontally

Through Thickness Shear Strain:
Single vs Multi pass

- Single pass with different reductions



- Multiple passes with 21% reductions

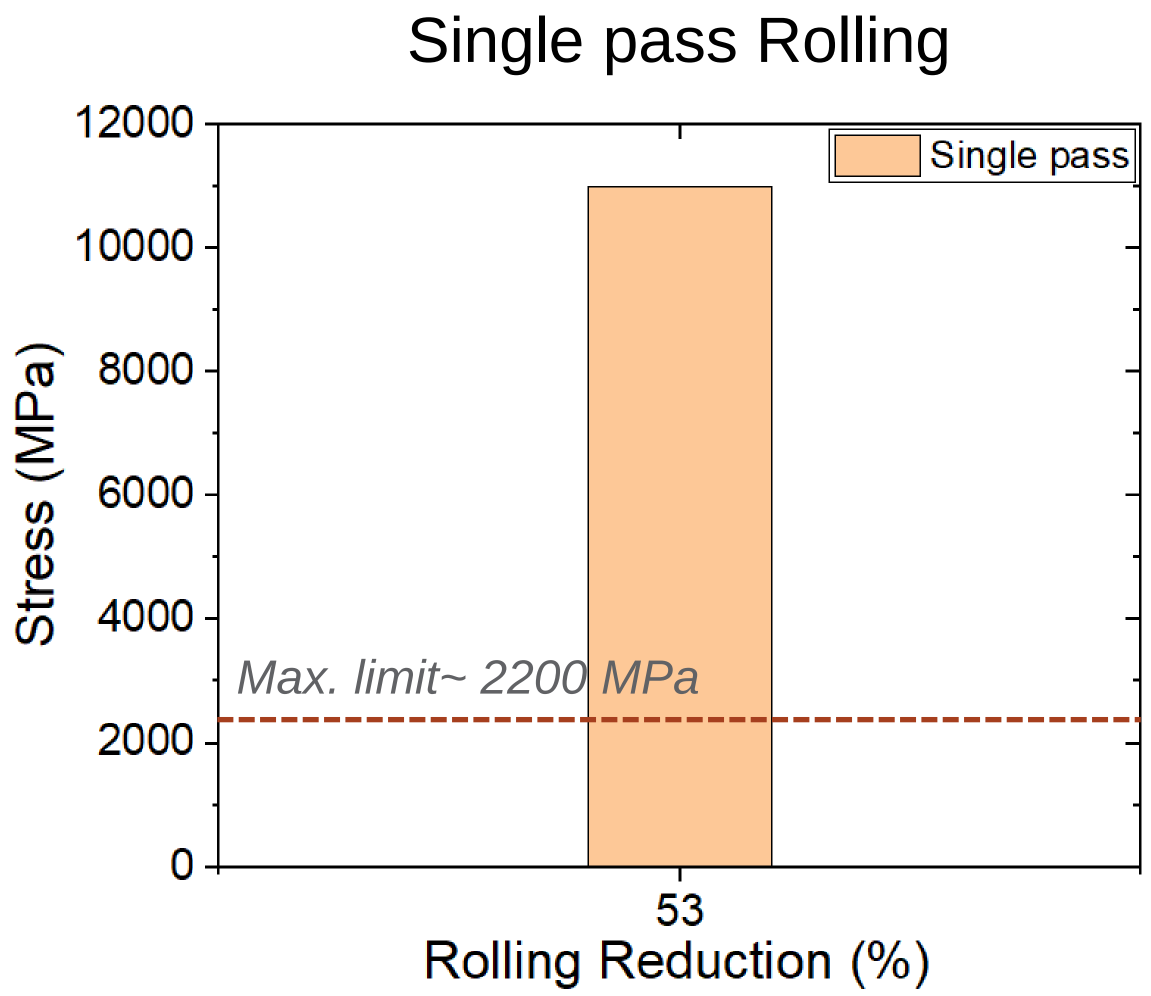


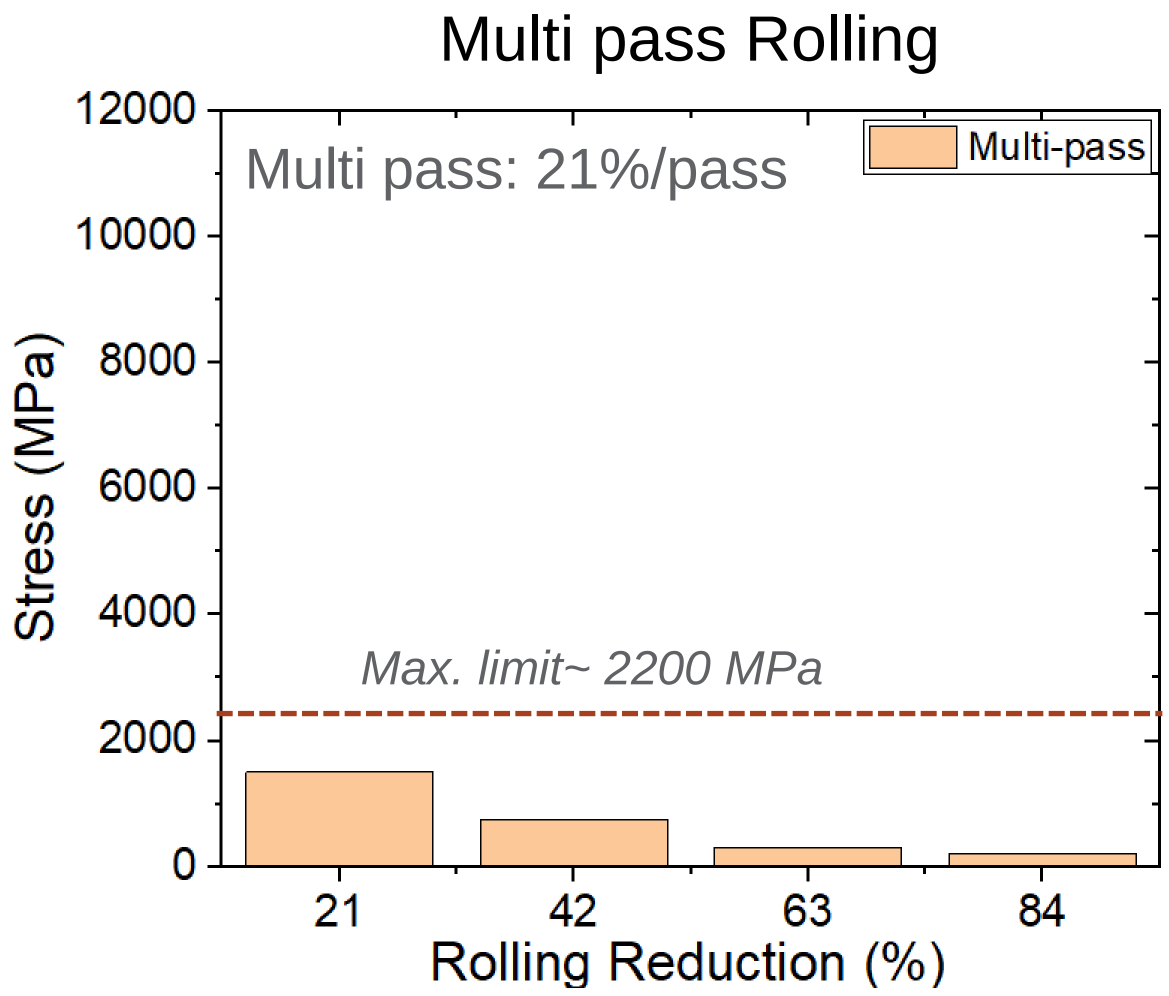
➢ No positive shear strain is obtained for multiple-pass case due to less aggressive reduction for each pass

➢ Rolled at 850°C

Fiber experienced stressed:
Single vs. Multi-pass Rolling

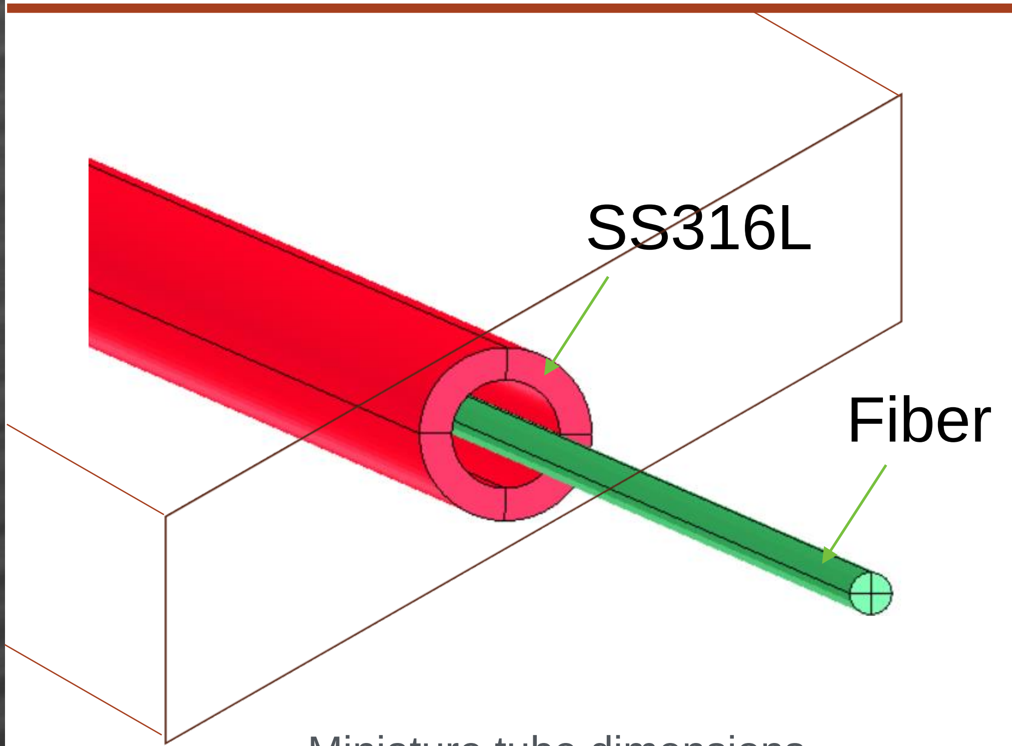
- Sapphire (Al_2O_3) fibers experienced maximum stress during rolling (Single vs Multi pass)



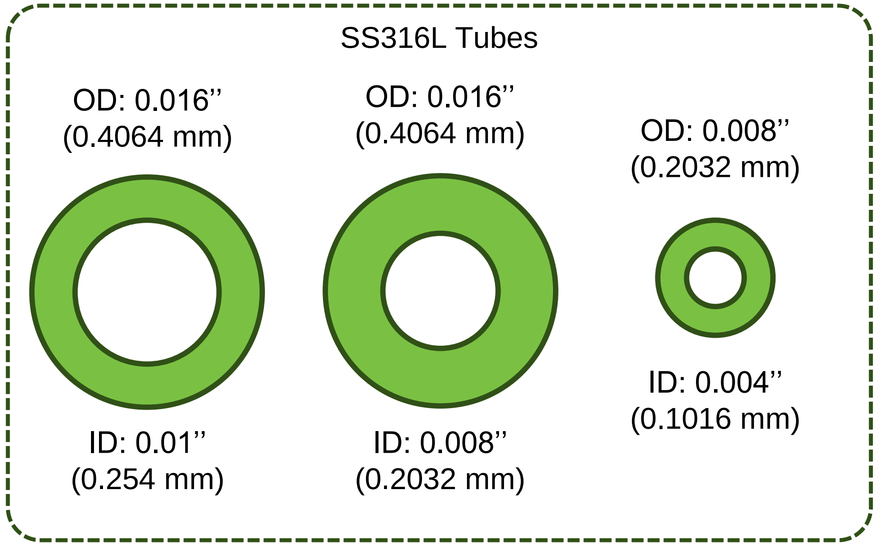


- Multi-pass rolling less aggressive and adopted for initial trials
- Dividing the reduction into multiple passes can significantly reduce the stress in the fiber.

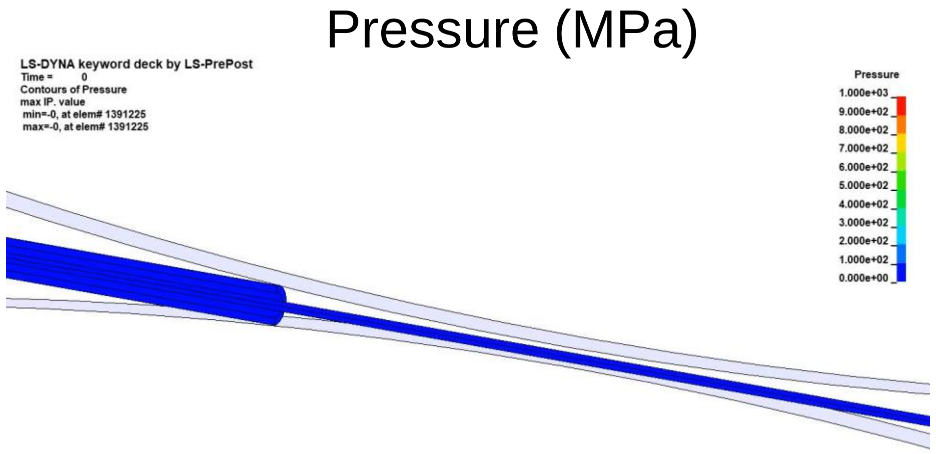
Model with Guided Tube:
For better adherence of fibers and matrix

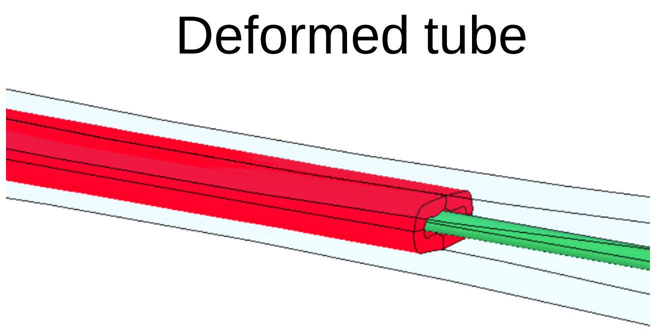


Miniature tube dimensions



50% reduction



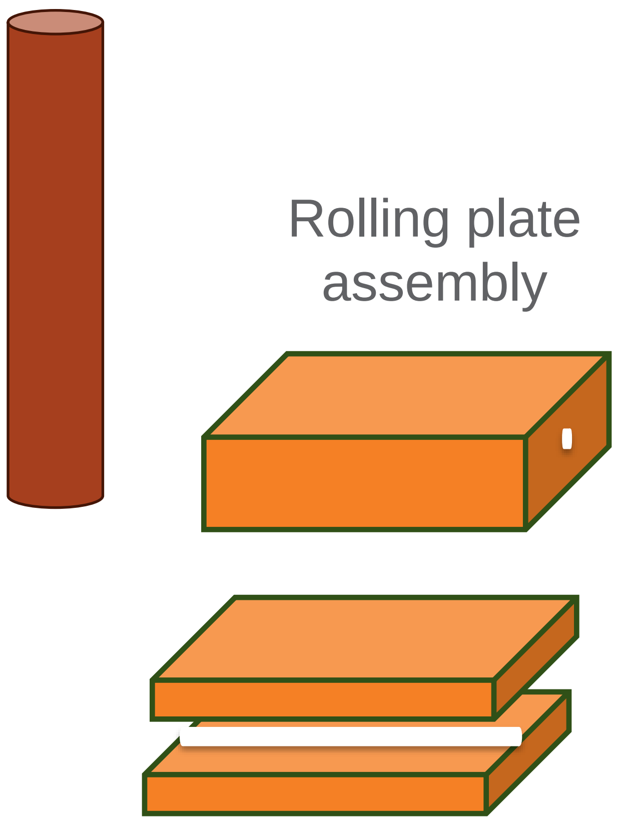


Insertion of 316 tube along with fiber in 316 matrix is beneficial for bonding and reduces stress

Approach 1: Fiber Assembly

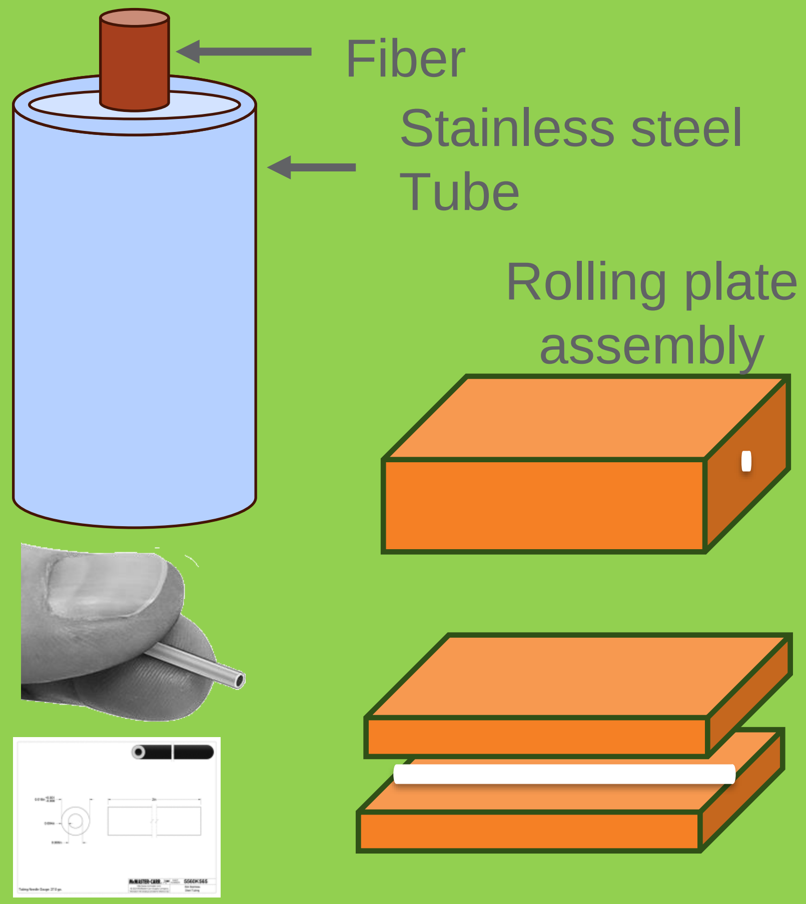
Fiber Assembly I

Bare fiber



Fiber Assembly II

Fiber inserted into miniature tube

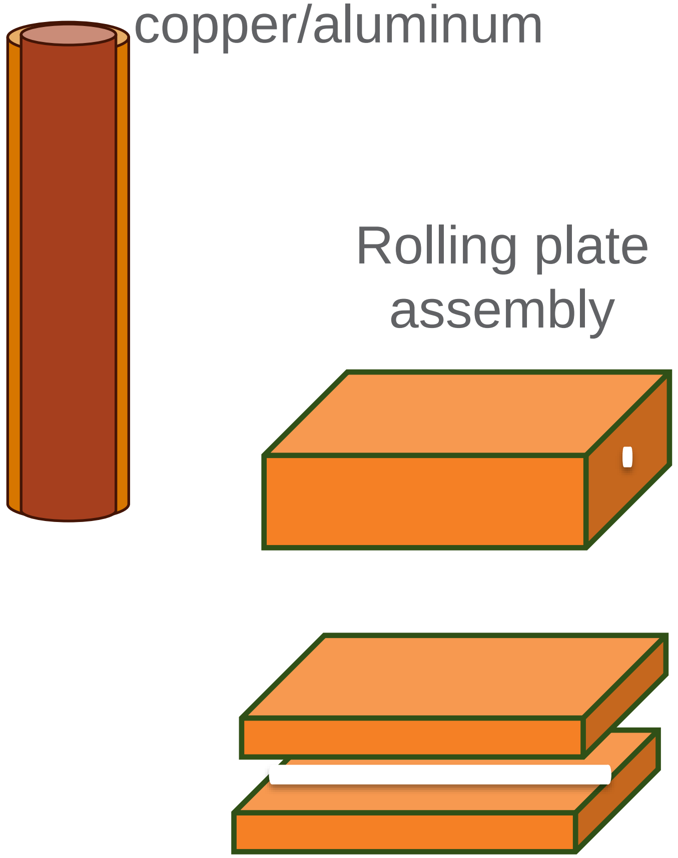


Tube outer diameter: 0.4 mm

Inner diameter: 0.25 mm

Fiber Assembly III

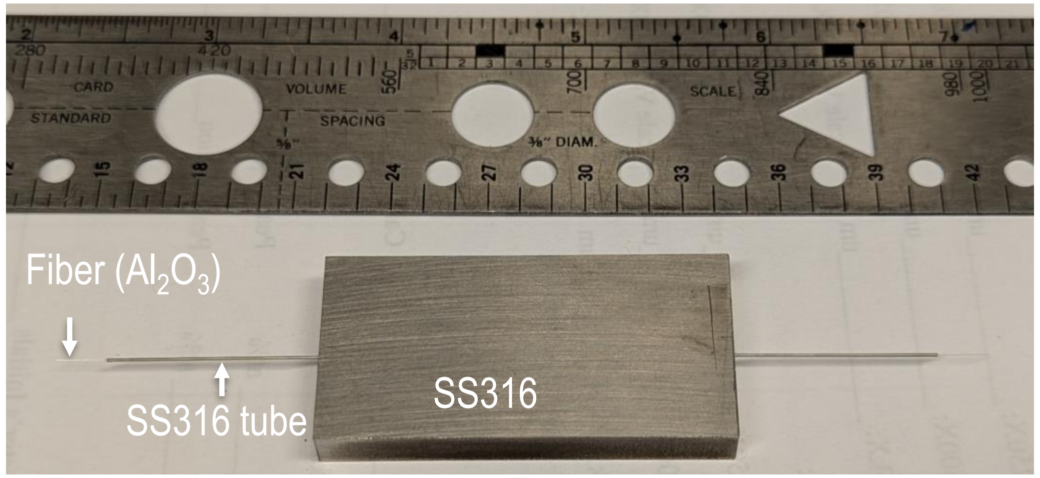
Fiber coated with copper/aluminum



Approach 1
Multi-pass Rolling: Up to 20% Rolled
(6.2 mm to 5.0 mm)

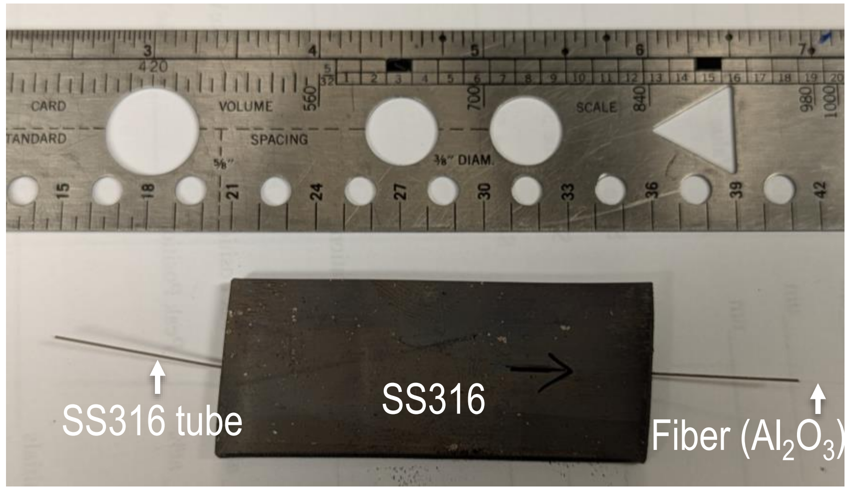
Embedded fiber:
- Rolling temperature: 900°C
- 5 min soaking time
- Total reduction: 20% (10%/pass)

Starting assembly

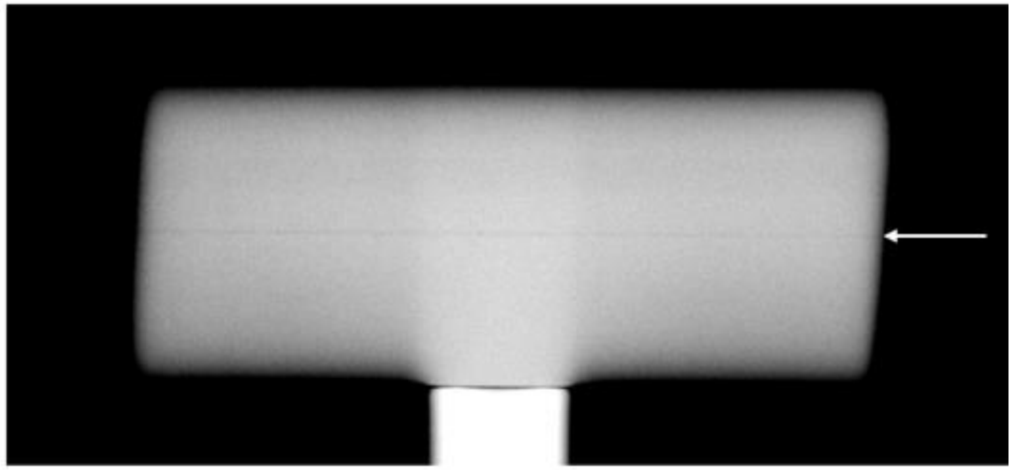


10% Rolled

20% Rolled



Radiography

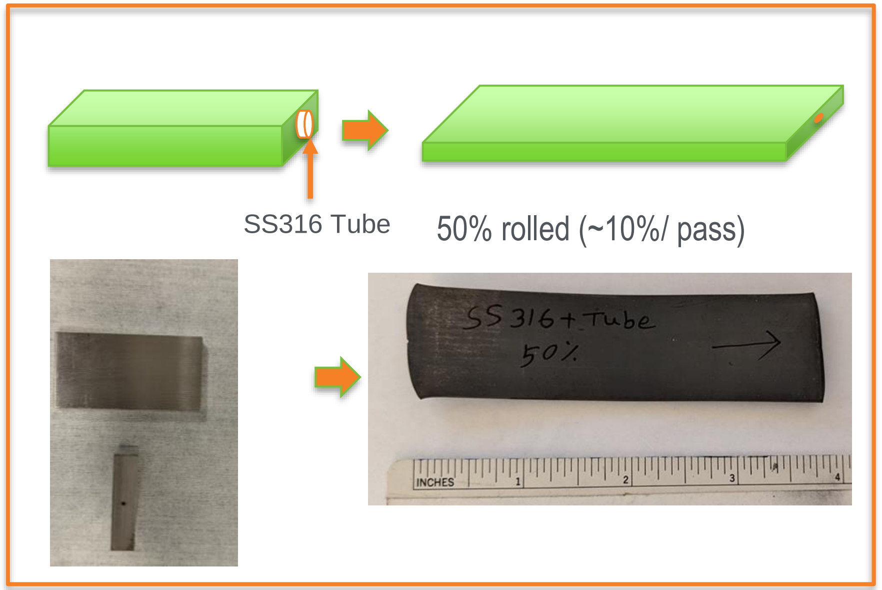


- **Fiber intact and visible after 20% reduction**

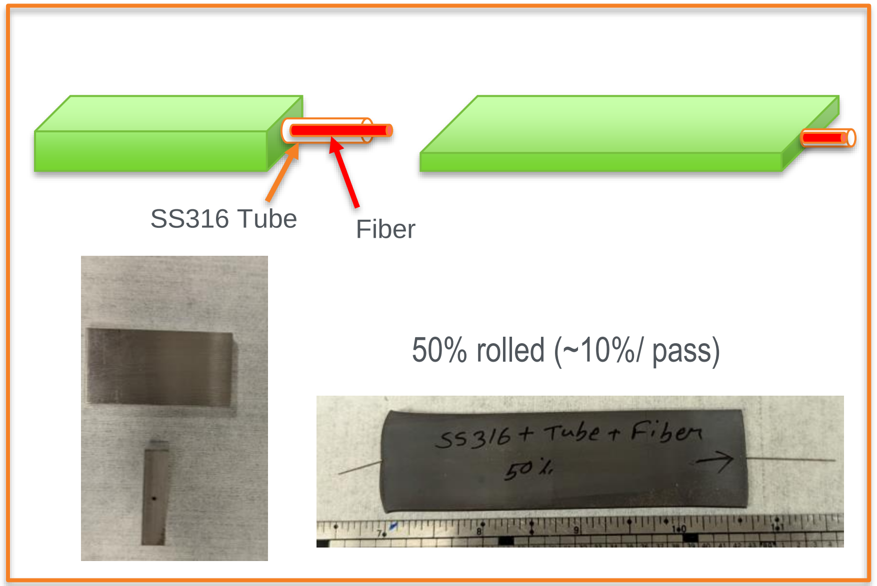
Approach 1
Multi-pass Rolling: Up to 50% Rolled
(6.2 mm to 3.1 mm)

#	Dimension	Multi pass reduction (%)	Temp (°C)	Soaking (min)	Drilled hole/Fiber	Observations
1	2”x1” (6.2 mm thick)	~50	800	15	w/o channel (Dummy plate)	Rolled, defects free
2	2”x1” (6.2 mm thick)	~50	800	15	w/ channel (0.5 mm dia) + Tube	Rolled, defects free
3	2”x1” (6.2 mm thick)	~50	800	15	w/ channel (0.5 mm dia) + Tube + long Fiber	Rolled, defects free
4	2”x1” (6.2 mm thick)	~50	800	15	w/ channel (0.5 mm dia) + long Tube + long Fiber	Rolled, defects free

Bare Steel + SS316 Tube



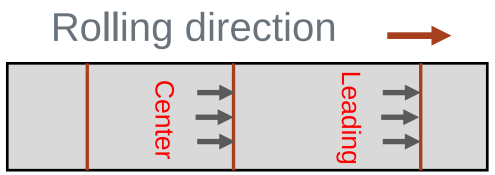
Bare Steel + long SS316 Tube + long Fiber



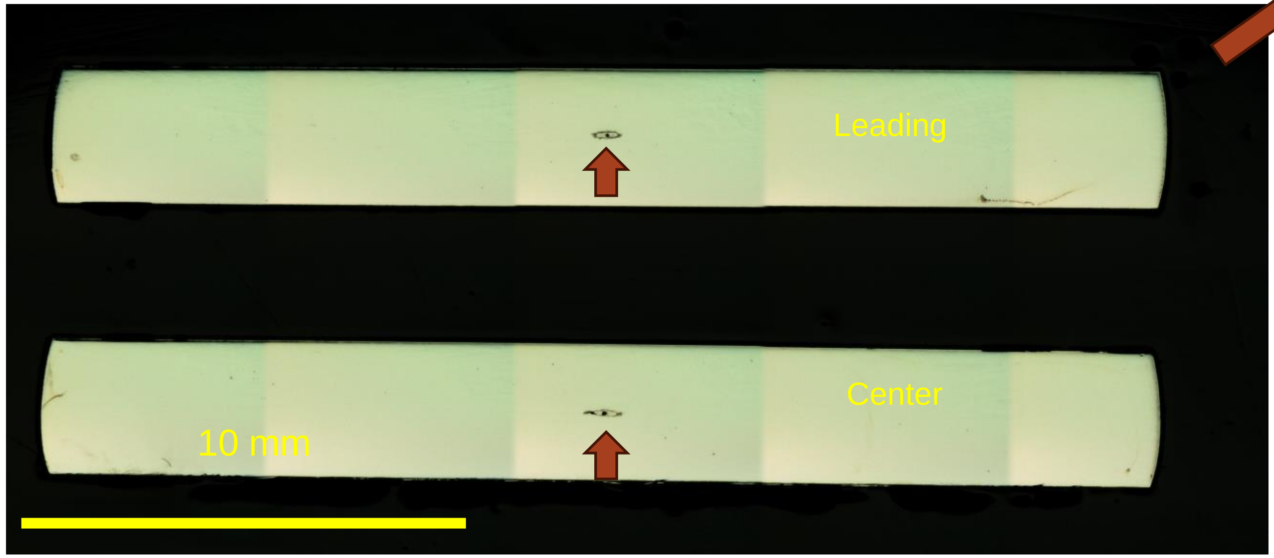
Fiber intact and adjusted to the length

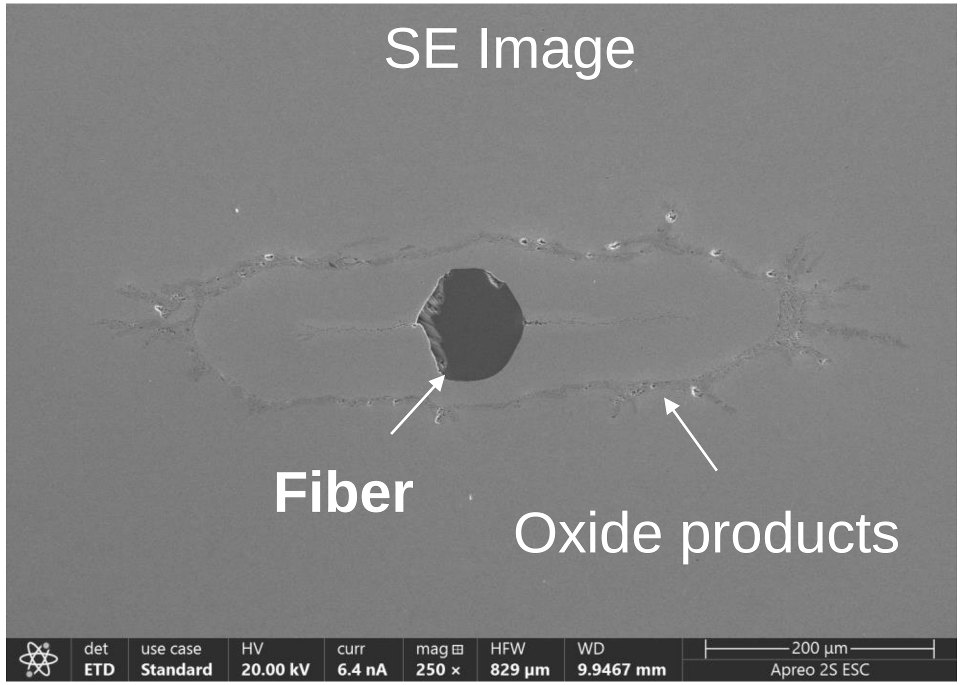
Approach 1
Cross-Sectional Microscopy: SEM Analysis

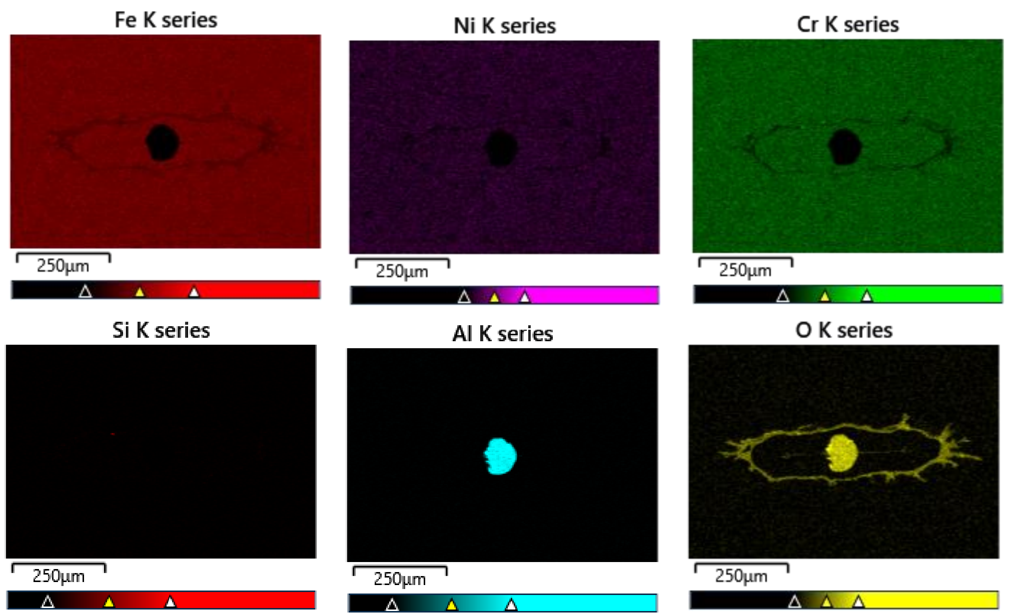
SS316 + long Tube+ long Fiber (800°C, ~50%)



Cross section view





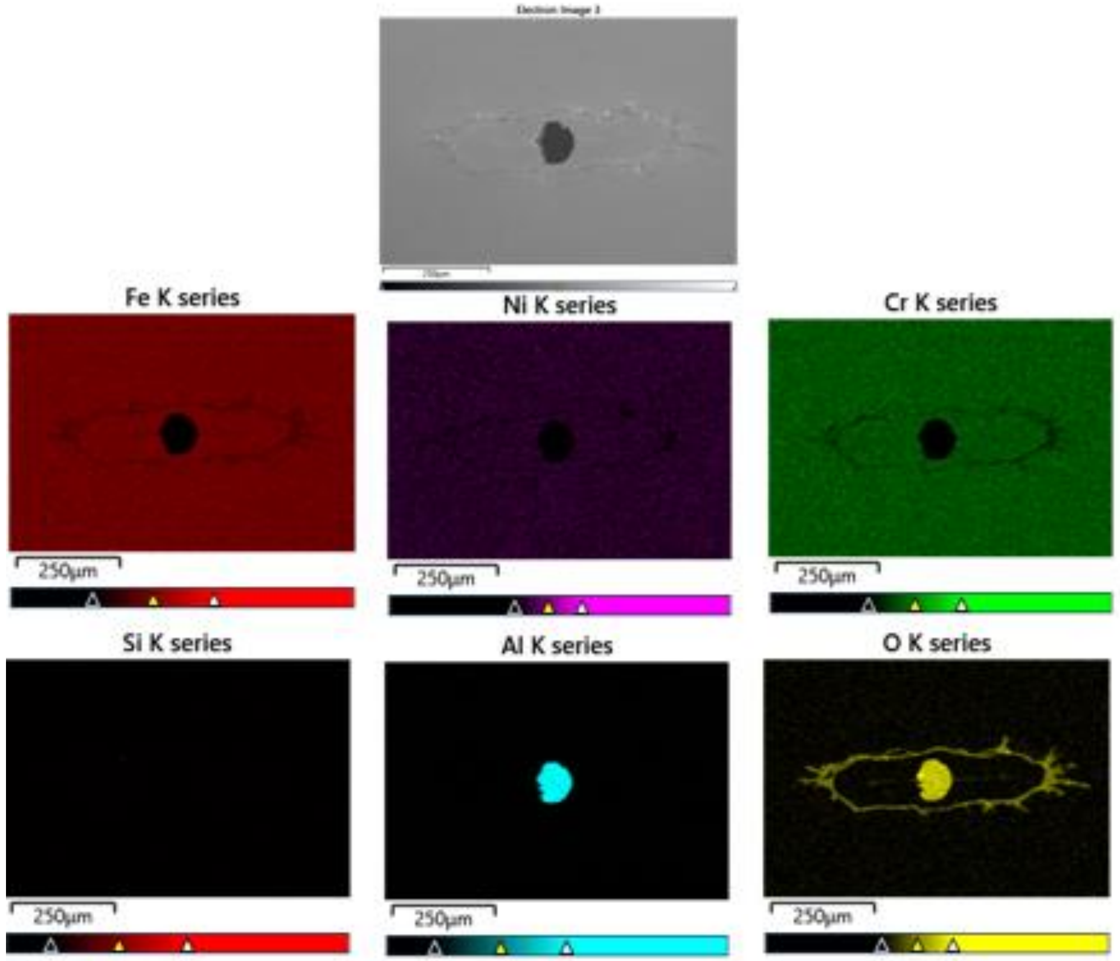


- ➢ *Fiber intact and adjusted to the length*
- ➢ *Oxide layer can be addressed by cleaning process and covers*

Approach 1: Summary of Results

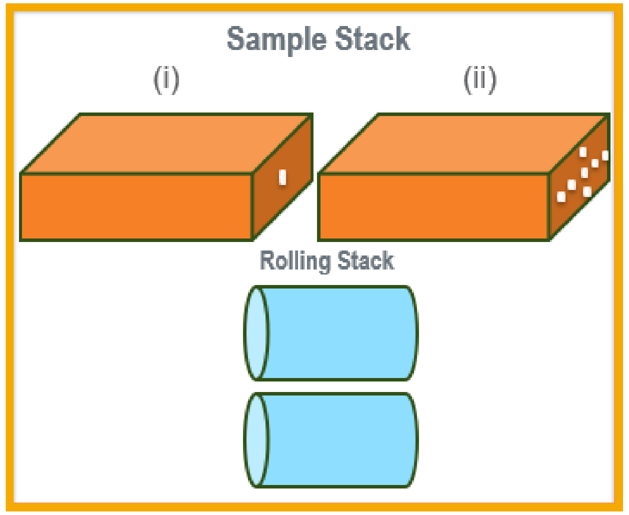
Performed targeted and successful experiments by utilizing modeling

- Demonstrated and validated the models
- Successfully showcased that we can embed sensor using the first approach
- Deformation of pre-drilled holes with vertical and horizontal orientations is studied with rolling reduction up to ~50% at rolling temperature of 850°C.
- The through thickness shear strain is studied with various reduction ratios.

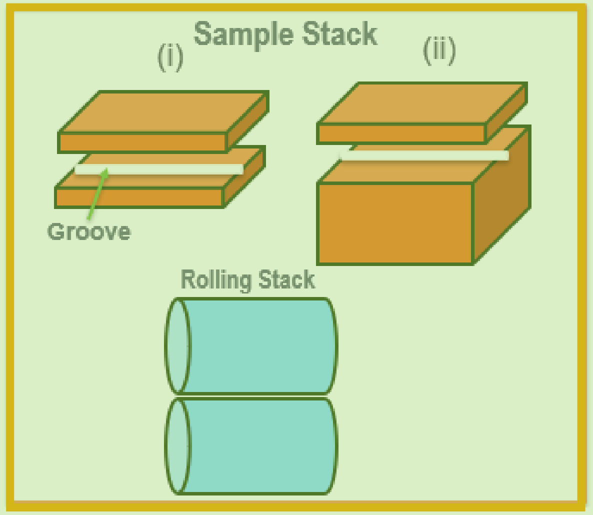


Approach 2
Diffusion Bonding of Two Plates with Rolls

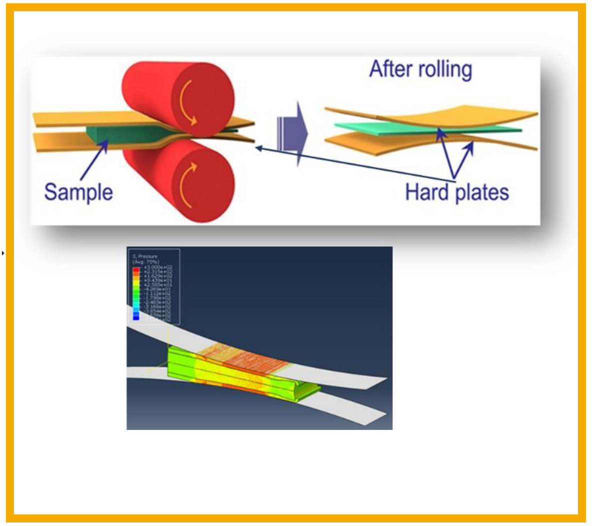
Approach 1



Approach 2



Approach 3



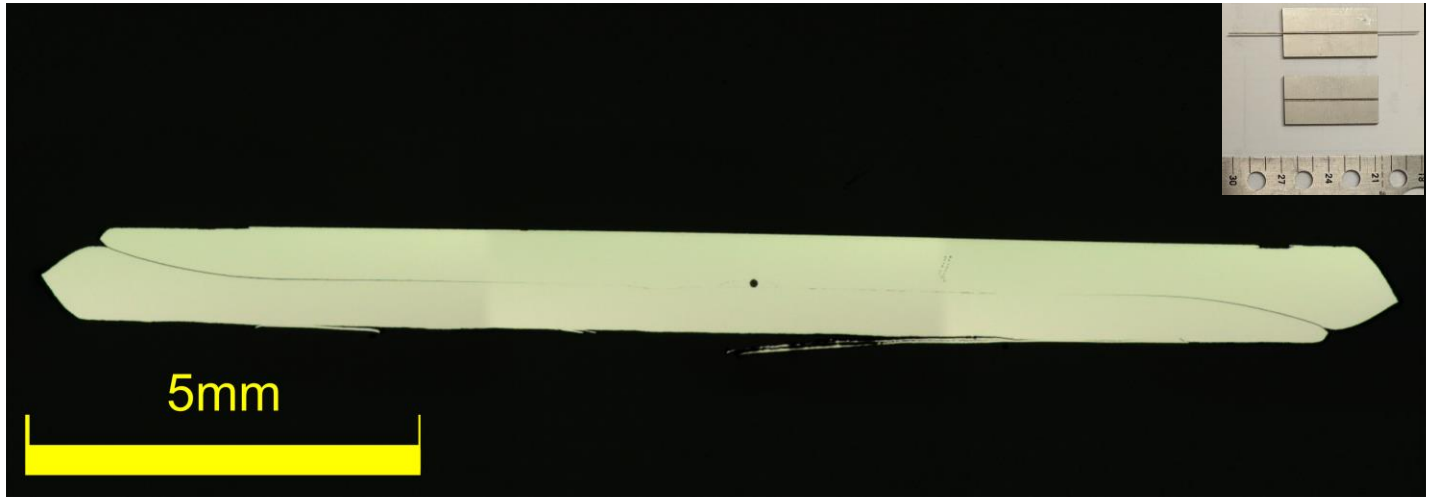
The concept of hot confined rolling to embed the sensor and minimize stresses at the center of the sheet to minimize stresses at the sensor location

The connections and Wires can be connected initially during the rolling process

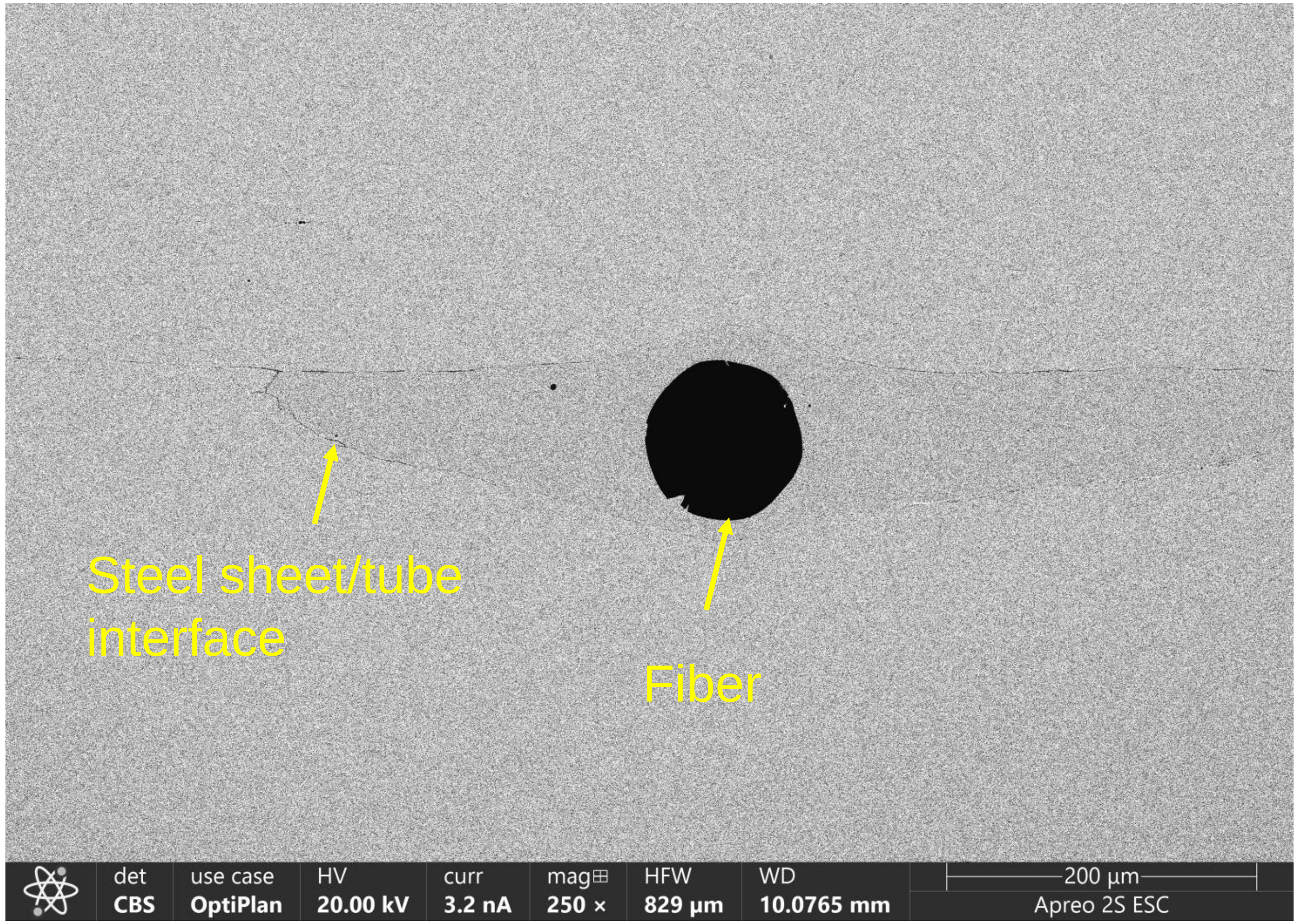
Approach 2: Cross-sectional Microstructure

#	Dimension	Single pass reduction (%)	Temp (°C)	Soaking (min)	Grooved/Fiber	Observations
5	1”x0.5” (1.5 + 1.5 mm thick)	~62	900	5	w/ groove + Tube + Fiber	Bonded

Optical



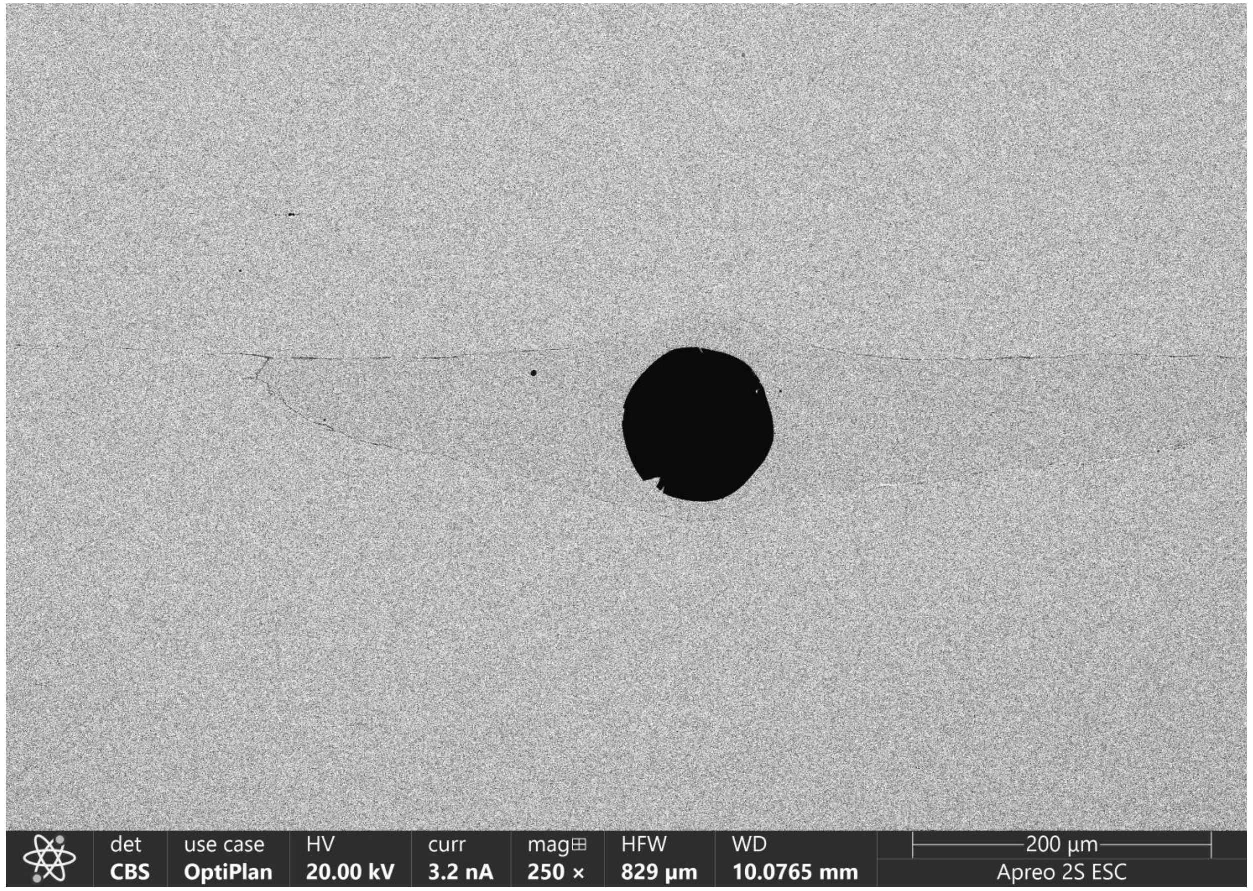
SEM



- ➢ Two sheets were metallurgically bonded
- ➢ Steel sheet/tube bonded nicely
- ➢ No fiber/tube interface defects observed

Approach 2: Summary of Results

- Successfully showcased embedding sensor using the second approach
- Analytical Model Predictions and calculations validated for bonding



Testing – Post Fabrication

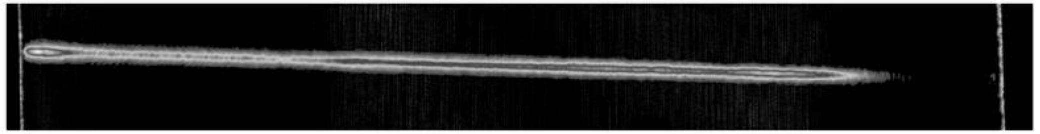
(1) NDE Testing: Ultrasonic test and Radiography

- Continuity of fibers after rolling
- Detect processing defects, such as voids, debonding, delamination etc.

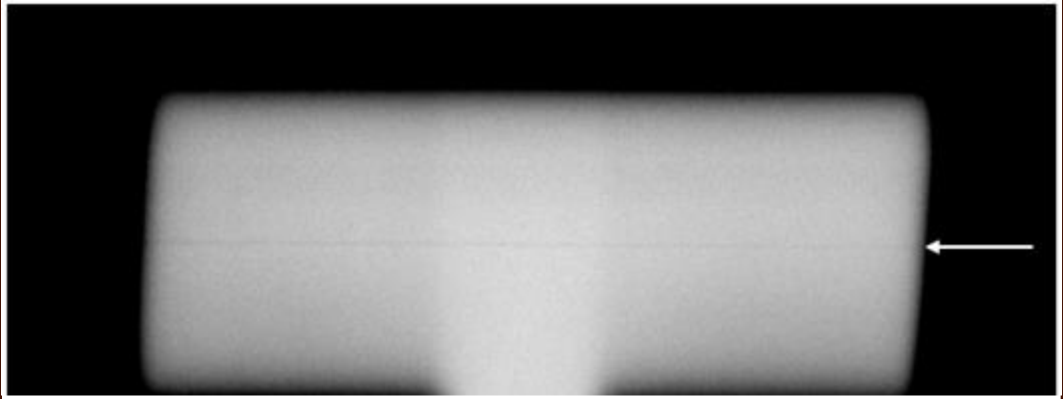
(2) Functionality testing: Wavelength attenuation/ amplitude of laser signal, dB

- Continuity of the optical fibers
- Performance of the fibers at elevated temperature and strain

NDE: Ultrasonic test



NDE: Radiography



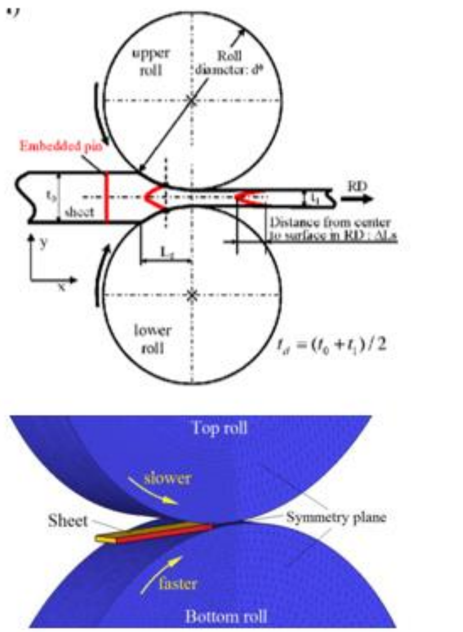
FY25 Plans

- Continue cross sectional microscopy- Examine the interface of fiber/matrix
- Continue non-destructive characterizations- UT, radiography and X-ray CT for fiber continuity study
- Fabrication with SiO_2 fiber Bragg grating (FBG)- Au or Cu or Al coating
- Testing the embedded fiber assemblies in ORNL with the help of Chris Petrie

Concept can be applied to other forms as well

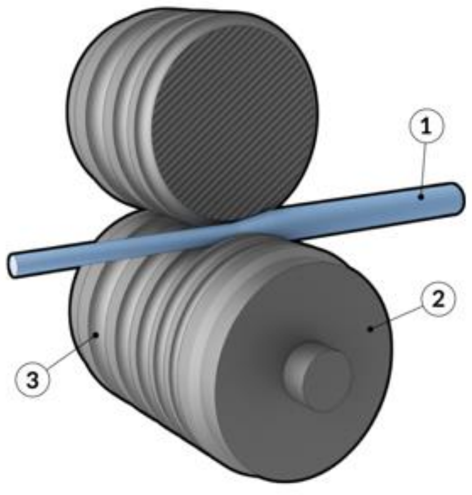
Rolling Sheet → Rod or Tube

Sheet rolling



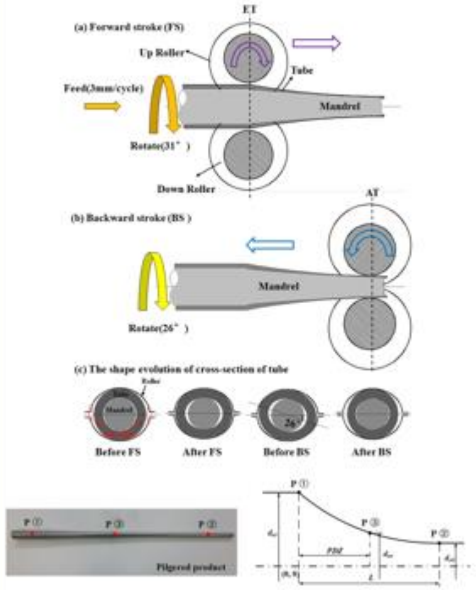
Metals 2020, 10(1), 91.
The 12th International Conference on Numerical Methods in Industrial Forming Processes, MATEC Web Conf., 80 (2016).

Rod rolling/ Groove rolling



1. https://www.manufacturingguide.com/en/rod-rolling

Tube rolling/ Pilger rolling



1. International Journal of Material Forming (2021) 14:533–545.
2. Appl. Sci. 2021, 11(23), 11265.

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Pacific Northwest
NATIONAL LABORATORY

Thank you