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Directed Energy Deposition Process of Corrosion Resistant Coating for Lead-Bismuth Eutectic Environment

Dr. Gidong Kim
KIMS, Korea
05 June 2024

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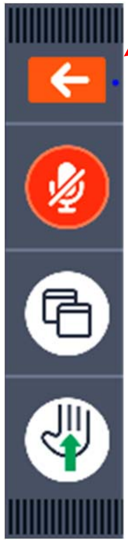

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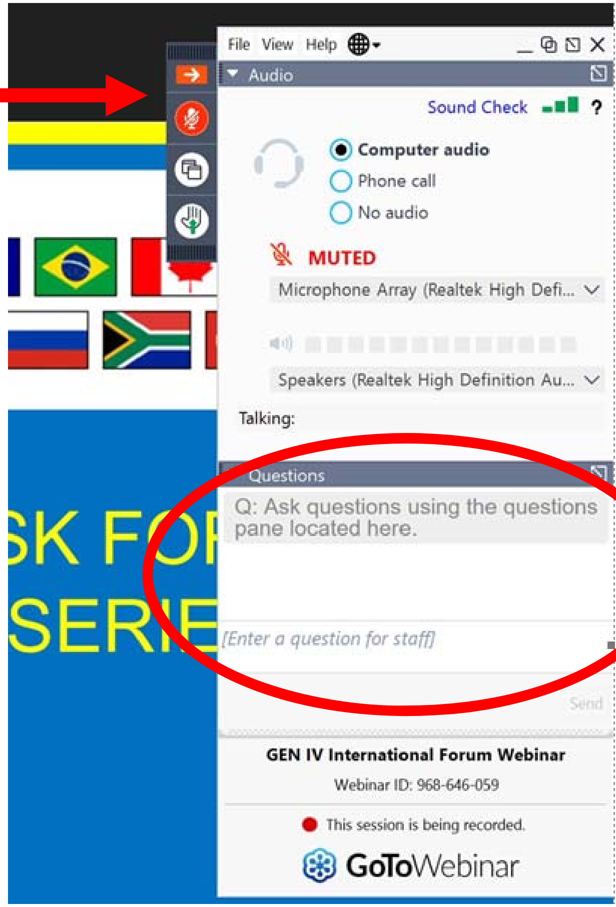

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ULSAN NATIONAL INSTITUTE OF SCIENCE AND TECHNOLOGY

KIMS Korea Institute of Materials Science

Meet the Presenter

Dr. Gidong Kim is a Senior researcher at the Korea Institute of Materials Science (KIMS). He earned his MSc in Materials Science and Engineering from Pusan National University (Republic of Korea) and PhD in Nuclear Engineering from Ulsan National Institute of Science and Technology (Republic of Korea).

His research interests include additive manufacturing, brazing, and the development of welding procedures (WPS/PQR) for industrial applications. Also, he is working as an Authorized Nuclear Inspector (ANI) to ensure the safety of nuclear power plants in Republic of Korea.

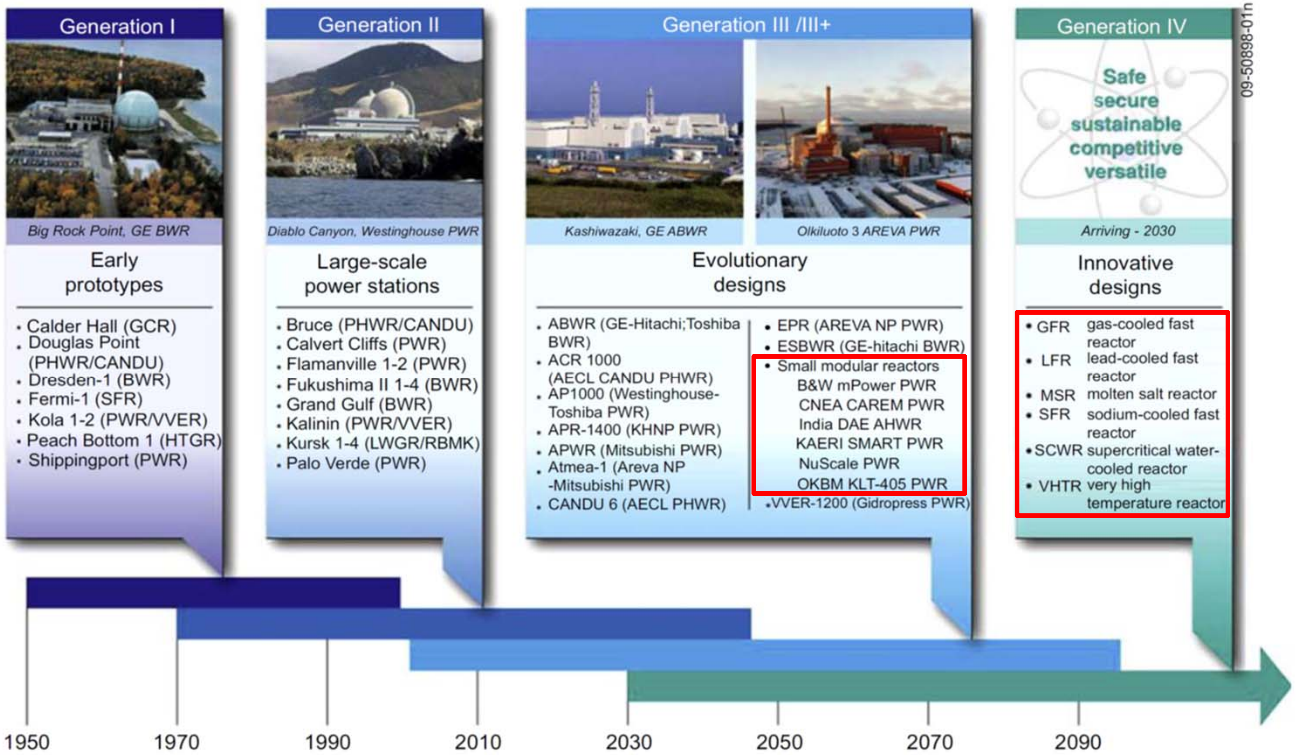
Recently, he has been conducting research on advanced manufacturing technologies (AMT) applicable to small modular reactor (SMR), including Laser Directed Energy Deposition (DED) and Powder Metallurgy Hot Isostatic Pressing (PM-HIP).

Dr. Kim is the popular vote winner of the 2023 Pitch your Gen IV Research competition.

Email: gdkim@kims.re.kr

Outlook

Introduction
Generations of nuclear reactors (1/2)



[1] Handbook of Generation IV Nuclear Reactors, 2016

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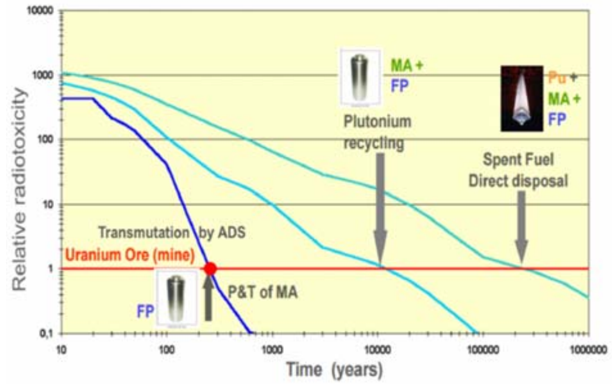
Introduction
Generations of nuclear reactors (2/2)

- **Generation III** commercial nuclear reactors using **thermal neutron** (0.025 eV)
  - **Near zero greenhouse gas** emission energy source
    (Covers 10 % of the global electricity demand)
  - However, 1) Uses ^{235}U (0.7% of the total natural U) as nuclear fuel → **limited resources**
    2) Produce huge amounts of high-level radioactive wastes → **disposal problems**

- **Generation IV** fast reactors using **fast neutron** (≥1 MeV)
  - High-energy neutron can '**breed**' the ^{238}U to fissile isotopes (^{239}Pu, etc.)
  - **Short-lived** (100's of years) **waste** forms
  - Through pyro-processing, '**closed fuel cycle**' could be achieved
    → Making nuclear power essentially **sustainable** and **renewable**

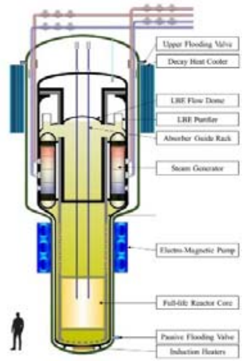
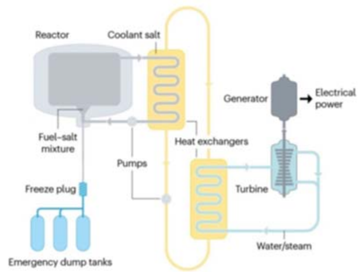


<Comparison of nuclear waste radiotoxicity before and after transmutation [2]>

[2] M. Salvatores, Nuclear Fuel Cycle Science and Engineering, 2012

Introduction

Representative model of Gen. IV SMR in Republic of Korea [3,4]

Main body	**KHNP**	**UNIST**	**KAERI**
Outline	 Innovative-SMR(i-SMR)	 MicroURANUS	 Fundamental Technology Development Project for MSR
Capacity	170 MWe × 4 unit	20 MWe	~ 100 MWth
Size	4.5 × 18 m	2.4 × 11 m	To be determined
Coolant	Light water	Lead-Bismuth Eutectic	Chlorides Molten Salts
Purpose	Power generation	Power generation Marine application	Marine application
Current status	Under standard design ('23 ~)	Concept design Completed('22)	Under concept design ('23 ~)
Note	Improved model of SMART KHNP, KAERI	Compact type of SNU URANUS	Core Fundamental Technologies for Carbon-Free Marine Systems

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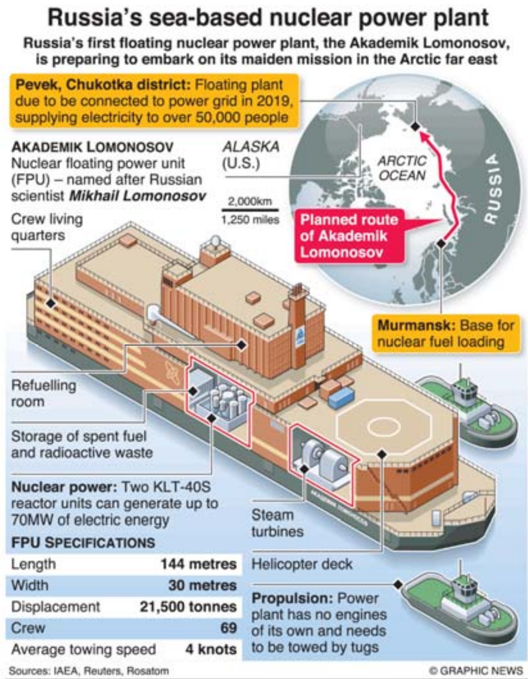
[3] Advances in Small Modular Reactor Technology Developments, 2022 [4] KAIST MSR Material Technology Workshop, 2023

Introduction
Necessity of marine propulsion reactors

- **Micro-modular reactor for marine application (ex., icebreaker)**
  - IMO (International Maritime Organization) strongly requires reducing the carbon intensity of all ships by 40% by 2030, compared to 2008
  - Most ships are unable to meet these regulations from now
  - The need for **nuclear-propulsion ships** is arising

- Historically, nuclear powered submarines and aircraft carriers have been built and operated.
  ; small area → **Compact and modulated design**

- From 2019, PWR type MMR (35 MW x 2) based icebreaker ship started operation
  - "nuclear Titanic" and "Chernobyl on Ice" ?
    → **Passive or inherent safety of SMR/MMR**
  : automatically shut down without external power and human operation



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[4] Matt Muenster, Breakthrough, 2022 [5] Introduction to Nuclear Power, 1996 [6] Mike Tyler, Graphic News, 2018

Introduction
Parameters and materials for generation III, IV reactors

Reactor type	Coolant	Pressure (MPa)	T_{in} / T_{out}	Neutron spectrum, Maximum Dose (dpa)	Cladding	Structural Materials	
						In-Core	Out-Core
PWR	**Water**	16	290/320	Thermal, ~80	Zr alloy	Stainless steels, nickel-based alloys	
BWR	**Water**	7	280/288	Thermal, ~7	Zircaloy	Stainless steels, nickel-based alloys	
VHTR	**Helium**	7	600/1000	Thermal, <20	SiC or ZrC coating and surrounding graphite	Graphites, PyC, Sic, ZrC, Vessel : F-M*	Ni-based superalloys, F-M, low-alloy steels
GFR	**Helium, supercritical CO_2**	7	450/850	Fast, 80	Ceramic	Refractory metals and alloy, ceramics, ODS* Vessel : F-M	Ni-based superalloys, F-M,
SFR	**Sodium**	0.1	370/550	Fast, 200	F-M or F-M ODS	F-M, 316SS	Ferritics, austenitics
LFR	**Lead or Lead-bismuth**	0.1	250/350, 600/800	Fast, 150	High-Si F-M, ODS, ceramics, refractory metals	High-Si F-M, ODS	High-Si austenitics, ceramics, refractory metals
MSR	**Molten salt**	0.1	700/1000	Thermal, 200	Not applicable	Ceramics, refractory metals, high-Mo, Ni-based alloy, graphite, Hastelloy N, XM-19	High-Mo, Ni-based alloys

- Gen IV reactors require adequate materials compatibility with their corresponding coolants

[7] Materials challenges for nuclear systems, 2010 [8] Materials Challenges for Advanced Nuclear Energy Systems, 2009

Introduction
Liquid Pb-Bi eutectic(LBE) cooled fast reactors

<Basic characteristics of selected reactor coolants [9]>

Coolant*	Atomic Mass	Relative Moderation	Neutron Absorption Cross Section at 1 MeV (mbarn)	Neutron Scattering Cross Section (barn)	Melting point (℃)	Boiling point (℃)	Chemical reactivity with air and water
Pb	207	1	6.001	6.4	327	1737	Inert
Pb-Bi	**208**	**0.82**	**1.492**	**6.9**	**125**	**1670**	**Inert**
Na	23	1.8	0.23	3.2	98	883	Highly reactive
H_2O	18	421	0.1056	3.5	0	100	Inert
D_2O	20	49	0.0002115	2.6	0	100	Inert
He	2	0.27	0.007953	3.7	-	-269	Inert

*Coolants for molten salt reactor have various type of Fluorine(F) or Chlorine(Cl) mixture (ThF_4, UF_4, UCl, PuCl, Li, Be, K, etc.). Melting points are varies in 400 ~ 500℃ and stay liquid while operation.
Good thermal property (passive safety) but corrosion of structural materials issue.

- **Lead-based heavy liquid metals (HLM)** : lead and **lead-bismuth eutectic** (**LBE**, $Pb_{44.5}Bi_{55.5}$)
  - **Primary coolant** for fast neutron reactors
  1) Exhibit **limited neutron moderation** and **absorption** ➔ **efficient fuel breeding**
  2) **Good heat transfer** capability ➔ enables the production of **compact, power-dense cores**
  3) **High boiling point** / **Chemically stable** with air and water / Decay heat can be removed by **natural convection** in accidental condition ➔ **Inherent safety**

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LBE is one of the suitable SMR coolant material

[9] Neil E. Todreas et. al., Nuclear Technology, 2004

Introduction
Materials for LBE cooled fast reactors

- **Candidate materials (structural / fuel cladding)** in liquid LBE-cooled fast reactors
  - **1) Ferritic / martensitic steels (BCC)**
    (9-12 wt.% Cr steels with 0.1-0.2 wt.% C and addition of Mo, V, etc.)
    - Good **mechanical properties** in elevated temperature (550 – 610 ℃)
    - Excellent resistance to **void swelling**
    - Low ductile-to-brittle transition temperature
    - Moderate corrosion resistance due to Cr content
    - Liquid metal embrittlement (**LME**) issue
  - **2) Austenitic stainless steels (FCC)**
    (STS 304L, 316L, 316 Ti, 15-15Ti(1.4970), etc.)
    - **Good performance** at relatively low operation temperature (350 - 475 ℃)
    - Irradiation-induced void swelling could occur at high irradiation doses
    - Liquid metal corrosion (**LMC**) issue
  - **3) Other materials**
    (Oxide dispersion strengthened steels, refractory metals, SiC matrix composites, etc.)
    - Although its superior properties, manufacturing process, and reliability should be verified

[10] Xing G et. al., Progress in Materials Science, 2022

Introduction
Degradation phenomena of materials on LBE cooled fast reactors

- **Candidate materials** (austenitic / ferritic steels) in LFR are **not fully compatible** with high temperature **liquid LBE** coolants
- **Major degradation phenomena on LFR materials**

  1) **Liquid metal corrosion (LMC)**
     - **Oxidation** : LBE penetrates into the grain / twin boundaries
     - **Dissolution corrosion** : selective leaching of **Ni**

     → **Dense / thin oxide layer can prevent LMC**
       Note : active oxygen control (Co ≈ 10^{-7} – 10^{-6} wt. %) is essential

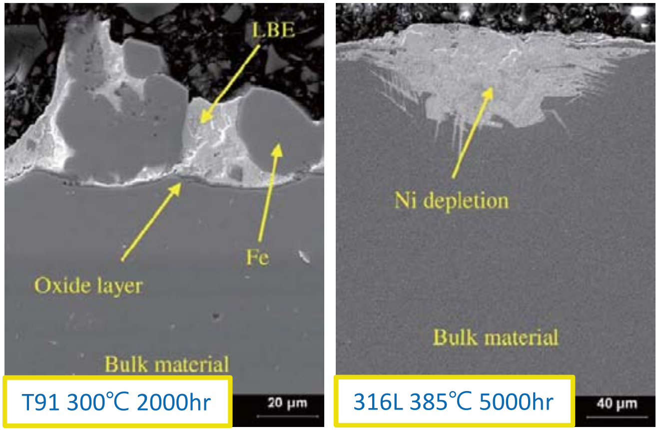
     → **Alloying elements** in candidate materials can produce **stable oxide layer**
       Note : mechanical / irradiation properties of bulk material must be verified

     → **Surface coatings** on candidate materials can produce **stable oxide layer**
       Note : reliability of coated layer and its properties must be verified

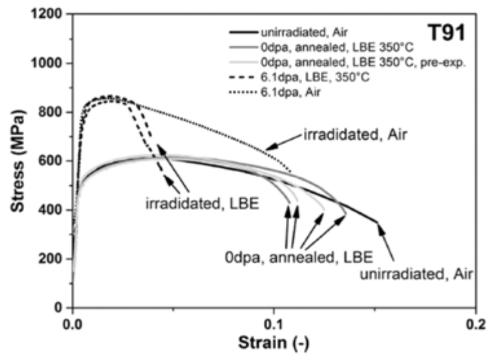
  2) **Liquid metal embrittlement (LME)**
     - Loss of ductility of materials in contact with liquid LBE below 450℃

     → Ferritic steels are more susceptible to LME than austenitic steels

     → Mechanism of LME is not clearly revealed



[11]



[10]

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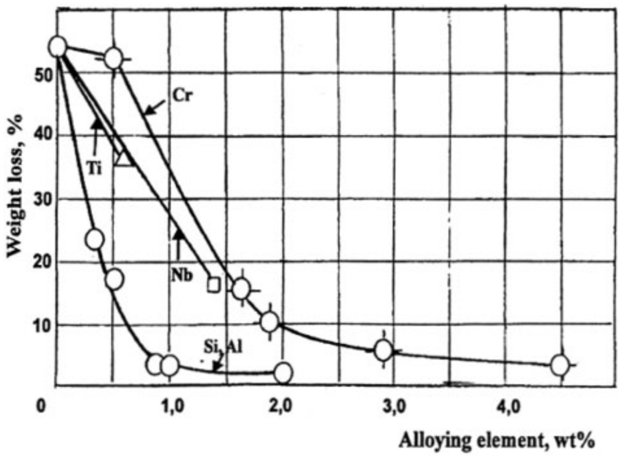
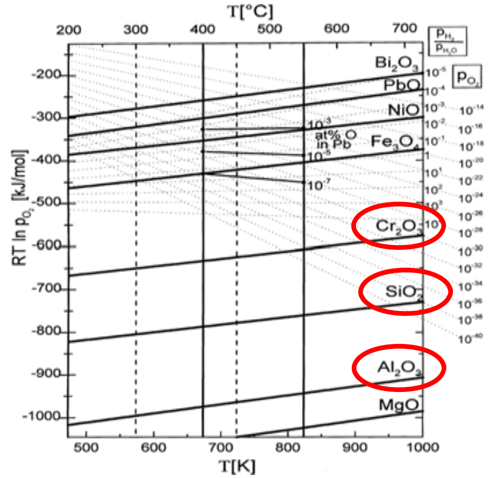
[10] Xing G et. al., Progress in Materials Science, 2022 [11] F.J. Martin-Munoz et.al., Journal of Nuclear Materials, 2011

Introduction
Major alloying element to prevent LMC





<Alloying element concentration vs. weight loss in LBE, [12]><Ellingham diagram for metal/metal oxide systems, [12]>

Cr : 12wt.% Max

Over 12% Cr : Ferrite and σ-Cr decomposition under 550 ℃ ➔ Intergranular corrosion, weakens alloy

Si : 2.5wt.% Max.

Over 2.5% Si :Susceptible to radiation embrittlement

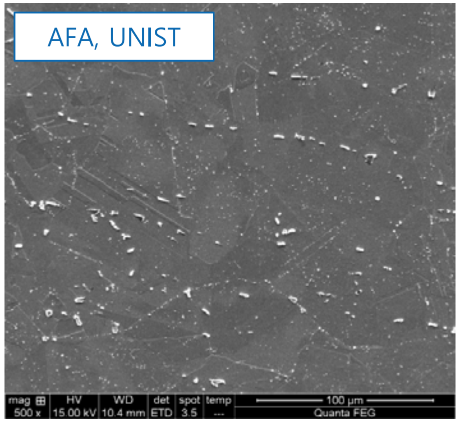
Al : 5.5wt.% Min.

Theoretical calculation results show that minimum 5.5% Al is need to produce Al_2O_3 layer
➔ Low weldability and castability, hydrogen embrittlement

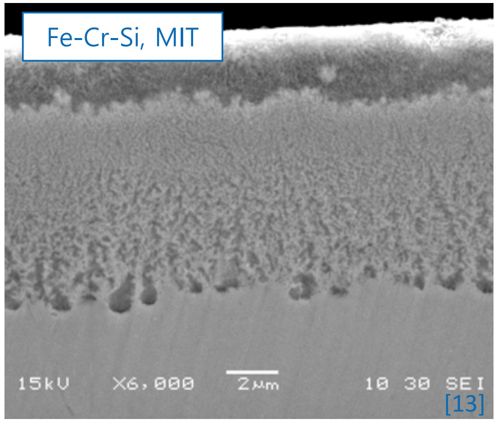
[12] I.V. Gorynin et al., Heavy Liquid Metal Coolant in Nuclear Technology, 1999

Introduction
Representative LMC resistant material

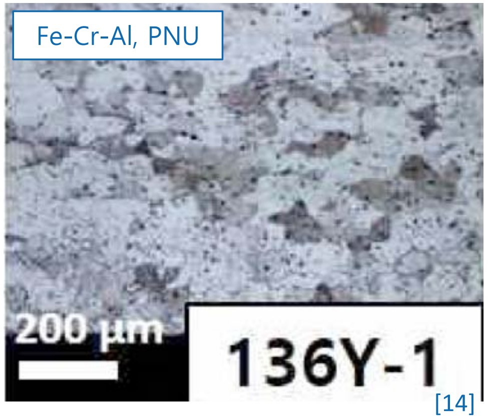
Alumina Forming Austenite (AFA)	Fe-Cr-Si	Fe-Cr-Al
• Corrosion resistance : Cr, Al, Ti	• Corrosion resistance : Cr, Si	• Corrosion resistance : Cr, Al, Y
• Austenitic phase (FCC) : good compatibility with austenitic base metal	• Ferritic / martensitic (BCC) phase : high strength	• Ferritic / martensitic (BCC) phase : high strength
• Possibility of Ni dissolution	• Irradiation embrittlement	• Low weldability



Fe-15Cr-18Ni-1.2Mo-1.54Mn-0.38Si-**3Al-0.4Ti**-0.1C



Fe-12Cr-**2Si**

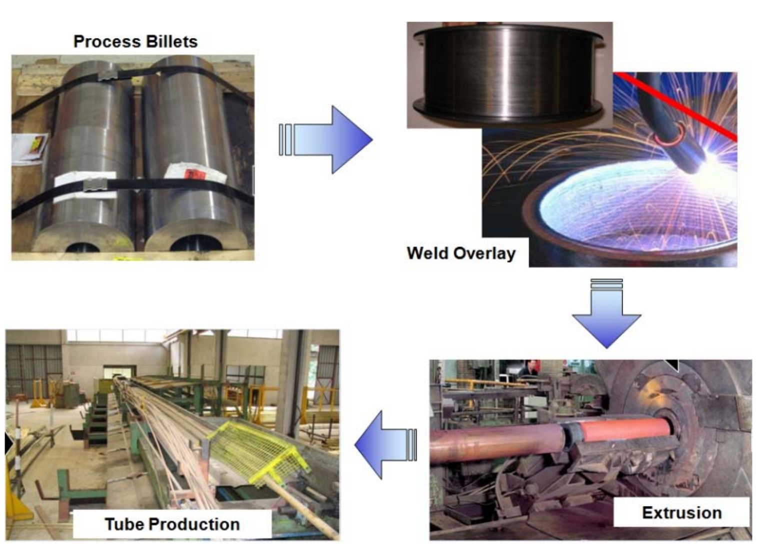


Fe-13Cr-**6Al-0.15Y**

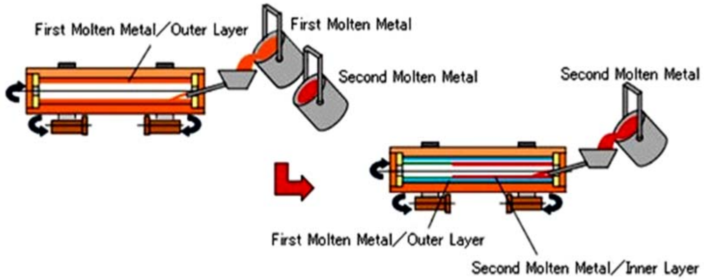
Various LMC resistant materials have been developed and evaluated by several researchers

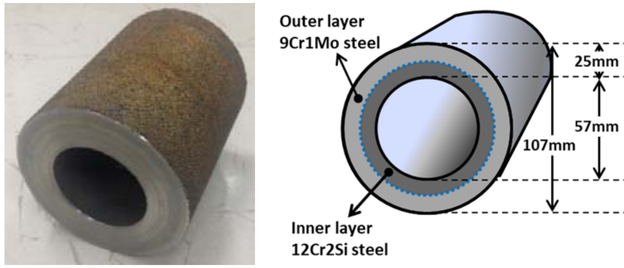
[13] R.G. Ballinger, SMR Workshop on MMR, 2016 [14] Bahn, Development of Fundamental Technology for FeCrAl-Based Cladding for Light Water Reactor Fuel to Improve Accident Tolerance, 2020

Missing parts in current study
Development of LMC resistant material – manufacturing barrier / Code register



<Weld overlay and extrusion / pilgering> [13]





<Dual-layered centrifugal casting billet> [14]

- Extrusion, pilgering, casting
  → Failed to reach final dimension
  (ex. 10 mm OD, 1 mm thickness)
- Single metal tube (EP-823(Fe-Cr-Si system), Russia)
  → Cannot be used in US, Korea
  (Not registered to ASME, KEPIC Code)

NB-2120 PRESSURE-RETAINING MATERIAL
NB-2121 Permitted Material Specifications

(*a*) Pressure-retaining material shall conform to the requirements of one of the specifications for material given in Section II, Part D, Subpart 1, Tables 2A and 2B, [15]

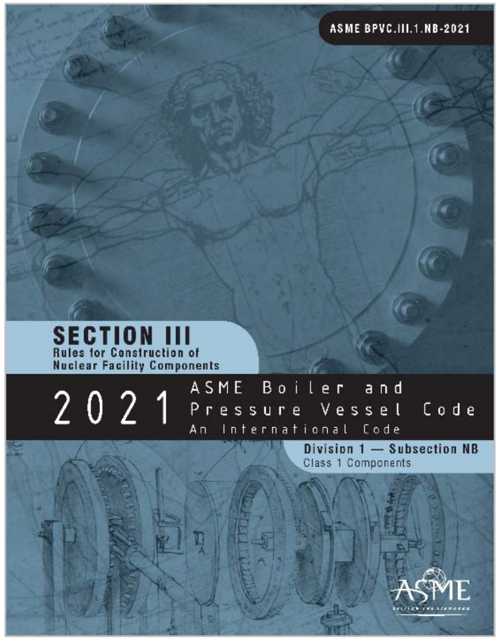
MNB 2120 PRESSURE-RETAINING MATERIAL
MNB 2121 Permitted Material Specifications

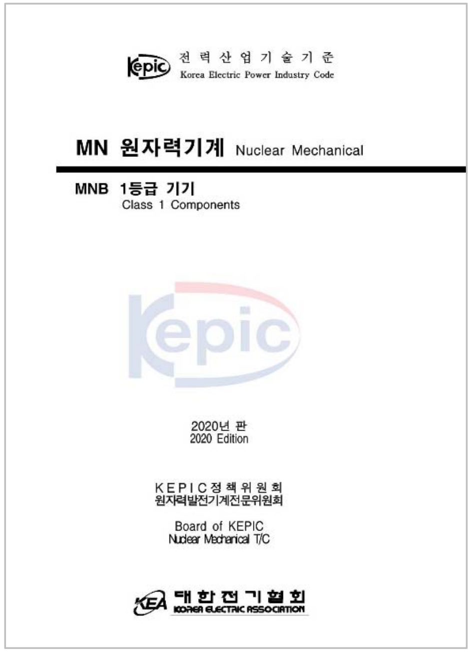
(1) Pressure-retaining material shall conform to the requirements of one of the specifications for material given in KEPIC-MDP, Appendices ⅡA and ⅡB, [16]

[13] R.G. Ballinger, SMR Workshop on MMR, 2016 [14] G Kim et al., ICONE22, 2014 [15] ASME BPVC NB, 2021 [16] KEPIC MNB, 2020

Approach

Development of LMC resistant material – manufacturing barrier / Code register





(f) The requirements of this Article do not apply to hard surfacing or corrosion-resistant weld metal overlay that is 10% or less of the thickness of the base material (NB-3122). [15]

MNB(2020 Ed.)

(6) The requirements of MNB 2000 do not apply to hard surfacing or corrosion-resistant weld metal overlay that is 10% or less of the thickness of the base material (MNB 3122). [16]

- Nevertheless, unregistered material also can be used if the clad material has 10 % or less thickness as base metal

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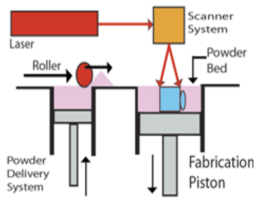
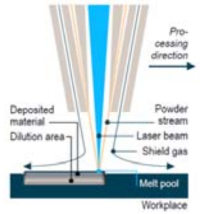
[15] ASME Boiler and Pressure Vessel Code, Section III NB, 2021 [16] KEPIC MNB, 2020

Approach
Metal additive manufacturing technology

1) Powder bed fusion (PBF)
  - Thermal energy selectively fuses regions of a powder bed
2) Directed energy deposition (DED)
  - Focused thermal energy is used to fuse materials by melting as the material is deposited

	POWDER BED FUSION	DIRECT ENERGY DEPOSITION
Schematic Diagram		
Build speed	5 ~ 20 cm³/h (40-160 g/h)	~ 70 cm³/h (Up to 0.5 kg/h)
Accuracy	+/- 0.02-0.05 mm	+/-0.125-0.25 mm
Detail capability	0.04-0.2 mm	0.5-1.0 mm
Surface quality	Ra 4-10 μm	Ra 7-20 μm
Max. part size	630mm x 400 mm x 500 mm	2,000 mm x 1,500 mm x 750 mm
Materials	Steel, Al,Ti, CoCr, Ni base alloy, bronze	Steel, Ti, Ni base alloy, ceramic
Typical application	Molds and die(tool inserts), Implants All types of component	Repair / modification of worn components surface coating

[17] KISTI Report(ISBN 978-89-294-0893-0 93550), 2016 [18] T. DebRoy et al., Progress in Materials Science, 2018

Approach

Metal additive manufacturing technology

1) Powder bed fusion (PBF)
 - Thermal energy selectively fuses regions of a powder bed
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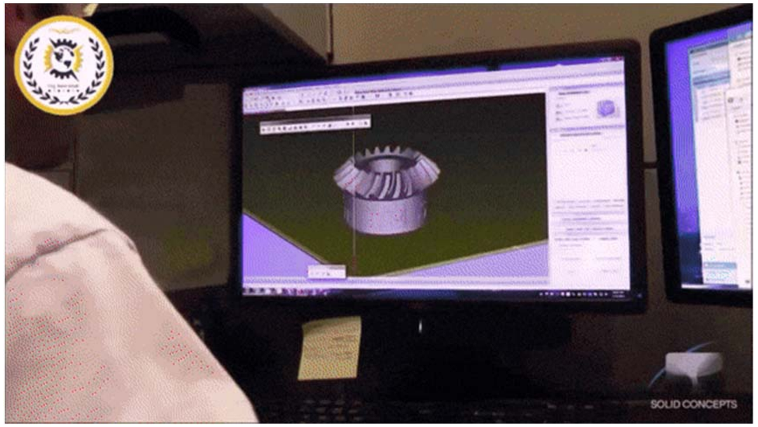
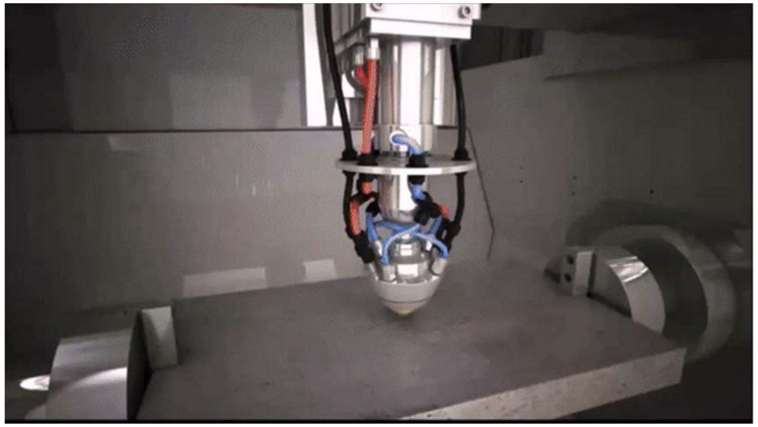
 - Focused thermal energy is used to fuse materials by melting as the material is deposited



<Powder bed fusion> [19]



<Directed energy deposition> [20, 21]

For accurate surface coating of metal AM on existing material, DED process can be selected

[19] Solid Concepts, Additive Manufacturing [20] BeAM Metal 3D Printing [21] RPM Innovations, INC., Additive Manufacturing

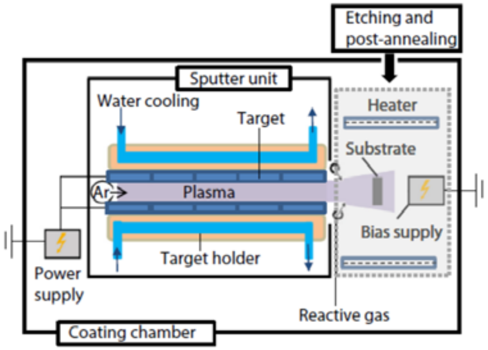
Approach

Surface coating process excluding DED

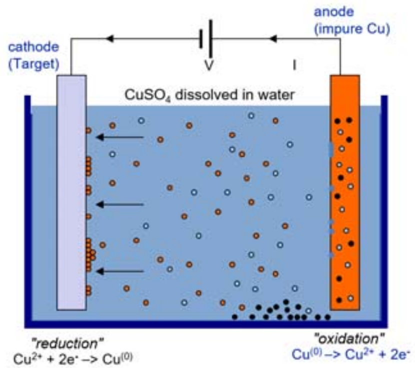
1) **Chemical vapor deposition (CVD) / Physical vapor deposition (PVD)**
 - Produce thin coated layer with **various target materials**
 - **Slow Deposition rate** (CVD: 11 μm/hr, PVD: 660 μm/hr) compared to other processes

2) **Electro plating / electroless plating**
 - Coating processes that have been applied to various industry parts (**proven**)
 - Require a large amount of **chemical solution** and **relatively slow deposition rate** (51 μm/hr)

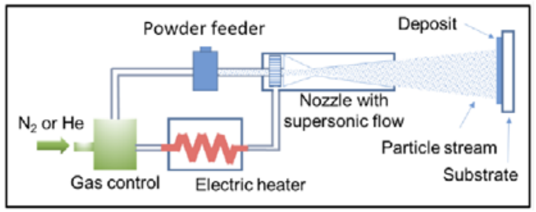
3) **Spraying process** (cold spray, thermal spray)
 - Generally show **massive deposition rate**
 - **Internal defect** such as porosity could exist and **limited mechanical bonding** with substrate



<High-speed PVD [23]>



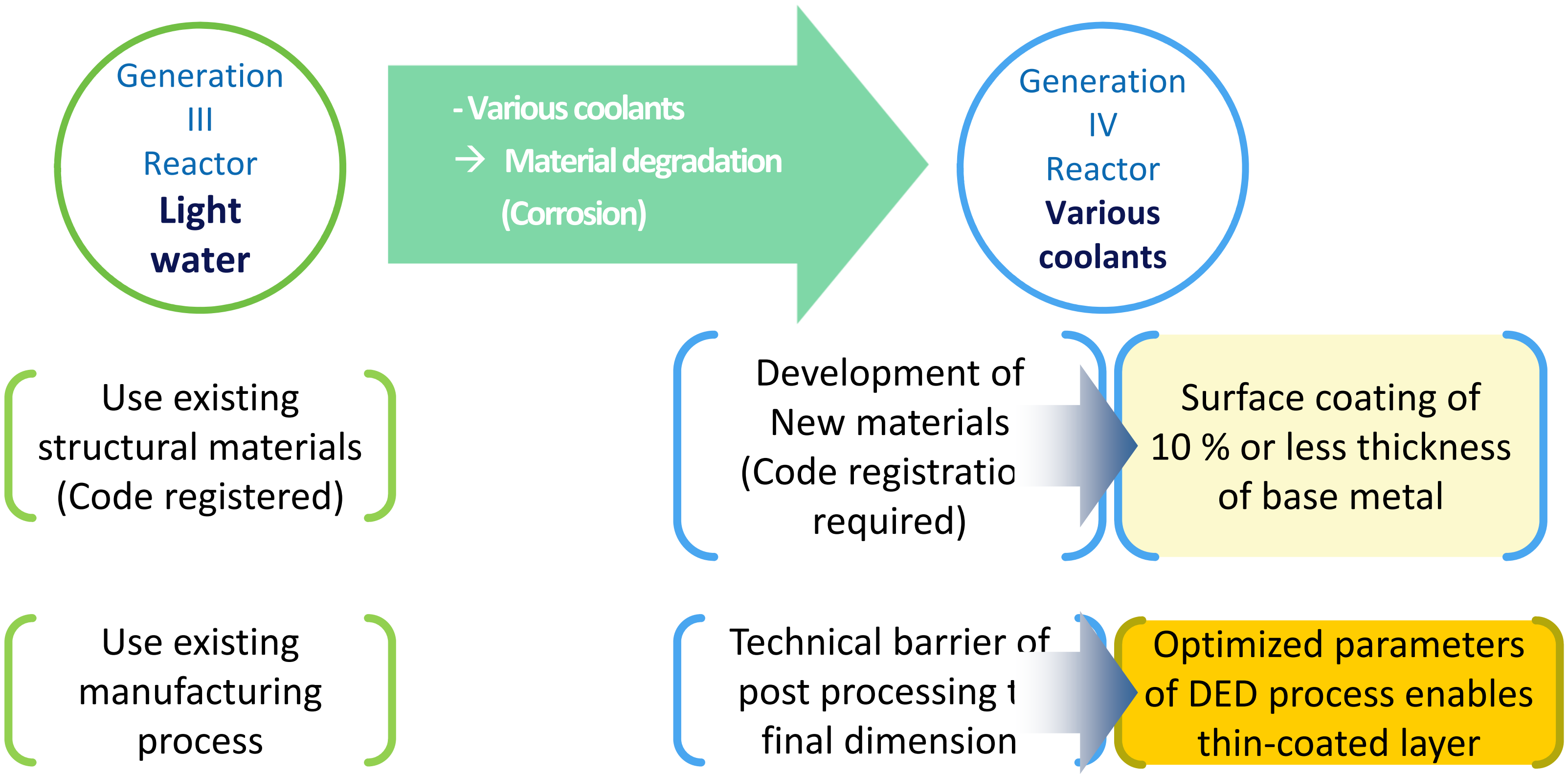
<Electroplating [23]>



<Cold spray [24]>

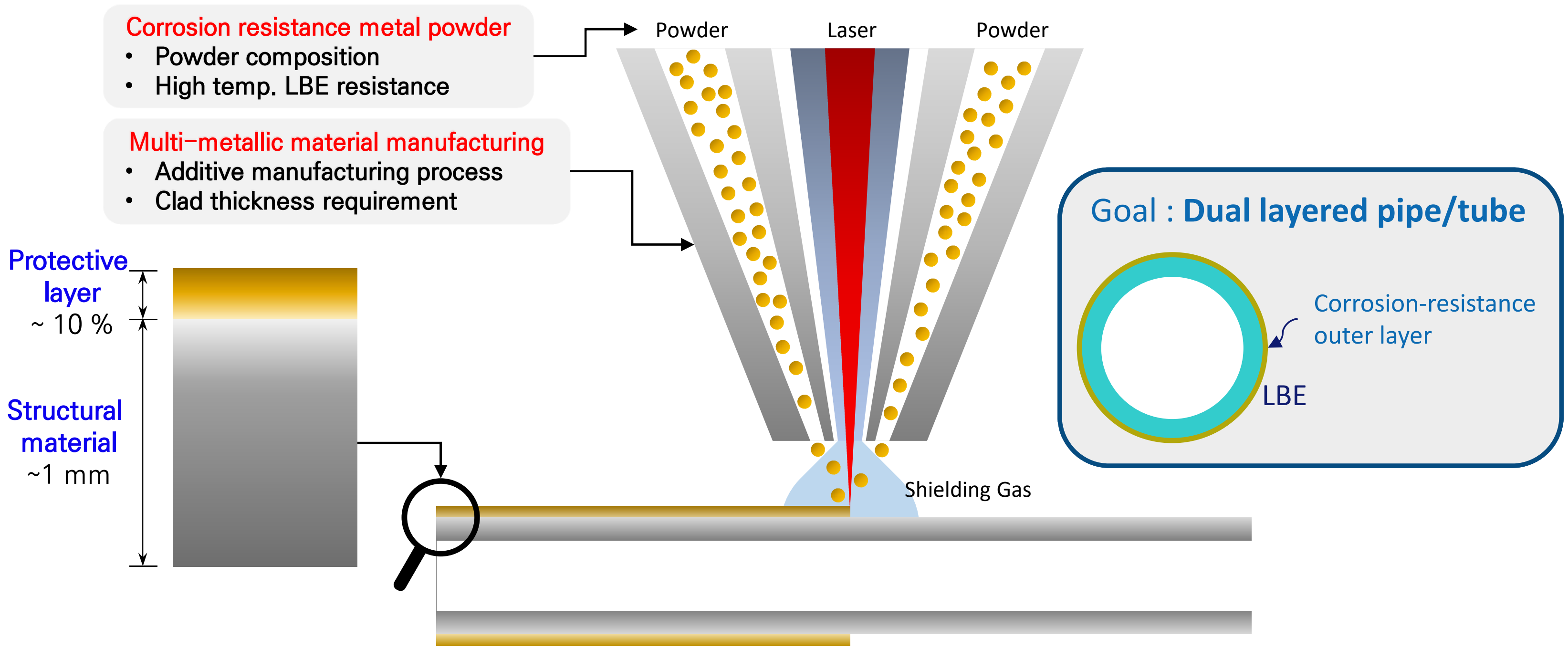
[22] T. Goto, Solid State Ionics, 2004 [23] Electroplating to make nanostructures [24] K. Ross, Cold Spray Process Details and Nuclear Applications, 2021

Approach


Generation
III
Reactor
Light
water
- Various coolants
→ Material degradation
(Corrosion)
Generation
IV
Reactor
Various
coolants
Use existing
structural materials
(Code registered)
Development of
New materials
(Code registratio
required)
Surface coating of
10 % or less thickness
of base metal
Use existing
manufacturing
process
Technical barrier of
post processing t
final dimension
Optimized parameters
of DED process enables
thin-coated layer

Objective

- **Surface coating of LMC resistant material __manufactured by DED process__**
  → **Overcome** the barriers of **__post processing__** and **__Code register__**

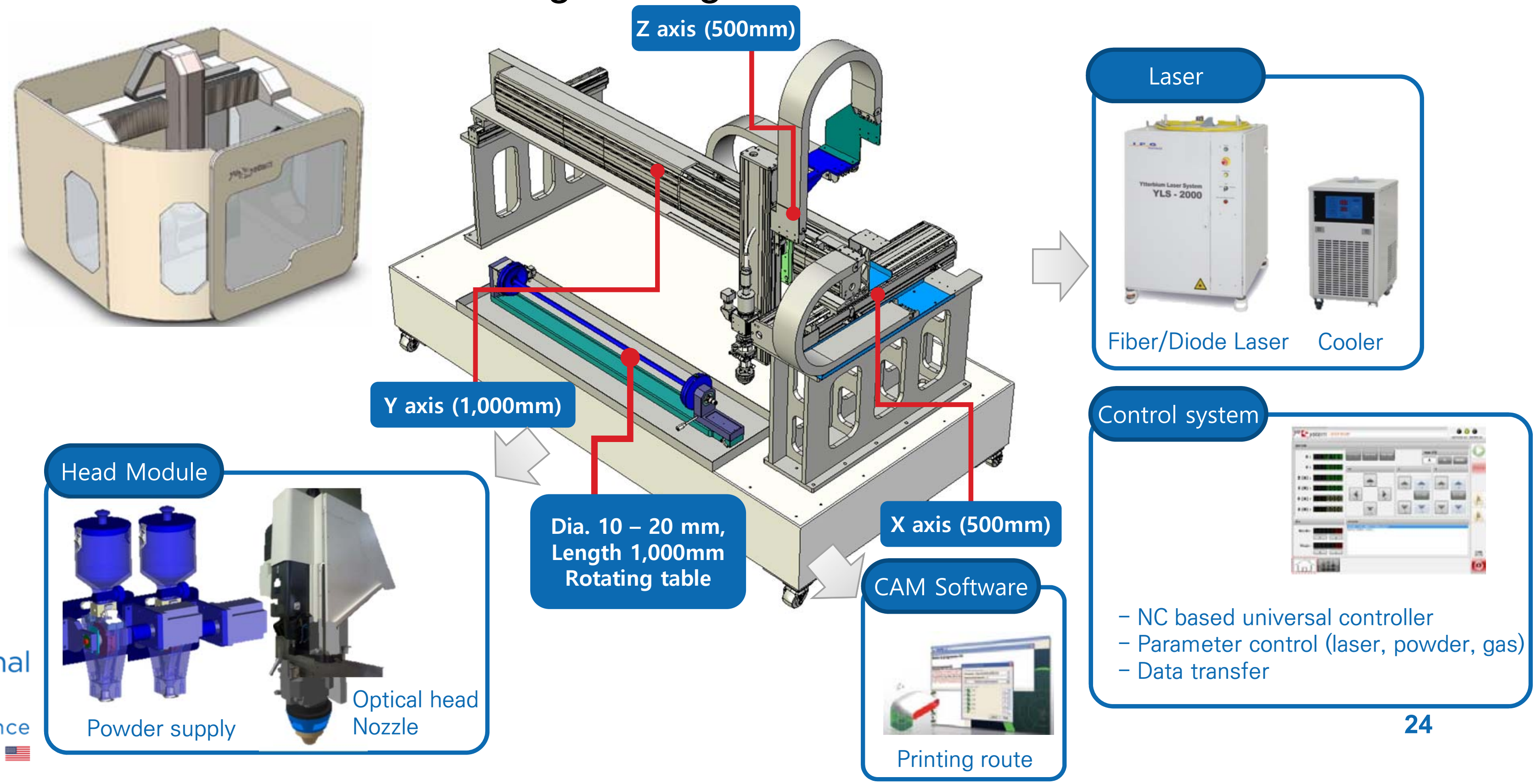


Study on LMC resistant clad material manufactured by DED process
- Microstructure examination, hardness, high temp. tensile test
- General corrosion, high temp. LBE exposure test

→ __Propose optimized DED parameters__ for LMC resistant materials and __report evaluation results__

Experimental procedures
Apparatus for DED process

- Existing DED equipment are not sufficient for the fuel cladding dimension (min. 1 m long tube)
  → Designed with consideration the fuel cladding coating



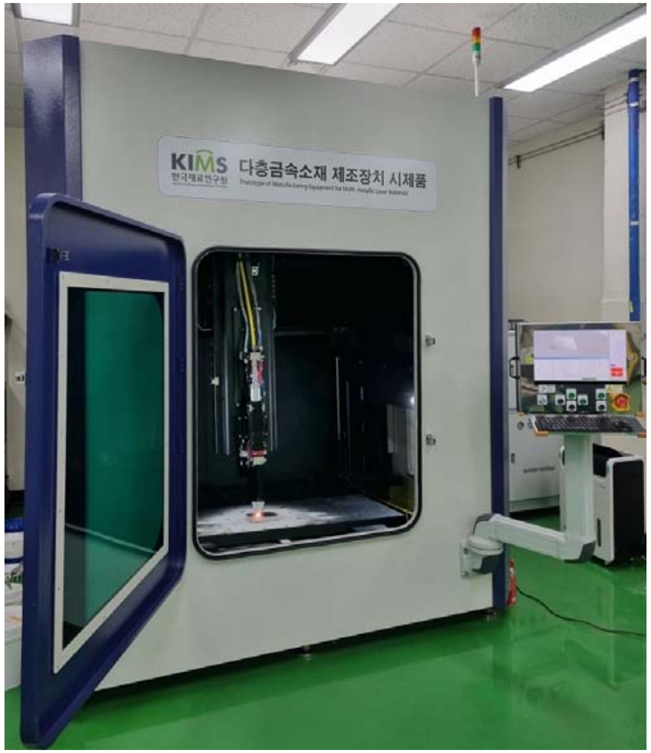
Experimental procedures
Apparatus for DED process - specification

Manufacturing Equipment for Multi-metallic Layer Materials	
○ **Laser generator** 1) IPG Fiber laser 2) Wavelength range : 1,030 - 1,080 nm 3) Max. output power : 1 kW	○ **Laser head** 1) Directed Energy Deposition, 1mm Beam 2) 3 Type Coaxial Nozzle
○ **External water cooling unit**	○ **Powder supply equipment** 1) Hopper : 3EA (0.5L) 2) Ar gas transfer 3) Powder feed rate : 0.1 - 6 g/min 5) Powder feed accuracy : (3 g/min condition) ± 0.5 g/min 6) Available powder size : 10 - 200 μm
○ **Main body** 1) Gantry type 3 Axis (X/Y/Z) + U Axis Machine 2) Build Size : 1,000 x500 x 500 mm 3) Repeatability : under ±0.1mm 4) Permission Size of work piece: 1,000 x 500 mm 5) Permission Mass of work piece: over 1,000 kg 6) Traverse speed : 20 m/min	○ **Software** 1) Universal control (Windows based HMI Program) 2) *.stl compatible and instant design software 3) Laser, transfer mode, powder, gas real time control

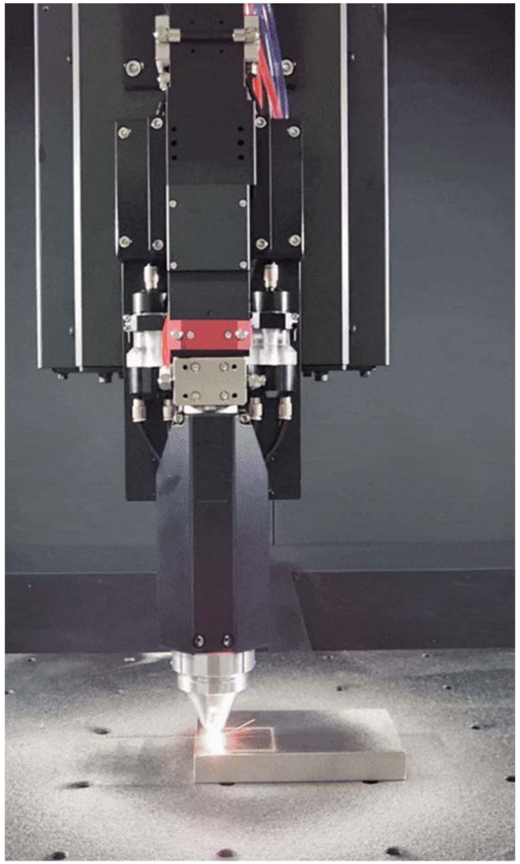
Experimental procedures
Apparatus for DED process

Gantry frame

Laser generator
(1 kW Fiber Laser)



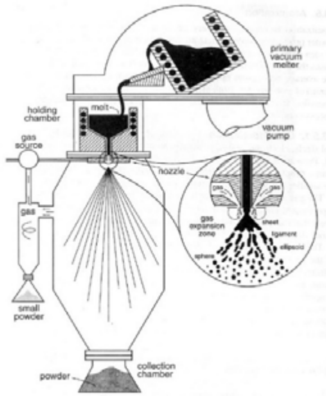
Outlook of the apparatus



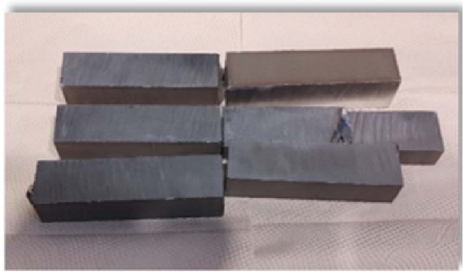
Laser head / nozzle

Experimental procedures
Powder manufacturing for DED process

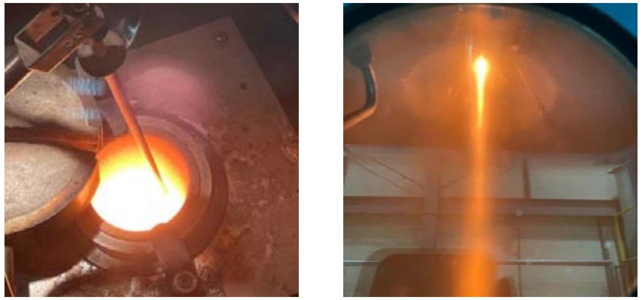
- Target alloy billets are produced by **vacuum induction melting** process
- Alloy billets are converted into metal powders using **gas atomizing process**
  1) Under 10^{-2} torr vacuum environment, melt the alloy billets using MgO crucible
  2) Overheating (1650℃), Ar gas spray (50 – 70 bar)
  3) Sieve to 45 - 150 ㎛ size
- **Powder characterization** : particle size, angle of repose, apparent density, tap density, Hausner Ratio, flowability



Overview of gas atomizing process



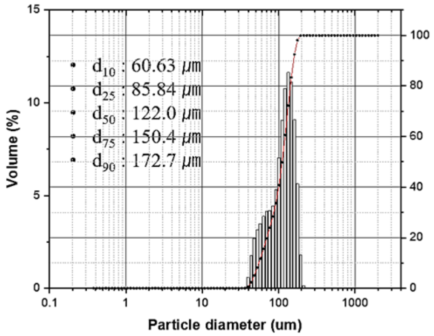
Alloy billets

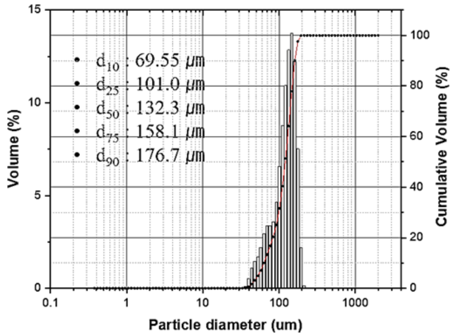


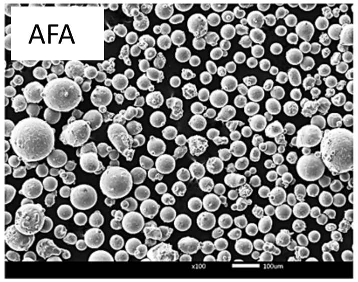
Gas atomizing to produce metal powder

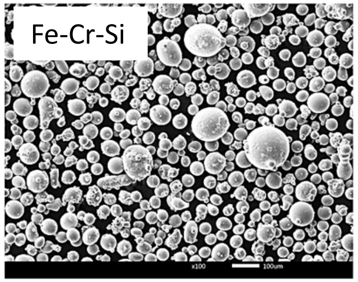
Sieve

Experimental procedures
Powder manufacturing for DED process



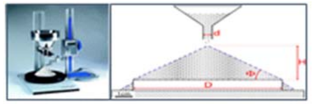
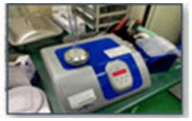






Average particle size : AFA: 132 ㎛, Fe-Cr-Si: 122 ㎛
STS 316L (commercial DED powder): 45 - 150 ㎛

	Fe	Cr	Ni	Mo	Mn	Si	Al	Ti	C	O
STS 316L	Bal.	16.80	10.4	2.20	1.60	0.94	-	-	0.019	-
AFA	Bal.	15.75	15.5	1.27	1.53	0.54	3.54	0.13	0.063	0.008
Fe-Cr-Si	Bal.	13.25	-	-	-	2.70	-	-	0.071	0.030
Substrate (STS316L)	Bal.	16.18	10.1	2.05	1.06	0.60	-	-	0.018	-

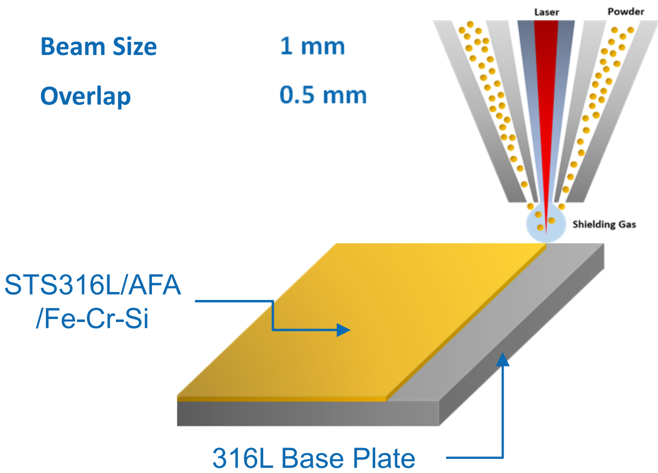
	Method	AFA	Fe-Cr-Si
Angle of repose (˚)		32.5	31.0
Apparent density (g/cm^3)	 ASTM B 212/417/329/703, MPIF 04	4.181	4.254
Tap density (g/cm^3)	 ASTM B 527, MPIF 46	4.762	4.854
Hausner Ratio	Hausner ratio / Flow character: 1.00-1.11 Excellent; 1.12-1.18 Good; 1.19-1.25 Fair; 1.26-1.34 Passable; 1.35-1.45 Poor	1.14	1.14
Flowbility (sec./50g)	 ASTM B 213, MPIF 03	13.54	12.94

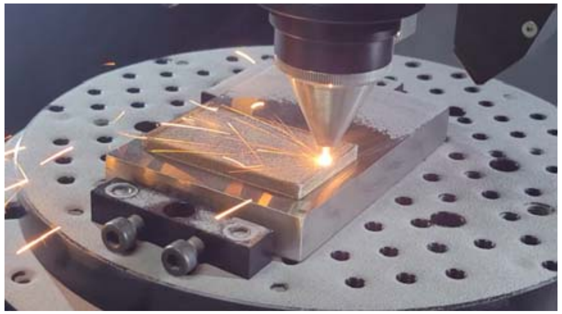
Powders of LMC resistant materials are produced and evaluated for the use of DED process

Experimental procedures
DED coated layer characterization - parameter optimization

Beam Size **1 mm**

Overlap **0.5 mm**



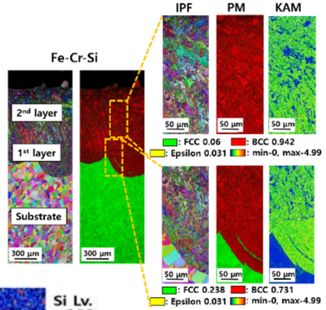


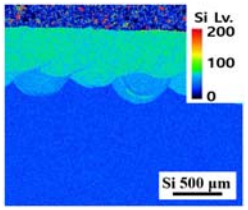
- Major parameters in DED : laser power(kW), laser scan speed(mm/s), powder feed rate(g/min)
- Laser power : 0.4, 0.5, 0.6 kW
- Laser scan speed : 12, 14, 16 mm/s
- Powder feed rate : 6.0 g/min (fix)
- Shield gas : Ar

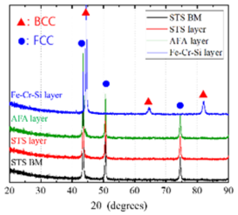
DED parameters

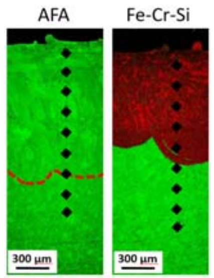
Condition	Laser power (kW)	Scan speed (mm/s)	Line energy input (J/mm)
1	0.4	12	33.33
2	0.5	12	41.67
3	0.6	12	50.00
4	0.4	14	28.57
5	0.5	14	35.71
6	0.6	14	42.86
7	0.4	16	25.00
8	0.5	16	31.25
9	0.6	16	37.50

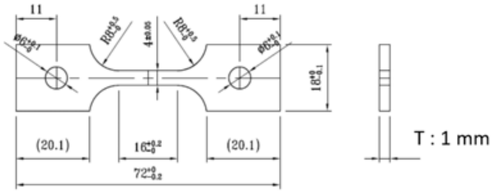
Experimental procedures
DED coated layer characterization – analysis of coated layer



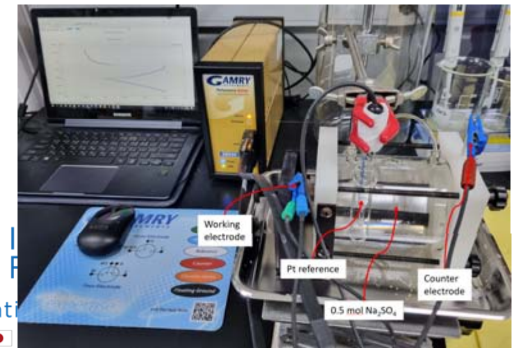


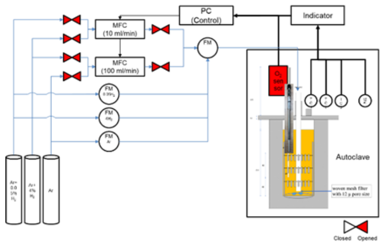


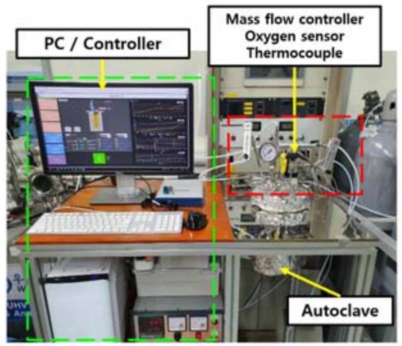




Microstructure	Mechanical
XRD SEM/EDS/EBSD EPMA	Hardness Tensile test (room temp. and high temp.)
Corrosion	**LBE exposure**
Potentiodynamic polarization test	High temp. LBE exposure test



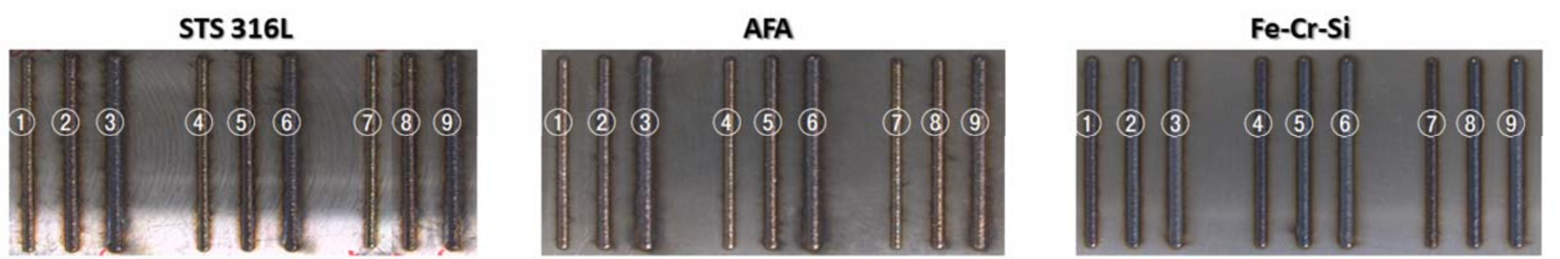


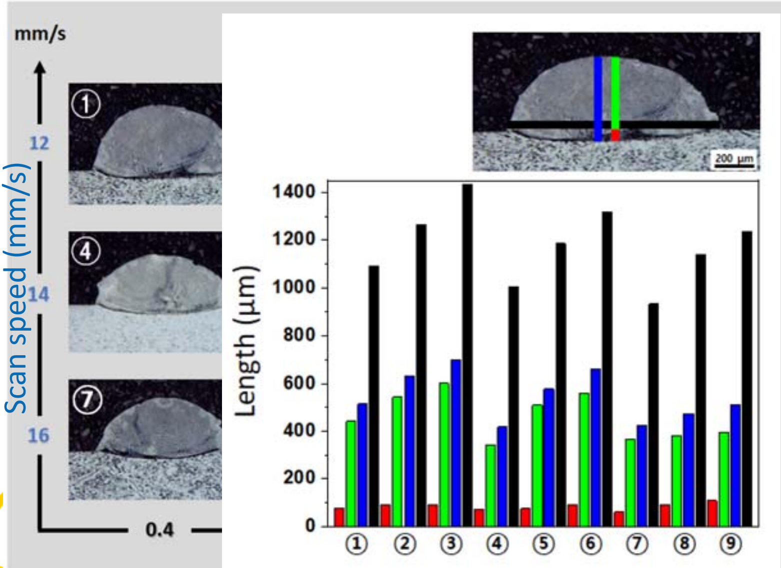


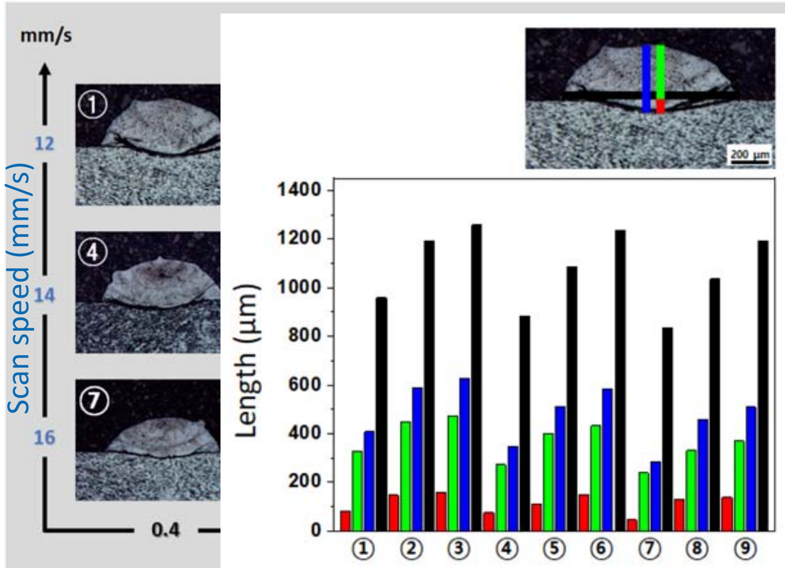
*Conducted in UNIST

[25] G Kim et al., Development of Fe–Cr–Si deposited layer manufactured by laser directed energy deposition process, JMRT, 2024

Results and discussions
DED coated layer characterization - parameter optimization



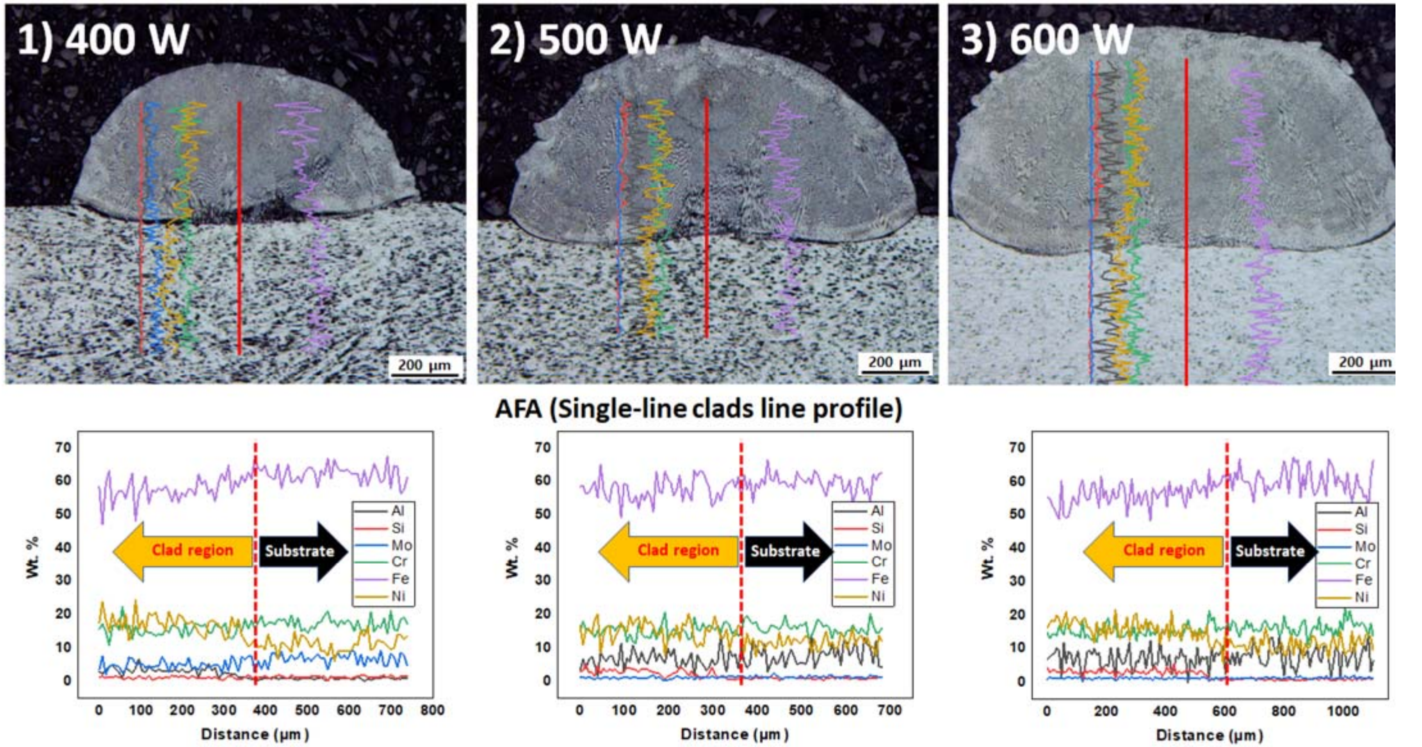




Dilution, width and height of single-line clad can be acquired (low dilution is preferred)

Results and discussions
DED coated layer characterization - parameter optimization

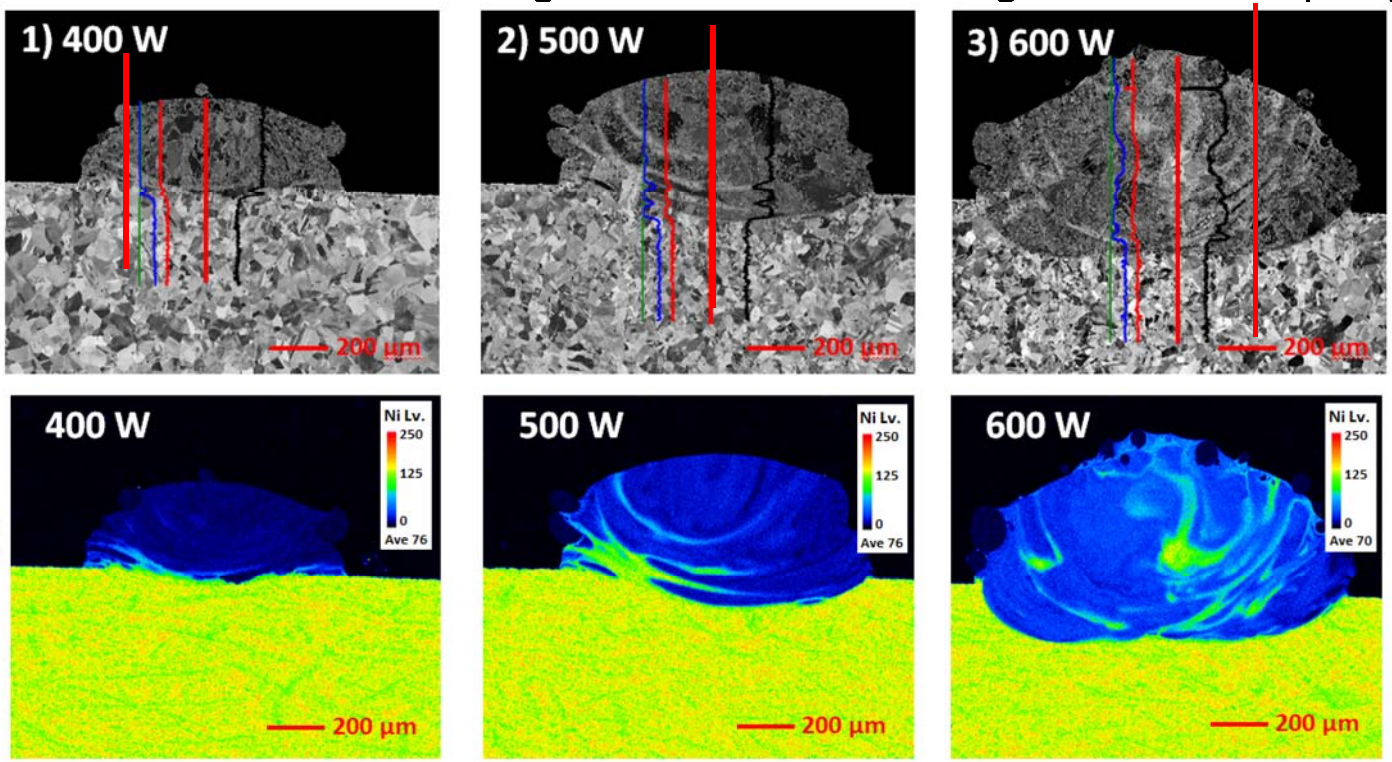
Component behavior of **AFA** single-line clads according to the heat input (EPMA)



Components of AFA clad and base metal (316L) show similar distribution

Results and discussions
DED coated layer characterization - parameter optimization

Component behavior of **Fe-Cr-Si** single-line clads according to the heat input (EPMA)

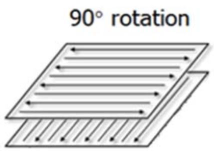


Fe-Cr-Si clad show the differential components distribution
(especially **Ni dilution**)

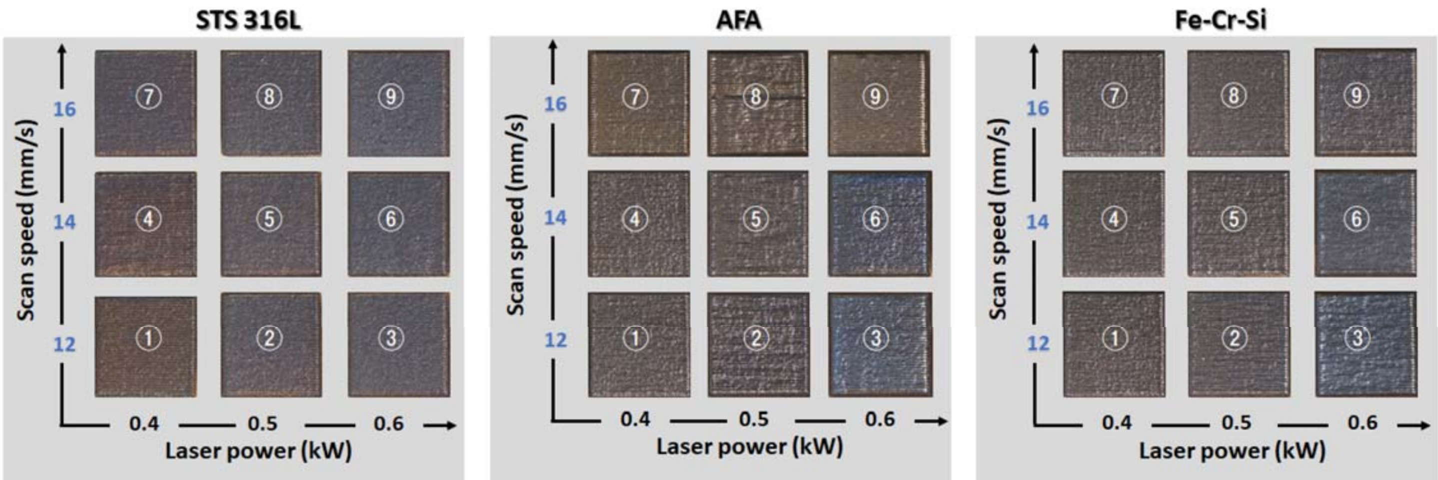
Results and discussions
DED coated layer characterization - parameter optimization

Surface coated layer characterization

- Laser power : 0.4, 0.5, 0.6 kW
- Laser scan speed : 12, 14, 16 mm/s
- Powder feed rate : 6.0 g/min (fix)



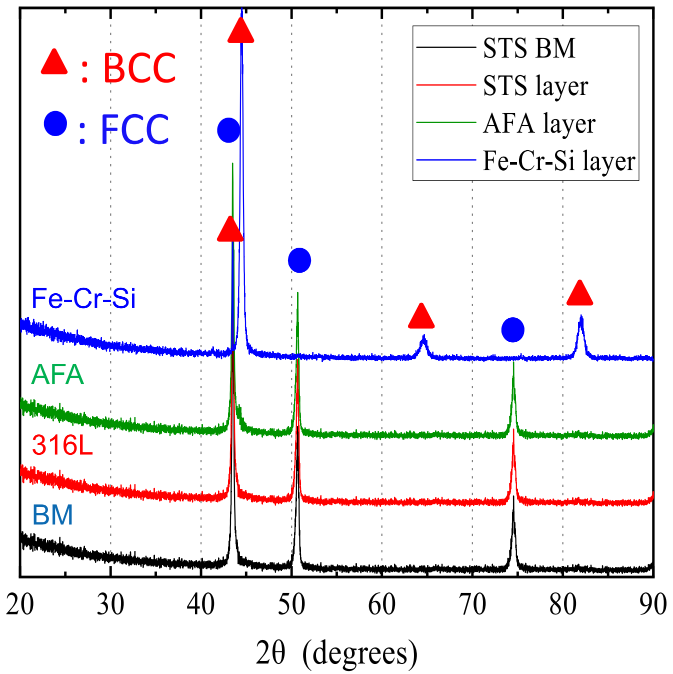
Zig-zag pattern
Two-layer
40 x 40 mm



Coated surfaces show the line-remaining morphology

Results and discussions
DED coated layer characterization - parameter optimization

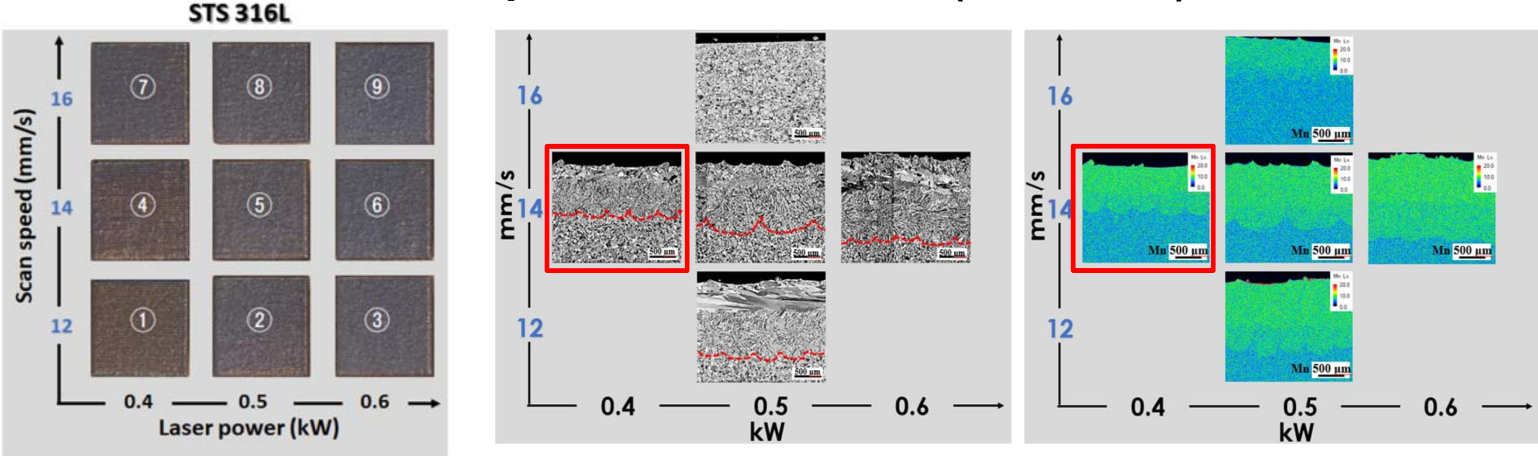
Surface coated layer characterization – surface XRD analysis

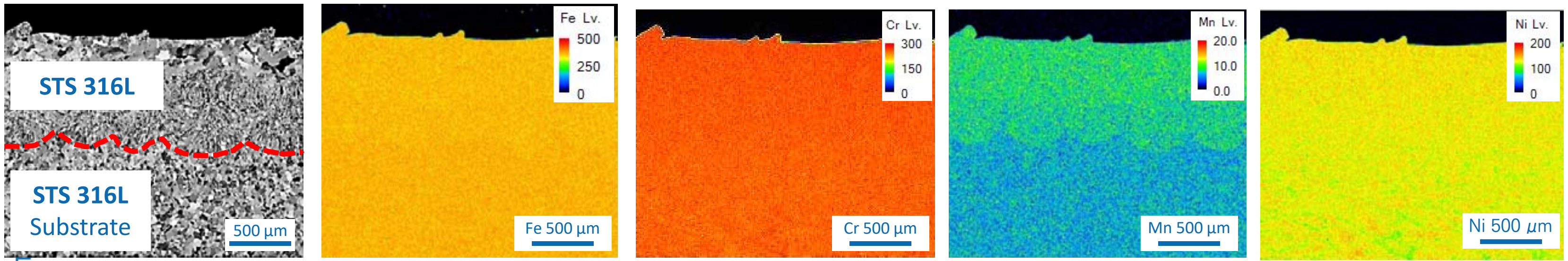


Substrate(316L), STS316L layer, AFA layer : FCC (austenitic)
Fe-Cr-Si layer : BCC (ferritic / martensitic)

Results and discussions
DED coated layer characterization - parameter optimization

Cross-section analysis of each deposition conditions (STS 316L)





No. ④ Condition : No defect, moderate deposited thickness

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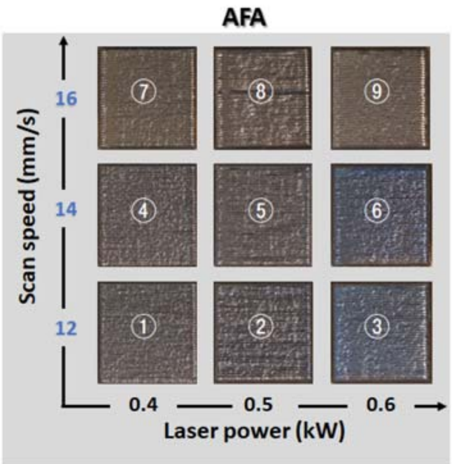
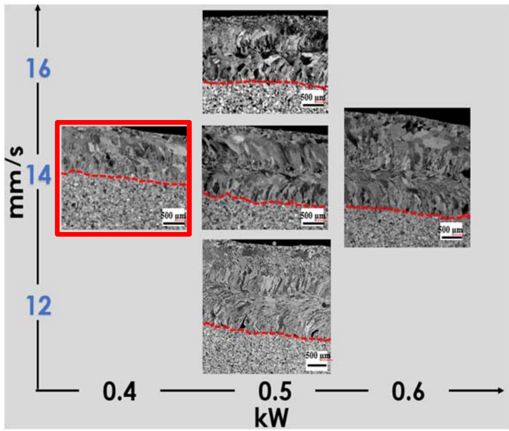
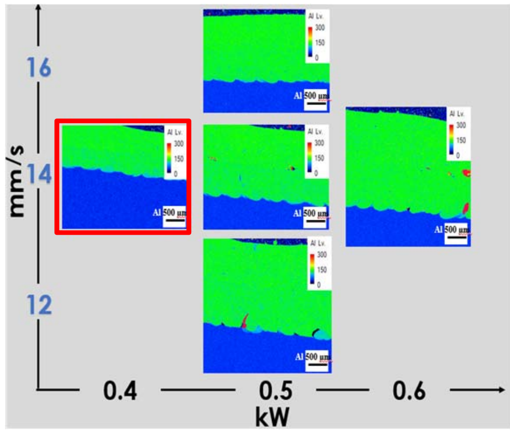
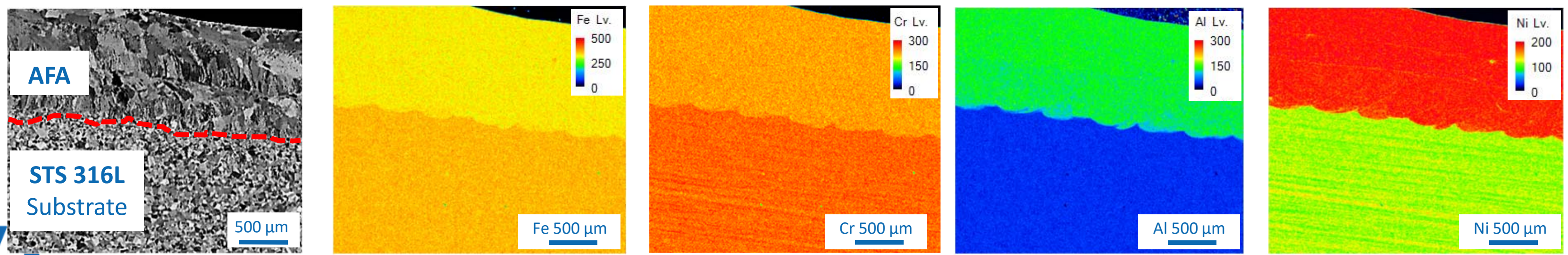
Results and discussions
DED coated layer characterization - parameter optimization

Cross-section analysis of each deposition conditions (AFA)





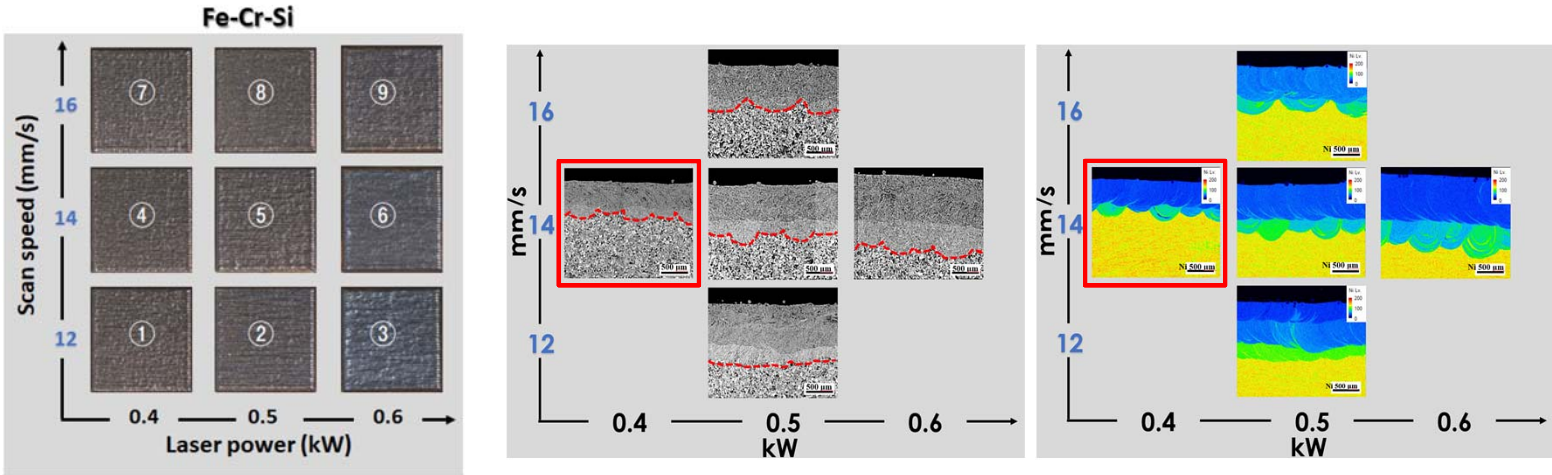


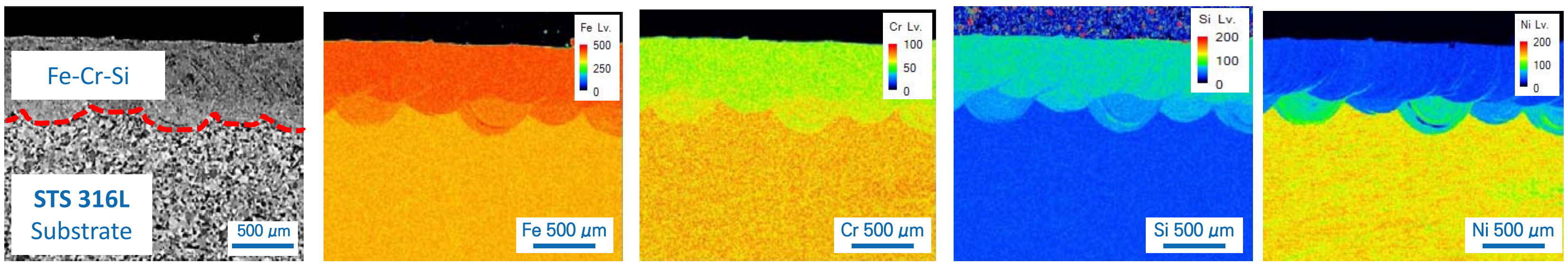


No. ④ Condition : No defect, moderate deposited thickness

Results and discussions
DED coated layer characterization - parameter optimization

Cross-section analysis of each deposition conditions (Fe-Cr-Si)

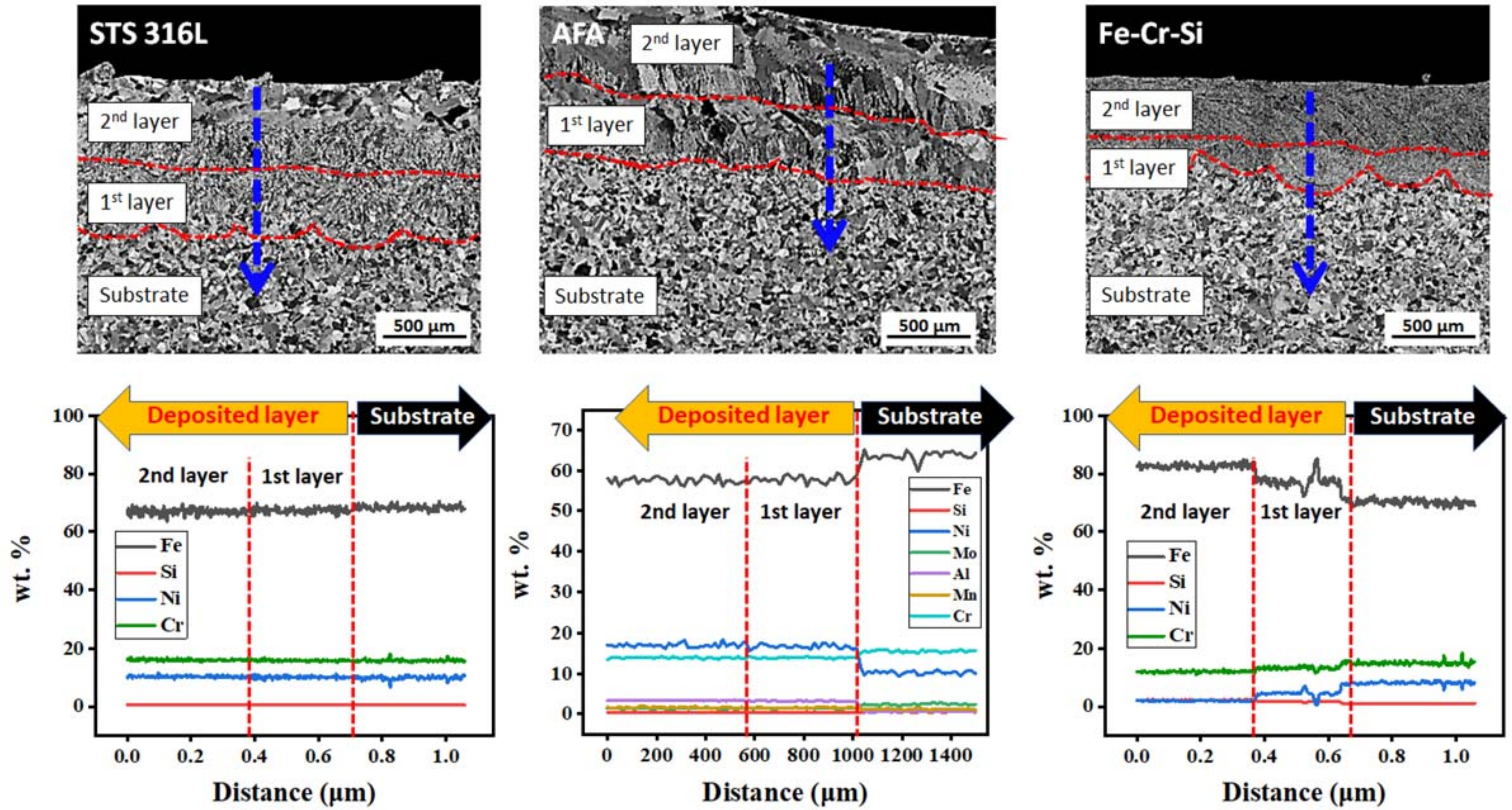




No. ④ Condition : No defect, moderate deposited thickness

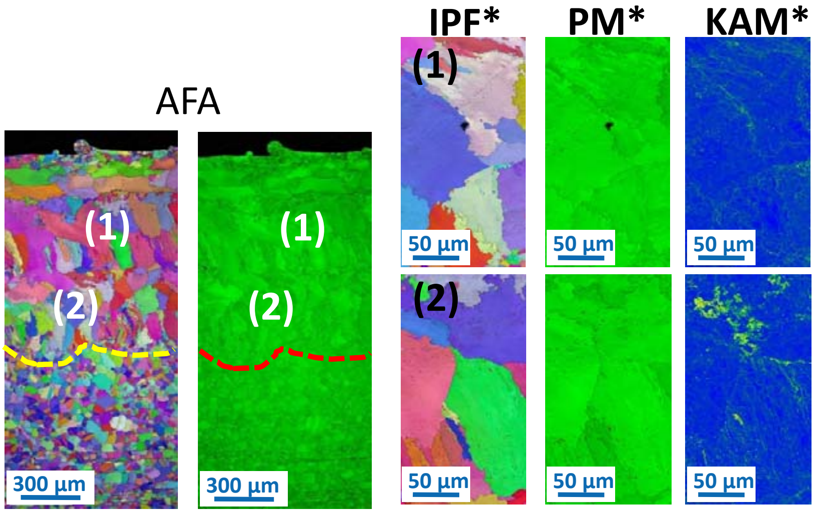
Results and discussions
DED coated layer characterization - microstructures

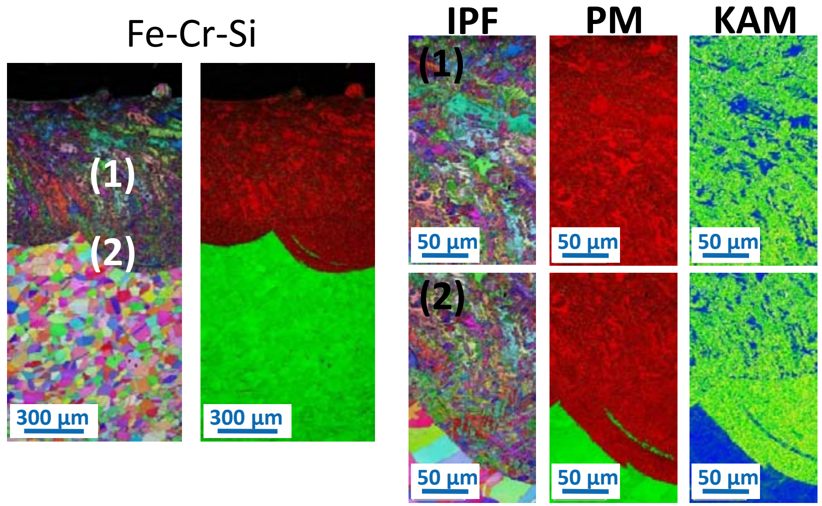
Composition line analysis of each clad material



Results and discussions
DED coated layer characterization - microstructures

Phase transformation behavior of coated layers (EBSD)





- AFA coated layer : FCC
- Fe-Cr-Si coated layer : BCC

1) First layer – deformation↑ & dislocation density↑
2) Second layer – deformation ↓ & dislocation density ↓

*IPF: inverse pole figure, PM: phase map, KAM: kernel average misorientation

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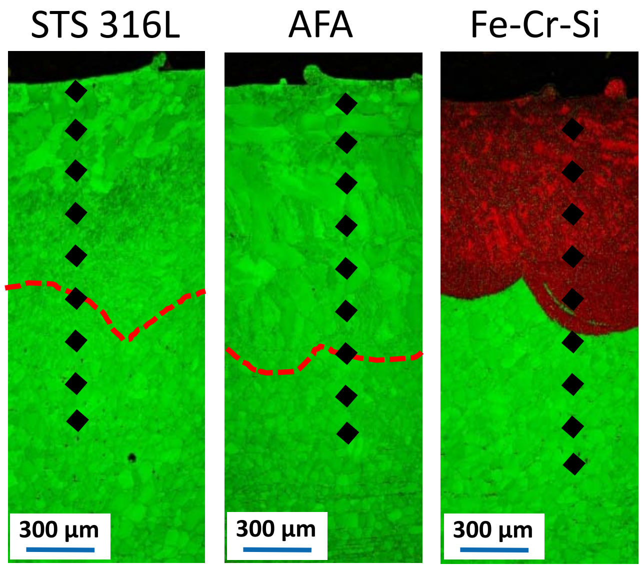
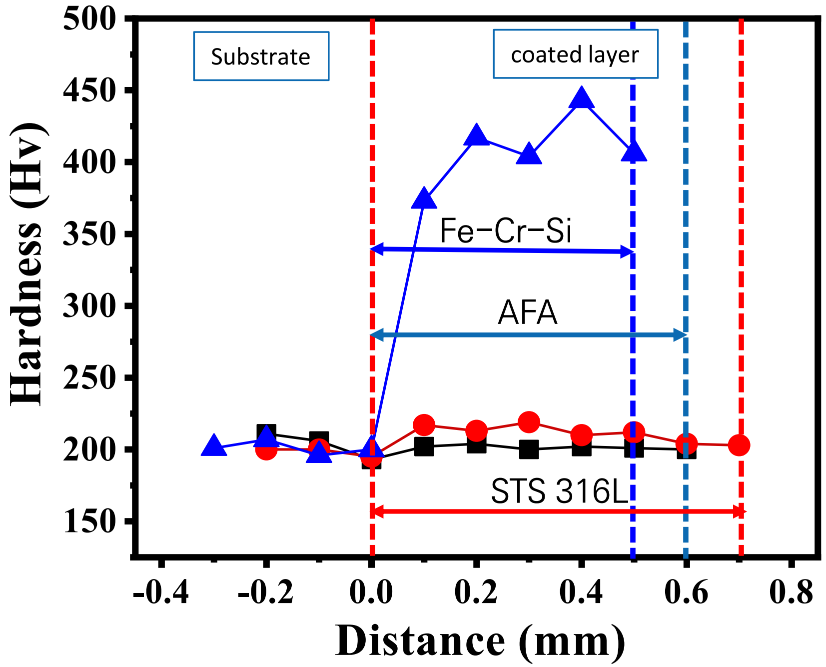
Results and discussions
DED coated layer characterization - hardness

Hardness distribution of coated layers (Vickers hardness test)



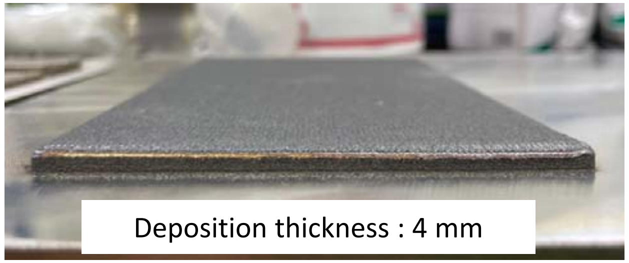


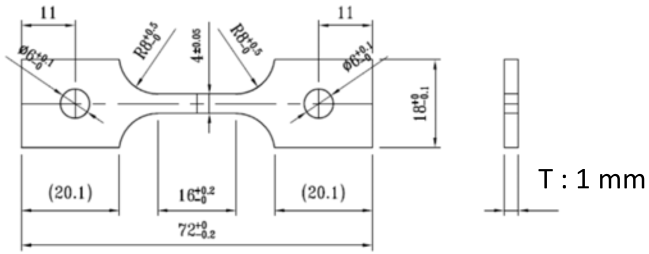
STS 316L, AFA : showed similar hardness throughout the layers including substrate (FCC phase)

Fe-Cr-Si : Ferritic / martensitic BCC phase shows over 400 Hv

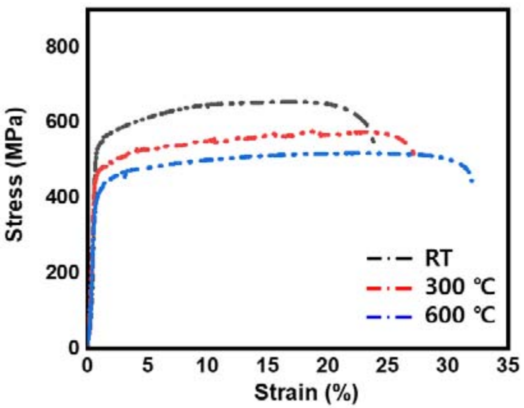
Results and discussions
DED coated layer characterization – tensile test

Tensile test of coated layers (R.T, 300 ℃, 600 ℃)

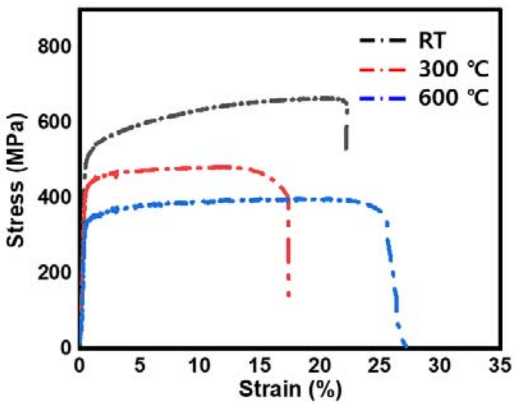




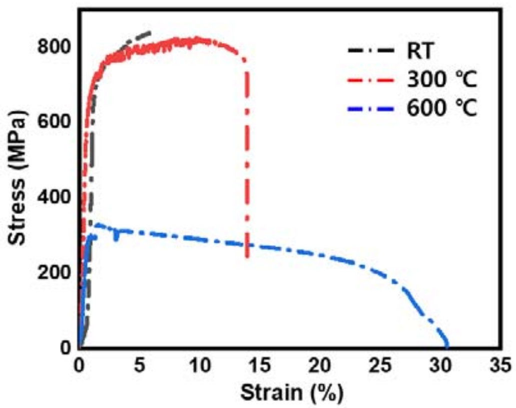
STS 316L



AFA



Fe-Cr-Si



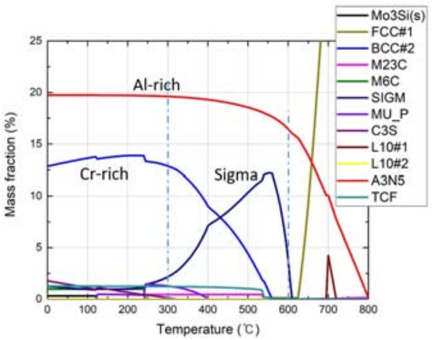
STS 316L, AFA : 600 ℃ results show lower tensile strength and higher elongation than 300 ℃ results

Fe-Cr-Si : Significant drop of tensile strength in 600 ℃ but elongation recovery occurred

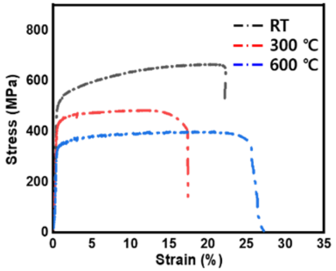
Results and discussions
DED coated layer characterization – tensile test

Tensile test: AFA

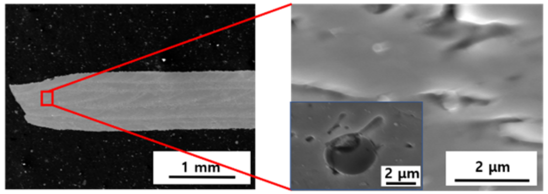
Coated layer	Temp. (℃)	YS (MPa)	TS (MPa)	E (%)
AFA	Room temp.	501	663	22.1
	300	442	481	17.4
	600	334	398	26.4

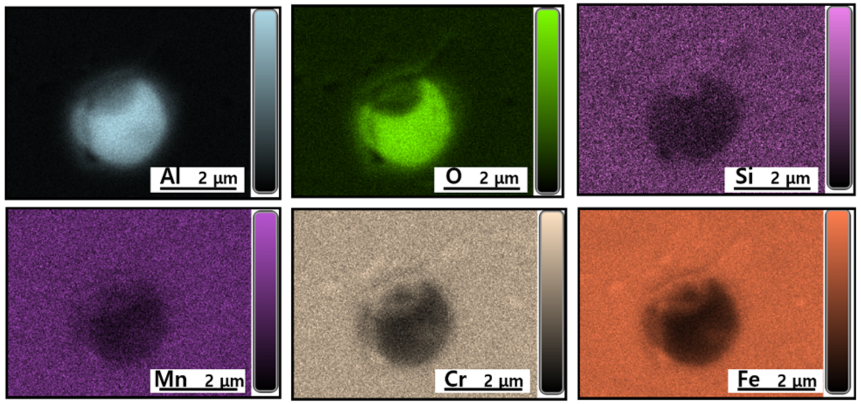


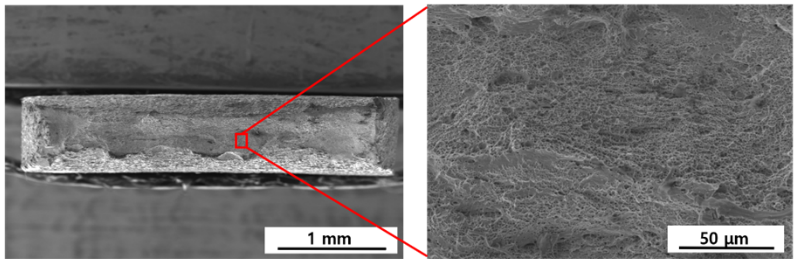
Equilibrium thermo-dynamic calculation (FactSage)



300 ℃ specimen







- High temp. : reduced Y.S, T.S ; but decreased elongation at 300 ℃
- Fracture surface of 300 ℃ specimen
  : huge oxide (Al_2O_3) formed
  ; may act as crack initiation site
- Fracture surface : dimple + cleavage

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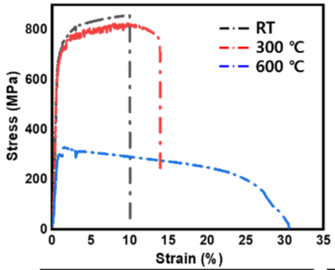
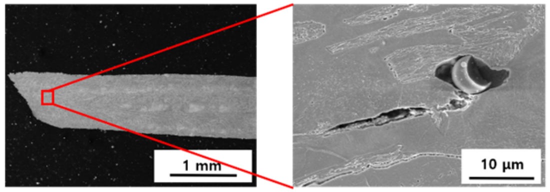
Results and discussions
DED coated layer characterization – tensile test

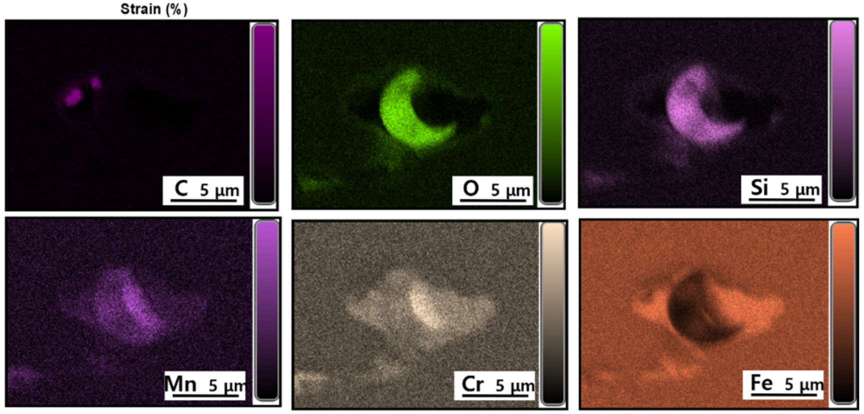
Tensile test: Fe-Cr-Si

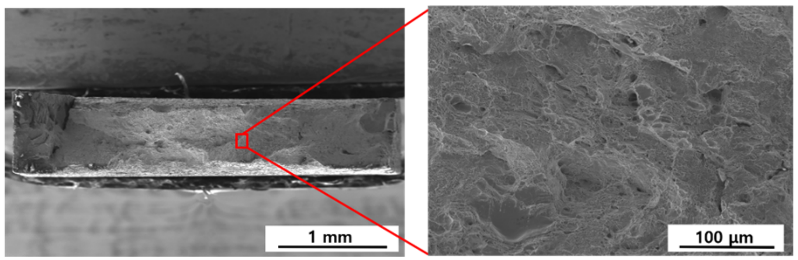
Coated layer	Temp. (℃)	YS (MPa)	TS (MPa)	E (%)
Fe-Cr-Si	Room temp.	681	857	10.1
	300	672	821	13.9
	600	260	327	30.5



300 ℃ specimen







- High temp. : reduced Y.S, T.S ; but decreased elongation at 300 ℃
- Fracture surface of 300 ℃ specimen : huge oxide (SiO_2) formed ; may act as crack initiation site
- Fracture surface : dimple + cleavage

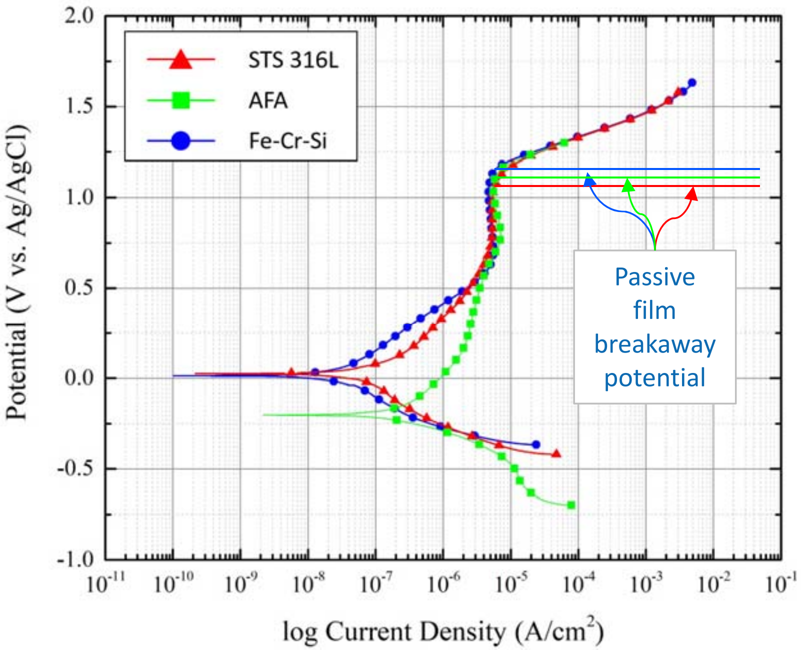
Results and discussions
DED coated layer characterization – general corrosion

General corrosion test of coated layers

- Potentiodynamic polarization test in 0.5M H_2SO_4 solution
  (test range : - 0.5 V ~ 1.5 V(open circuit), 0.833 mV/sec)

Tafel fitting parameters of the test

Coated layer	E_{corr} (mV vs. Ag/AgCl)	I_{corr} (nA/cm^2)	Corrosion rate (mm/yr)
STS 316L	24.4	111.0	0.051
AFA	-203.0	270.0	0.123
Fe-Cr-Si	14.6	38.2	0.017



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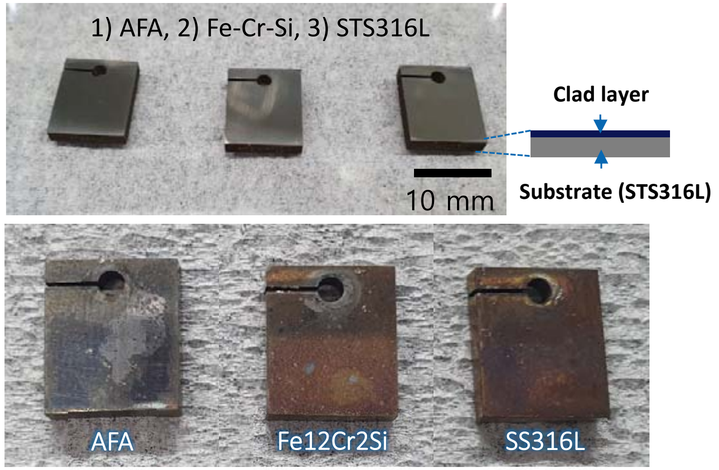
AFA : lower corrosion potential and higher corrosion rate than 316L and Fe-Cr-Si
Fe-Cr-Si : lowest corrosion rate, passive films are more stable than 316L and AFA

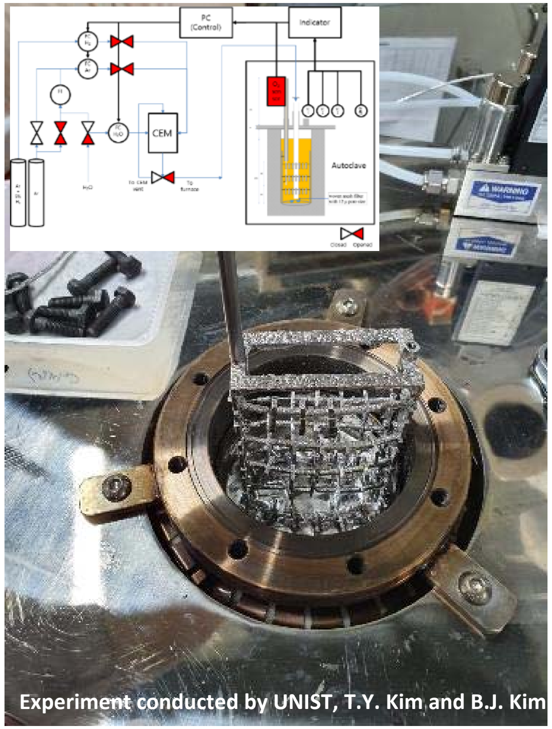
Results and discussions
DED coated layer characterization – LBE exposure test

High temperature LBE exposure test

- Specimen size : 10 x 12 x 2t mm
- Temperature : 550 ℃
- Oxygen concentration : 1e-7 wt.%
- Exposure time : 500 hr, 2000 hr



500 hr exposure

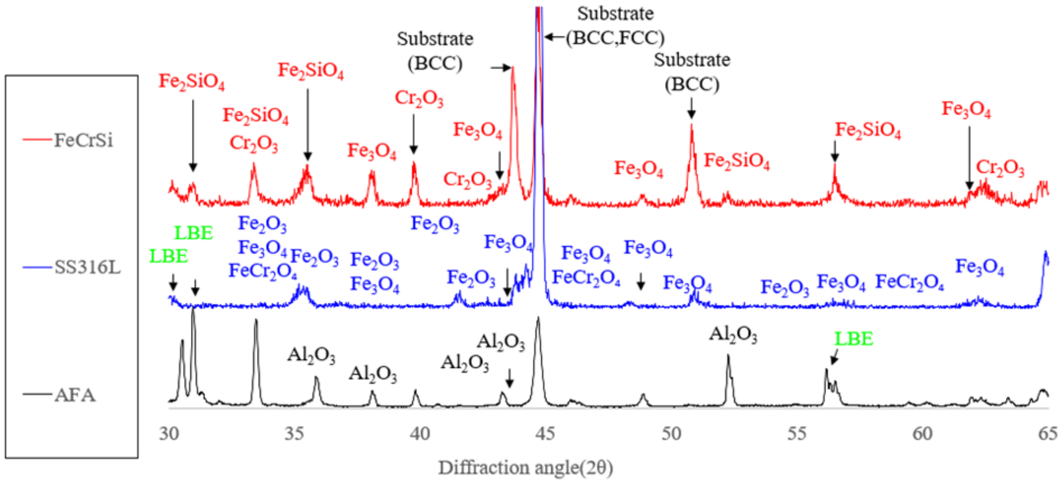


Analysis : XRD (phase identification)
TEM (microstructure evaluation of oxide layer and corrosion area)
EDS (Chemical composition of the oxide layer and corrosion area)

Results and discussions
DED coated layer characterization – LBE exposure test

High temperature LBE exposure test

- XRD Phase analysis after 2000 h exposure



XRD results of STS 316L/AFA/Fe-Cr-Si coated specimen exposed for 2,000 hours in LBE environment at 550 ℃

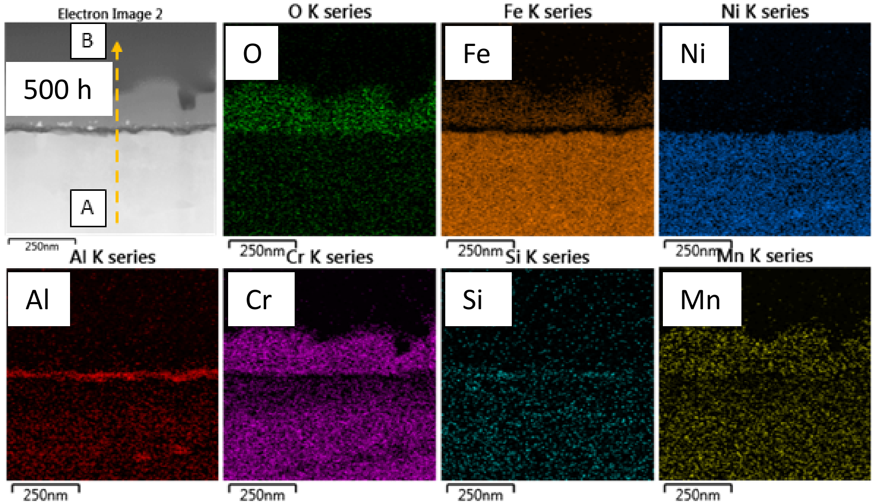
- STS 316L : Fe_3O_4, $FeCr_2O_4$ and FCC phase peak (general peak of STS 316L)
- AFA : Al_2O_3, Fe_3O_4, $FeCr_2O_4$ and FCC phase peak (Al-oxide formed)
- Fe-Cr-Si : Fe_3O_4, Cr_2O_3, Fe_2SiO_4 and BCC/FCC phase peak (Fe_2SiO_4: assumed as Si oxide)

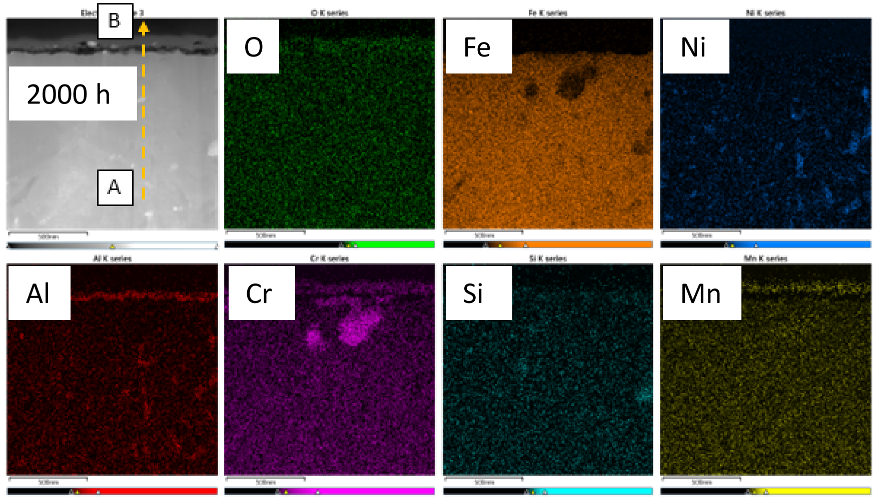
Similar surface phase constitutions as bulk materials

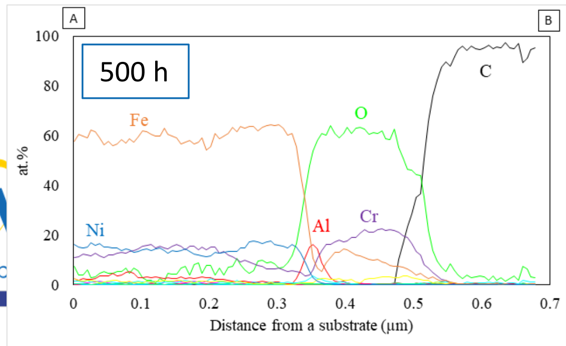
Results and discussions
DED coated layer characterization – LBE exposure test

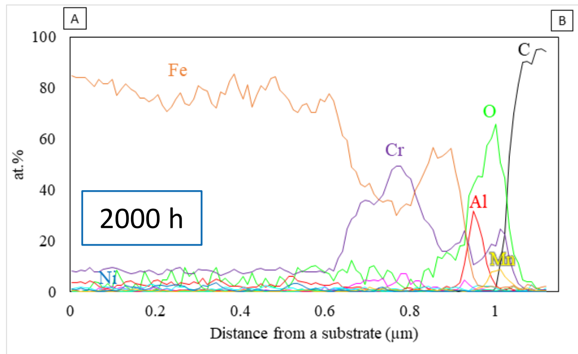
High temperature LBE exposure test

- TEM analysis after 500 , 2000 h exposure (AFA)





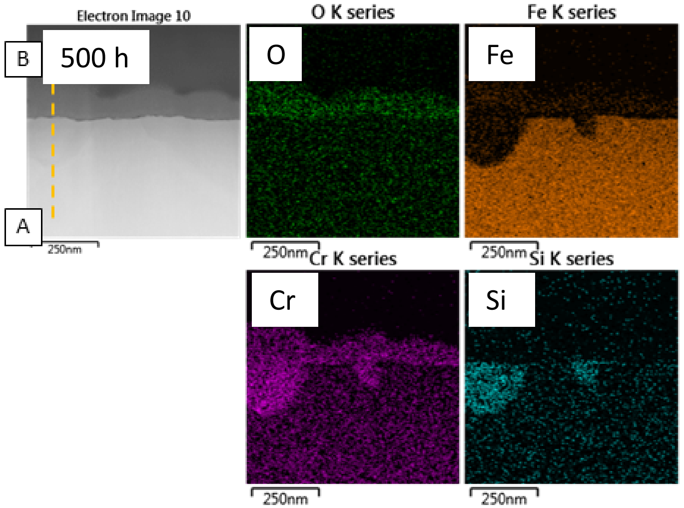


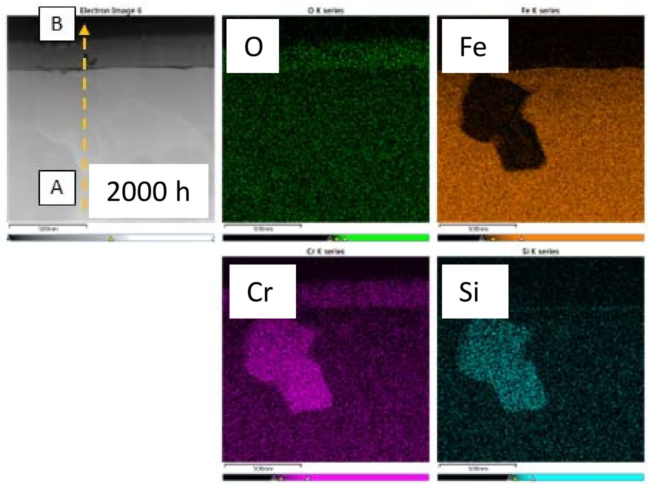


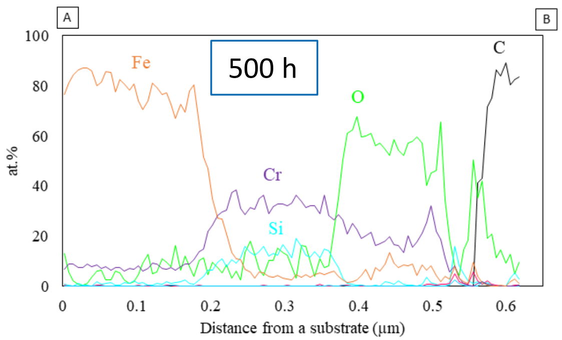
Results and discussions
DED coated layer characterization – LBE exposure test

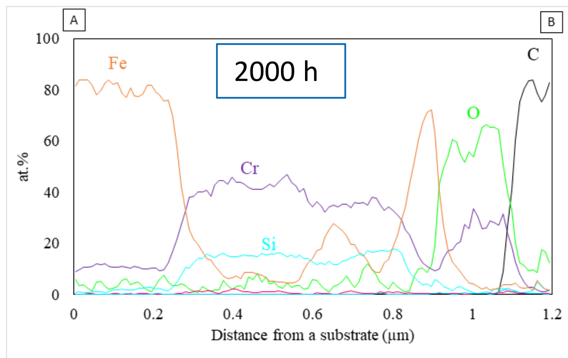
High temperature LBE exposure test

- TEM analysis after 500 , 2000 h exposure (Fe-Cr-Si)









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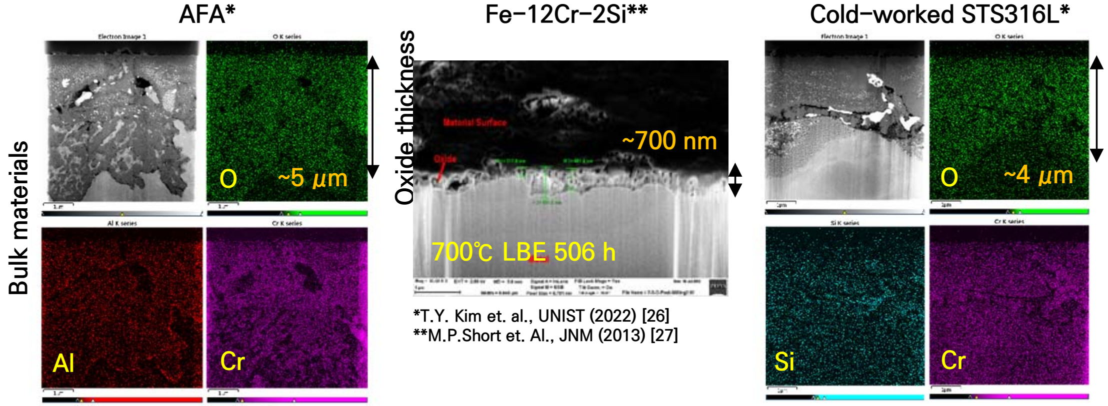
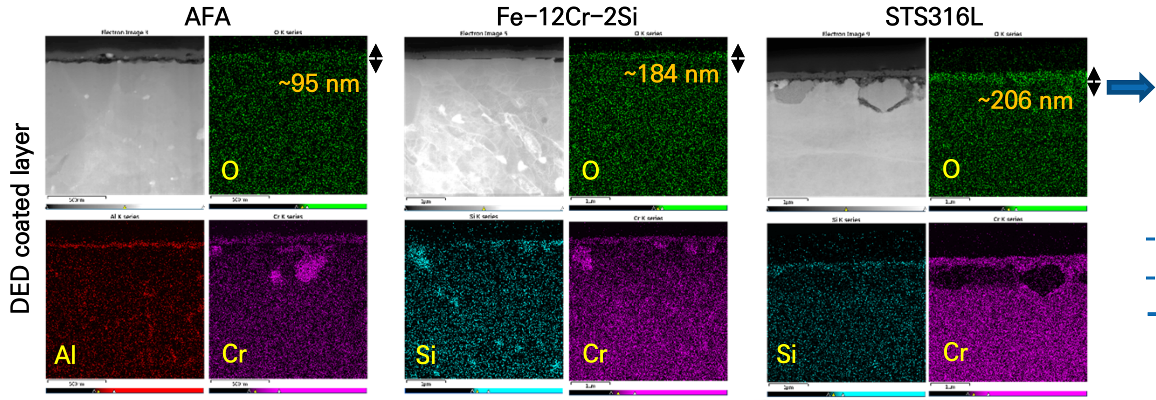
Results and discussions
DED coated layer characterization – LBE exposure test

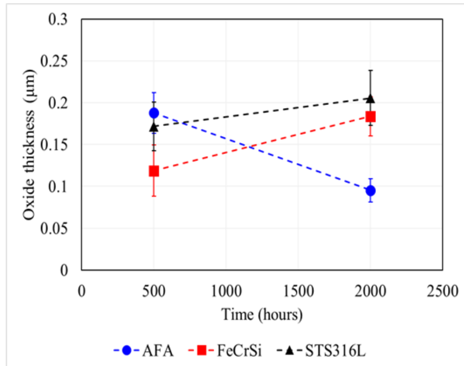
High temperature LBE exposure test - Comparison with bulk material



Bulk specimen exposed in LBE environment at high temperature



TEM-map EDS results of DED coated specimen exposed for 2,000 hours in LBE environment at 550 ℃



- Thin and dense oxide layer
- Slow oxide growth rate

→ **Leads to longer material life**

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[26] Experiment data from UNIST, [27] M. P. Short et. al., Journal of Nuclear Materials, 2013

Conclusion
Optimization of DED process parameters

Suggest the optimized DED process parameter

Metal powder

- Particle size (㎛) : 45 - 150 in diameter (average : 120 - 130)
- Angle of repose (˚) : max. 33
- Hausner Ratio : 1 - 1.1
- Flowability (sec./50g) : max. 14

DED parameter

- Laser power : 0.4 kW
- Laser scan speed : 14 mm/s
- Powder feed rate : 6.0 g/min
- Overlap between the interpass : 0.5 mm

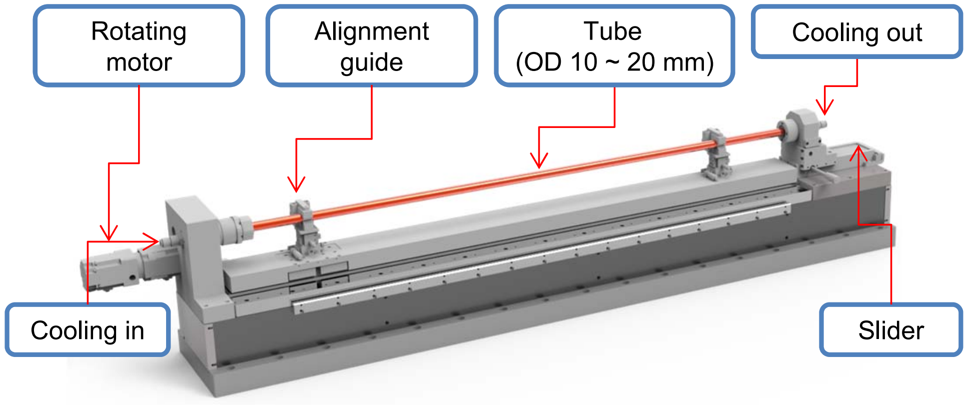
DED parameters

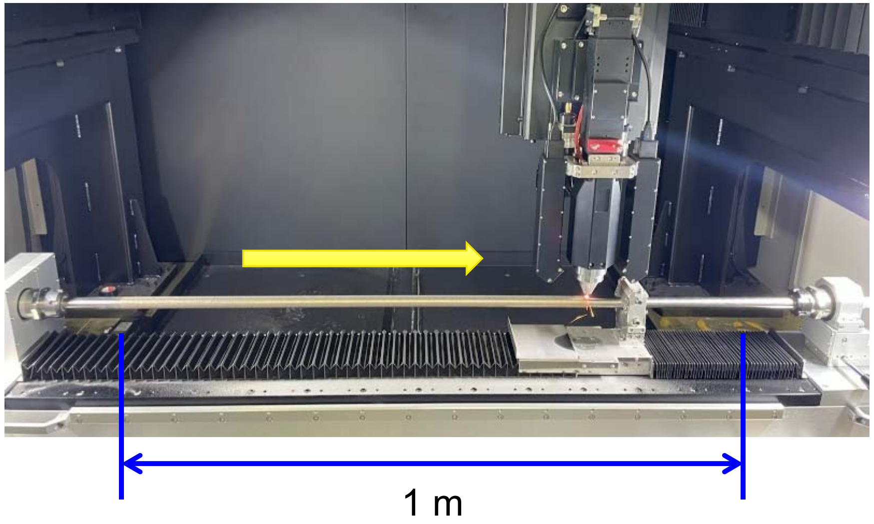
Condition	Laser power (kW)	Scan speed (mm/s)	Line energy input (J/mm)
1	0.4	12	33.33
2	0.5	12	41.67
3	0.6	12	50.00
4	0.4	14	28.57
5	0.5	14	35.71
6	0.6	14	42.86
7	0.4	16	25.00
8	0.5	16	31.25
9	0.6	16	37.50

Optimized DED parameters (including powder requirements) were suggested for **defect-free** and **restricted base metal dilution**

Conclusion
DED coating to final dimension pipe

Rotating fixture for tube coating : overcome the barriers of **post processing** and **Code & Standard**



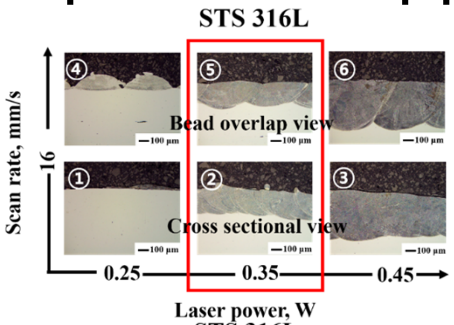


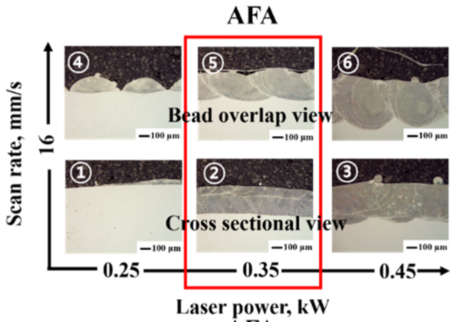
DED equipment for fuel cladding surface coating has been prepared

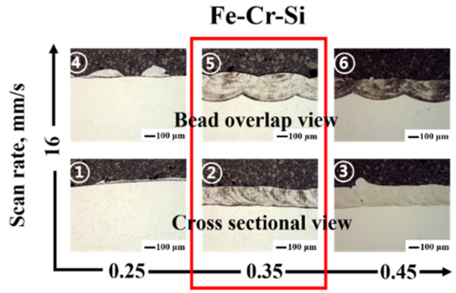
Conclusion
DED coating to final dimension pipe

Coating parameter optimization for pipe outside

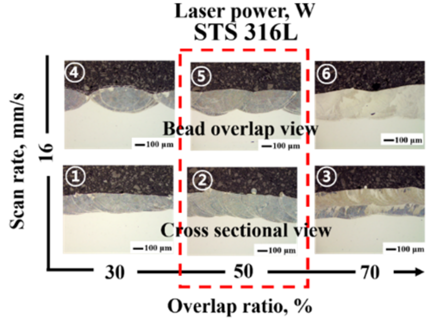
Laser output

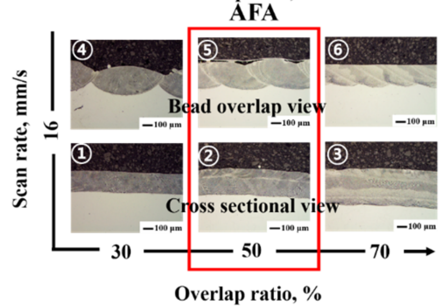


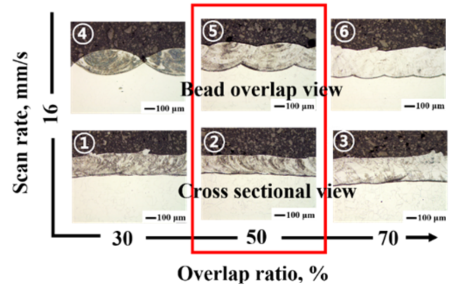




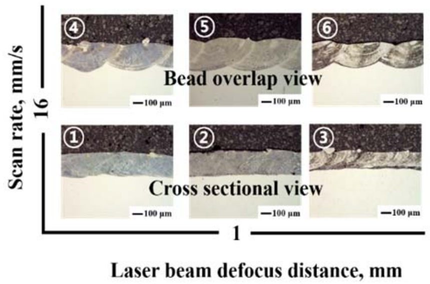
Overlap ratio

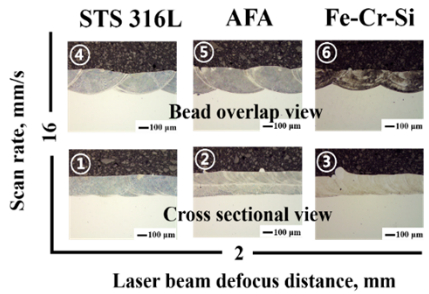






Laser beam defocus





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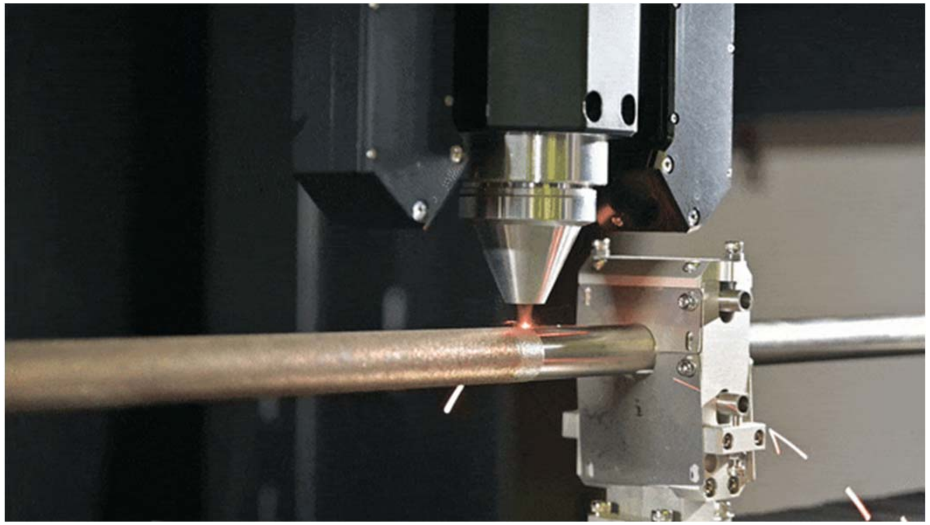
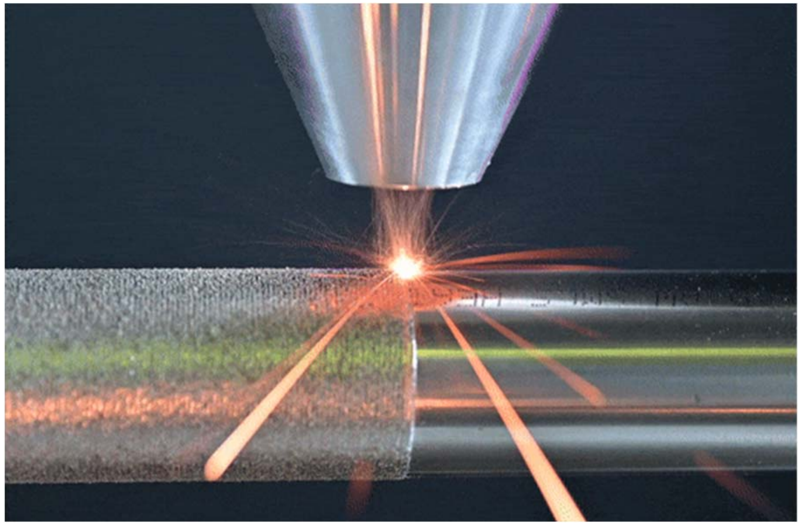
Conclusion
DED coating to final dimension pipe

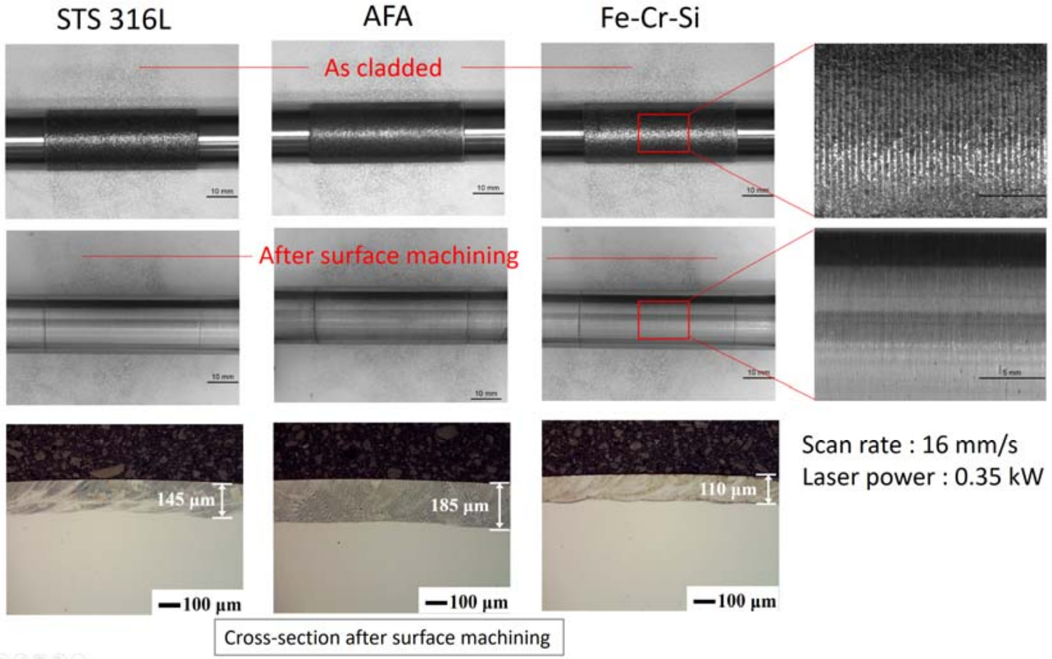
- **Overcome the barriers of post processing and Code & Standard**

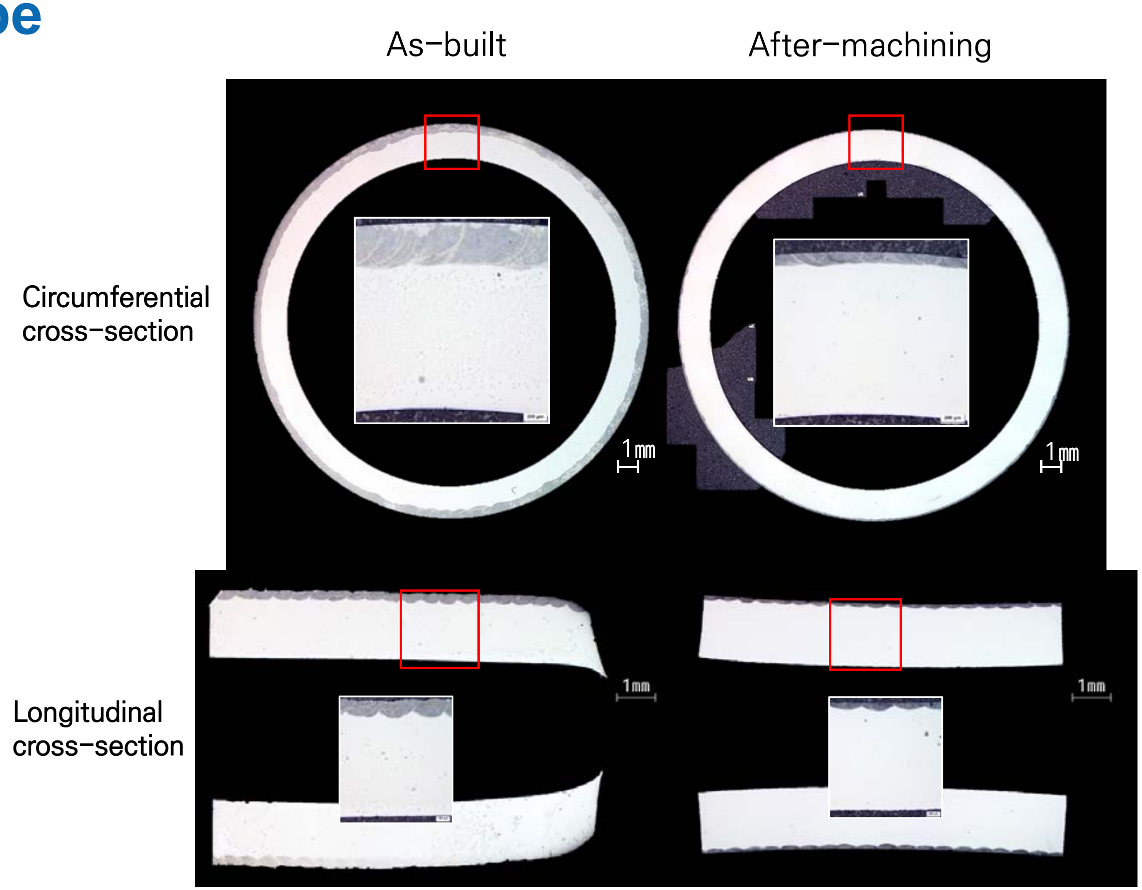




Feasibility verified to build **thin-surface coating** of **LBE corrosion resistant materials** on the **final size of fuel cladding** using rotating tube DED process with **optimized process variables**

Conclusion
DED coating to final dimension pipe





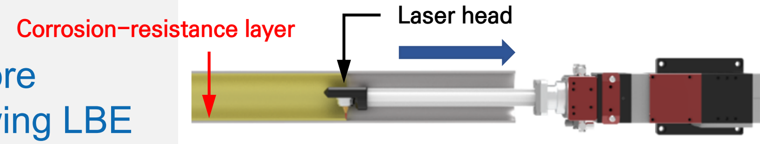
Summary and future work

- **Proposal of methodology for surface coating technology using DED technique**
- **Optimization of coating layer manufacturing process of LBE corrosion resistant materials**
- **Conducting various property evaluations of the DED coated layer**

- Microstructures and composition evaluation
- Mechanical properties : Hardness, (high-temp.) tensile test
- Corrosion property
- High-temp. LBE exposure test

Implications and future work

- Various data could be the basis for the use of coated layer to materials for SMR
- For Code registration of developed materials, more mechanical properties are required (more high temperature tensile data, creep and fatigue data, etc.)
- To predict the life of the coated layer, more detailed LBE exposure test (including flowing LBE test) shall be conducted and verified
- The methodology for inner coating shall also be developed



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Upcoming Webinars

Date	Title	Presenter
31 July 2024	Online monitoring development in support of the nuclear fuel cycle	Amanda Lines and Sam Bryan, PNNL, USA
28 August 2024	International Molten Salt Research in Support of MSR Development	Aslak Stubsgaard, Denmark Isabelle Morlaes, France Ed Pheil USA Markus Piro, Canada Jeremy Pearson, USA
September 2024	Overview and Update of Sodium Fast Reactor Activities within the Gen IV International Forum	Yoshitaka Chikazawa, JAEA, Japan

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