High-Pressure Engineering of Sm-Based Oxypnictide Superconductors: A Route to High-Performance Superconductivity

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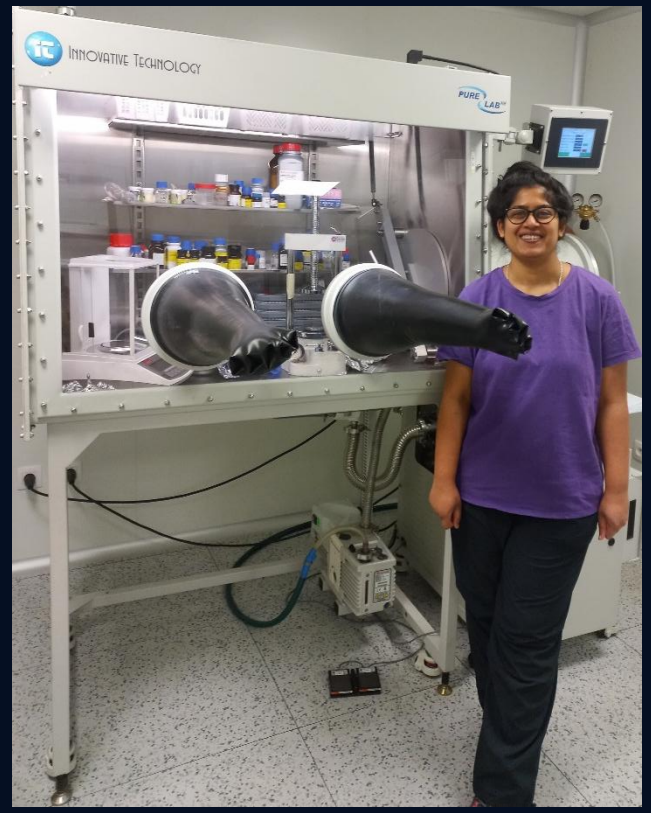
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High pressure: a tool to form materials and tune their properties

Pressure changes the thermodynamic and kinetic conditions of synthesis—and can also alter the properties of an existing material.

01 MATERIAL Phase Formation

Pressure + Temperature

Change phase stability, reaction pathways, element incorporation and defect formation.

Reactants → Target phases → Material

02 TUNE MATERIAL PROPERTIES

Pressure-dependent structure

Can change microstructure and, in suitable systems, electrical, magnetic, mechanical or superconducting behaviour.

- Structure
- Transport
- Magnetism
- Superconductivity

KEY MESSAGE **High pressure is both a synthesis tool and a way to tune material behaviour; the outcome depends on pressure, temperature, composition and processing route.**

Two ways to apply pressure in materials research

Pressure may be used to change a material during synthesis, or to probe its properties after it has been prepared.

01 PRESSURE DURING GROWTH / SYNTHESIS

Growth pressure

Pressure is applied while the material forms, together with controlled temperature and reaction conditions.

Purpose: influence phase formation, composition and microstructure.

02 PRESSURE DURING CHARACTERIZATION

Applied / external pressure

Pressure is applied to an already prepared sample while measuring its response under pressure.

Purpose: probe pressure-dependent structure and properties.

GROWTH PRESSURE apply during the sample growth process | CHARACTERIZATION PRESSURE = tests how it responds

Why apply pressure during synthesis?

Pressure acts while the material forms—changing the reaction pathway, not just testing the prepared sample.

PRESSURE + TEMPERATURE
Drive solid-state reactions

PHASES + COMPOSITION
Influence phase formation and element incorporation

MICROSTRUCTURE
Shape density, defects and grain connectivity

01

Stabilise desired phases

Pressure can favour phases that are difficult to obtain at ambient pressure and alter competing impurity phases.

02

Tune composition & defects

Pressure changes reaction kinetics and can influence elemental incorporation, defect populations and homogeneity.

03

Improve current pathways

With suitable processing, densification and grain-boundary connectivity can improve transport and superconducting current.

KEY IDEA

Growth pressure is a synthesis variable: it can engineer structure and connectivity—and thereby influence superconducting transition temperature T_c and critical current density J_c.

- Outcomes depend on pressure, temperature, dwell time and synthesis route.

Three pressure-growth techniques. Why use all three?

Different pressure environments and processing times let us tune phase formation, densification and microstructure.

01

High gas pressure and high-temperature synthesis

Argon gas pressure
Up to ~1.5 GPa

Explore pressure-assisted phase formation and elemental incorporation during synthesis.

WHY: Large sample space and study how growth conditions change the material itself and

02

Spark plasma sintering

Uniaxial pressure
Up to ~45 MPa

Rapid heating and short dwell times promote densification while limiting prolonged thermal exposure.

WHY: improve density and grain connectivity efficiently.

03

Cubic-anvil high-pressure synthesis

Solid pressure medium
Up to ~6 GPa

Access much higher pressures to explore phase stability and microstructural changes; in-situ and ex-situ routes can be compared.

WHY: test the effects of a wider pressure range and processing route.

WHY THREE? **To compare complementary routes—not assume one pressure method is best for every goal.**

Especially focused on high temperature superconductor • Maximum machine capability is not the same as the pressure used in each experiment.

From applied pressure to superconducting performance

01

APPLY Pressure and Temperature

Pressure medium,
load and temperature

02

PHASE FORMATION

Reaction pathway,
phase stability

03

MICROSTRUCTURE

Densification,
porosity, grain contacts

04

SUPERCONDUCTING RESPONSE

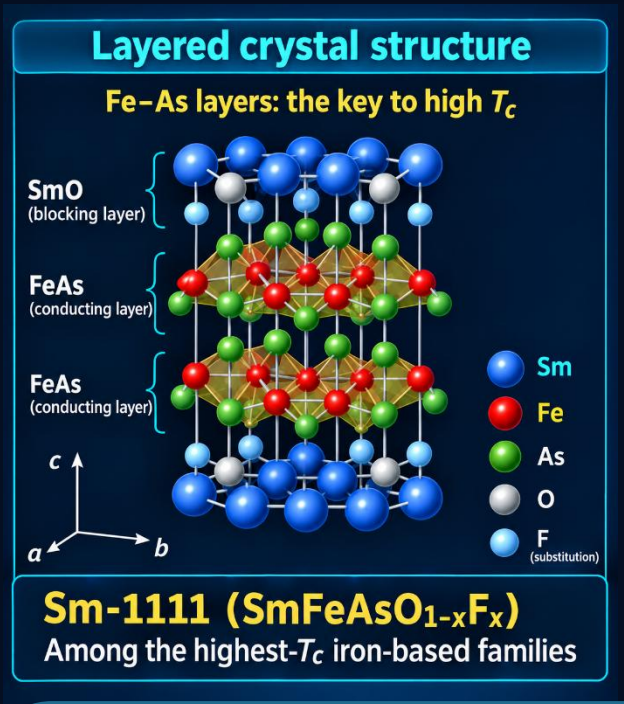
Tc, Jc and
vortex pinning

KEY IDEA

The goal is not simply maximum pressure or maximum density — it is the right balance of phase quality, grain connectivity and vortex pinning and overall the superconducting properties.

Interpretation framework for this study; measured outcomes should be established from the experimental data on subsequent slides.

Iron based high T_c superconductors (FBS): 1111 family



$RE_1Fe_1As_1O_1$: *RE*1111; *RE* = rare earth *RE* = La, Ce, **Sm**, Gd

- High transition temperature T_c ~ 58 K for FBS
- High critical current density J_c~10^6 A/cm^2
- T_c ~ 58 K for F doped Sm1111 (for ~20% F-doping)
- Doped superconductors
- High upper critical field > 70 T
- Low anisotropy ~3-6

Shiv J. Singh et. al. Crystals 12, 20 (2022)

Supercond. Sci. Tech. 25, 113001 (2012)

Supercond. Sci. Tech. 27, 044002 (2014)

- Relatively high T_c and high upper critical fields
- High J_c requires dense, phase-pure samples with good grain connectivity
- Synthesis conditions influence phase formation, F incorporation and microstructure

Pressure-assisted synthesis of $SmFeAsO_{0.8}F_{0.2}$ (Optimal F doping content)

Tune phases and microstructure

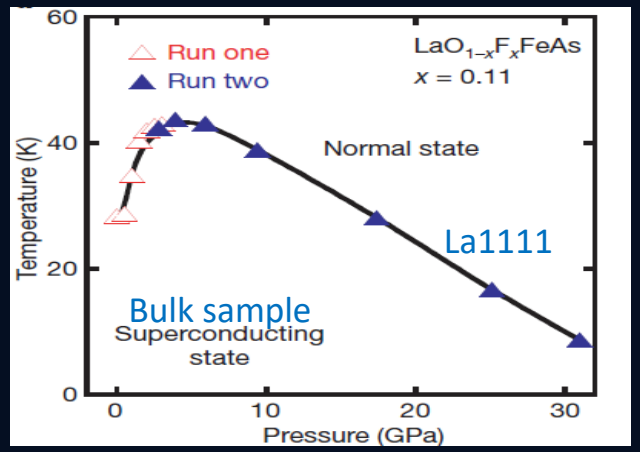
- ✓ Control phase formation and promote F incorporation
- ✓ Improve densification and grain connectivity
- ✓ Enhance superconducting performance, especially practical J_c

High T_c alone is not enough: pressure-assisted synthesis aims to engineer a dense, well-connected microstructure for high practical J_c.

Reported few studies on high pressure techniques for FBS

1. A lot of studies show **that applied external pressure studies improve the superconducting properties** where the samples are prepared by CSP.

Nature **453**, 376 (2008)



2. Very few reports based on the few compositions show **the positive effect of high-pressure synthesis (HP-HTS) and improved the sample quality** with suitable sized samples.

Phys. Rev. B **86**, 214509 (2012)

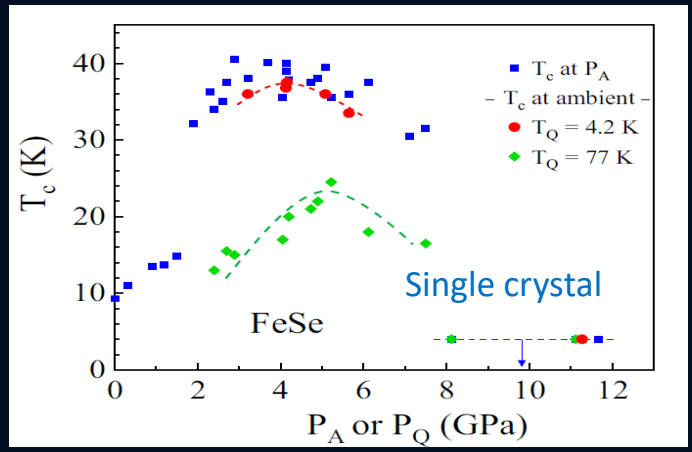
Appl. Phys. Lett. **105**, 102602 (2014)

Single crystal Growth of SmFeAs(O,F) (Sm1111)
CSP (crystal size 10 µm; flux: CSCl) (no pressure)

HP-HTS (crystal size 300 µm, flux: KCl/NaCl)

3. A recent high-pressure study opens a new possibility of **pressure quenching at various temperatures through HP for FBS to obtain the high superconducting properties at ambient pressure**.

PNAS **118**, e2108938118 (2021)



Motivation

Three connected objectives: optimize synthesis, understand defects, and identify the most effective processing route.

01

OPTIMIZE SYNTHESIS

Optimize $SmFeAsO_{0.8}F_{0.2}$ (Sm1111) using different pressure techniques to control phase formation and defects.

SYNTHESIS

02

UNDERSTAND DEFECTS

Use structural, microstructural and superconducting characterization to determine how processing-induced defects affect T_c and J_c.

CHARACTERIZATION

03

COMPARE PROCESSING ROUTES

Compare different pressure techniques with conventional synthesis (CSP) to identify the most effective route.

COMPARISON

Main AIM | Link processing conditions to phase formation, microstructure and superconducting performance.

Four synthesis routes applied to the same composition

We investigate **$SmFeAsO_{0.8}F_{0.2}$ (Sm1111)** using three pressure-assisted techniques and a conventional ambient-pressure reference.

01

HP–HTS

High gas pressure and high-temperature gas synthesis

Argon gas pressure

Explore pressure effects during high-temperature synthesis.

SAME TARGET COMPOSITION

02

SPS

Spark plasma sintering

Uniaxial pressure

Promote rapid densification with short processing times.

SAME TARGET COMPOSITION

03

CA–HP

Cubic-anvil high-pressure synthesis

Solid pressure medium

Access higher synthesis pressures; compare in-situ and ex-situ routes where applicable.

SAME TARGET COMPOSITION

RE F

CSP–AP

Conventional synthesis process at ambient pressure

Ambient pressure

Reference sample for comparison with pressure-assisted processing.

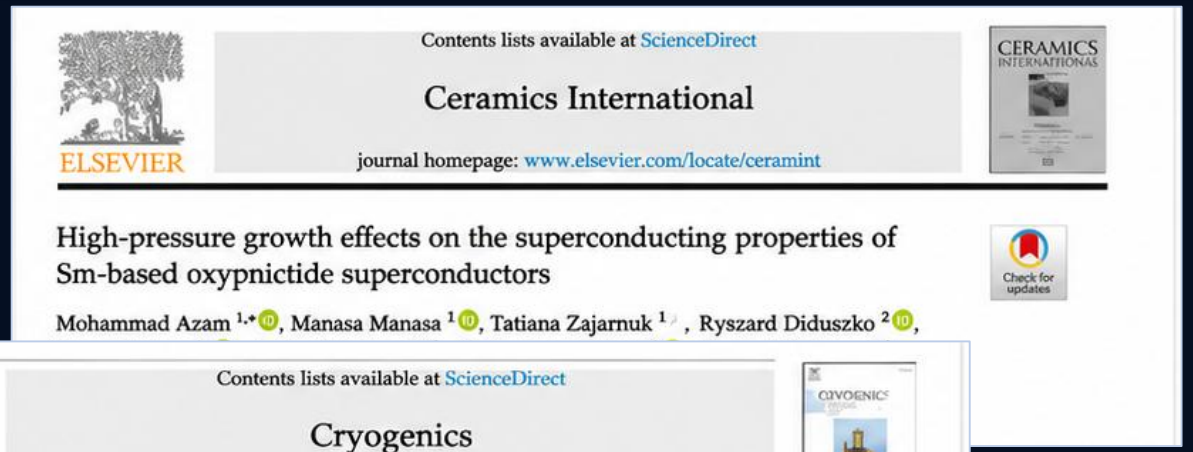
SAME TARGET COMPOSITION

STUDY DESIGN **Compare phase formation, microstructure, T_c and J_c to identify how each processing route affects superconducting performance.**

CSP–AP = conventional solid-state synthesis at ambient pressure. Technique capability ranges and actual experimental conditions should be distinguished.

Pressure-assisted synthesis of Sm-1111

Published studies motivate three pressure-processing routes for $SmFeAsO_{0.8}F_{0.2}$.

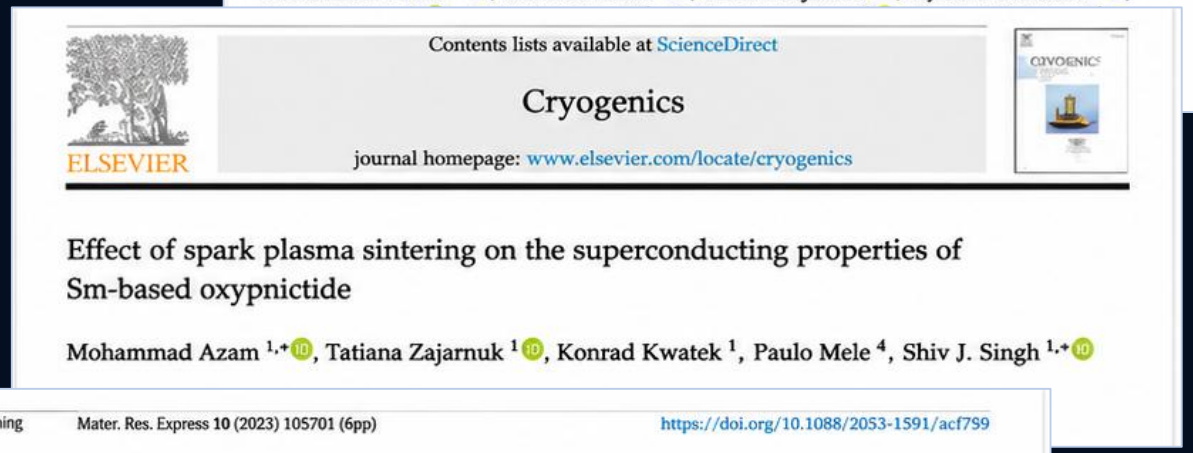

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High-pressure growth effects on the superconducting properties of Sm-based oxypnictide superconductors

Mohammad Azam [1,*], Manasa Manasa [1], Tatiana Zajarnuk [1], Ryszard Diduszko [2],


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Cryogenics

journal homepage: www.elsevier.com/locate/cryogenics

Effect of spark plasma sintering on the superconducting properties of Sm-based oxypnictide

Mohammad Azam [1,*], Tatiana Zajarnuk [1], Konrad Kwatek [1], Paulo Mele [4], Shiv J. Singh [1,*]


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PAPER

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Optimization of superconducting properties of F-doped SmFeAsO by cubic anvil high-pressure technique

Mohammad Azam [1,*], Tatiana Zajarnuk [1], Hiraku Ogino [2] and Shiv J Singh [1,*]

Mater. Res. Express 10 (2023) 105701.

High Gas Pressure (HP-HTS)

Optimal HP-HTS condition:
0.5 GPa, 900 °C, for 1 h

Spark Plasma Sintering (SPS)

Optimal SPS condition:
45 MPa, 900 °C, 10 min

Cubic Anvil (CA-HP)

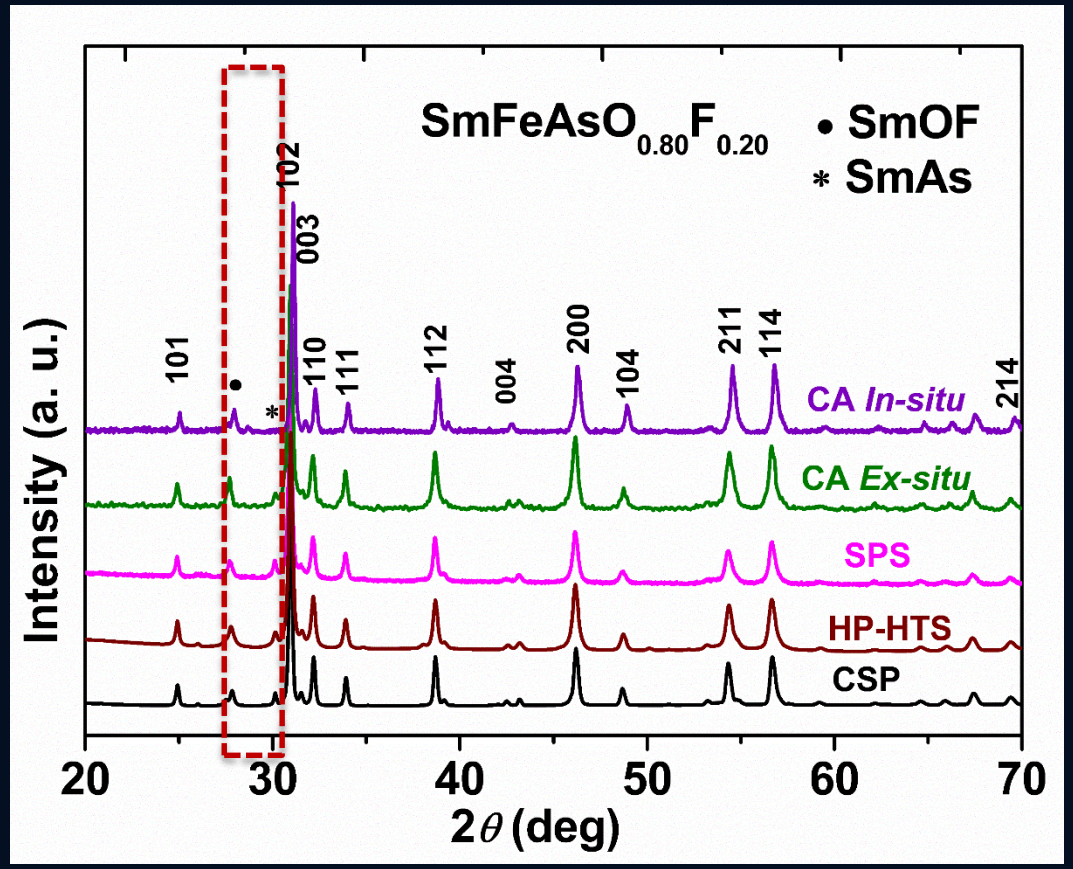
***Ex-situ* optimal condition :**
0.5 GPa, 900 °C, 1 h

***In-situ* optimal condition :**
4 GPa, 1400 °C, 1 h

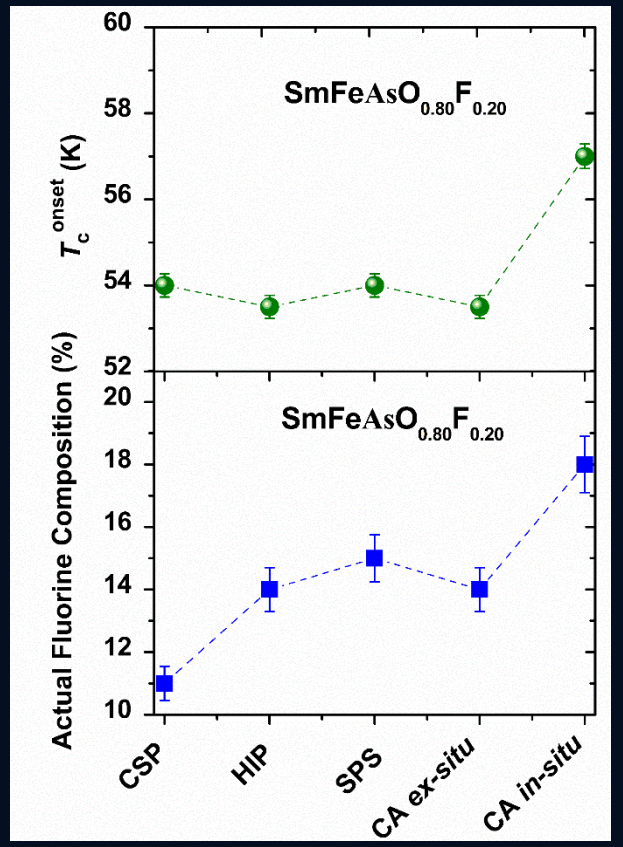
Structural and superconducting transition of optimized Sm-1111

Comparison of $SmFeAsO_{0.8}F_{0.2}$ synthesized by different pressure-processing routes

Structural analysis



T_c and Actual Fluorine content



KEY MESSAGE | Pressure-processing route is associated with changes in phase formation, T_c and fluorine incorporation.

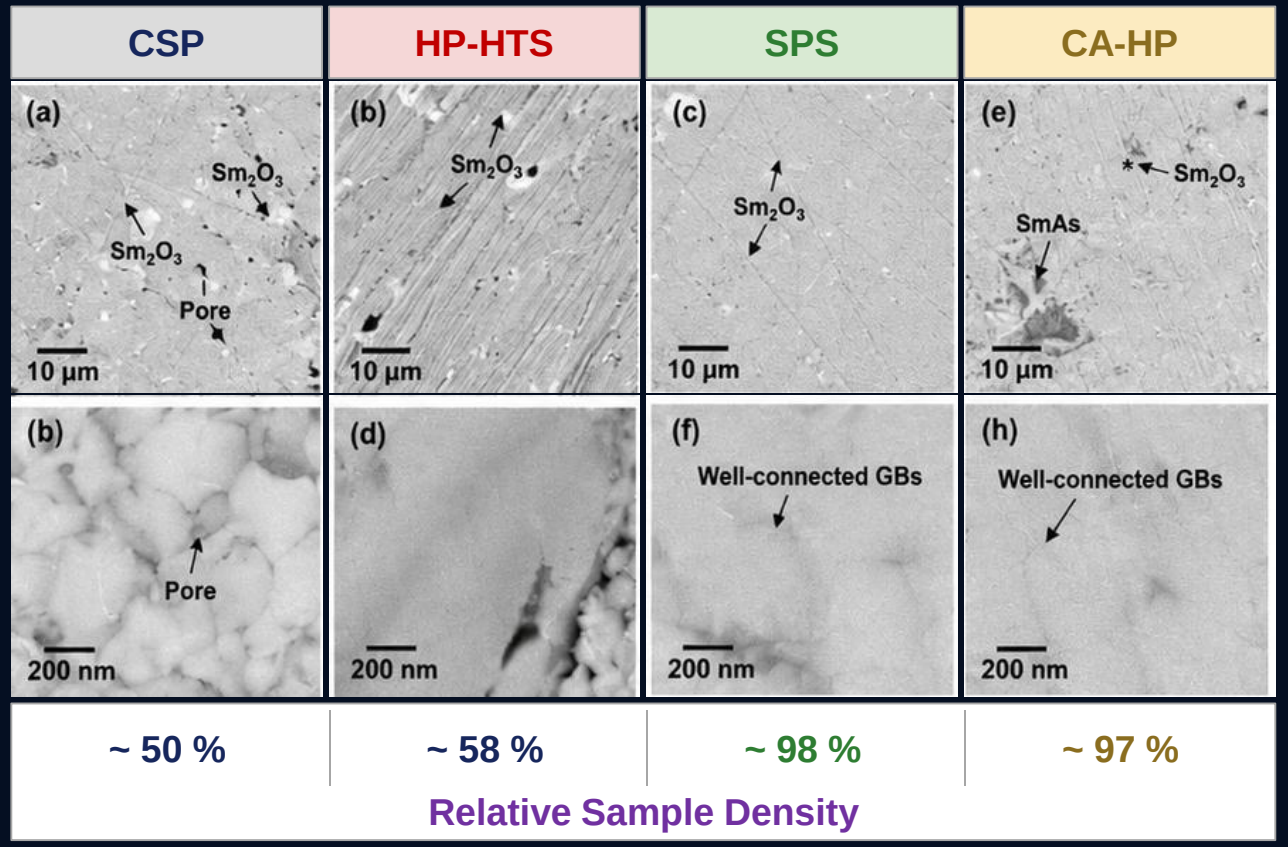
Azam *et al.* *Ceram. Int.* **51**, 13734–13751, (2025)

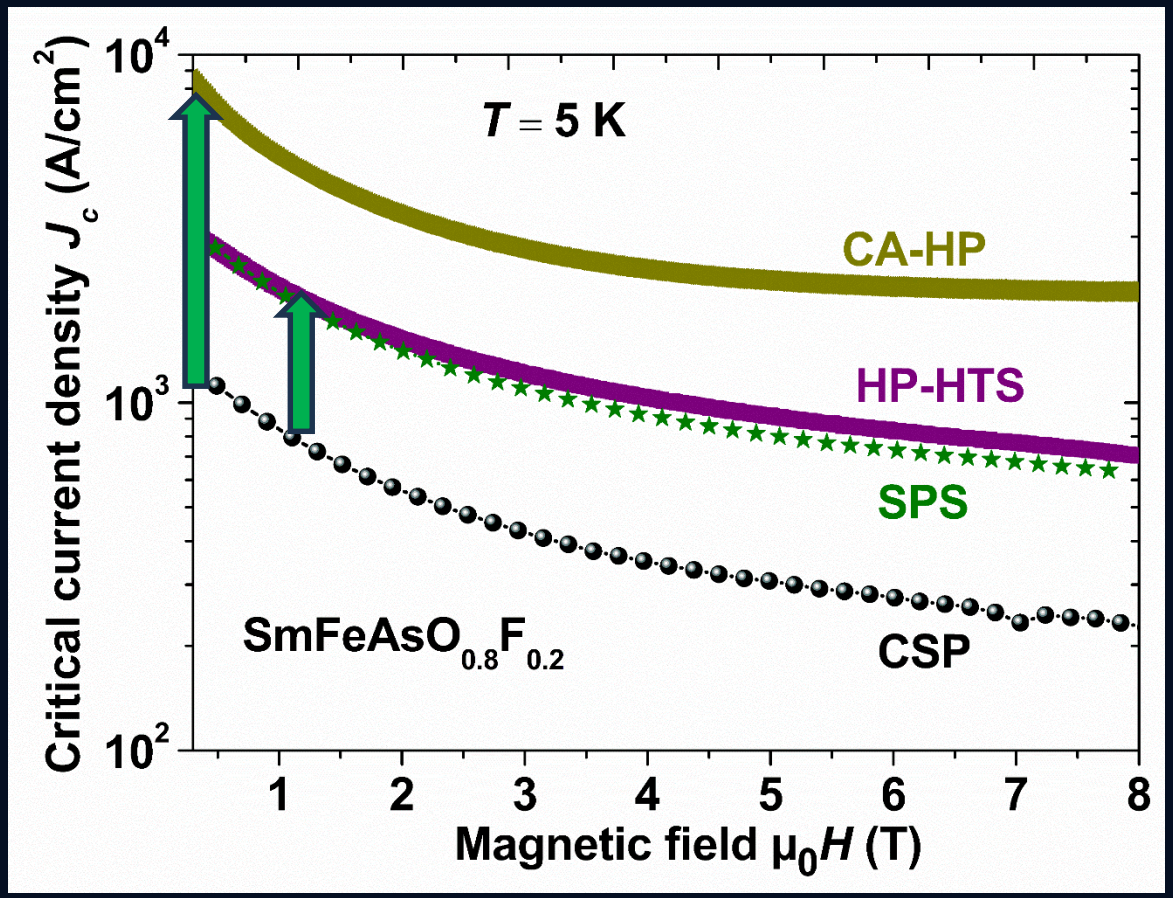
Azam *et al.* *Cryogenics* **150**, 104125 (2025)

Azam *et al.* *Mater. Res. Express* **12**, 11600 (2025)

Microstructure controls practical current-carrying performance

$SmFeAsO_{0.8}F_{0.2}$: comparing grain connectivity, relative density and field-dependent critical current density.





High density helps, but grain connectivity, phase constitution and defects also influence J_c.

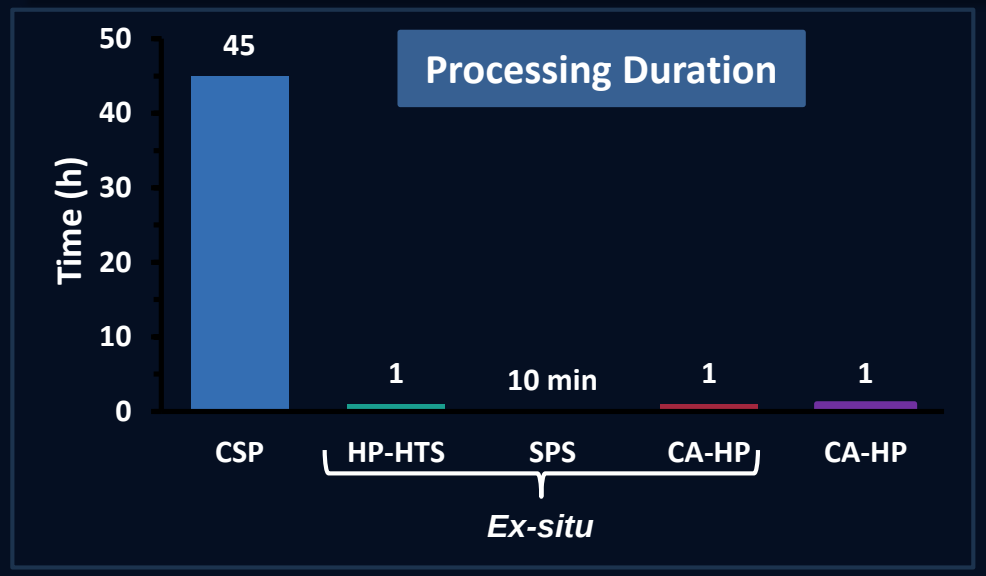
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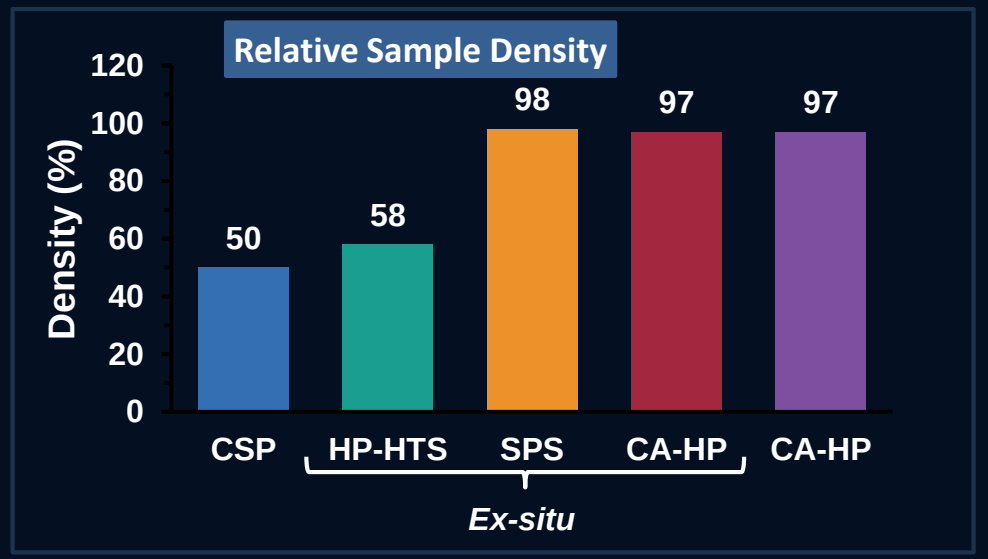
Azam *et al.* *Mater. Res. Express* **12**, 11600 (2025)

Processing route shapes superconducting performance

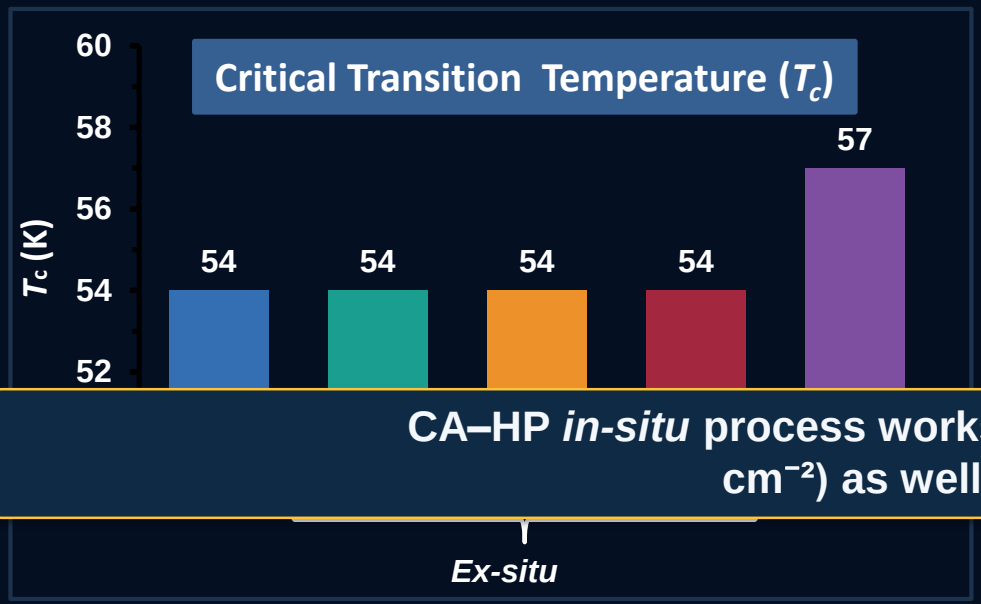
Three pressure-assisted techniques and an ambient-pressure reference: how do density, T_c and J_c compare?

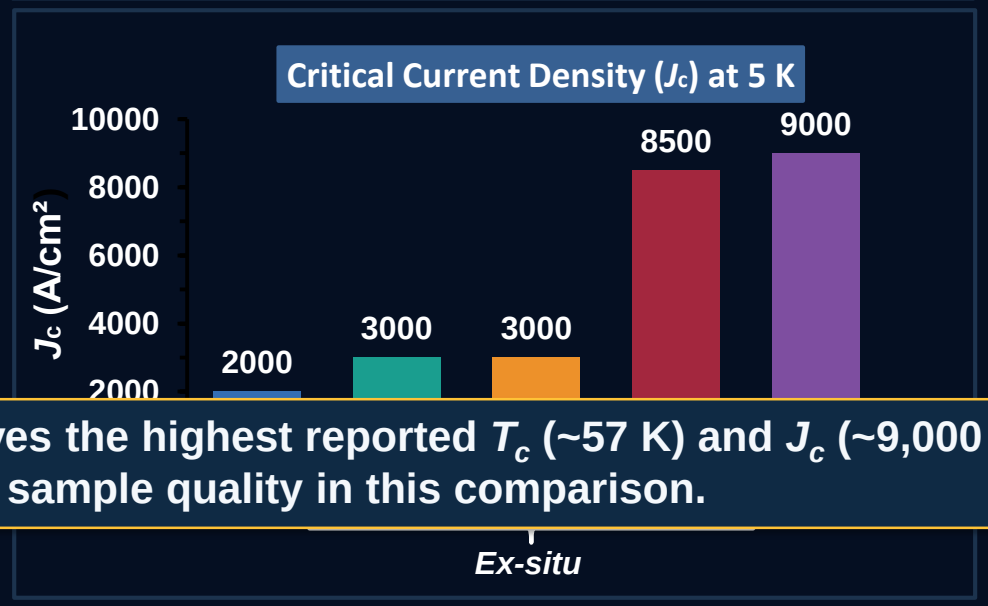


Azam et al. Cryogenics **150**, 104125 (2025)



Azam et al. Ceram. Int. **51**, 13734–13751, (2025)





Azam et al. Mater. Res. Express **12**, 11600 (2025)

CA–HP *in-situ* process works well and gives the highest reported T_c (~57 K) and J_c (~9,000 A cm^{-2}) as well as improved sample quality in this comparison.

CONCLUSIONS

High-pressure engineering of $SmFeAsO_{0.8}F_{0.2}$ (Sm1111)

HP–HTS, SPS and CA–HP were compared with conventional synthesis process (CSP) at ambient pressure.

01 PROCESSING CONTROLS SAMPLE QUALITY

Relative density increases from ~50% (CSP) and ~58% (HP–HTS) to ~98% (SPS) and ~97% (CA–HP).

02 CA-HP GIVES THE STRONGEST J_c

J_c rises from ~2,000 A cm^{-2} (CSP) to ~8,500 A cm^{-2} (CA–HP ex-situ) and ~9,000 A cm^{-2} (CA–HP in-situ).

03 IN-SITU CA-HP ALSO RAISES T_c

T_c is ~54 K for CSP, HP–HTS, SPS and CA–HP ex-situ, reaching ~57 K for CA–HP *in-situ*.

PROCESSING ROUTE → PHASE / MICROSTRUCTURE → CONNECTIVITY + PINNING → J_c AND T_c

Take-home: CA–HP *in-situ* gives the strongest overall result in this comparison; pressure processing must balance phase quality, density, connectivity and vortex pinning.

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Thank you for your kind attention

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