INL/EXP-24-76296-Revision-0

Electric Field Assisted Sintering (EFAS) Capabilities at INL

March 2024

Changing the World's Energy Future

Gabriel O Ilevbare, Jorgen Fredrick Rufner, Xinchang Zhang, Michael D McMurtrey, Andrew James Gorman, Tate Patterson, Ryann Bass

Idaho National Laboratory

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Prepared for the
U.S. Department of Energy
Under DOE Idaho Operations Office
Contract DE-AC07-05ID14517

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Electric Field Assisted Sintering (EFAS) Capabilities at INL

The Ohio State University Visit

18th January 2024, Idaho Falls, Idaho, USA

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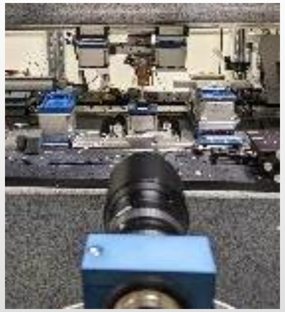
INL/PRO-XX-XXXXX

INL AM Capabilities Include

- Welding Technology Based Techniques:
  - Gas tungsten arc welding (GTAW) wire arc additive manufacturing (WAAM)
  - GTAW multi-wire arc additive manufacturing (mWAAM) with three wire additions for functionally-graded components
  - Hybrid/tandem laser arc additive manufacturing
  - Direct Energy Deposition Laser
- Plasma Jet Printing (Direct write system)
- Powder Bed Printing
- LENS
- Digital Light Printing (DLP)
- **Electric Field Assisted Sintering Capabilities – EFAS (SPS)**
  - Nano EFAS (Beamline Capable)
  - Micro EFAS (DCS-5; 5-Ton)
  - Fuji Dr. 515 (5-Ton)
  - DCS-25-10 (25 Ton)
  - DCS-800 (800-Ton)
  - Continuous EFAS

Electrifying Industrial Manufacturing- Sintering

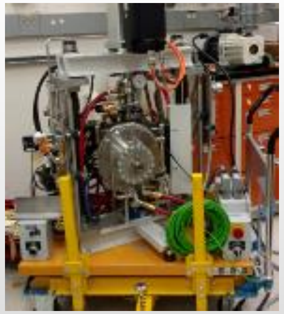
Center of Excellence for
Electric Field Sintering Sciences



Nano-EFAS

Real Time Chemical and Structural Evolution

5 Ton



DCS-5

In-situ Analysis of Microstructural Evolution During Materials Processing

25 Ton

DCS 25-10

Advanced nuclear fuels fabrication

800 Ton

DCS-800

Industrial Scale-up

CEFAS

Converts Traditional Batch Process to Continuous Process

Cold Sintering & Cold Flash Sintering

Uses Fluids to Lower Sintering Temperature and Speed

Metals, Ceramics and Composites

EFAS Applications and Benefits

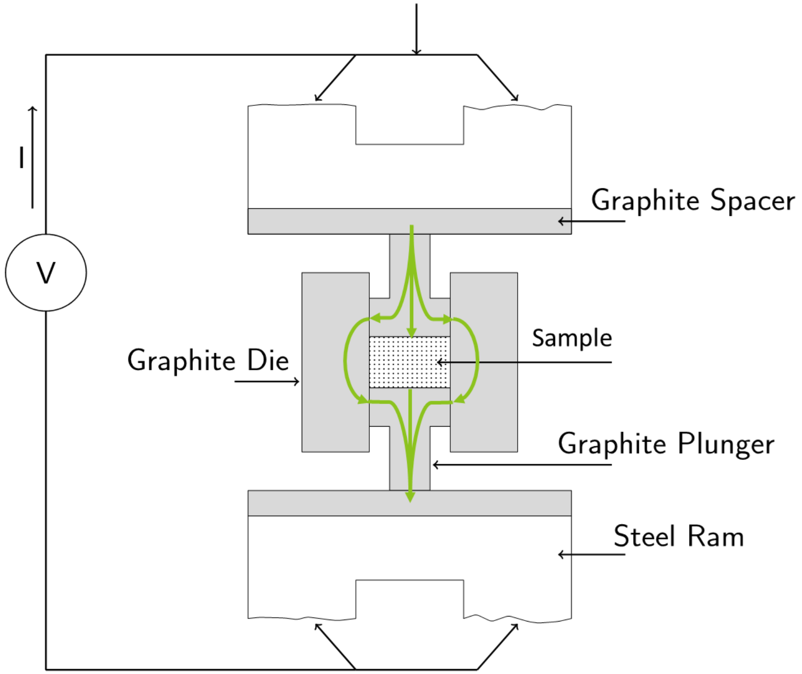
- Relatively fast sintering times on the order of minutes to hours (1+ hours for 12” samples)
- ***Cost savings of up to 90% per part compared with conventional methods***
- Usable on many materials classes including ultrahigh temperature ceramics (reaches 2500 ^{o}C)
- Net shaping/Net Near Shaping of parts
- Application of Simultaneous Pressure
- Usually produces fully dense parts with reduced final grain sizes compared with traditional methods
- Smoother surface finish and more tolerance friendly technique
- Application is broad-based across industries: Energy Generation, Automotive, Aerospace etc.
- Ability to manipulate heating profile to suit material and intended outcomes
- Effective for scaled prototyping of new systems and materials

Manufacturing Method/Cost Energy Comparison		
	Energy Use	Cost for 5,000 Parts
Hot Pressing	1.83kW/g	$5.7 Million
EFAS	0.1kW/g	$0.52 Million
EFAS Savings	**1.82kW/g**	**$5.2 Million**

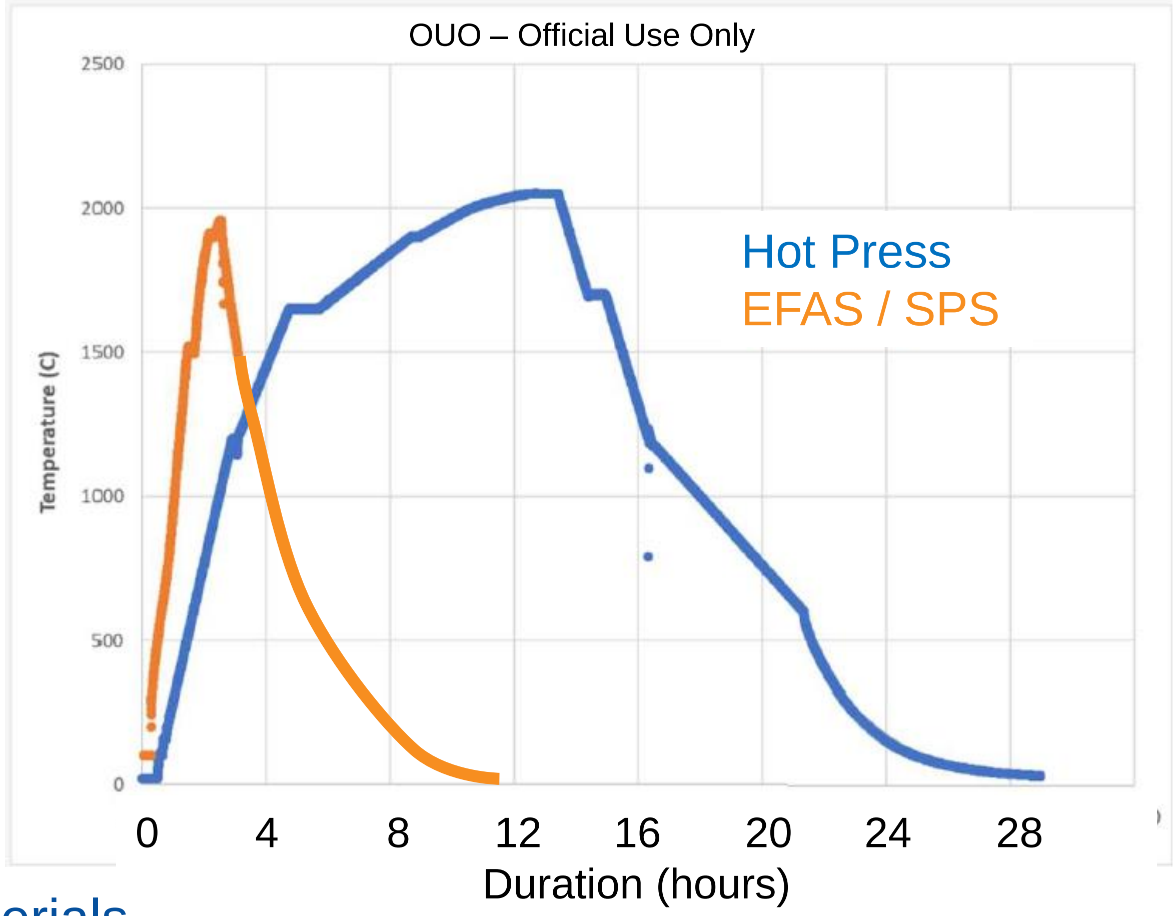
What is EFAS and why do people care about it?

Joule Heating

Current Density



Schematic of EFAS technique rams, tooling, and powder[1]

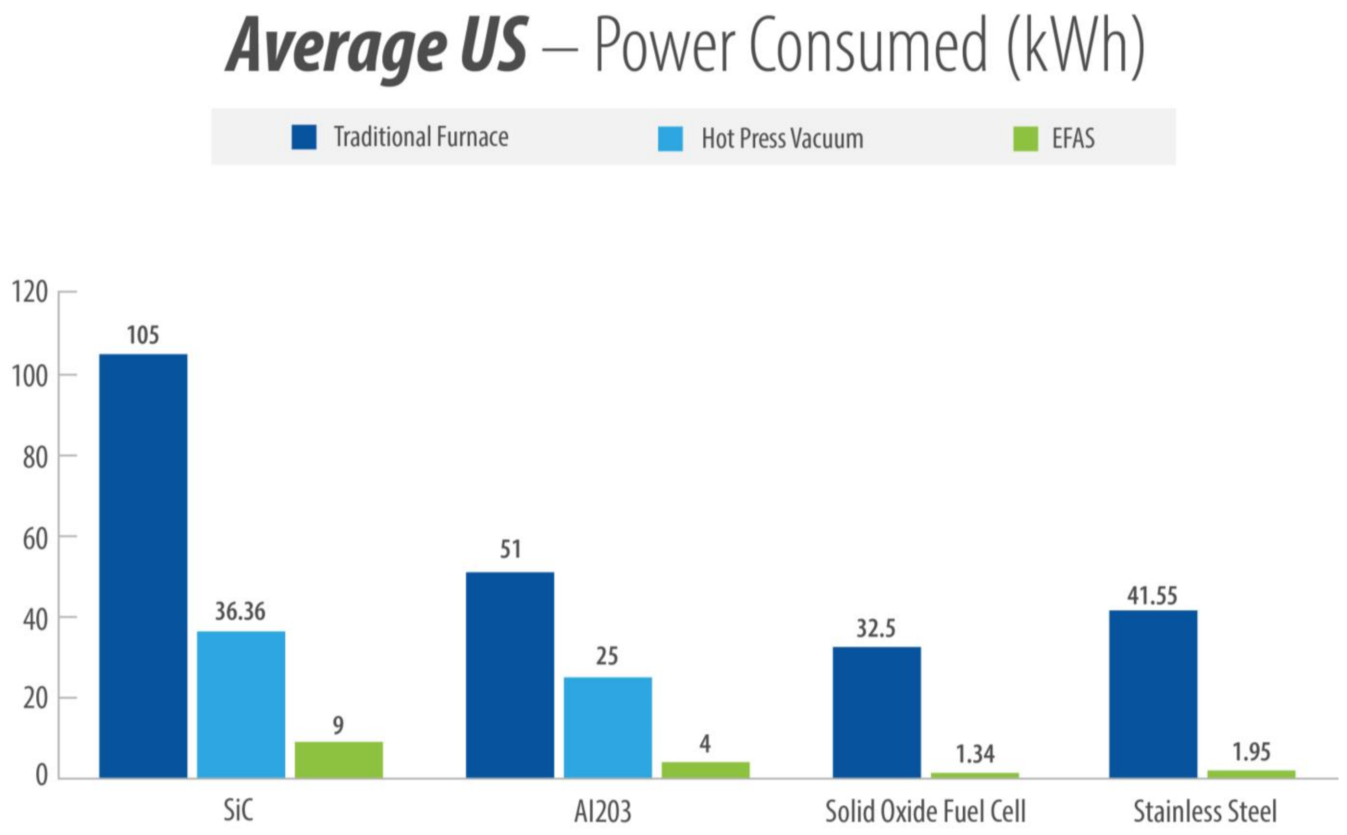


- Exceptional at consolidating difficult to process materials.
- 60-70% faster than closest technological “cousin”.
- Direct heating of sample/mold ensemble.
- Proven at small/research scale → scale up @ INL (DCS-800).

INL Electric Field Assisted Sintering (EFAS)

Highly efficient → reduced power, cost, and CO_2 through electrified materials manufacturing

- Decreased time/cost:
  - from traditional sintering ≈ 90%
  - from hot pressing ≈ 70%

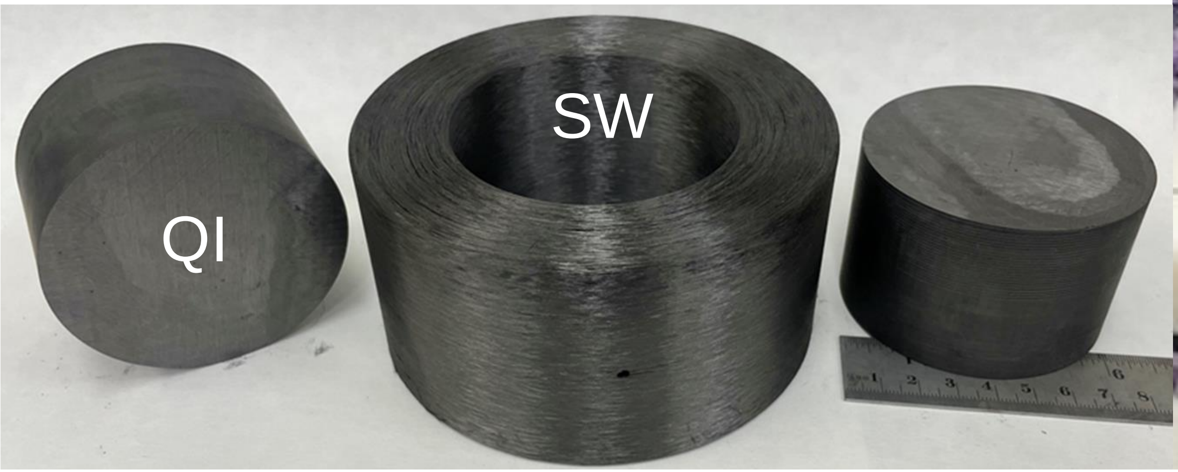


CO_2 Emissions for a single part consolidation (kg)				
	Traditional Furnace	Hot Pressing	EFAS	% Change
SiC	40.7	14.1	3.5	- 91.4%
Al_2O_3	19.8	9.7	1.6	- 91.9%
SOFC	12.6	--	0.5	- 96.0%
SS	16.1	--	0.8	- 95.0%

ARPA-E Revolutionary Ironmaking Industry & Proposers' Day 2023-June-15 Chicago IL

Large Scale Graphite Tooling

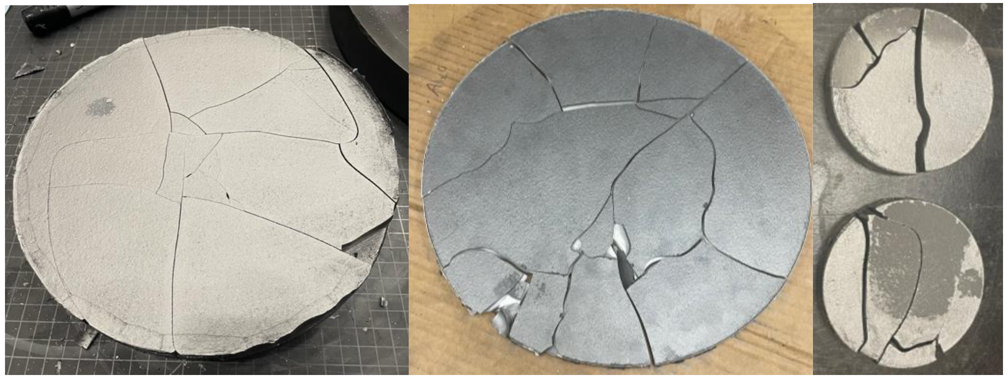
- However, scaling up to industrially relevant sizes brings challenges.
- Large graphite tooling:
  - CTE
  - Thermal gradients
  - Suboptimal quality at the largest sizes
  - LONG procurement lead times
  - Expensive.
  - Fails catastrophically (brittle fracture).
- C-C tooling can be designed for strength and thermal properties:
  - Fiber directionality mechanically, thermally, electrically
  - Spirally wound (SW) fiber orientation for high hoop strength
  - Quasi-Isotropic (QI) fiber layering for high compressive strength



Scaling up processing

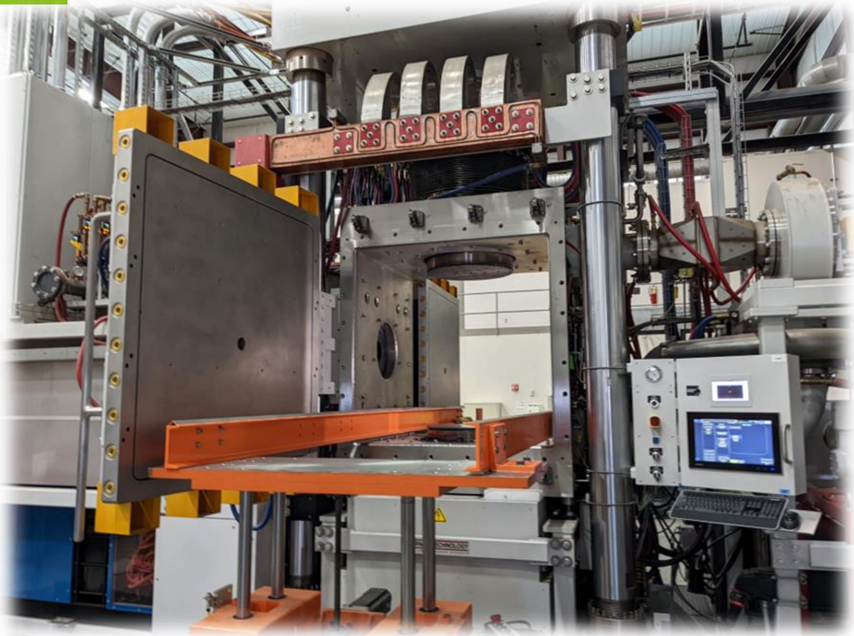
- Poor heat distribution can cause thermal expansion and contraction mismatches which lead to stresses in ceramic parts
  - Has been observed in ceramic parts from 3-12” in diameter
    - Can be solved with graphite tooling, but special processing and tooling configurations must be used.

12” 12” 3”

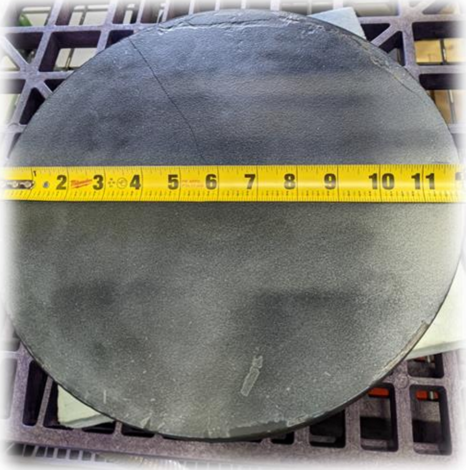


Intact 12” diameter ceramics

EFAS Scale Up
DCS-800



12” diameter Al_2O_3



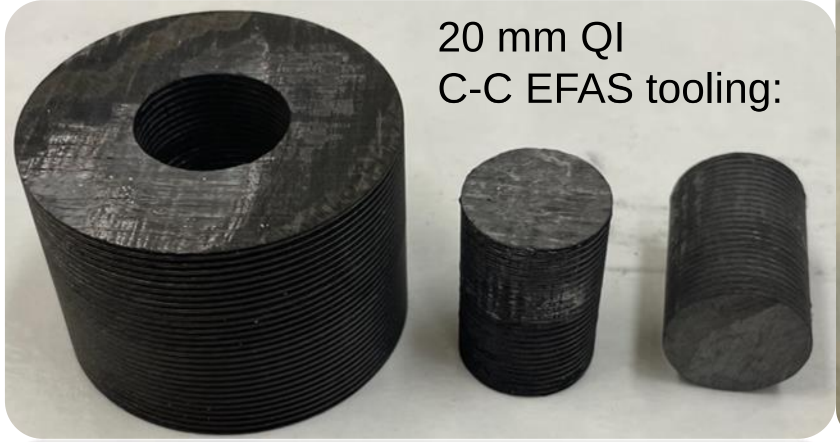
10” diameter Ni-WC

20in I.D. / 36” O.D die set
~ 1200lbs empty

Engineering High Efficiency C-C Tooling for EFAS

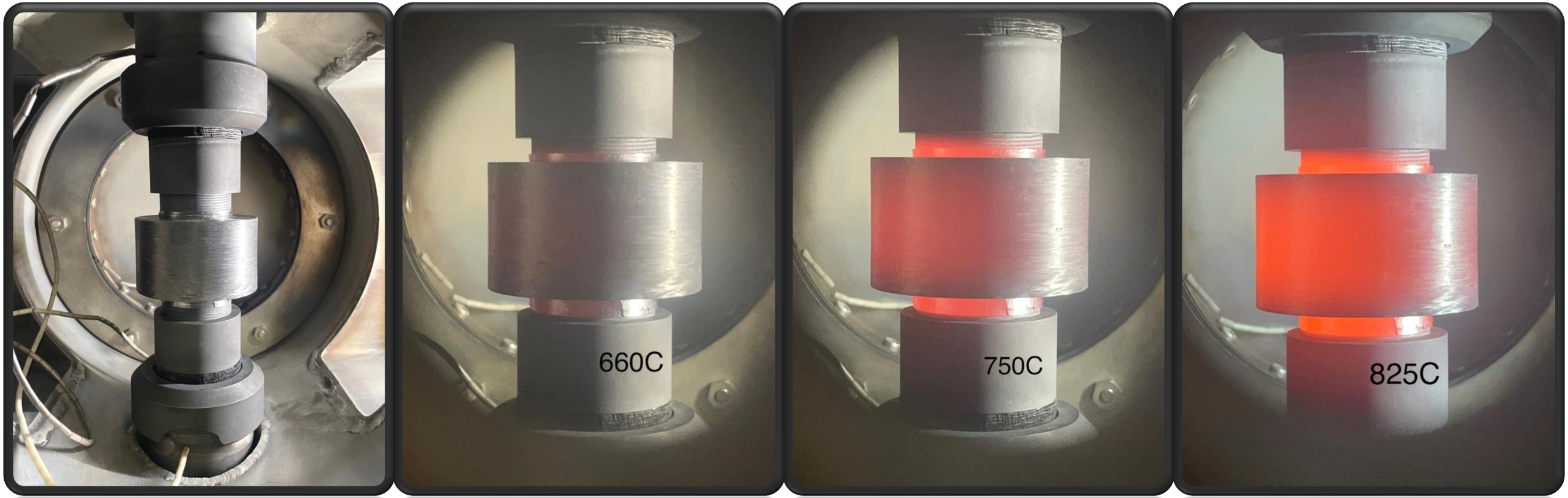
- The high resistivity Z-direction of the C-C, when aligned with the axis of current flow, more effectively generates heat.
- Low thermal diffusivity of the Z-direction traps heat where it is generated
- High diffusivity in the X/Y plane ensures temperature uniformity across the tooling

20 mm QI
C-C EFAS tooling:



32 mm SW
C-C EFAS Die:
(punches remain
QI type)

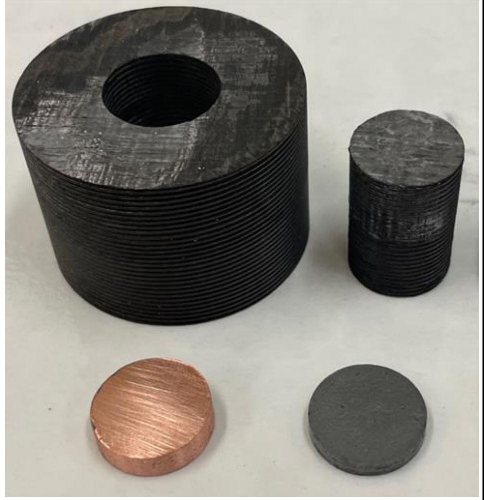
32 mm Spiral Die Heating; Cu Sintering



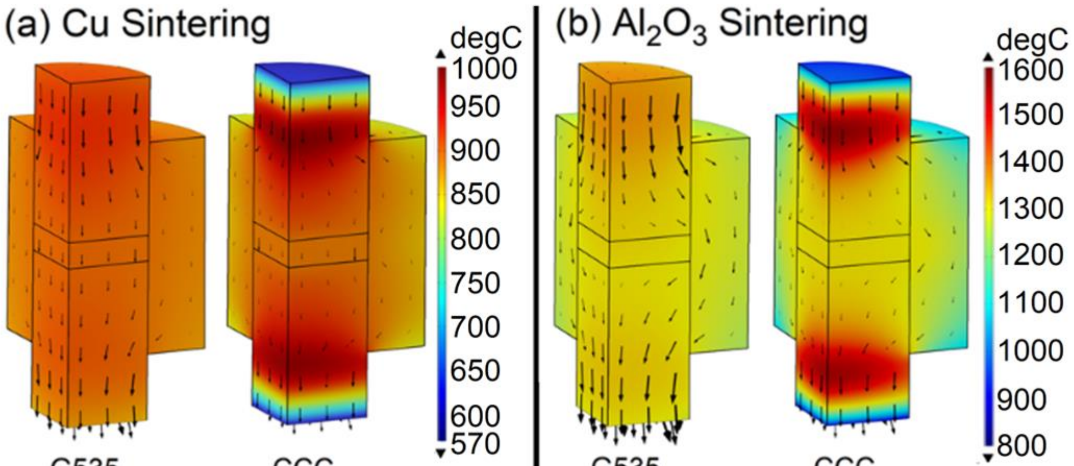
Carbon-Carbon vs. G535 Graphite Tooling

20 and 32mm diameter

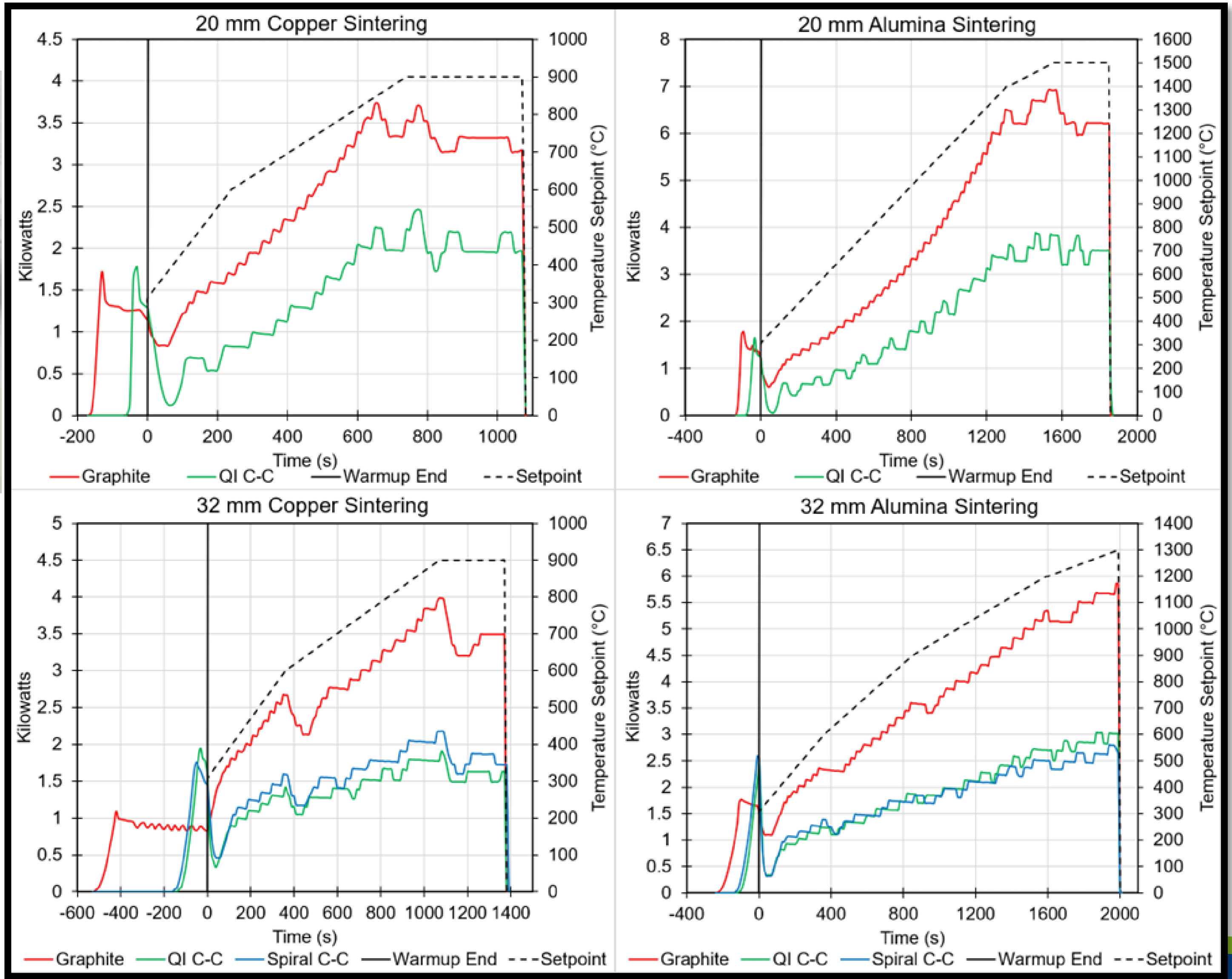
- Average **48.3%** less energy
- Ram temperatures stay **37.3%** colder.



COMSOL Modeling:

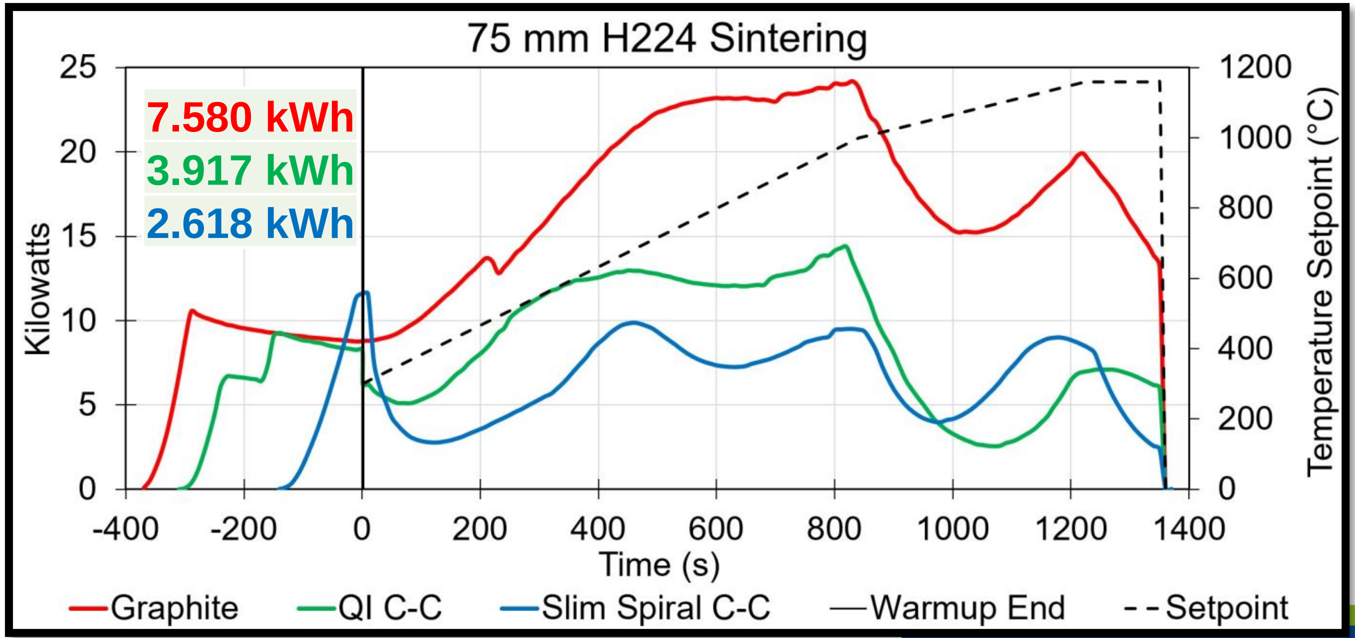


1290 A 550 A 1500 A 783 A



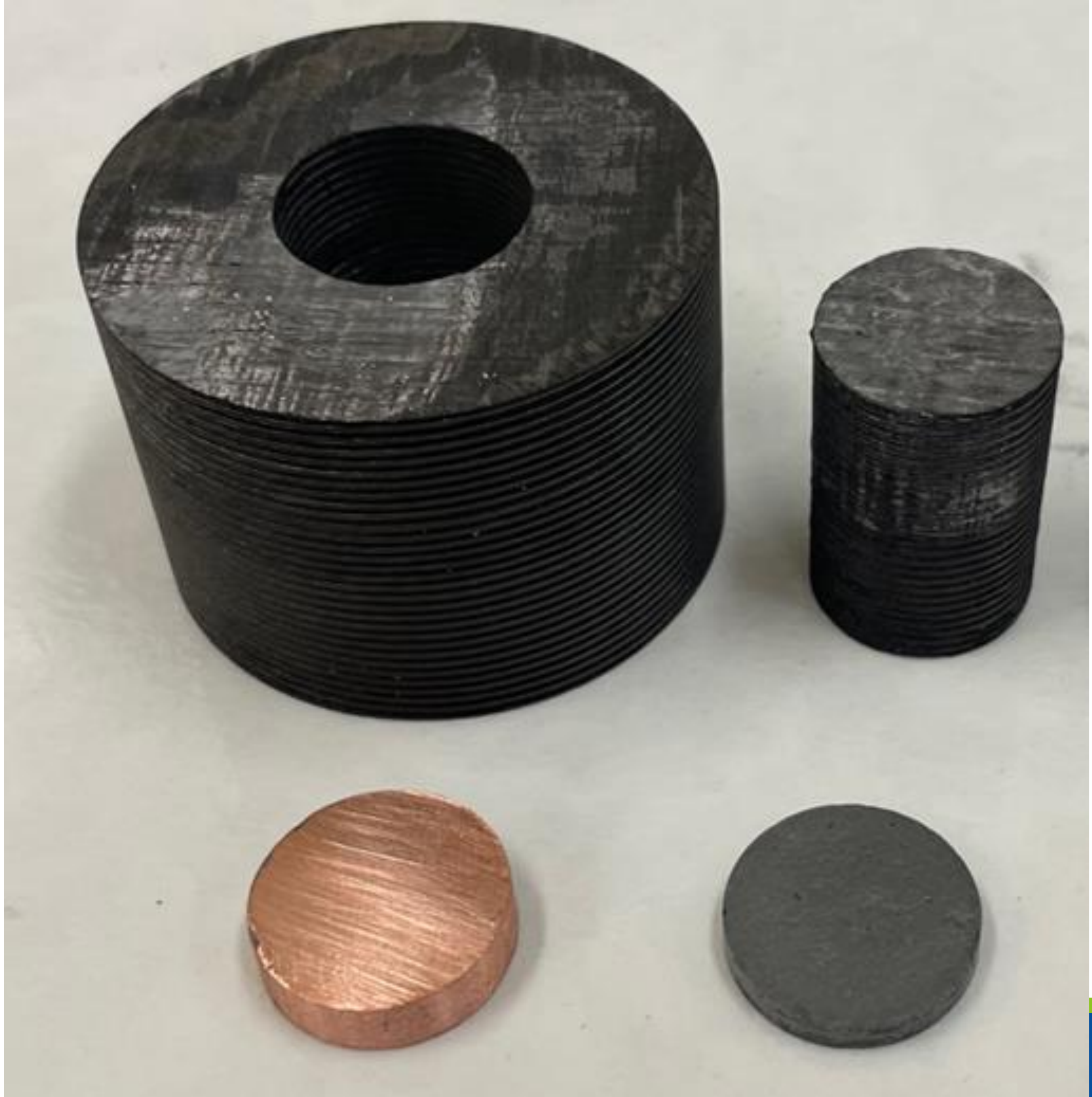
Energy need reduction continues with increasing die size

- 75 mm Spiral (SW) die made with thinner walls further reduces energy consumption:
  - QI compared to graphite reduces energy consumption by 48.3%,
  - Slim Spiral reduces energy consumption by 65.5%
  - Ram temperatures reduced by 36.1%, 51.0% respectively.



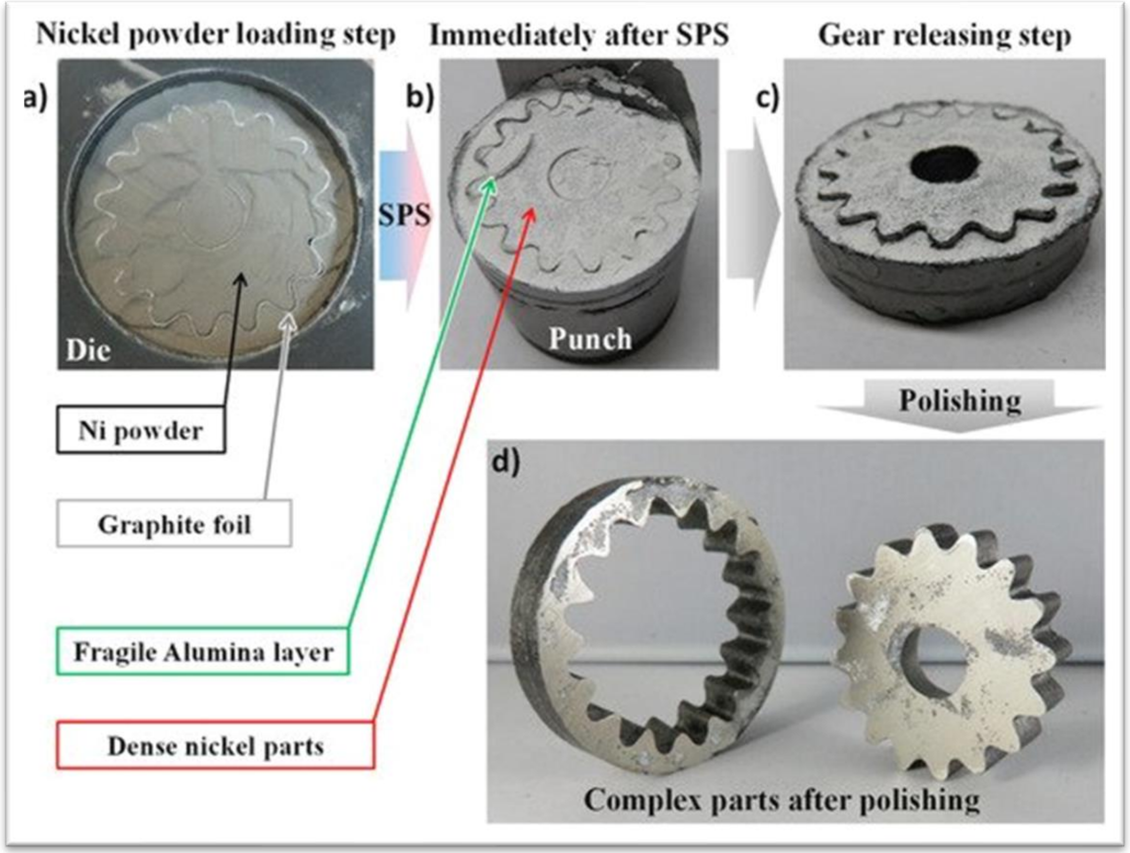
Summary- C-C Tooling

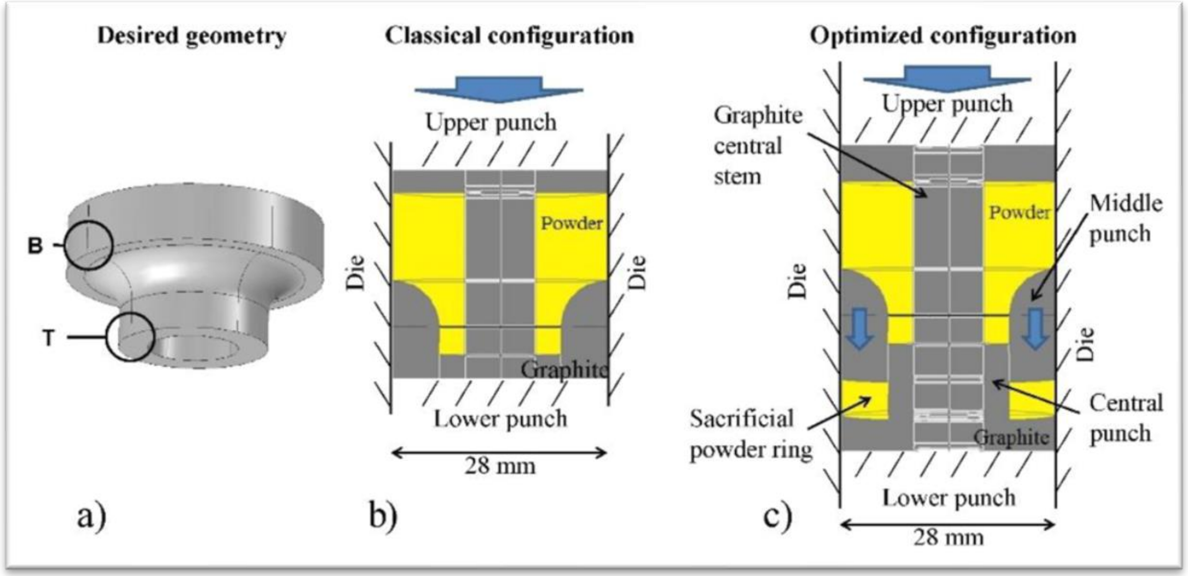
- C-C fabricated tooling leveraged the anisotropic properties to make stronger and more energy efficient tooling.
- Future work involves tailoring the fiber orientations to create targeted heating zones, thermal gradients.

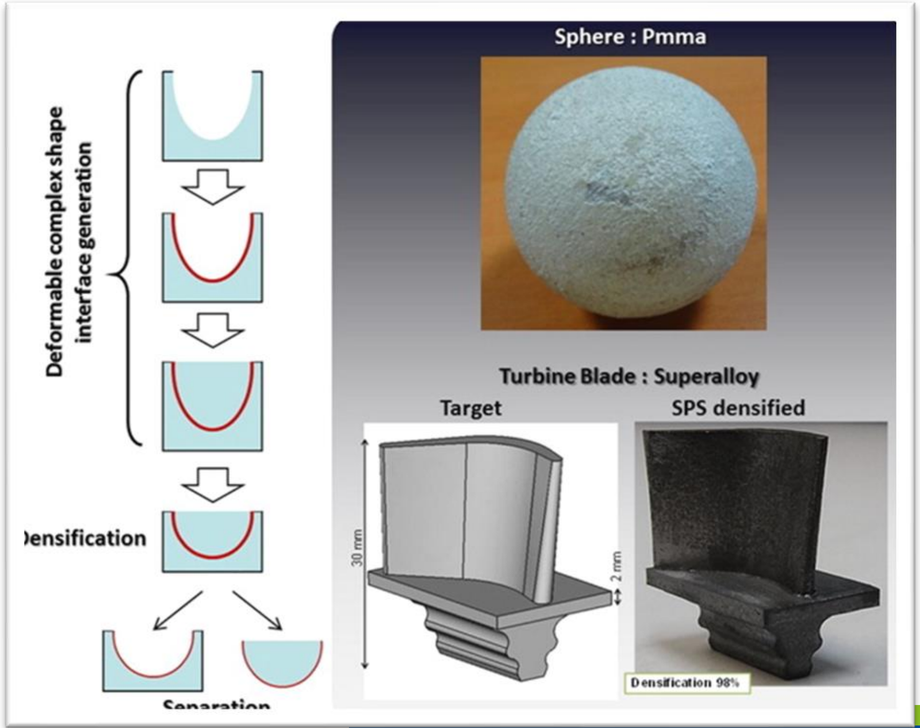


Path Forward for EFAS

Complex and Net Shaping







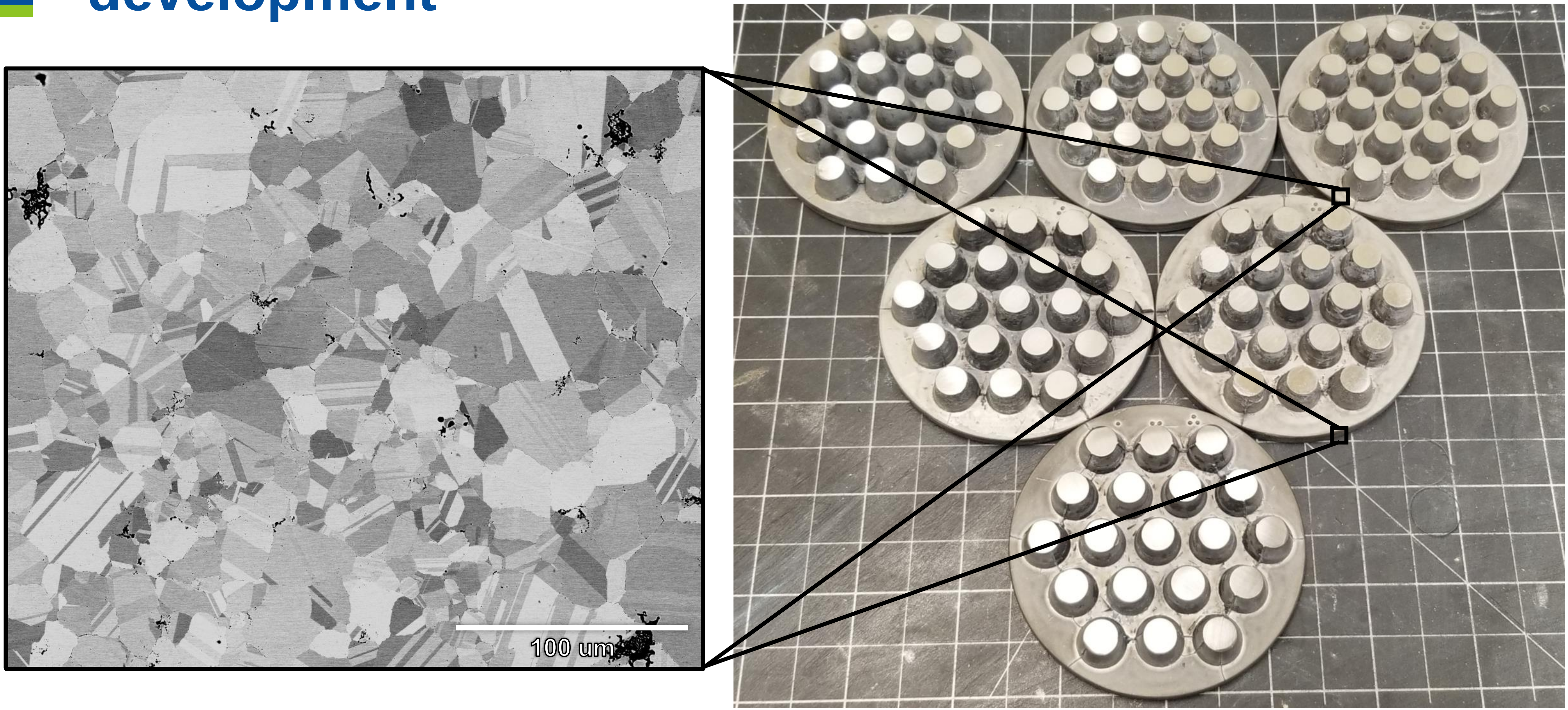
Maniere, et al., 2017, deformed interfaces approach, https://doi.org/10.1111/jace.15752
Maniere, et al., 2019, complex shapes, **https://doi.org/10.3390/ma12040557**

Advanced geometries

- Custom tooling and process strategy development to make high temperature ceramic, near net shaped parts.

This area is critical to develop for accelerated industrial EFAS adoption.

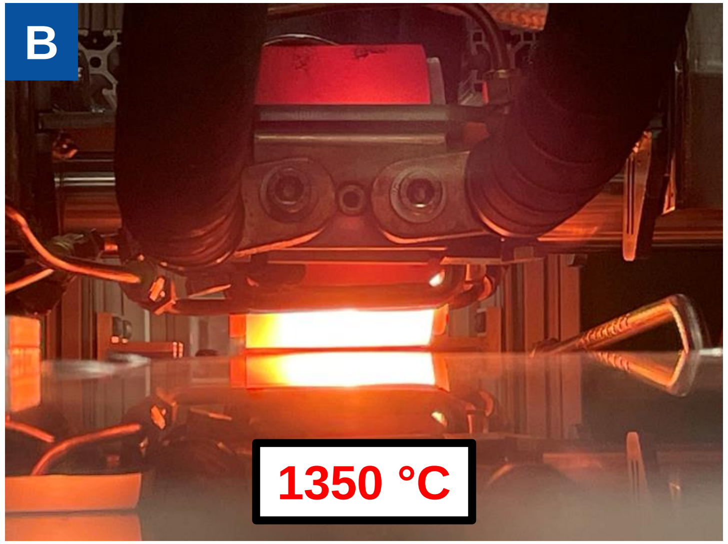
High throughput tooling strategies for rapid alloy development

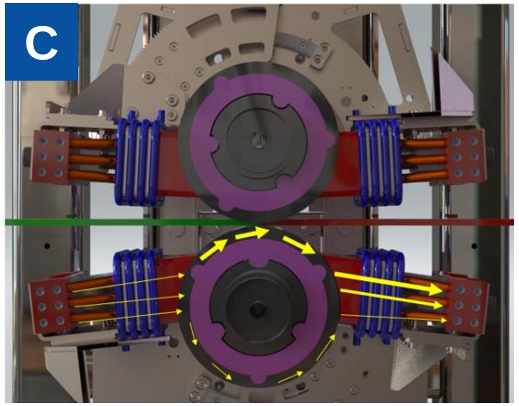


EFAS research into large scale production at INL

Continuous Electric Field Assisted Sintering (CEFAS)

- The DCS-800 is the largest EFAS instrument in the world available for users
- 150,000 amps / 800 tons of uniaxial pressure
- 8”-24” diameter **samples**

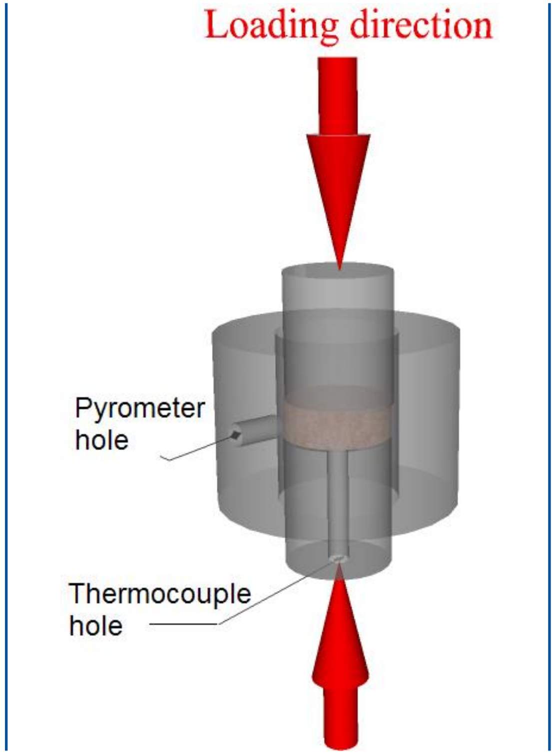


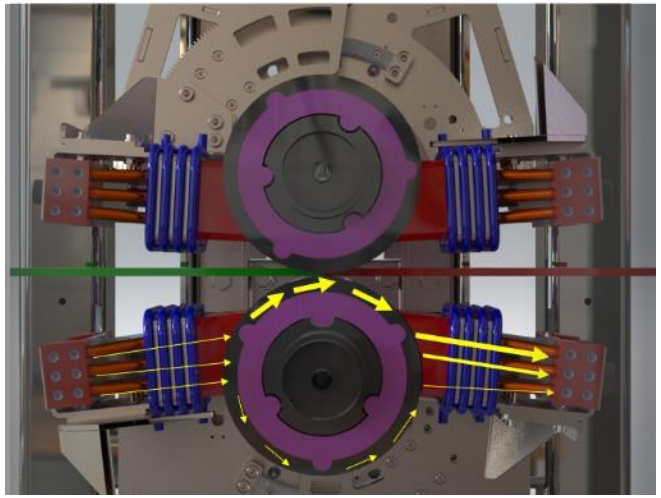


(A) The Continuous Electric Field Assisted Sintering (CEFAS) is our take on how to take EFAS from a batch to a continuous process. The instrument has reached operating temperatures up to 1350 °C and first samples will sinter in January/February 2024 **(B)** . Instead of samples compressing between two rams, samples will pass between two charged rollers. Electric current brushes will carry up to 6,000 amps across each roller which will heat up to 1350 °C at the roller contact point.

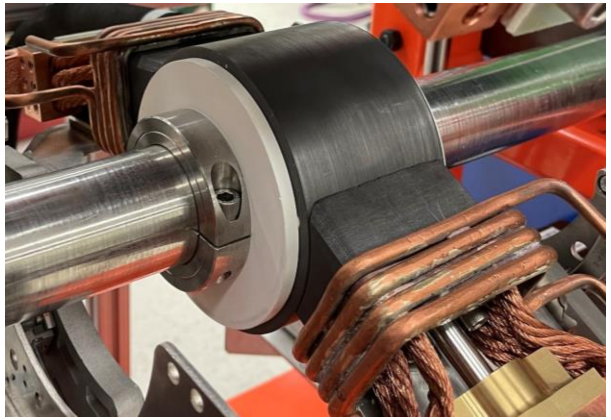
Continuous EFAS Research

- Higher part throughput and lower manufacturing cost
- Ideal for high aspect ratio, graded, multi-layered, or thinner material, i.e. ceramic SOEC/SOFCs

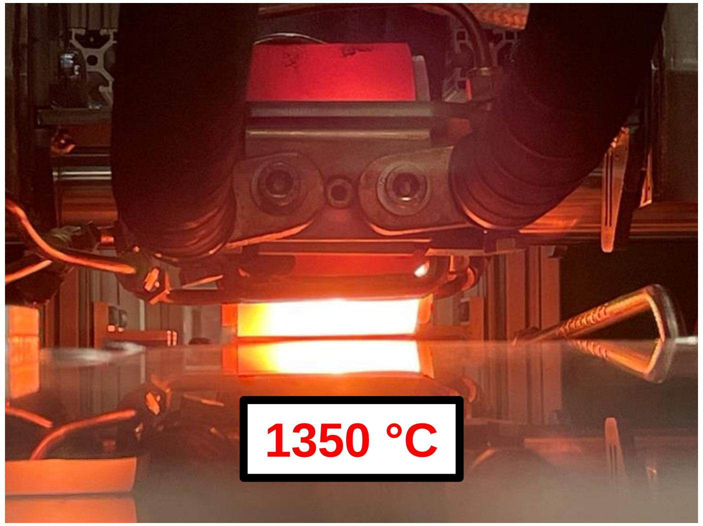




CEFAS Prototype



Continuous electric-field assisted sintering (CEFAS)

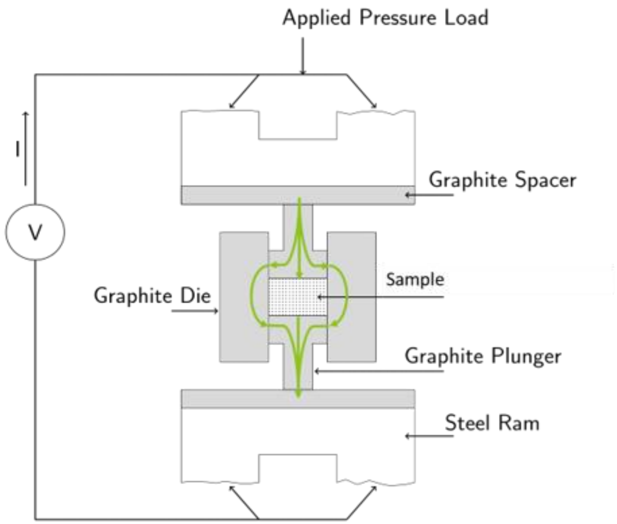


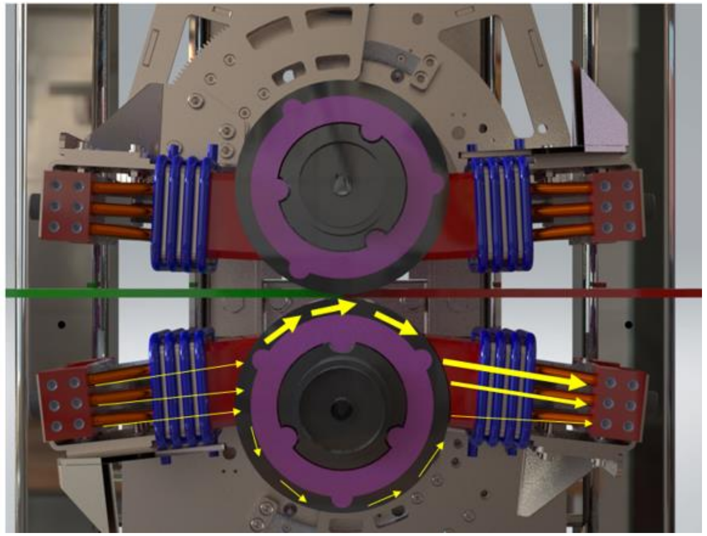
	CEFAS
Production type	Continuous
Forces	Uniaxial and shear
Current Max Operating Temp	1350 °C 1650 °C - end of project target
Heating rates	> 1000 °C/min
Sample length	Unlimited
Sample width	≤ 3”
Heating rates	1000 °C/min +
Processing time*	0.15 – 15 in/min

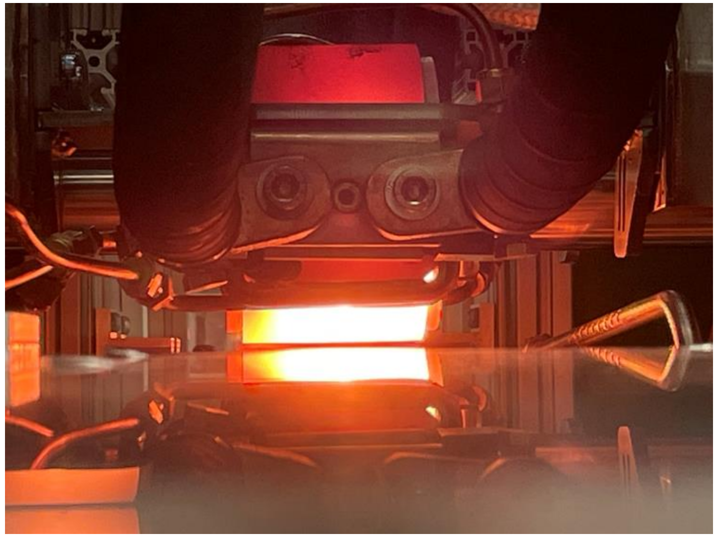
*Processing times highly dependent on thickness and material

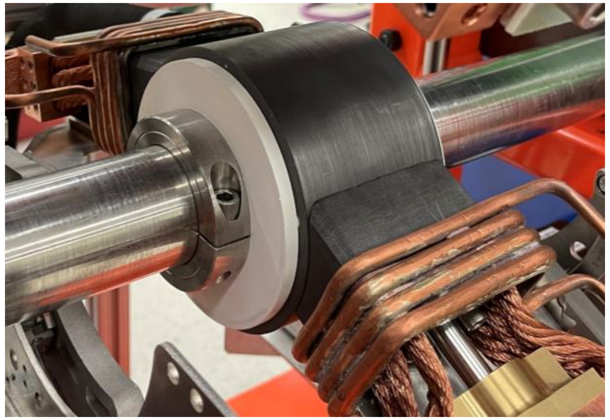
CEFAS vs EFAS

	EFAS	CEFAS
Production type	Batch	Continuous
Forces	Uniaxial	Uniaxial and shear
Max temperature	2500 °C	1350 °C 1650 °C - end of project target
Heating rates	100 °C/min	1000 °C/min +
Processing time	**30 min** for 0.8” dia. disc **(0.5 in²)**	**30 seconds** for 3” x 0.8” plate **(2.4 in²)**









Examples of Processed Materials

- SiC / B_4C
- C-C / C-SiC
- 316 SS
- Haynes282
- High Entropy Alloys
- Oxide Dispersion Strengthened steel
- Y_2O_3 / Al_2O_3
- B4C / SiC / TiN
- Ni-WC (Cermets)
- Ni-SiC Cermet

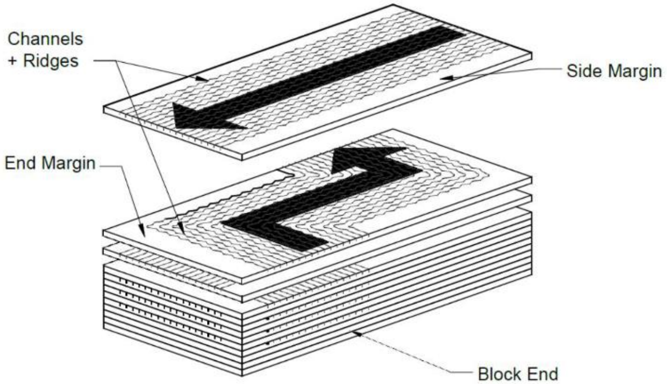
Cf-SiC Cladded with ZrB2

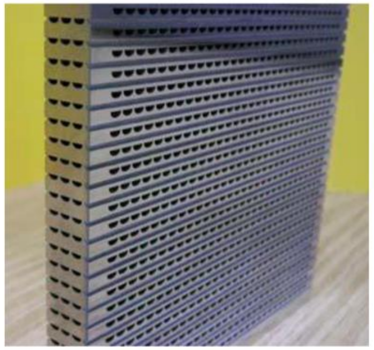
Compact Heat Exchangers: *Background and Significance*

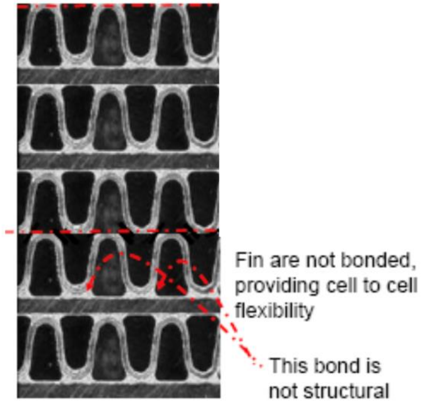
- Compact heat exchangers (CHXs) are of interest to many industries including nuclear, solar, and fossil fuel owing to their high thermal efficiency, low pressure drop, and great flexibility.
- Alloy 617 is the preeminent candidate material for the intermediate heat exchanger in very high-temperature gas-cooled reactors.
- Diffusion bonding (DB) is a particularly adept technique for fabricating CHXs.


[1]

Silver: Printed circuit heat exchanger
Black: conventional shell and tube heat exchanger



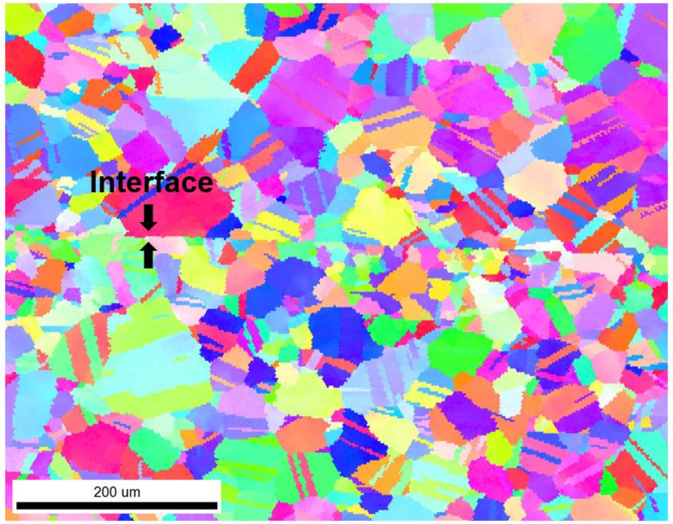

[2]



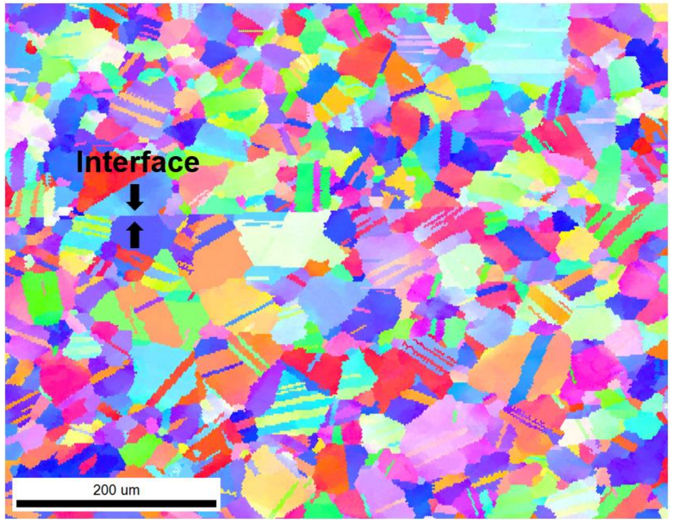
[1]

Diffusion Bonding of Alloy 617 using EFAS

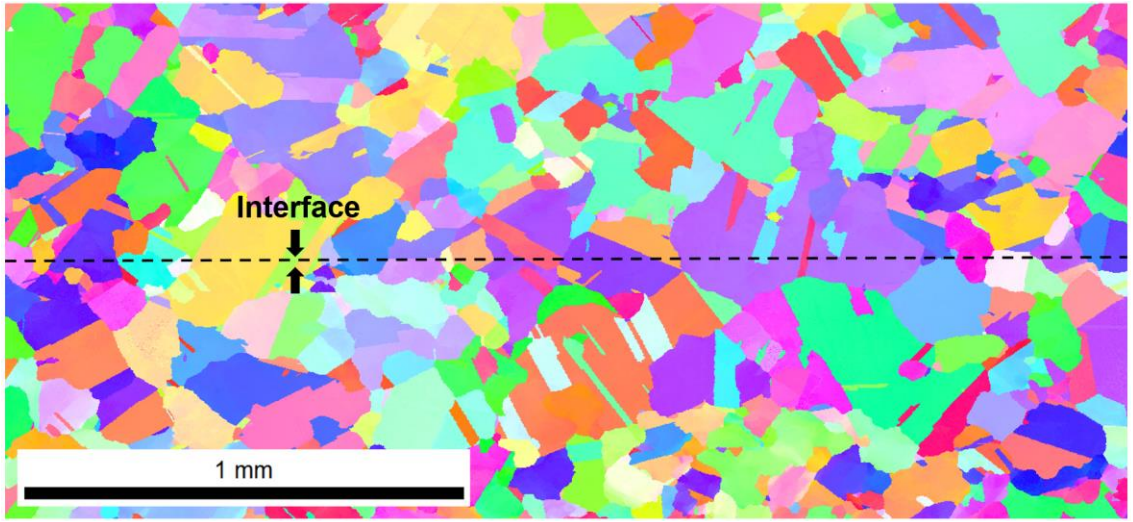
- Limited grain boundary migration across the interface in EFAS-1050°C and EFAS-1100°C. Significant grain boundary migration across the interface (~88.5%) in EFAS-1150°C
- Optimizing the temperature can result in substantial GB migration across the diffusion-weld interfaces



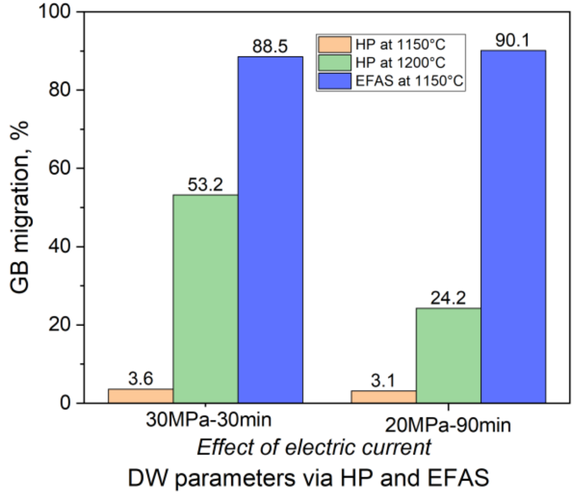
EFAS, 1050°C, Center



EFAS, 1100°C, Center

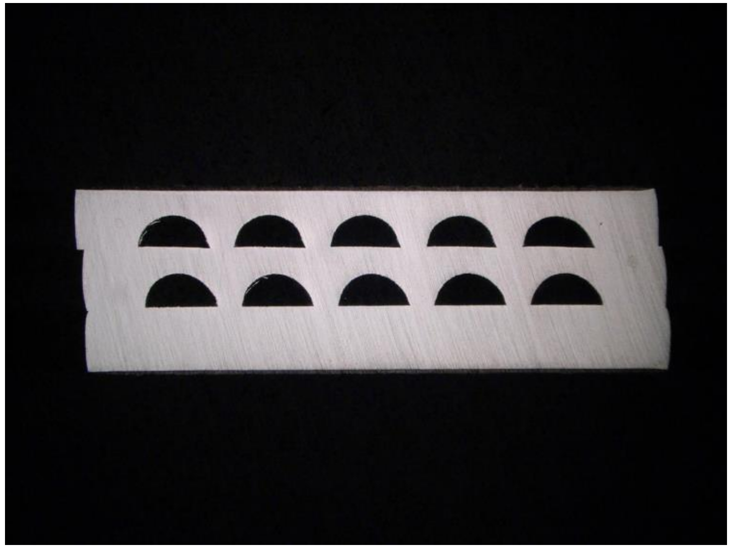


EFAS, 1150°C, Center

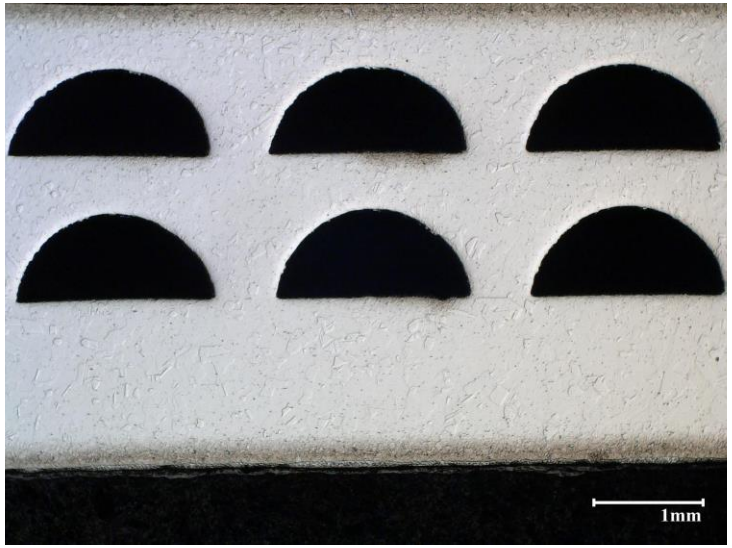


Under similar conditions GB Migrations is superior in EFAS compared with hot pressing (HP)

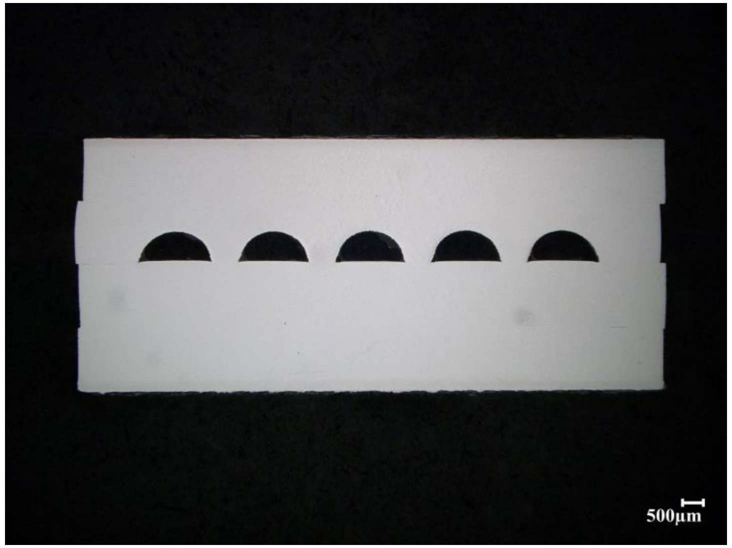
Diffusion Bonded CHX Channels of Alloy 617 using EFAS



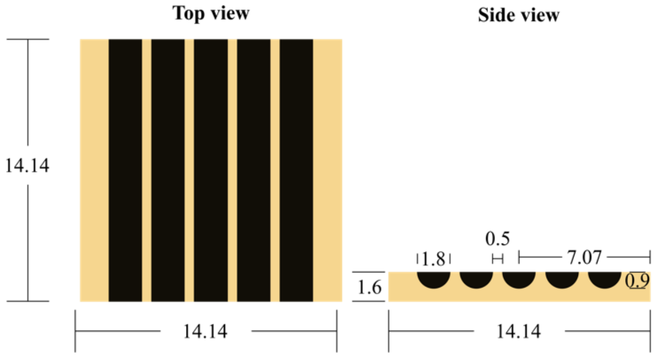
1050°C-10MPa-10min (3 Alloy 617 sheets)



1150°C-10MPa-10min (3 Alloy 617 sheets)



1150°C-10MPa-10min (4 Alloy 617 sheets)

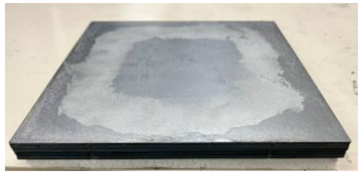


Schematic of channels

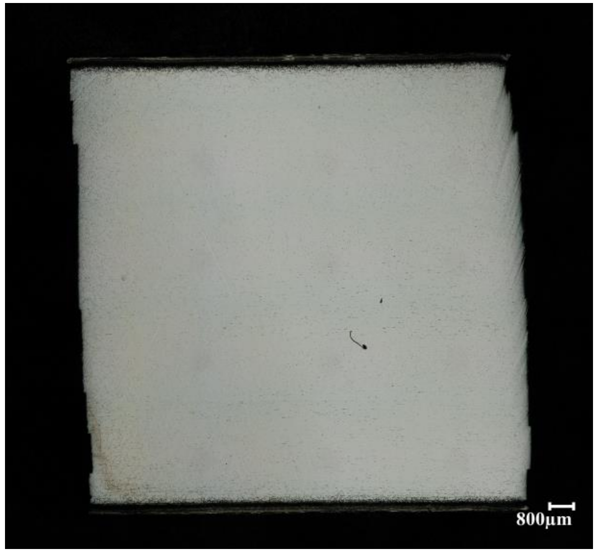
EFAS Diffusion Bonding Process Scale-up

- **Challenges:** 1) Small-scale DB parameters are not optimized for process scale-up
  2) Temperature distribution in the large-scale samples is not uniform

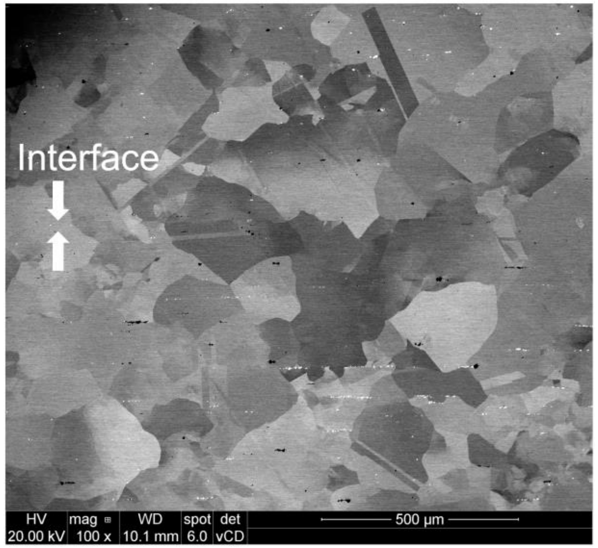
GB migration across the interface was achieved in small scale samples but not in large scale samples while both were fabricated at the same temperature (1125°C)



Small scale (Alloy 617 size: 14 × 14 mm²)



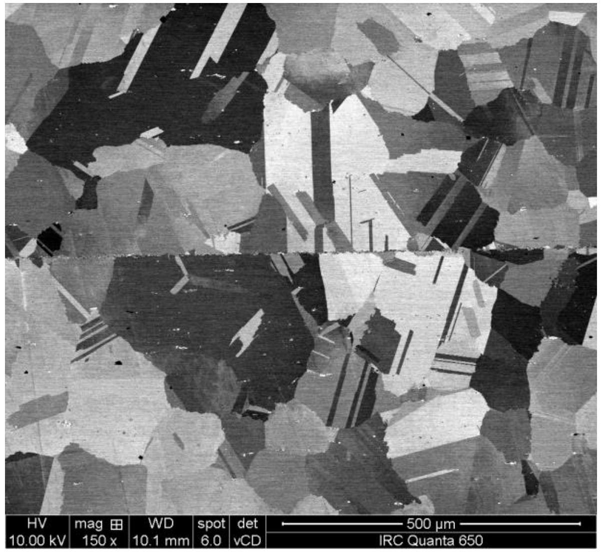
11 sheets of small-scale Alloy 617 (14 × 14 mm²) diffusion-welded via EFAS at 1125°C-10MPa-30min



Good grain boundary migration across the DB interfaces

Scale-up (Alloy 617 size: 216 × 216 mm²)

10 sheets of large-scale Alloy 617 (216×216 mm²) diffusion welded by DCS-800 EFAS at 1125°C-20MPa-30min



Limited grain boundary migration across the DB interfaces

EFAS and CEFAS Capability Take Away

- INL’s EFAS capabilities provide full spectrum, multi-scale, offering from research to industrial scale
- Major advantages of EFAS/CEFAS compared with conventional methods include:
  - ***Extremely fast*** processing speeds
  - Up to ***90% Energy Savings***
  - Exotic microstructures
- It provides access to:
  - Difficult to manufacture materials,
  - In some cases, the only pathway (e.g., some extremely high temperature ceramics)

Idaho National Laboratory

Battelle Energy Alliance manages INL for the U.S. Department of Energy's Office of Nuclear Energy. INL is the nation's center for nuclear energy research and development, and also performs research in each of DOE's strategic goal areas: energy, national security, science and the environment.