Intrinsically Tough and Affordable Ceramics Today (INTACT)

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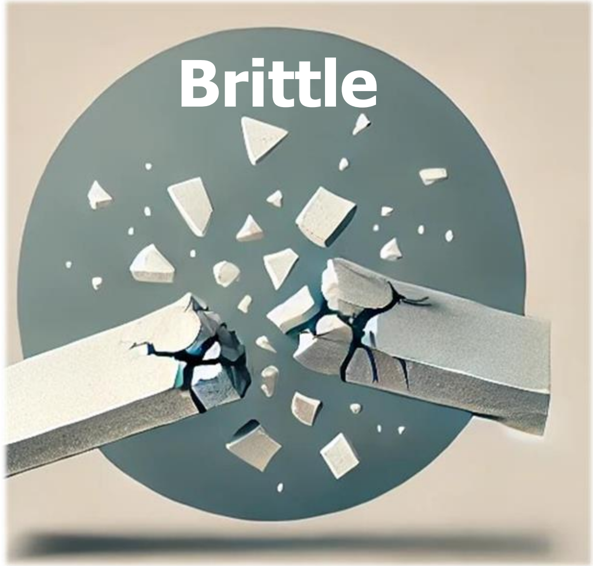
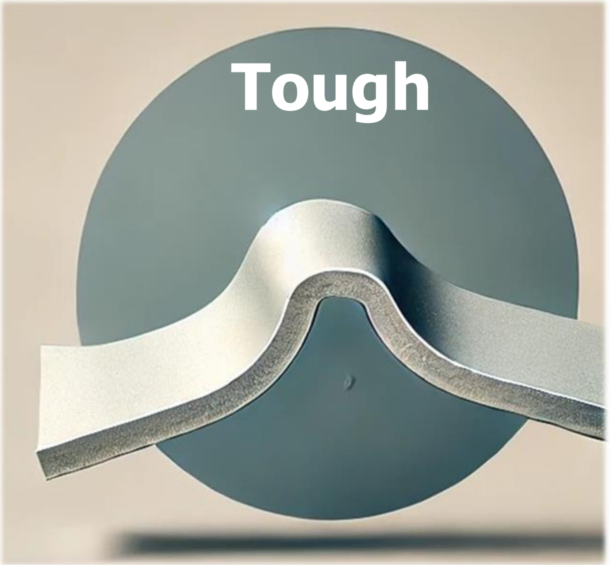


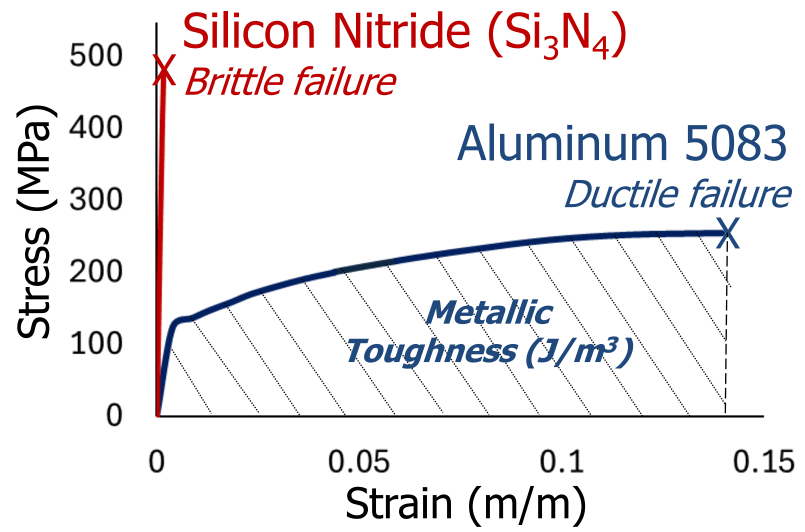


Disruption Opportunity
Information Session
January 21, 2025

Metals vs. Ceramics for Structural Applications

Key Properties	Metals	Ceramics
Ductility	High (>10%)	Low (<1%)
Fracture Toughness	High (>30 MPa$\sqrt{m}$)	Low (<5 MPa$\sqrt{m}$)
Compressive Strength	Moderate (~1 GPa)	High (~10 GPa) [a]
Elastic Modulus	Moderate (~200 GPa)	High (~400 GPa)
Density	Moderate (5-9 g/cm^3)	Low (2-6 g/cm^3)
Max. Use Temperature	Moderate (<1200°C)	High (2000°C+)
Corrosion Resistance	Highly susceptible [b]	Generally inert

[a] Brittle failure mode in ceramics leads to 1/10th strength asymmetry in tension (~1 GPa)
[b] Alloy and environment dependent



Intrinsic toughening mechanisms are active metals – can similar mechanisms be activated in ceramics?

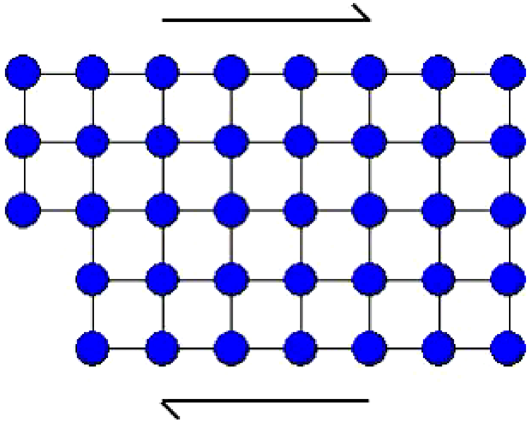
INTACT will explore approaches to engineer tough ceramics

- 10x stronger, 2x stiffer, ½ density relative to metals
- Capable of operating at 2x higher temperatures
- Able to survive harsh environments

INTACT addresses the Achilles heel of ceramics (low toughness) to open a new class of structural materials

Why are Metals Ductile and Ceramics Brittle?

Rigid lattice translation
Shear stress: G/5
(16 GPa for steel)

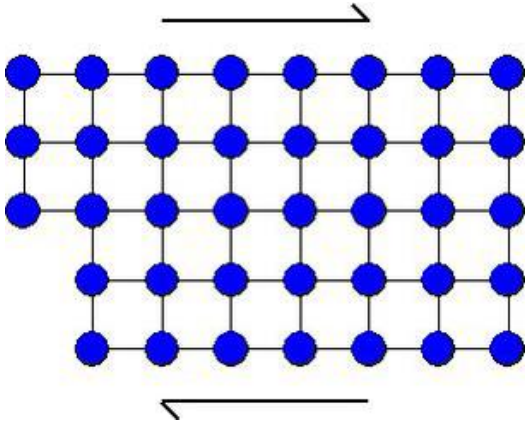


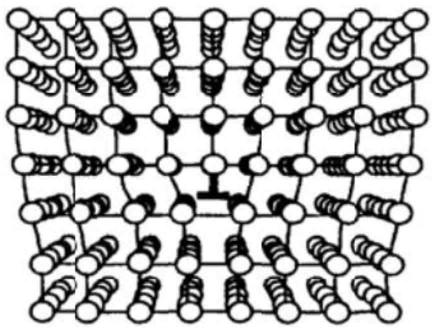
https://www.doitpoms.ac.uk/tlplib/dislocations/printall.php

vs.

Dislocation motion
Shear stress: G/180
(400 MPa for steel)

Fundamental mechanism of plasticity in metals

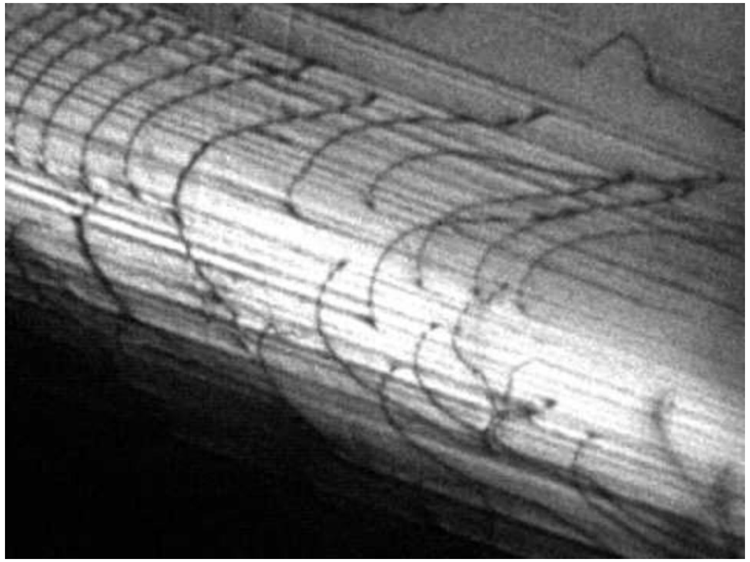




Shear stress required to *nucleate* and *glide* dislocations

	Nucleation (GPa)	Glide (GPa)
Metals	0.1-1	0.01-0.5
Ceramics	10-30	0.1-10

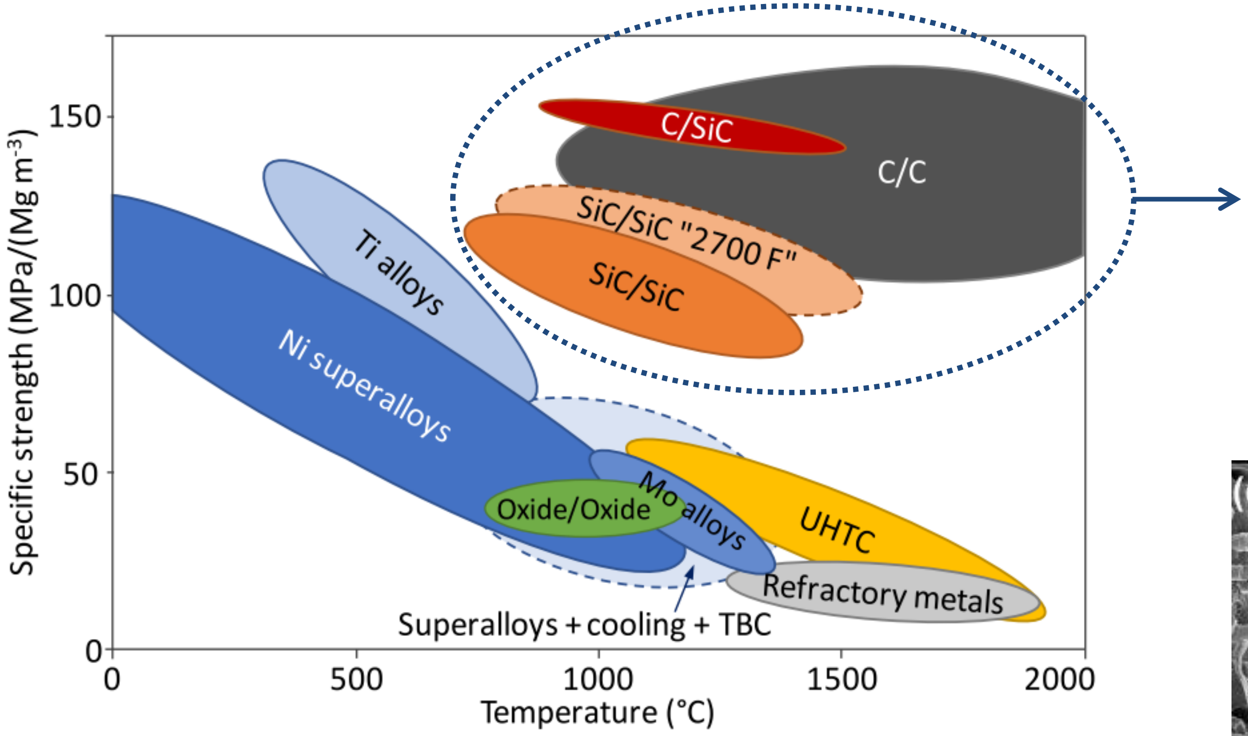
Dislocation activity is limited in ceramics,
but it is not absent



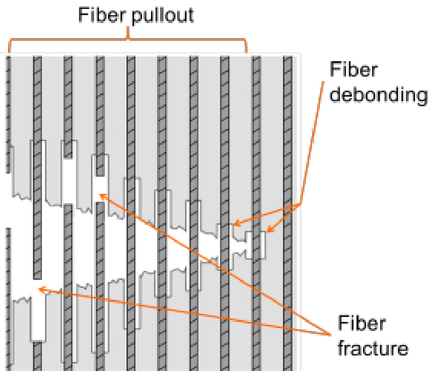
Dislocation glide experimentally observed in 304 stainless steel

G: Shear modulus (GPa)

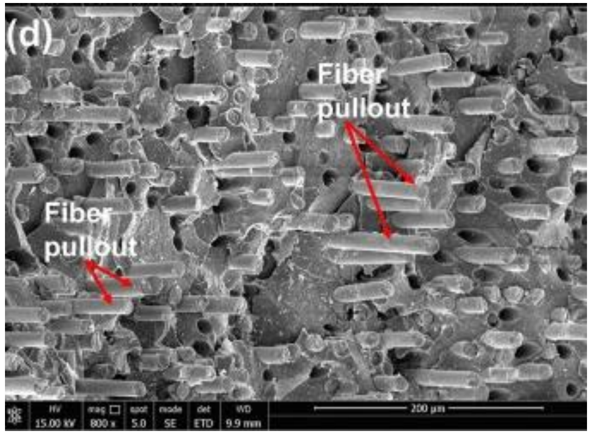
Ceramic Matrix Composites – Today's Solution to Ceramic Brittleness

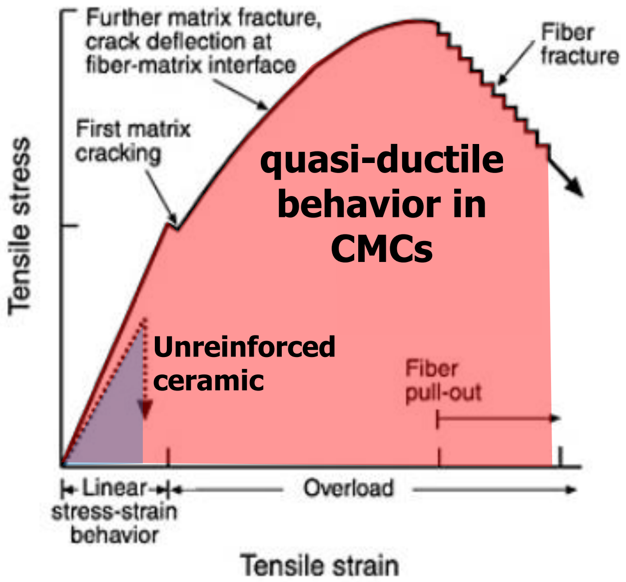


P. Fenetaud et al., Open Ceram, 15, 100396, 2023



Individual fiber fracture energy $\propto d_f^2 \sigma_f^2 L_f / E_f$





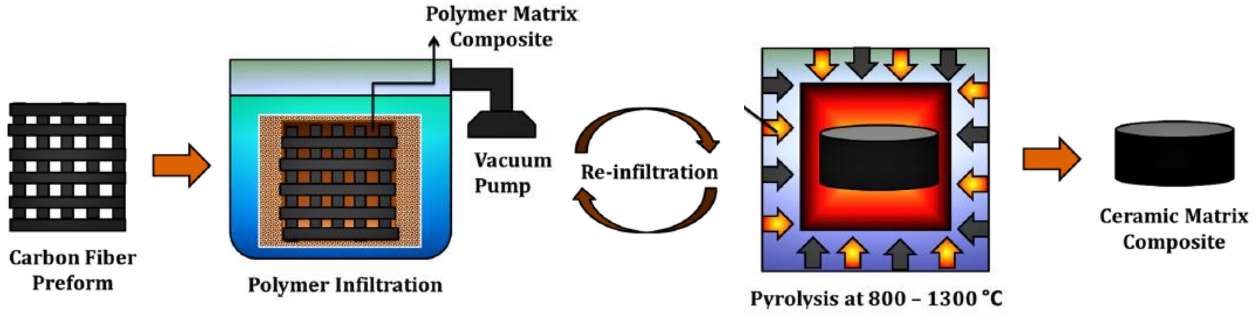
Liu H., Appl. Compos. Mater., 29(3), 2022
Beaumont, AFOSR Report, TR-79-0489, 1979

Fibers are added to impede cracking in ceramics leading to quasi-ductility

Fracture toughness values approaching 30 MPa$\sqrt{m}$ are possible

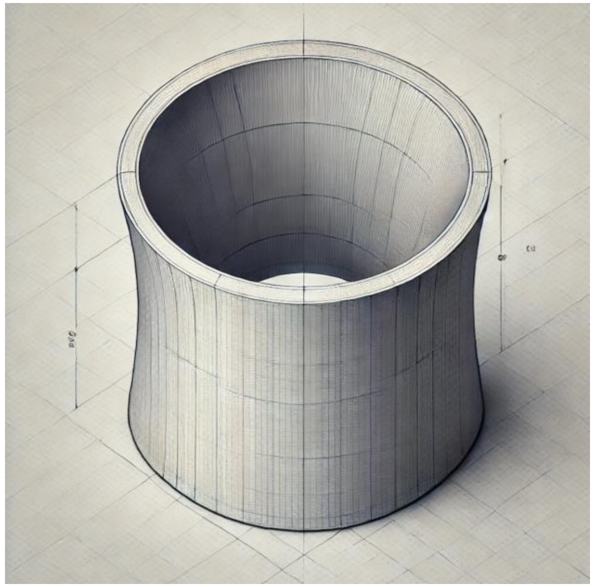
UHTC: Ultra-high temperature ceramics
CMCs: Ceramic Matrix Composites

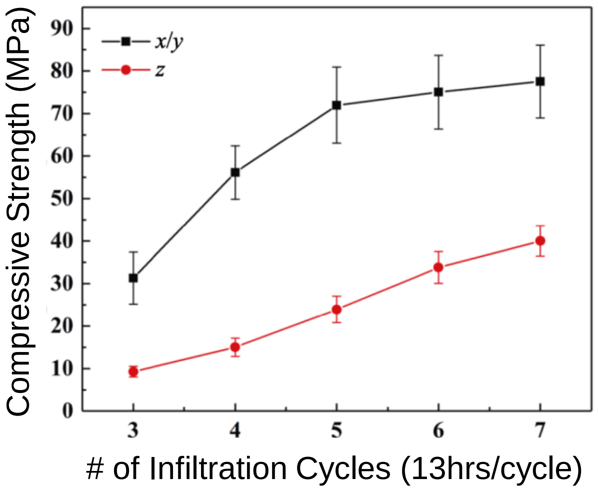
Polymer Infiltration and Pyrolysis (PIP) Process



Adapted from: Radhika et al., Silicon, 4:10141–10171, 2022

Multistep process bottlenecked by the need to infiltrate dense fiber preforms





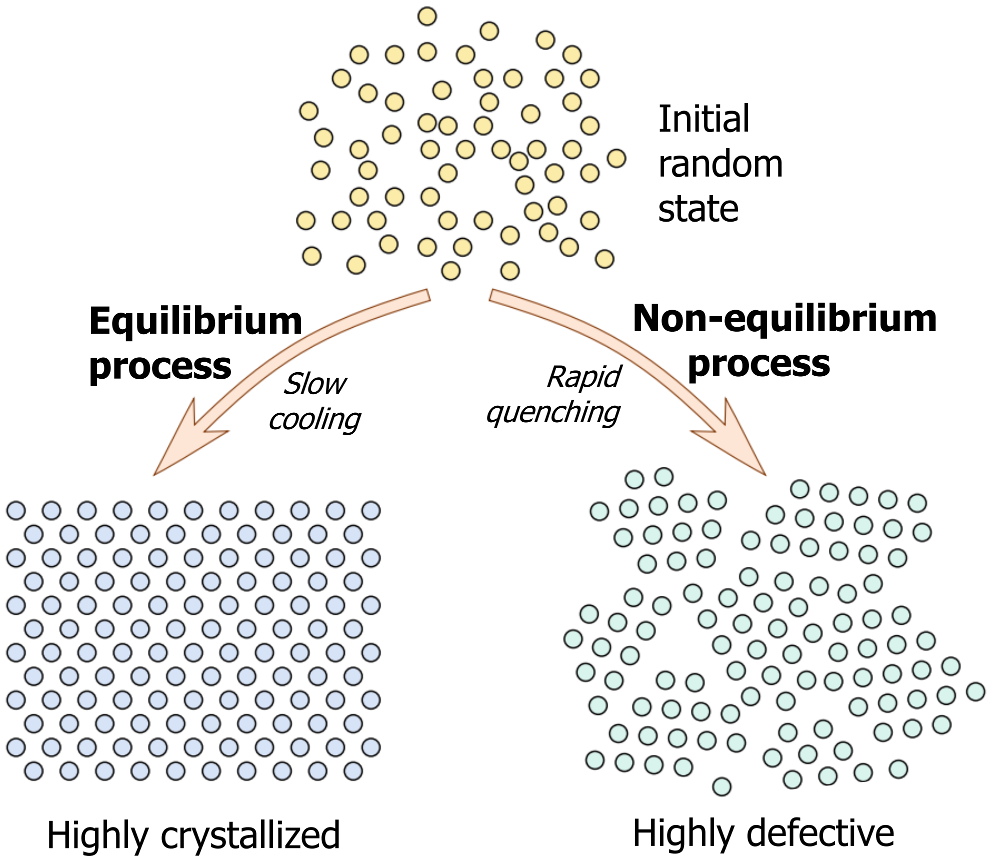
Lyu, et al., J. Advanced Ceramics, 11, 2022

- Infiltration steps >50% of manufacturing cycle time
- Densities >90% are difficult to achieve
- Material properties are heterogeneous based on fiber layup
- Limited to simple shapes – radii of curvature constrained by fibers ($R_{min} \propto E_f d/2\sigma_f$); typical R_{min}>2mm

CMC manufacturing is complicated
Lead times of 6 months or more and 10's of $k are typical, even for simple shapes

AFRL: Air Force Research Laboratory
RDE: Rotating Detonation Engine

INTACT’s New Approach – Explore **Non-Equilibrium** Processing



Adapted from: Taillard, Design of Heuristic Algorithms for Hard Optimization, Springer, 2023, p.156

Today: Equilibrium Ceramic Processing

- Ceramic powders are densified into solid shapes by sintering at high temperatures for extended time with slow heating/cooling rates
- e.g., typical Si_3N_4 sintering cycle: 1800°C/5hrs, 5°C/min cooling
- Yields near-equilibrium material (low defect population)

INTACT will Explore Non-Equilibrium Processing

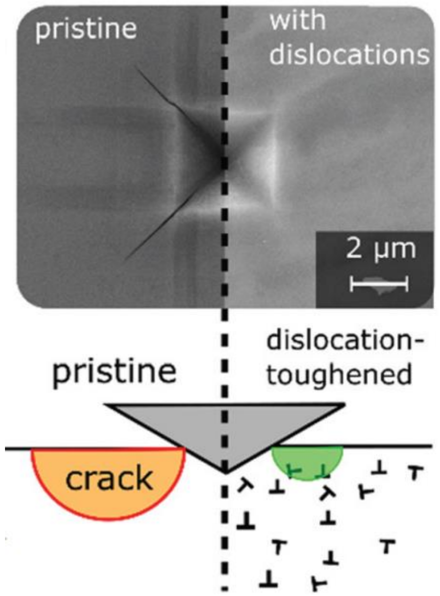
- Thermal processing routes enabled by, e.g., laser or electron beam methods (10^3-10^6 °C/s cooling rates typical)
- Introduction of mechanical work during production (e.g., forging)
- Other methods promoting *atomistic-scale* toughening

Toughness may be engineered into monolithic ceramic materials using non-equilibrium processing techniques

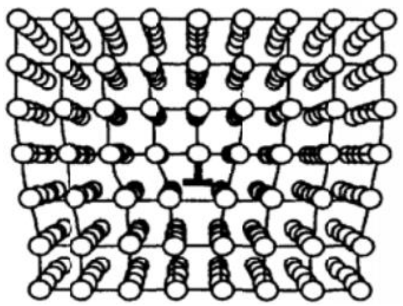
Emerging Possibilities in Thermomechanical Processing

Mechanical

Activating dislocations for plasticity



Porz, et al., Materials Horizons, 8 (2021)

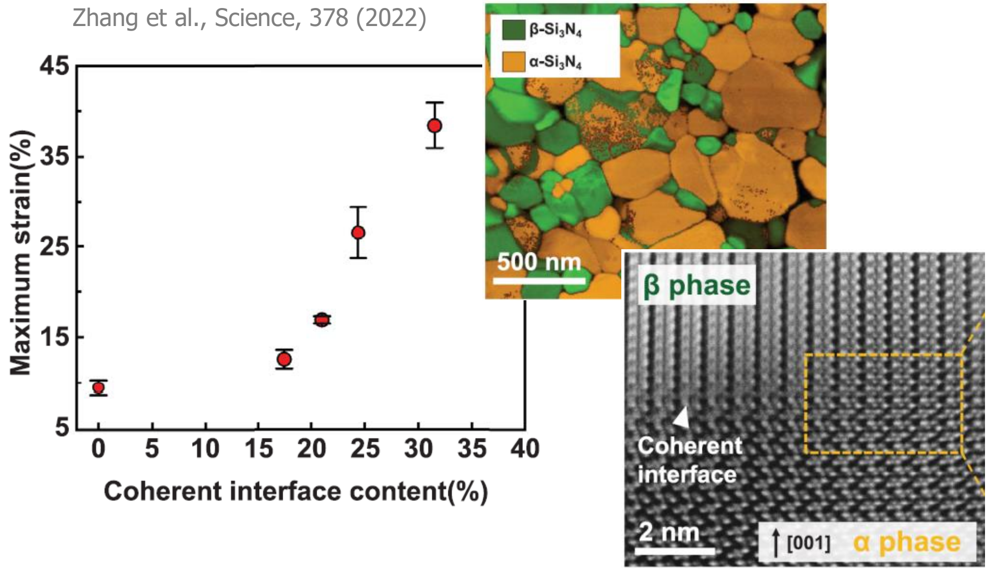


- Dislocation content increased from to 10^{10} to 10^{14} m^{-2} in $SrTiO_3$ through surface deformation
- <u>18x</u> increase in toughness due to dislocation motion; no cracking observed around indent
- *Caveat: Demonstrated in 5μm surface layer*

Thermal

Leveraging precise thermal control during processing

Zhang et al., Science, 378 (2022)



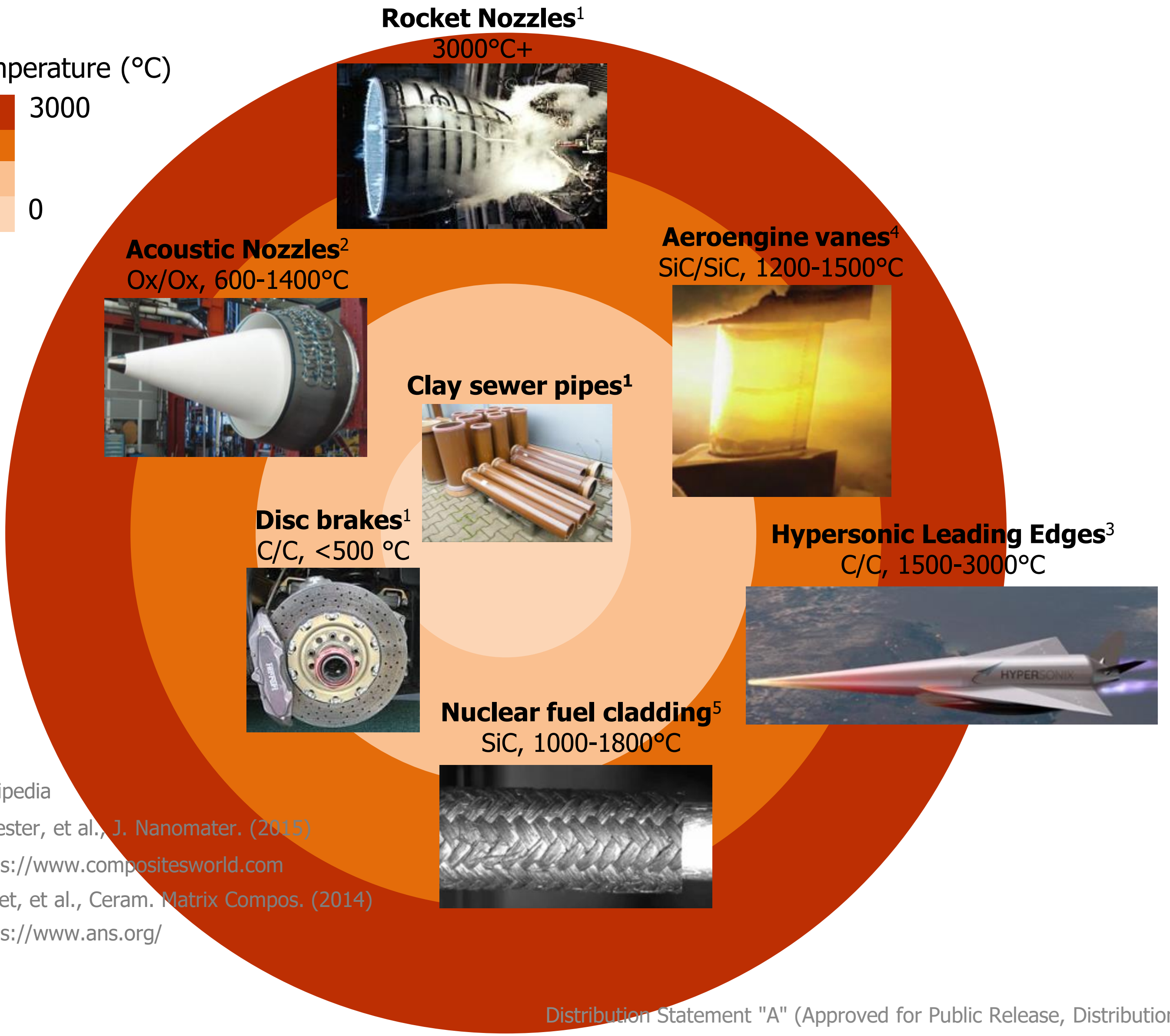
- α/β dual phase Si_3N_4 achieved through spark plasma sintering (1500°C/5min, 150°C/min heating/cooling)
- Plasticity enabled through stress-induced bond switching at coherent α/β interfaces; <u>40%</u> ductility observed
- *Caveat: Demonstrated on nano-pillars*

Recent results hint toward the possibility of intrinsically toughening bulk ceramics through engineered defects

INTACT Application Areas & Impact

Wide range of applications across temperatures

Service temperature (°C)
3000
0



(1) Wikipedia
(2) Silvester, et al., J. Nanomater. (2015)
(3) https://www.compositesworld.com
(4) Spriet, et al., Ceram. Matrix Compos. (2014)
(5) https://www.ans.org/

<u>If successful, INTACT would</u>:

- Disrupt the highly specialized CMC manufacturing industry
- Broaden the industrial base for high temperature ceramic structures
- Reduce component lead times from months to days
- Enable production of advanced high temperature structures at scale

INTACT could have broad impact across a range of DoD and commercial applications

Program Details

INTACT Program Structure, Metrics, and Requirements

Program Structure

Phase 1 (12 mos.)
Coupon-level material development to meet property metrics

→ *Performer Go/No-Go*

Phase 2 (12 mos.)
Challenge part demonstration and property optimization

Program Metrics

Metric	Units	Phase 1	Phase 2
Flexural strength @ 1200°C [a]	MPa	300	500
Fracture toughness [b]	MPa·$m^{1/2}$	15	30
Cycle time [c]	hours	48	24

[a] As measured by ASTM C1211 (or equivalent)
[b] As measured by ASTM C1421 (or equivalent) at room temperature
[c] Total time from start of production to near net shape, excluding post-process machining & inspection

Material Requirements

- Bulk material density ≤6 g/cm^3
- Flexural modulus >100 GPa at room temp
- Strength and fracture toughness must be measured at initial production and after a 1200°C/24hr anneal

Challenge Part Requirement

Performers must propose a challenge part for Phase 2. The proposed challenge part geometry must be larger than a 1x1x10 cm envelope and contain features that are difficult or impossible to realize in conventional fiber CMC manufacturing (e.g., tight radii of curvature, thin-walled sections, and/or internal geometric features)

CMC: Ceramic Matrix Composites

INTACT Milestones

Phase	Month	Milestone
Phase 1	**Month 1**	Kick-off meeting; Roles and effort level assigned
	Month 4	First bulk (>1mm) test coupon produced
	Month 6	Preliminary strength and fracture toughness testing
	Month 9	Material microstructure stability assessed; Preliminary manufacturing process plans (MPPs) drafted
	Month 12	Strength and fracture toughness test results with repeatability; Material datasheet drafted; Phase 1 final report
Phase 2	**Month 13**	Phase 2 kickoff meeting
	Month 17	Material property stability assessed
	Month 20	Process sensitivity study to define manufacturing window; Expanded mechanical property testing
	Month 23	Challenge part manufacturing demonstration complete
	Month 24	Final report including process, properties, future R&D, and industry adoption pathway

INTACT Deliverables

Performers will be expected to provide, at a minimum, the following deliverables:

- **Monthly presentation slides** highlighting progress, challenges, and future direction
- Comprehensive, stand-alone **written reports** for Months 4, 6, 9, and 17 project milestones
- Draft (Month 9) and final (Month 23) **manufacturing process plans**
- A draft (Month 12) and final (Month 20) **material datasheet**
- Comprehensive **manufacturing demonstration report** at Month 23
- Phase 1 completion (Month 12) and project completion (Month 24) **reports**

What is NOT in scope?

INTACT is not constrained to any particular ceramic material composition or processing approach.

However, proposed approaches relying on *any of the following toughening mechanisms* will be deemed out of scope:

- Architected material solutions where toughness is derived through mechanical design and/or structural geometry
- Engineered residual stress distribution (surface and/or bulk) to delay the onset of cracking
- Any other process relying on traditional CMC toughening mechanisms, or any other extrinsic mechanism, operating at a characteristic length scale exceeding 1μm

The purpose of INTACT is to explore intrinsic atomistic-scale toughening mechanisms in bulk ceramics

Tips for Success

- **Use graphics** to clearly articulate your breakthrough idea up front.
- Succinctly state **what is novel in your approach** and why you think it will be successful.
- Detail the ceramic material(s) and processing method(s) you will investigate and **why you selected these**.
- Thoroughly **address every metric** and requirement. Be quantitative. Simply stating "we will meet the metric" is not sufficient.
- Include an **objective risk assessment** and credible mitigation plans.
- Include specific examples on the **potential contribution** of your proposed approach to DARPA's mission.
- **Limit regurgitation** of assertions from the solicitation.
- Follow all proposal instructions, **use the provided templates**, and include all required attachments.
- Strategies focusing heavily on the <u>method</u> of discovery (e.g., artificial intelligence driven autonomous research) and lacking sound theoretical materials science underpinnings **will be reviewed unfavorably**.
- Include preliminary experimental results, modeling, simulation, or theoretical justification to **support any assertions in your proposal**.

INTACT Key Dates

- INTACT Program Announcement released: 15 JAN 2025
- Proposer profiles due: 24 JAN 2024 at 4:00pm EST
- Last day to submit questions to INTACT@darpa.mil: 7 MAR 2025
- Proposal due date: **14 MAR 2025 at 4:00pm EST** *(no exceptions!)*
- Anticipated program start date: 12 MAY 2025

Contracting Overview

John Bauer

DARPA

Non-Traditional Award Instruments

DSO DEFENSE SCIENCES OFFICE

❑ Research OTs – 10 U.S.C. § 4021

- A legally binding instrument other than a procurement contract, grant, or cooperative agreement for performing basic, applied, or advanced research & development
- Result of the effort is a study/whitepaper
- Can be awarded off the following solicitation types:
  - Broad Agency Announcement (BAA)
  - Program Announcements (DARPA-Specific)
  - Exploration Announcements (DARPA-Specific)

❑ Prototype OTs – 10 U.S.C. § 4022

- A legally binding instrument other than a procurement contract, grant, or cooperative agreement used for prototype projects proposed to be acquired by the DoD
- Result of the effort is a prototype
- Can be awarded off the following solicitation types:
  - Broad Agency Announcements
  - Program Solicitation (DARPA-Specific)

❑ What doesn't apply to OTs?

- Cost Accounting Standards
- FAR/DFARS/Agency specific acquisition regulations

❑ What laws still do apply?

- Laws that would apply to anyone doing business in the U.S. (e.g. environmental laws, import/export control)

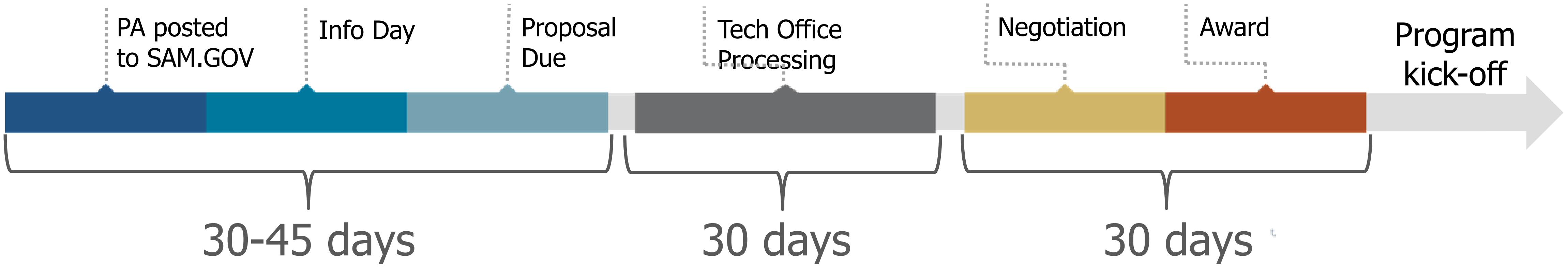
Interested in learning more?

Check out CMO's DARPAConnect Corner Webinar on Understanding Other Transactions:

How Long Does Funding Take?

Rapid Exploration Process Overview

Average Timeline from Announcement to Award for Program Announcements (PAs)



- All awards are OT for Prototypes
- Larger volumes of high-risk, high-reward, early-stage ideas.
- Artificial Intelligence Exploration (AIE): I2O
- Disruption Opportunity (DO): DSO
- Microsystems Exploration (μE): MTO

- AIE: $1M/award; $5M/topic
  - Processing: < 90 days
- DO/μE: $2M/award; $10M/topic
  - Processing: < 120 days

- Two-phase Projects
  - Phase 1 – Feasibility Study
  - Phase 2 – Proof of Concept
- AIE: NTE 18 months
- DO/μE: NTE 24 months
- Does not include follow-on acquisition post Phase 2

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