Winter 3-10-2016

Field effects during consolidation of metallic powders

Brandon McWilliams
US Army Research Laboratory, brandon.a.mcwilliams.civ@mail.mil

Field effects during consolidation of metallic powders

Brandon McWilliams and Jian Yu

Army Research Laboratory, Weapons & Materials Research Directorate, Aberdeen Proving Ground, MD

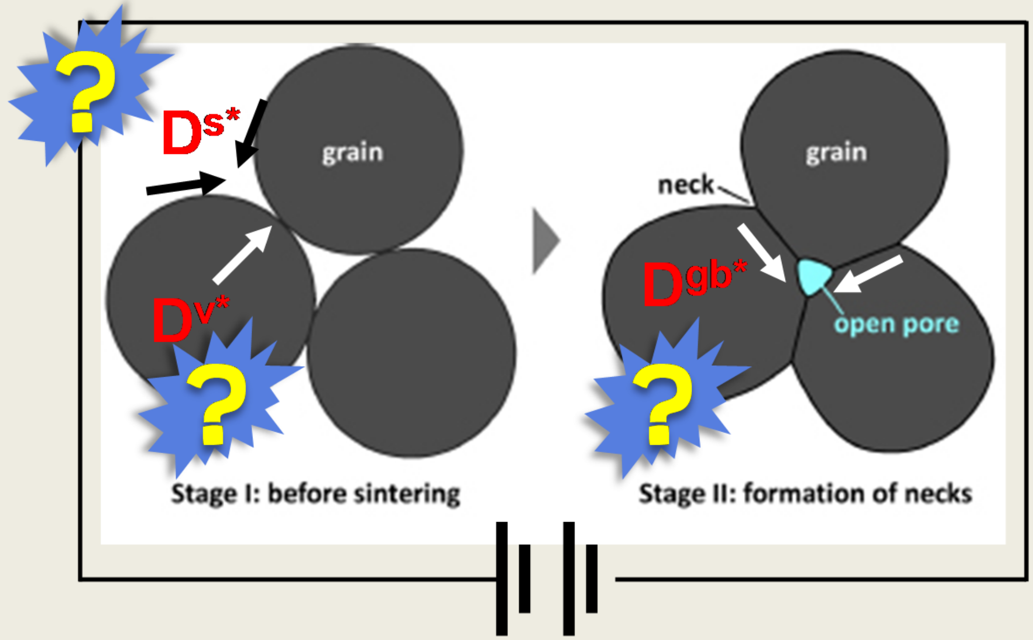
brandon.a.mcwilliams.civ@mail.mil

Electric Field Assisted Sintering and Related Phenomena Far From Equilibrium
March 6-11, 2016 Tomar, Portugal

In situ determination of enhanced sintering kinetics due to applied fields

Research Objectives

Develop an ***in situ*** methodology to investigate **sintering kinetics** of powder compacts under the influence of an **applied field**.



Challenges

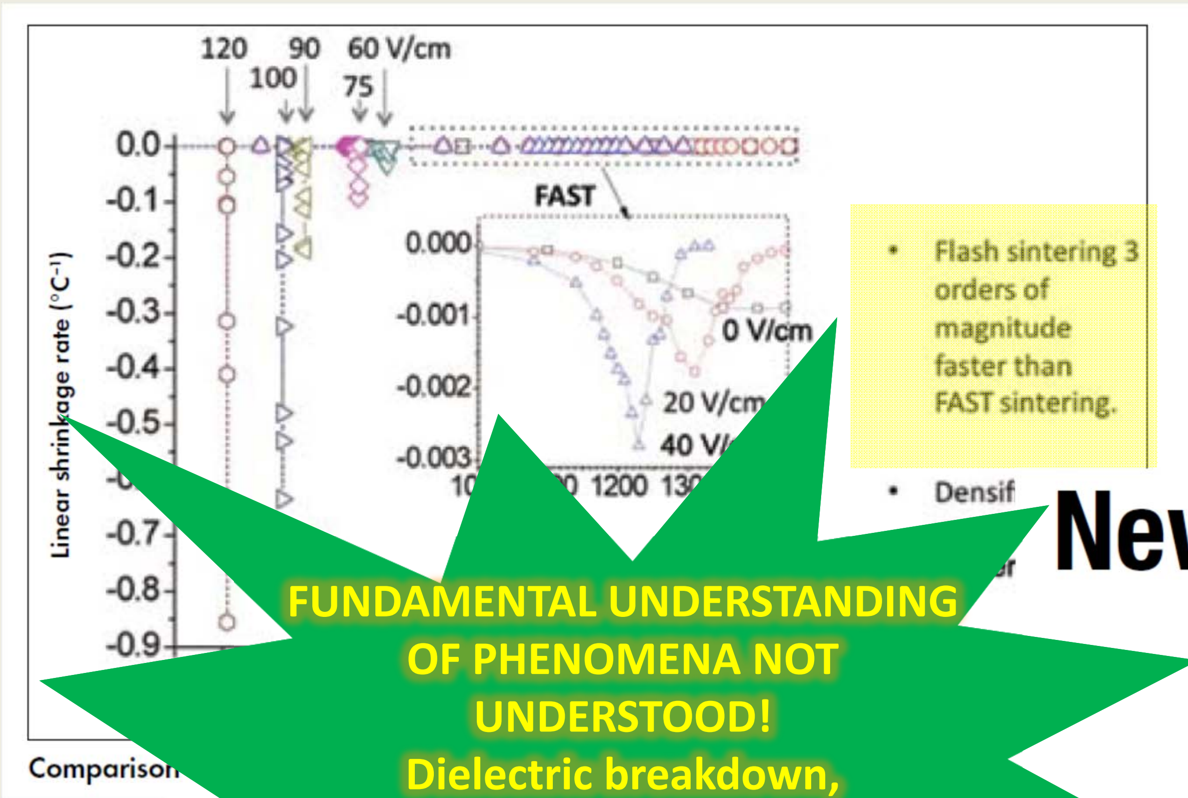
Develop understanding of **underlying mechanisms** contributing to mass transport at the grain scale; develop field **enhanced constitutive models** for process modeling of sintering kinetics

Impact

Exploit field enhancement(s) for net shape manufacturing of next generation ceramics and metals; enhanced process models for virtual manufacturing to promote **rapid transition** of technologies to Warfighter

B. McWilliams, J. Yu, and A. Zavaliangos, Fully coupled thermal-electric-sintering simulation of electric field assisted sintering of net-shape compacts, *J. Mat. Sci.* 50 (2015) 519-530.

What is flash sintering?



- Flash sintering 3 orders of magnitude faster than FAST sintering.
- Densif

- No pressure
- Constant voltage
- Constant heating rate (10°C/min)

FUNDAMENTAL UNDERSTANDING OF PHENOMENA NOT UNDERSTOOD!
Dielectric breakdown, conductivity f(T), photoemission, electromigration, Joule heating, … ??

New paradigm prophecy

April 2013

By Peter Wray

Rishi Raj explains the discovery of flash sintering and electrical fields and other field effects will revolutionize ceramic manufacturing.

American Ceramic Society Bulletin, Vol. 92, No. 3

U.S. ARMY RDECOM

Relation to SPS/FAST processing

ARL

SPS – current, heat, pressure → Complex!

But it works!

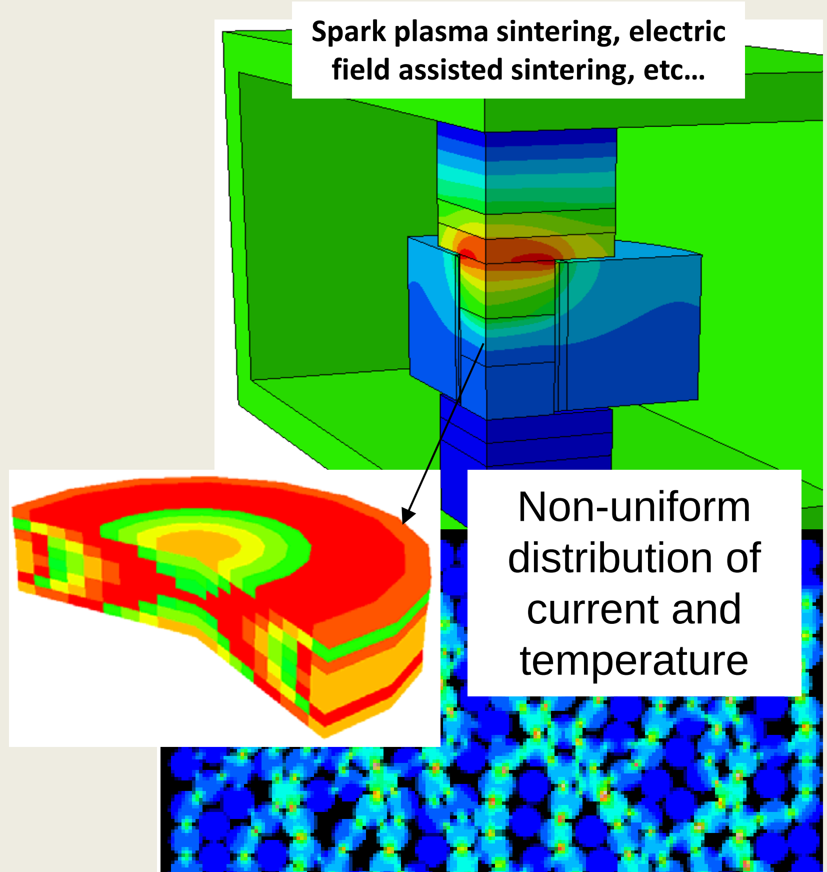
- **Ability to sinter "difficult" materials and at "lower" temps**

Why? TBD!

- **Mechanisms poorly understood for metals and ceramics**

<u>GOAL</u>: Answer: What is the effect of the electric field?

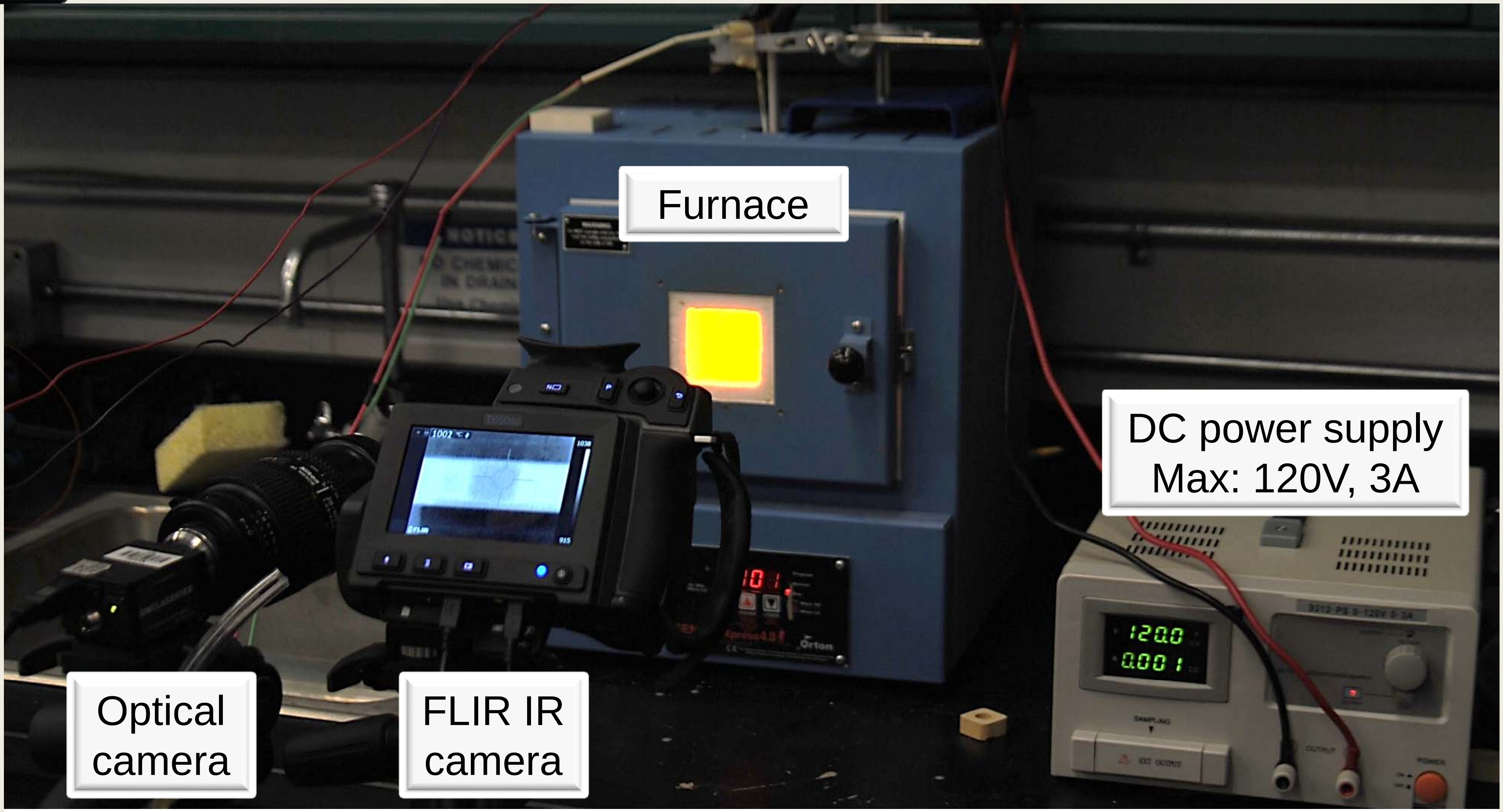
- **Design <u>controlled</u> experiment to determine and quantify effect of electric field *<u>and/or</u>* electric current on sintering kinetics and microstructure evolution**



- **Controlled current path**
- **NO stress/pressure**
- **Uniform heating**

Method	Typical aplied current	Typical voltage across specimen	Typical E field across specimen
Field assisted sintering	1000s of A	1 - 10V	< 10 V/cm
Flash sintering	mA to A	100-1000V	100s to 1000s V/cm

Flash sintering processing infrastructure



- **Full field DIC** strain measurement, **temperature** (furnace thermocouple close to sample, and electric **current**.
- FLIR IR camera (optional) for full field sample temperature and high temperature DIC strain measurements

Innovative metal powder processing using applied DC fields

Aluminum 5083

Cold Isostatic Pressed (CIP)

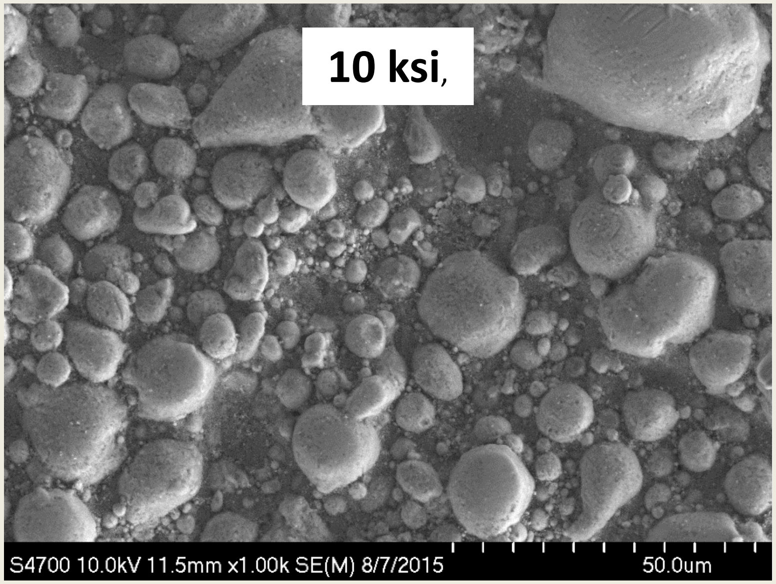
- 72 and 138 MPa to study effect of starting green density/microstructure

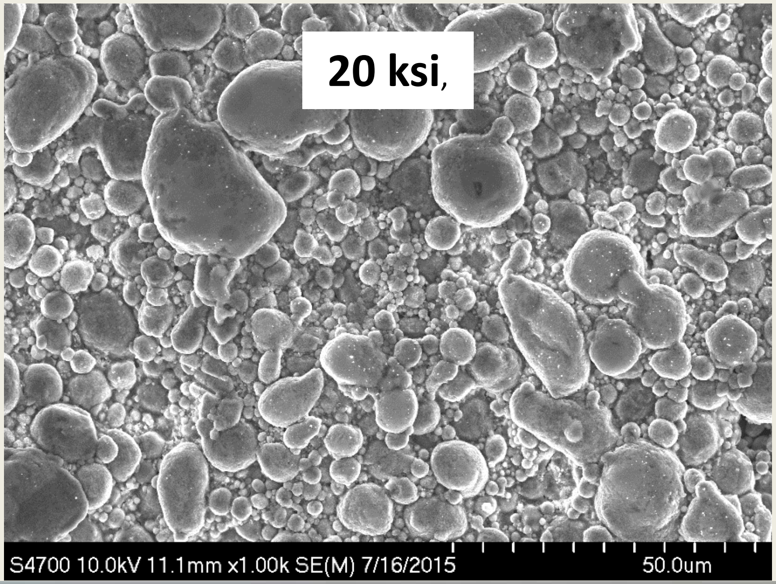
4 °C/min to 550°C, hold for 90 min

Argon atmosphere

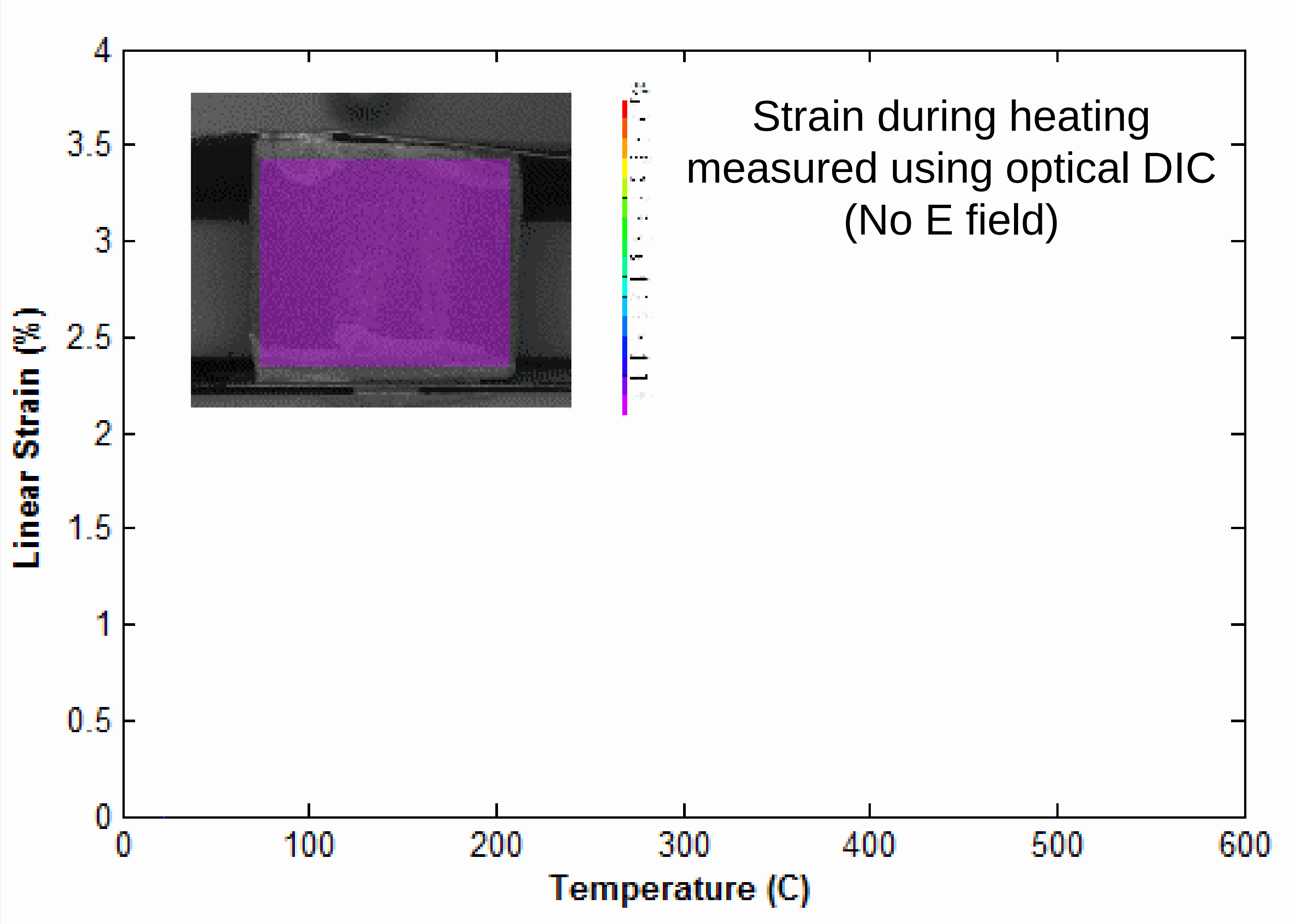
In-situ strain using optical DIC

NO Current			
Starting density	Heating rate (°C/min)	Max temp (°C)	Hold time (min)
10 ksi	4	550	90
20 ksi	4	550	90

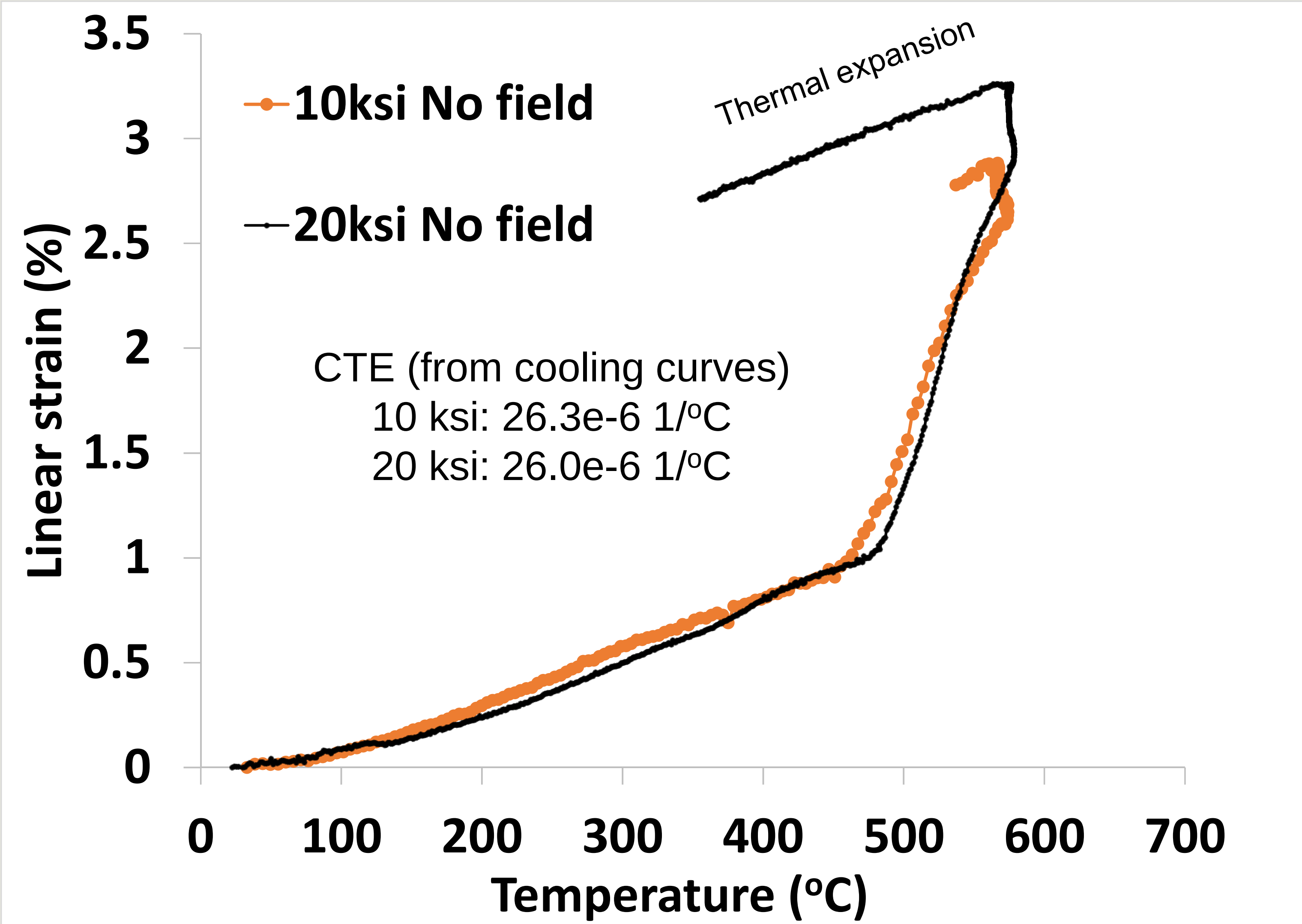




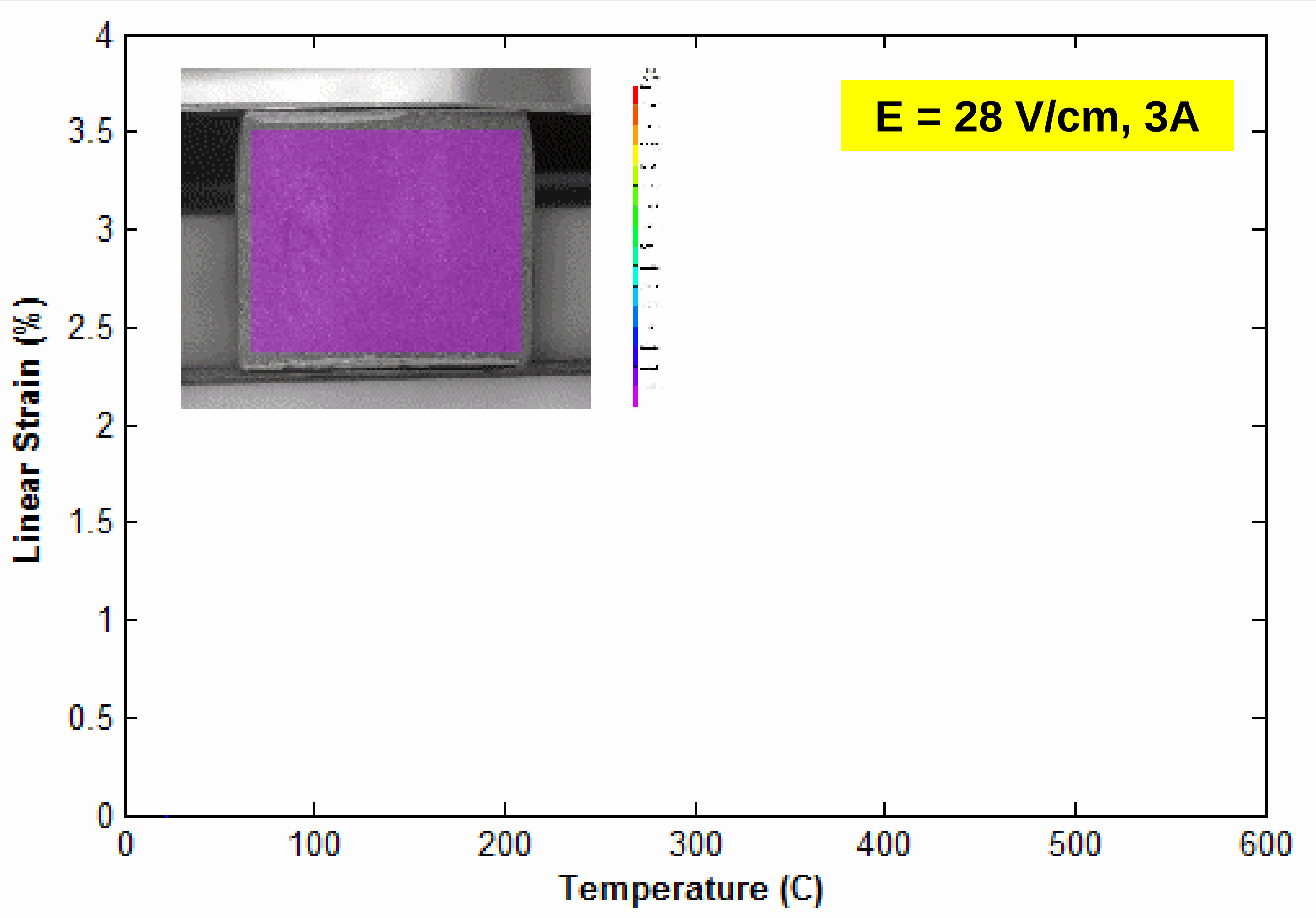
In-situ measurement of sintering kinetics: No applied field



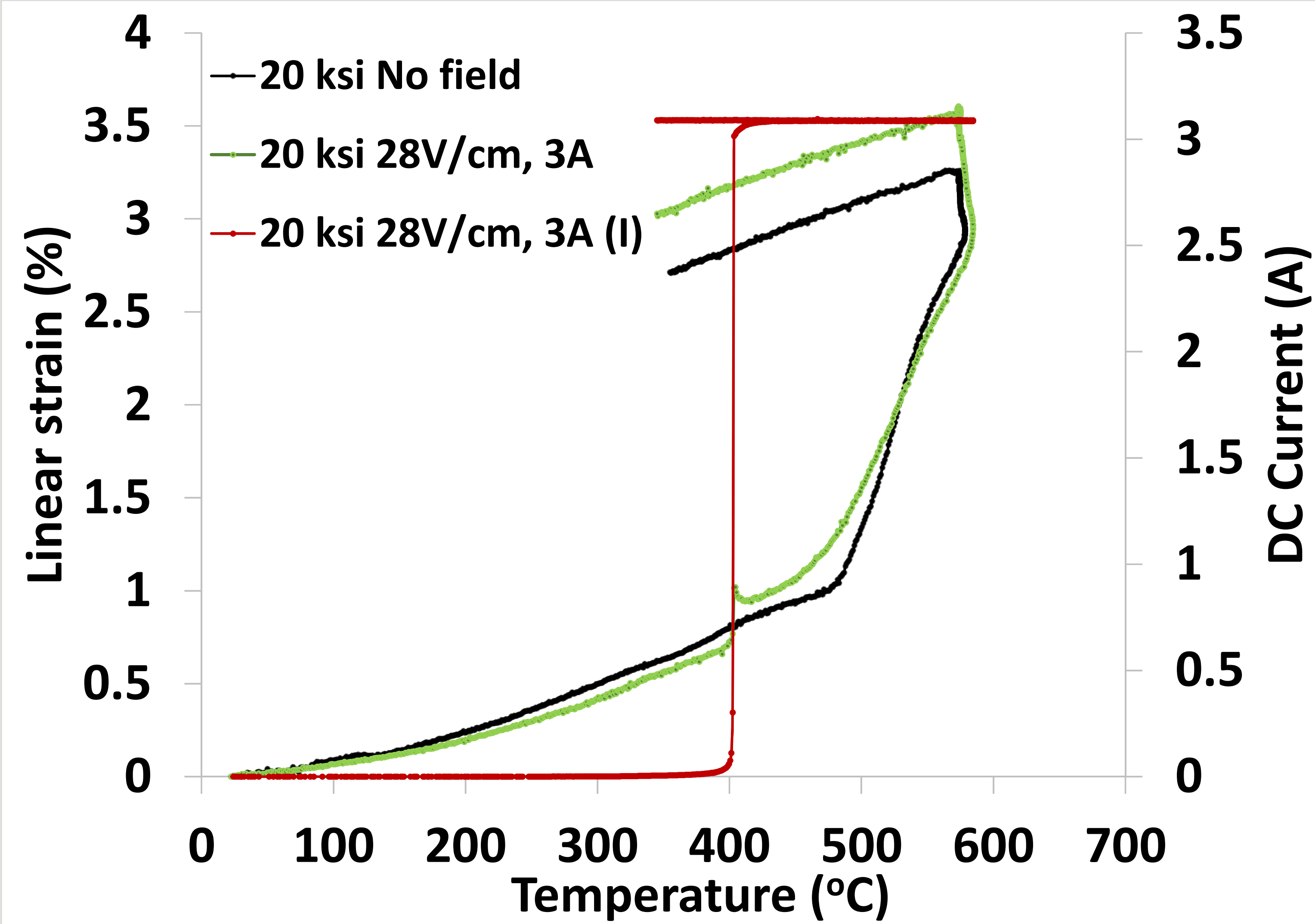
In-situ measurement of sintering kinetics: No applied field



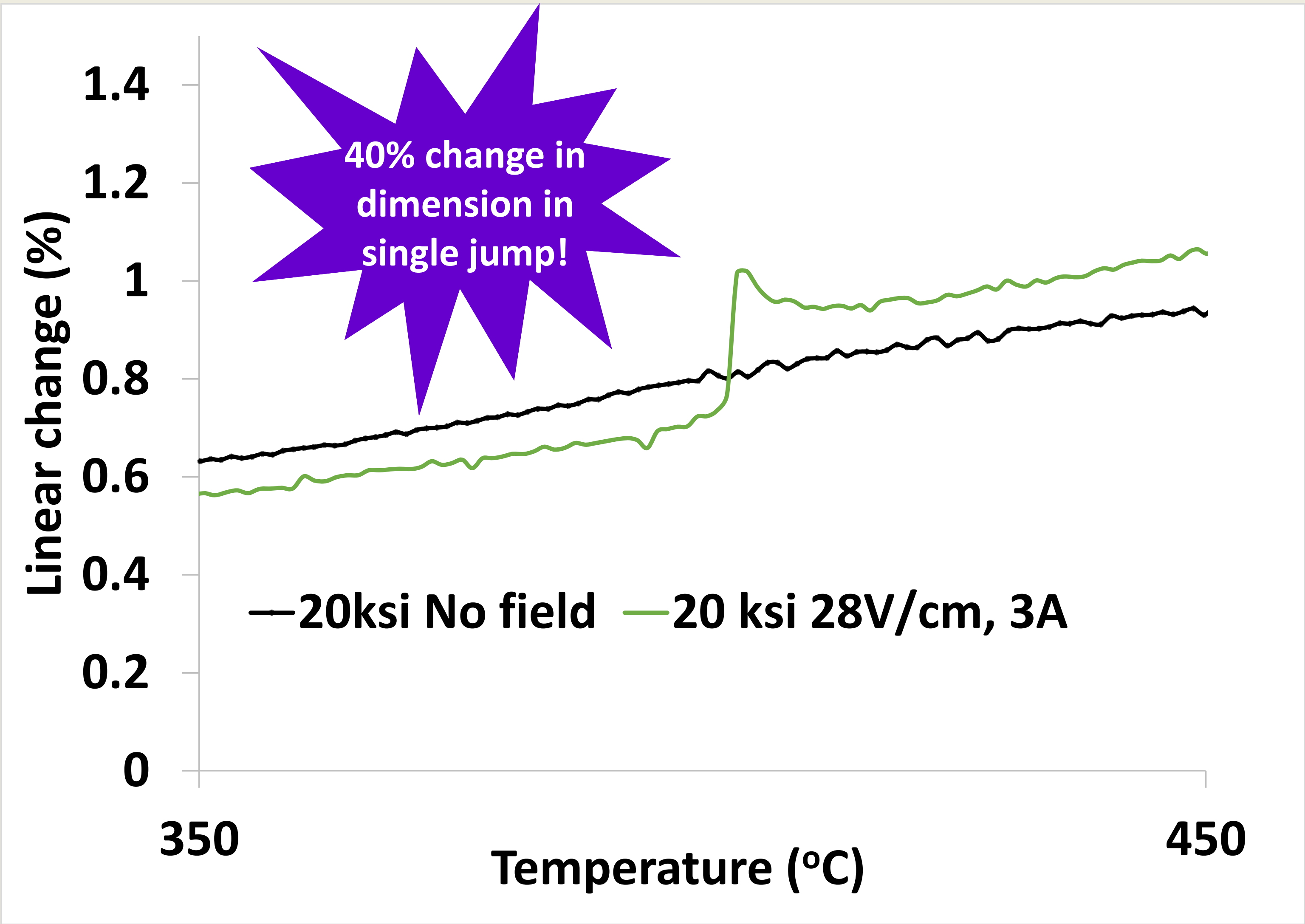
In-situ measurement of sintering kinetics: E = 28 V/cm



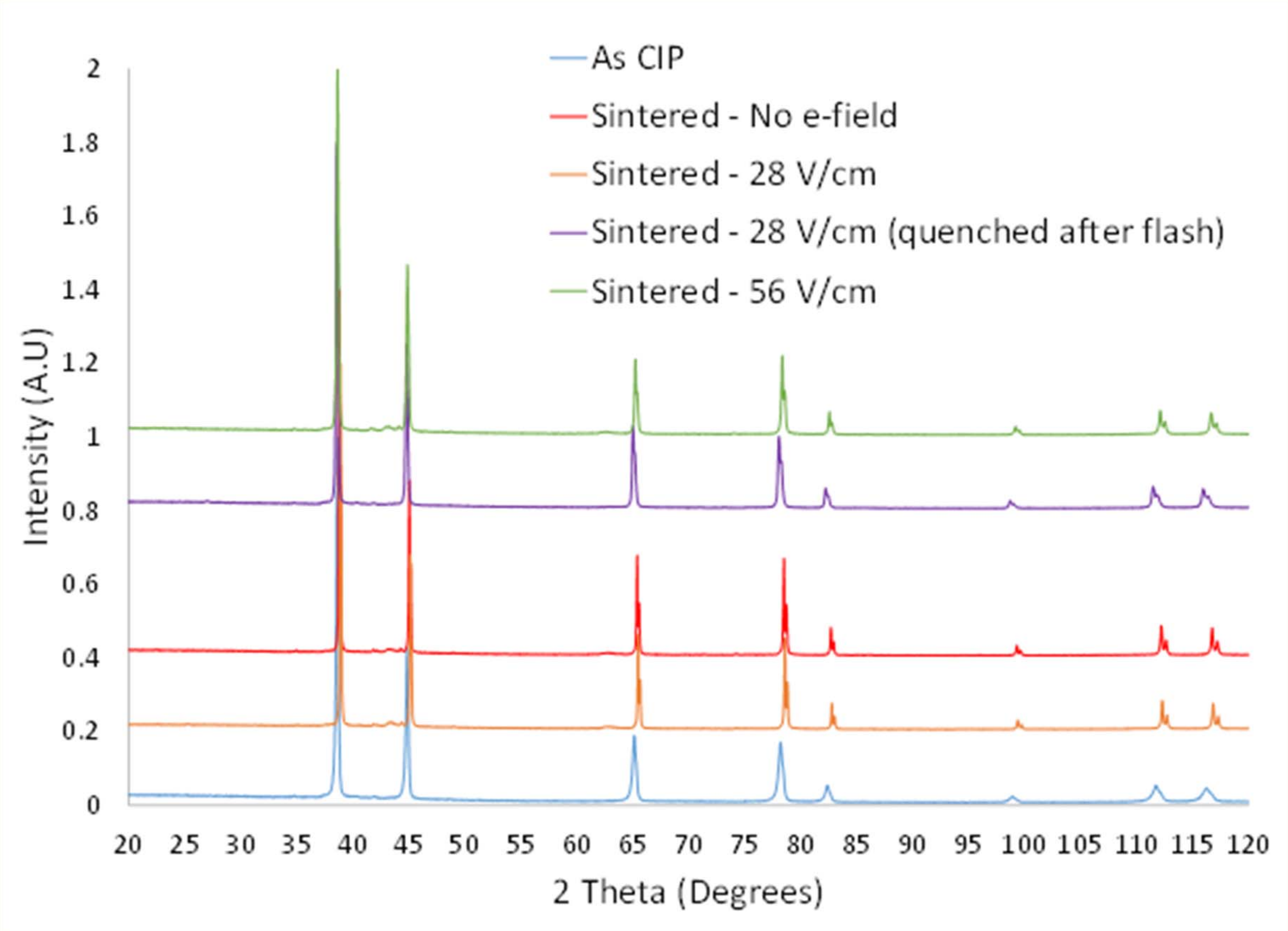
In-situ measurement of sintering kinetics: comparison



Evidence of "flash" phenomena in metal powder processing

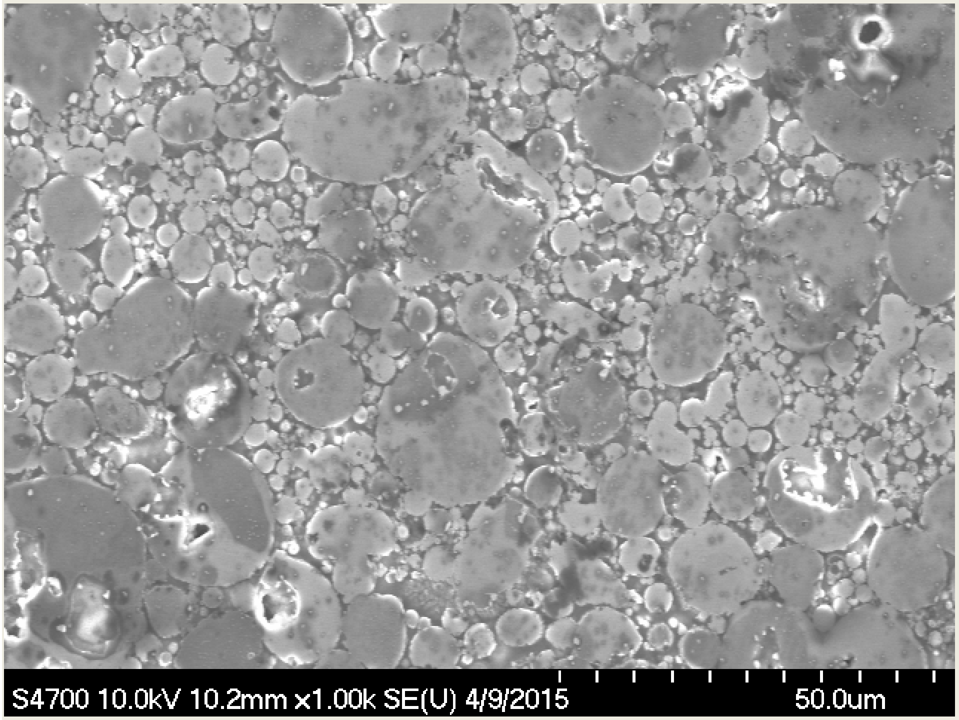


XRD

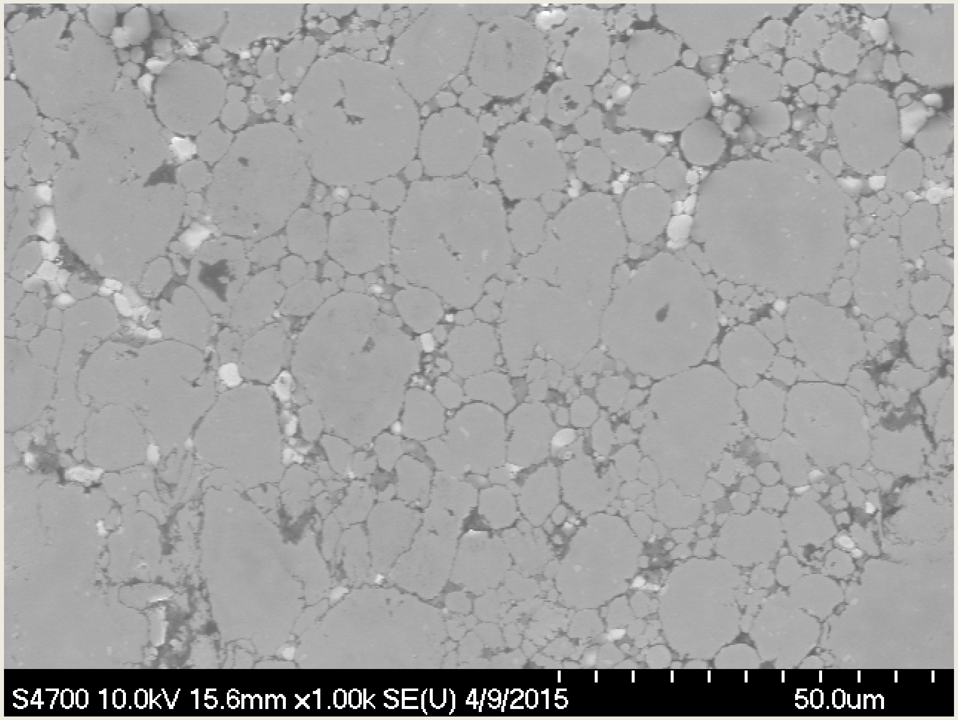

As CIP
Sintered - No e-field
Sintered - 28 V/cm
Sintered - 28 V/cm (quenched after flash)
Sintered - 56 V/cm
Intensity (A.U)
2
1.8
1.6
1.4
1.2
1
0.8
0.6
0.4
0.2
0
20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100 105 110 115 120
2 Theta (Degrees)

Linking processing to microstructure

No electric field



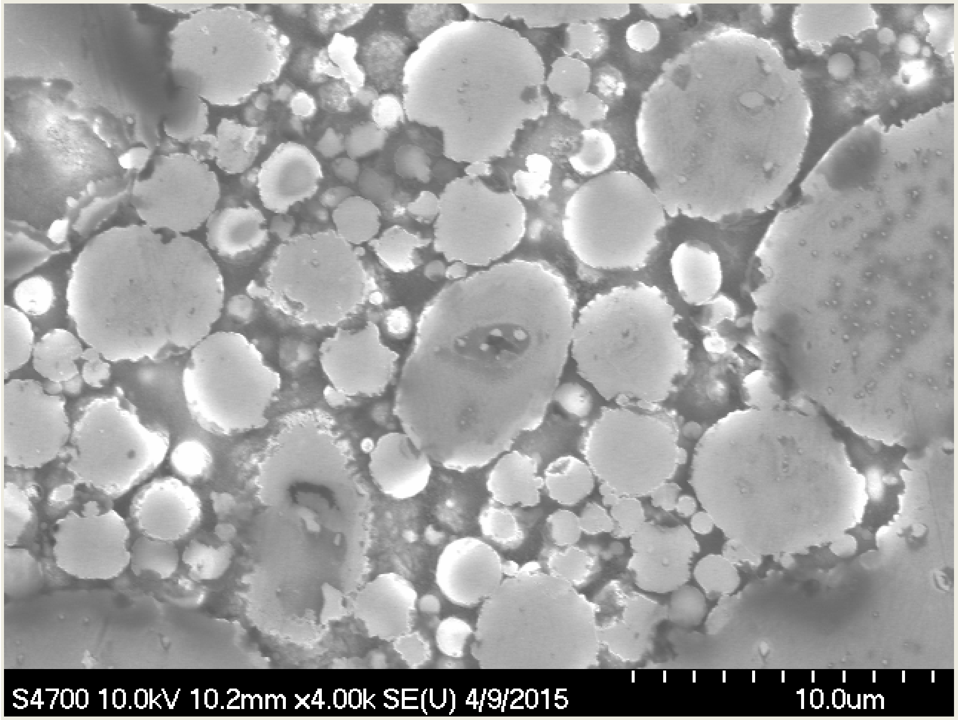
28V/cm, 3A (sample edge)



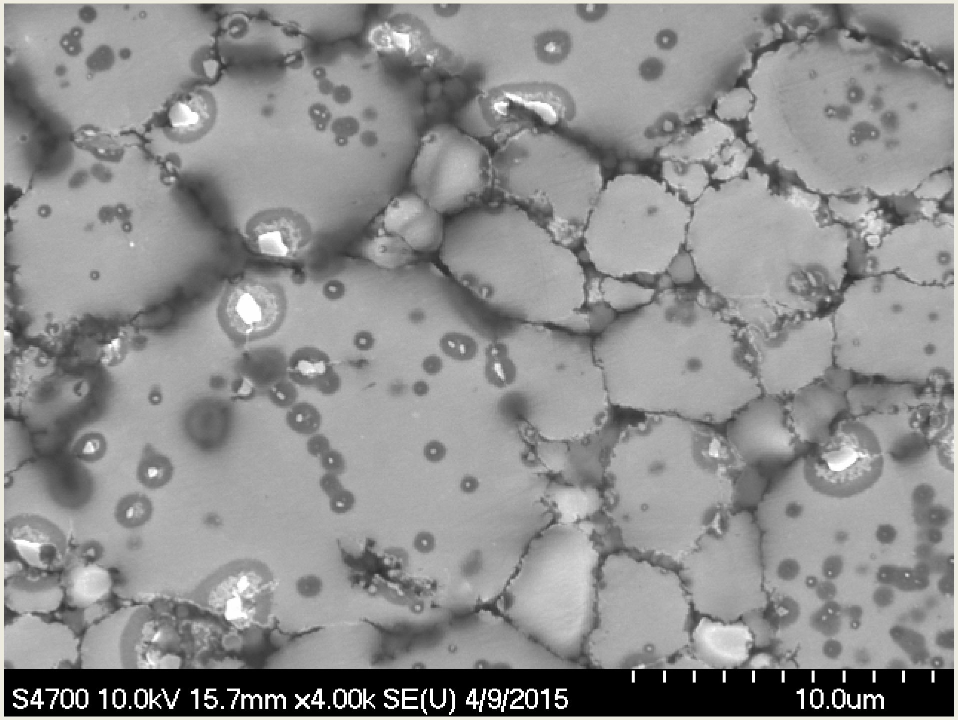
10 ksi CIP + sinter polished surfaces (1000x)

Linking processing to microstructure

No electric field



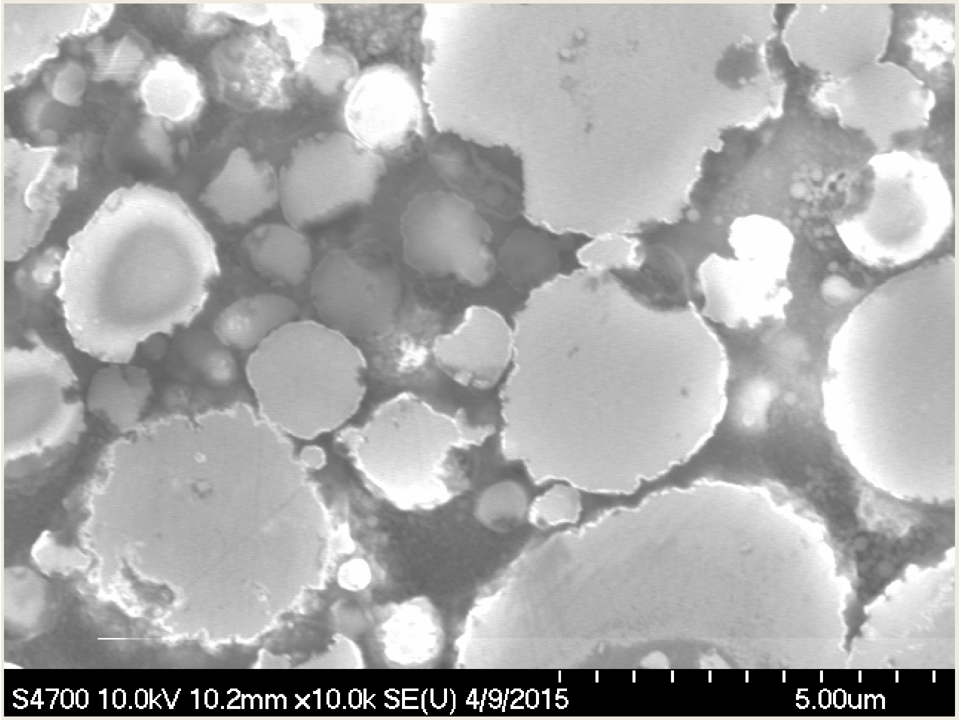
28V/cm, 3A (sample edge)



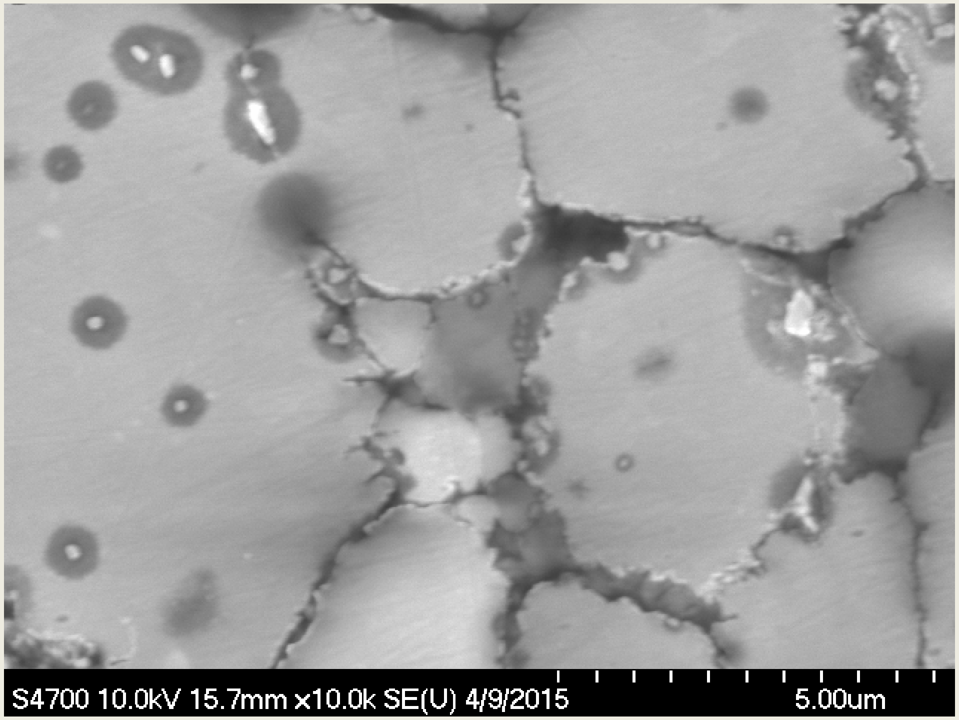
10 ksi CIP + sinter polished surf (4000x)

Linking processing to microstructure

No electric field



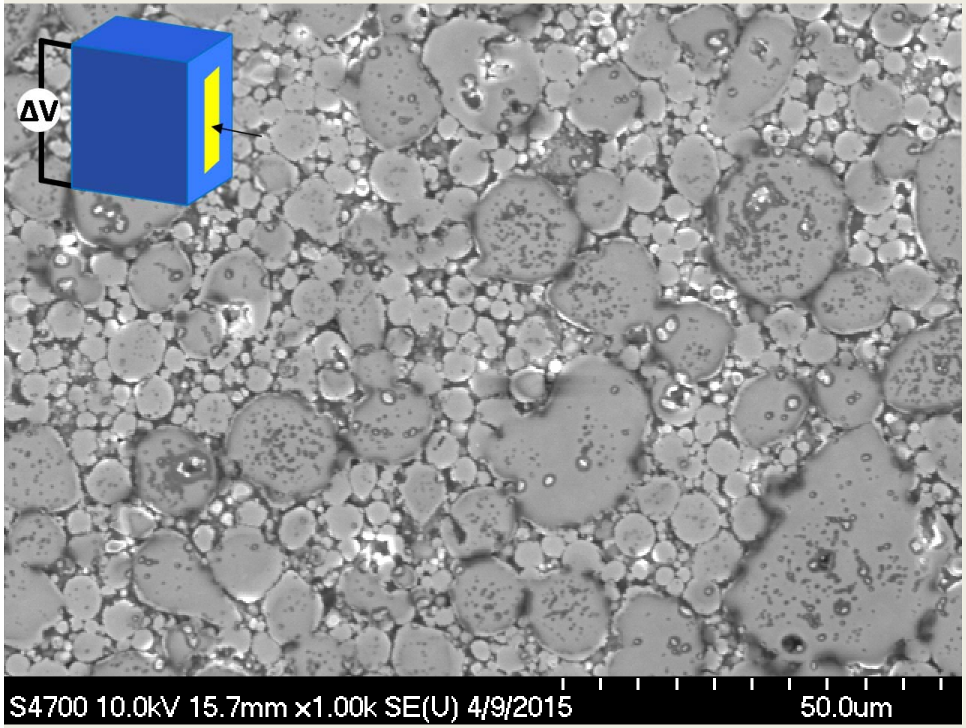
28V/cm, 3A (sample edge)



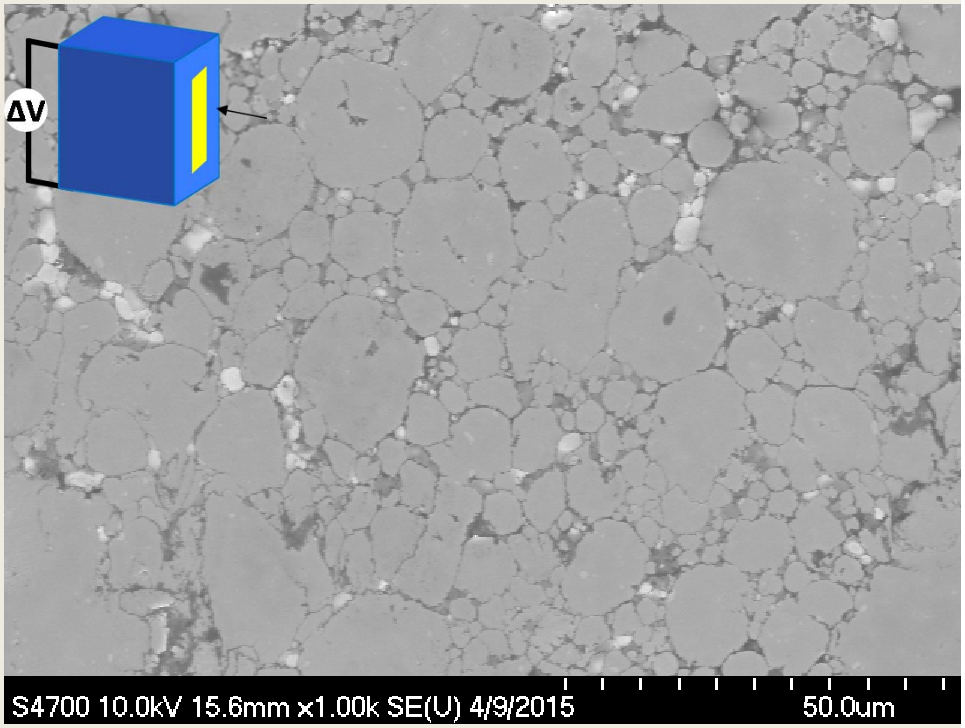
10 ksi CIP + sinter polished surf (10000x)

Observations on sample heterogeneity

Specimen center



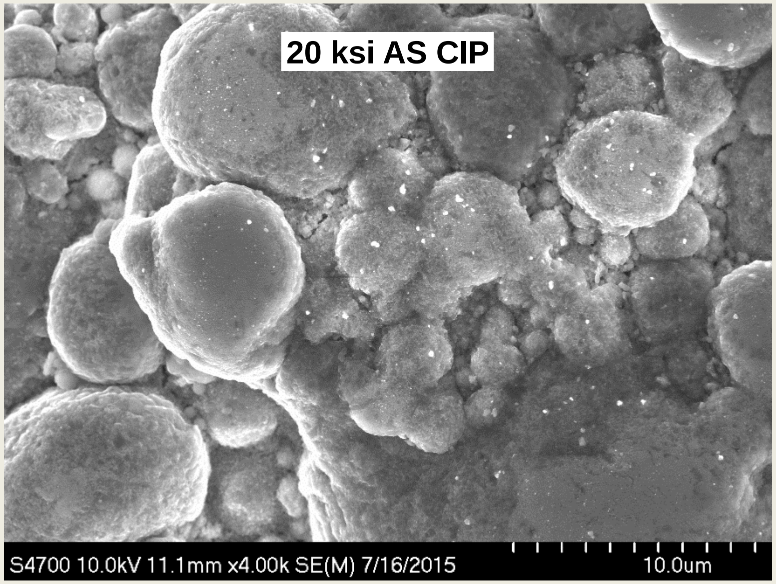
Specimen edge

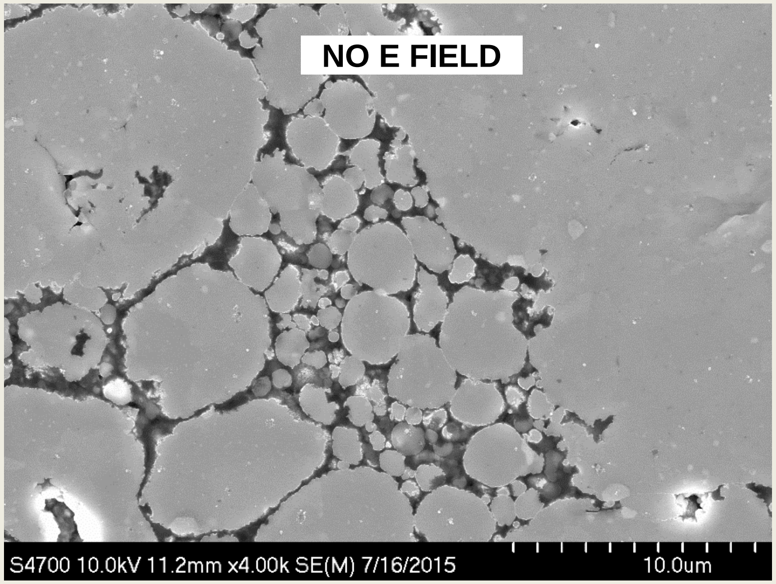


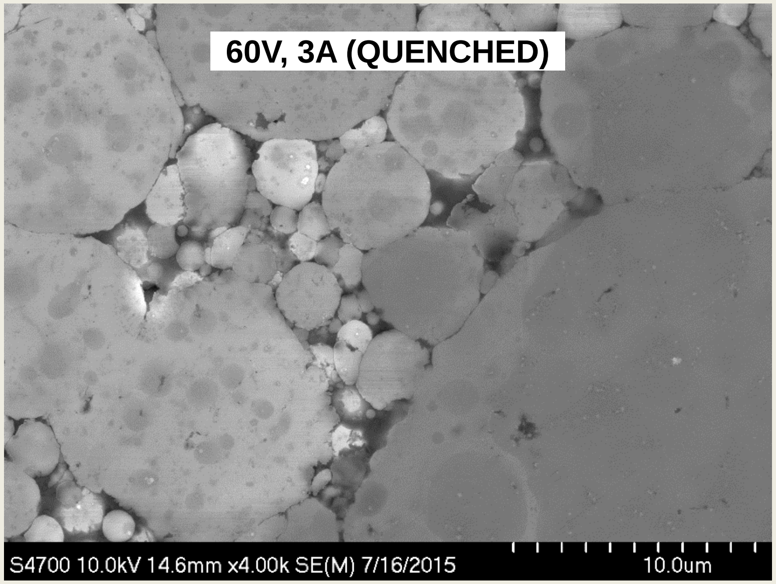
"Edge" = denser

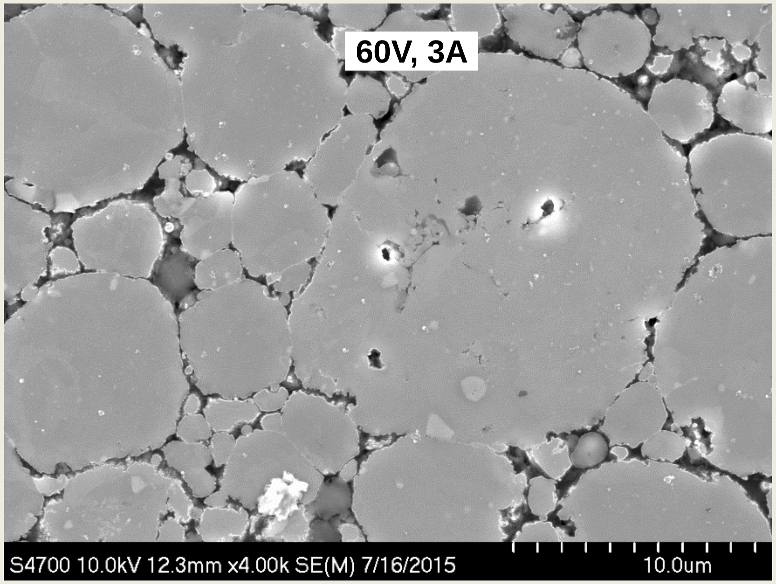
10 ksi CIP + sinter (28V/cm), 3A) polished surf

Quenching after flash event

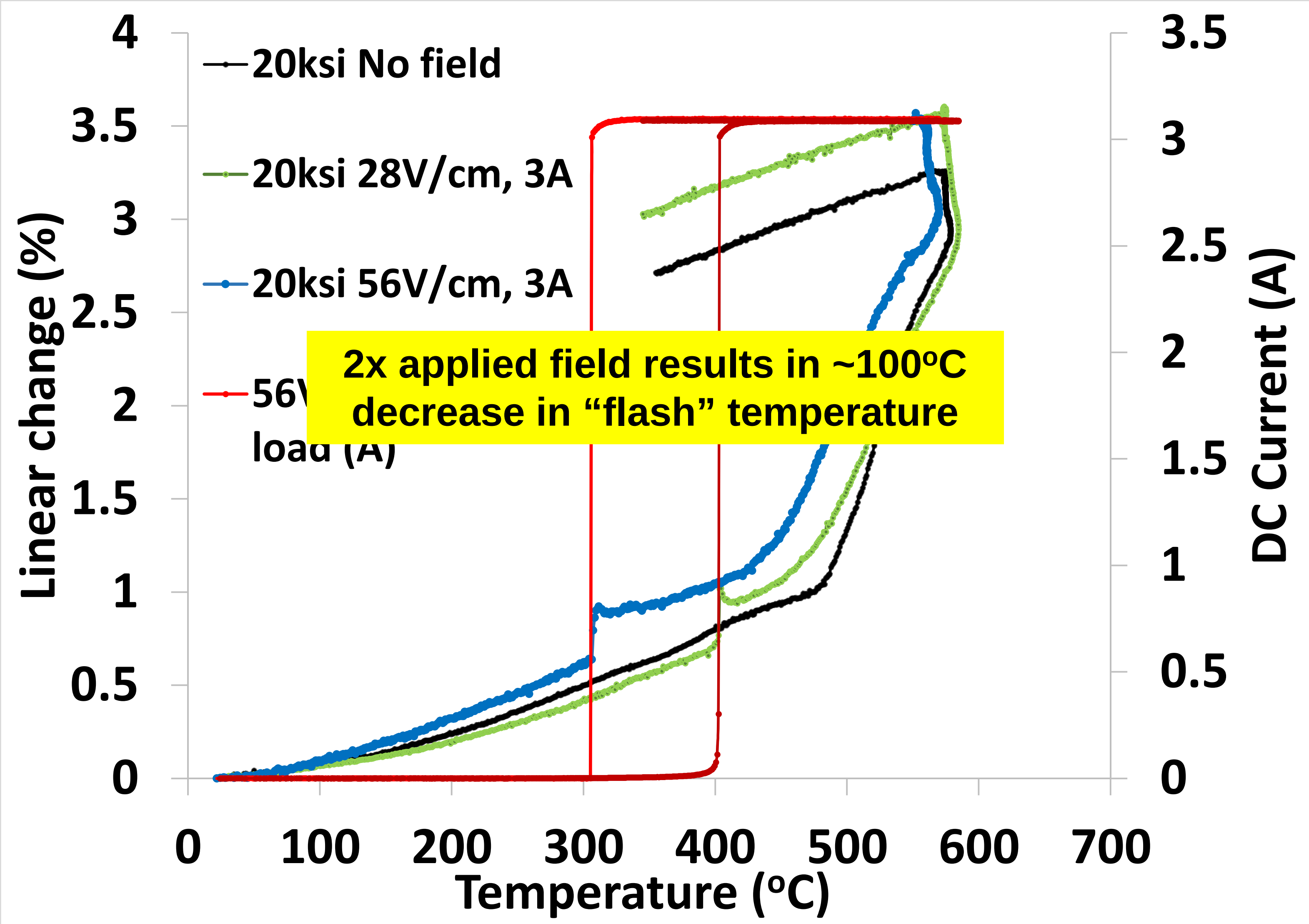






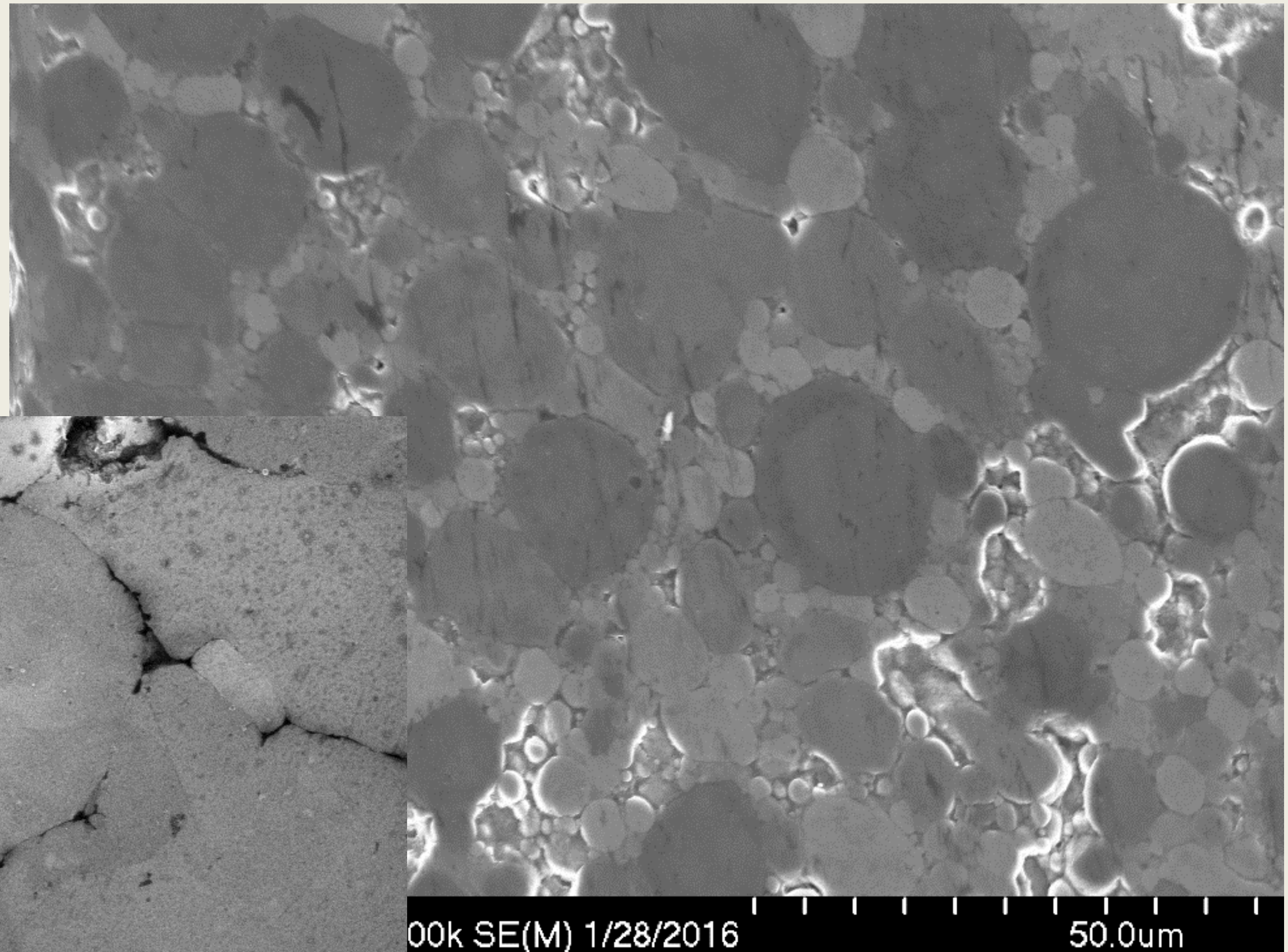


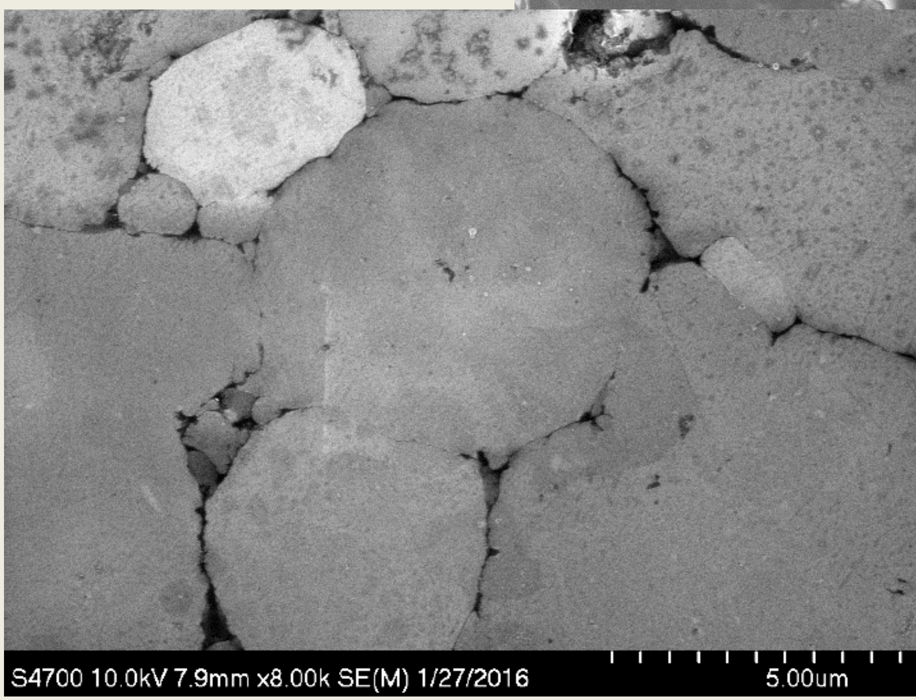
Effect of field processing parameters on sintering kinetics



How low temperature is possible?

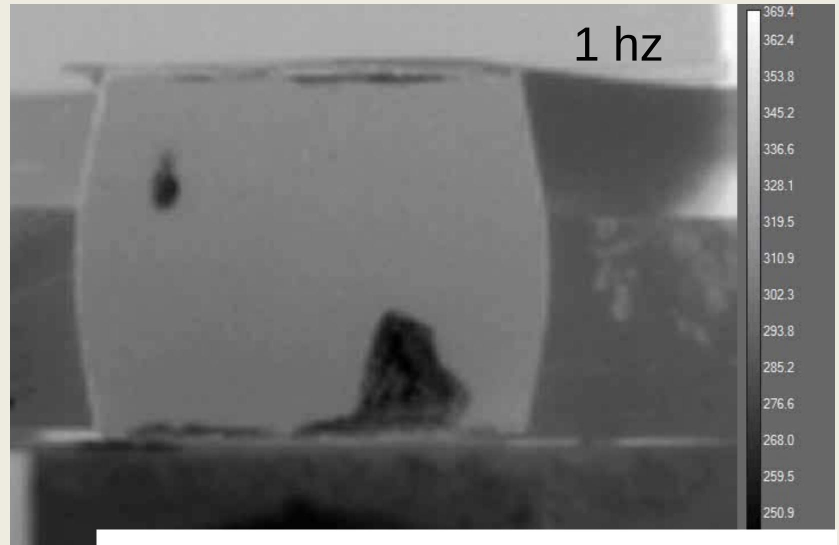
AA5083 sintered under electric field with NO additional heating (room temperature experiment)



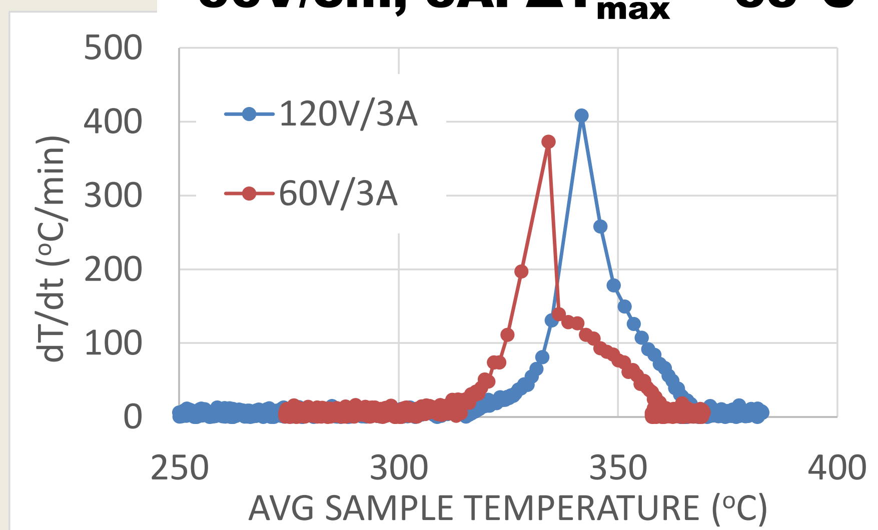


Quantifying the effect of Joule heating

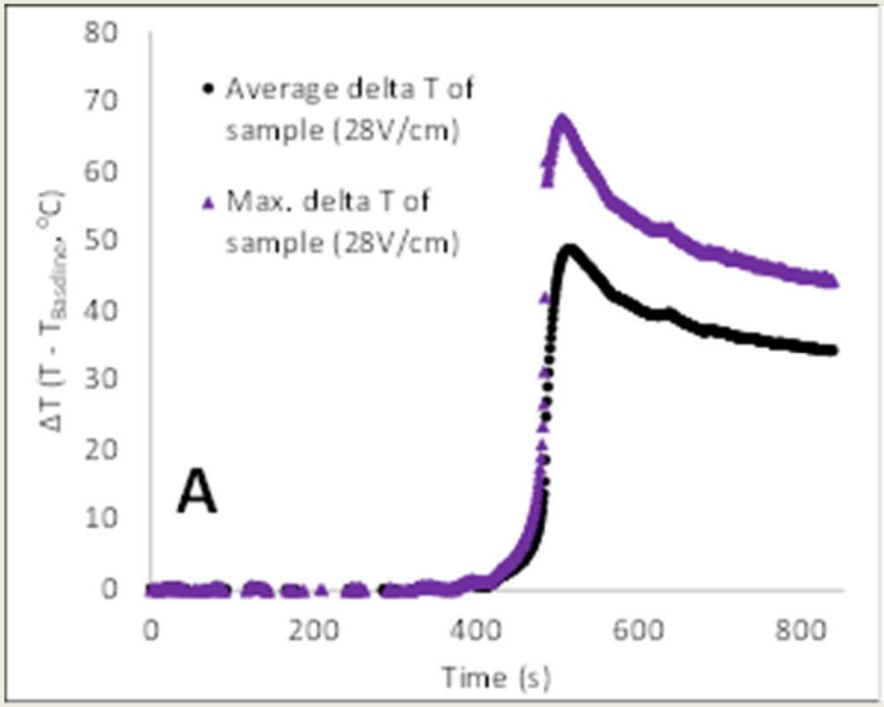
- **Run away Joule heating often cited in literature to explain "flash" in ceramics**
- **Could "field effect" be resistive Joule heating in the sample resulting in a higher sintering temperature than the non-field sample?**
  - **Sample non-conductive prior to flash**
- **Local temperatures at particle contacts could be much higher than bulk but would expect to see evidence of melting**
- **IR camera for full field temperature measurements during "flash"**

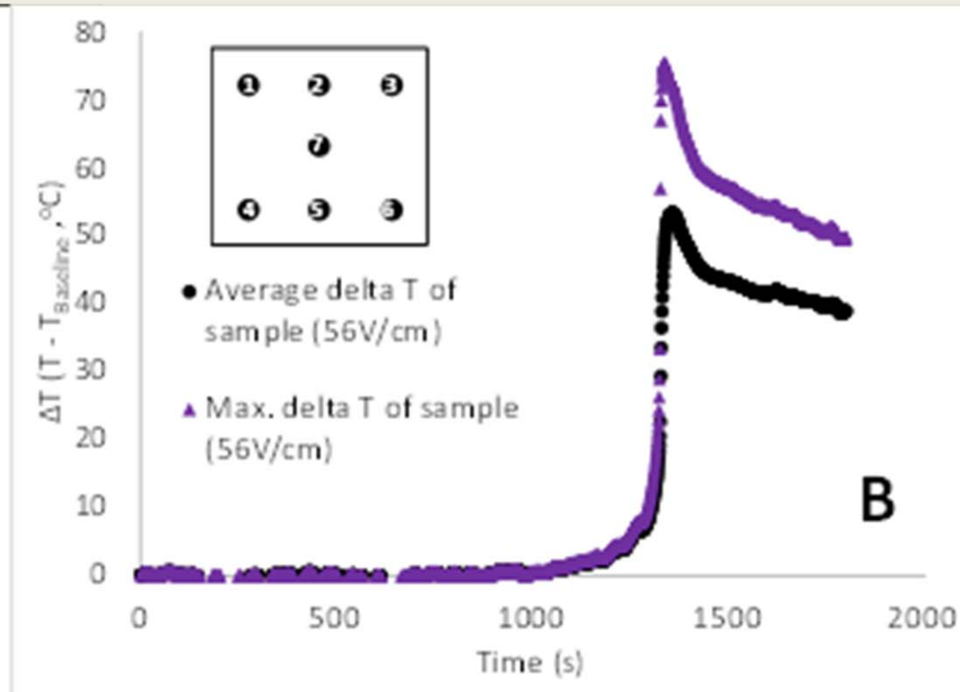


28V/cm, 3A: ΔT_{max} = 49°C
56V/cm, 3A: ΔT_{max} = 53°C



ΔT of sample during flash

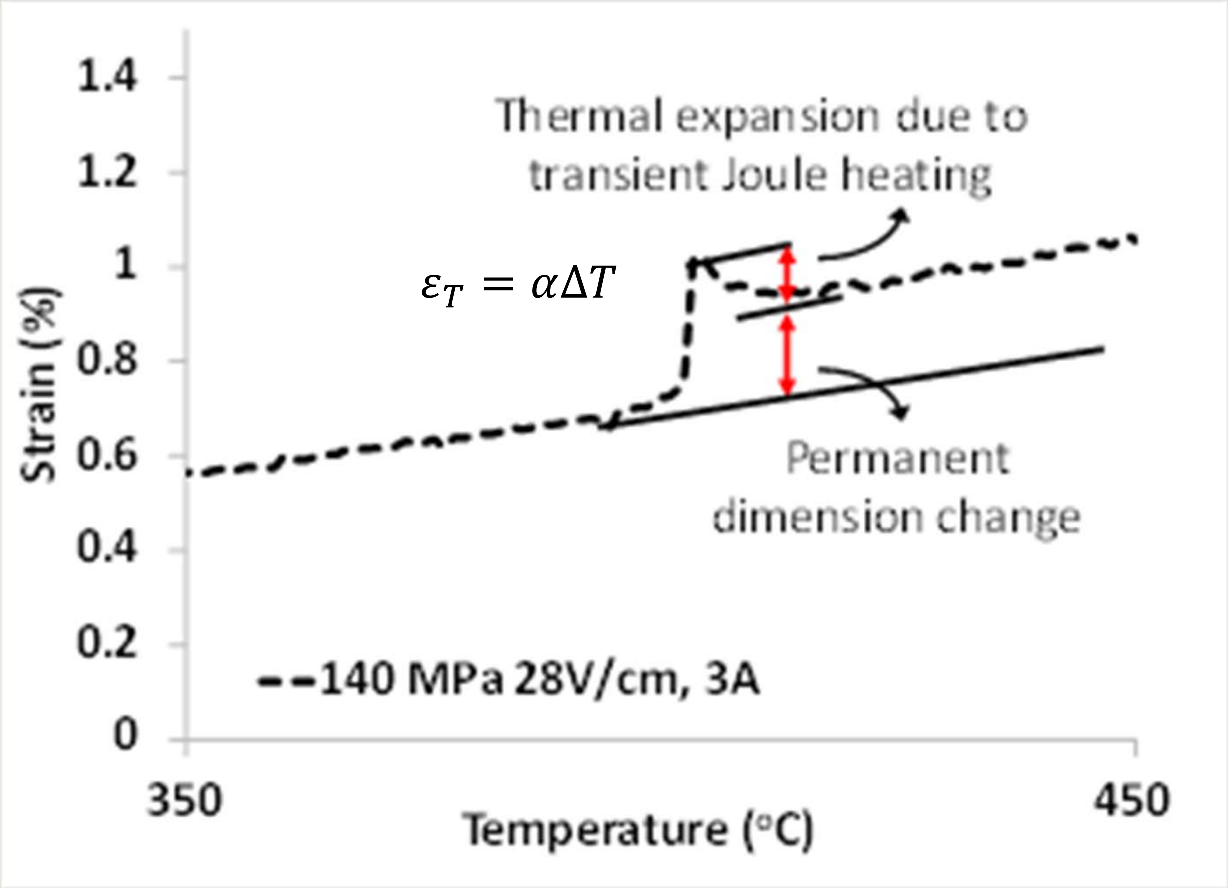




Max. observed ΔT = ~75ºC
T = ~475 ºC

Experiment is current limited so Joule heating is about the same regardless of initial applied field strength

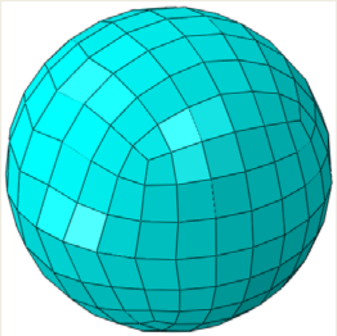
Quantification of thermal strain

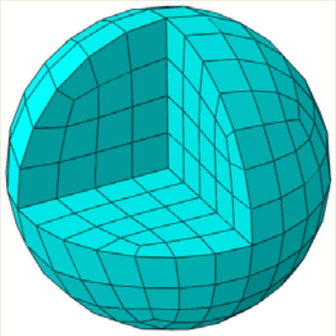


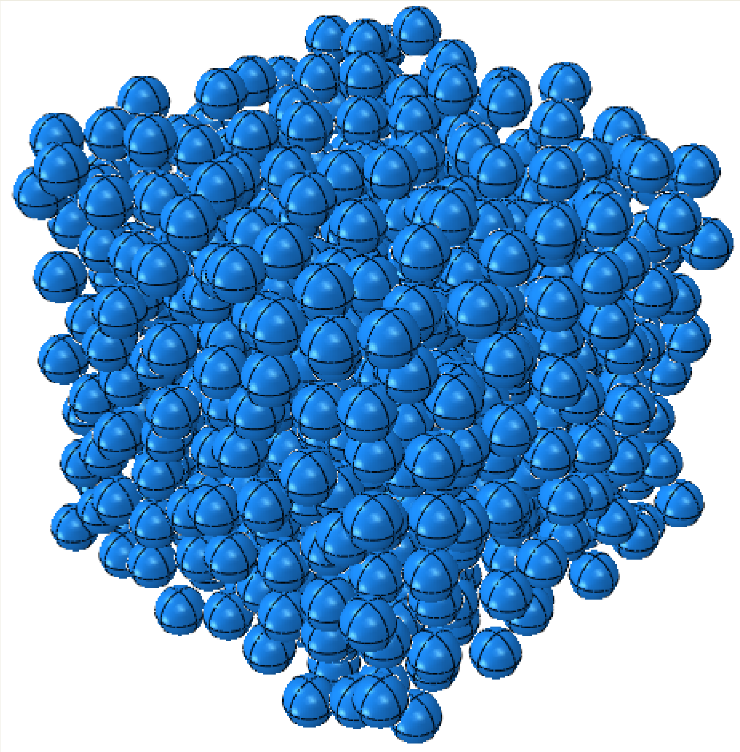
Discrete particle modeling

How do local fields and current densities develop?

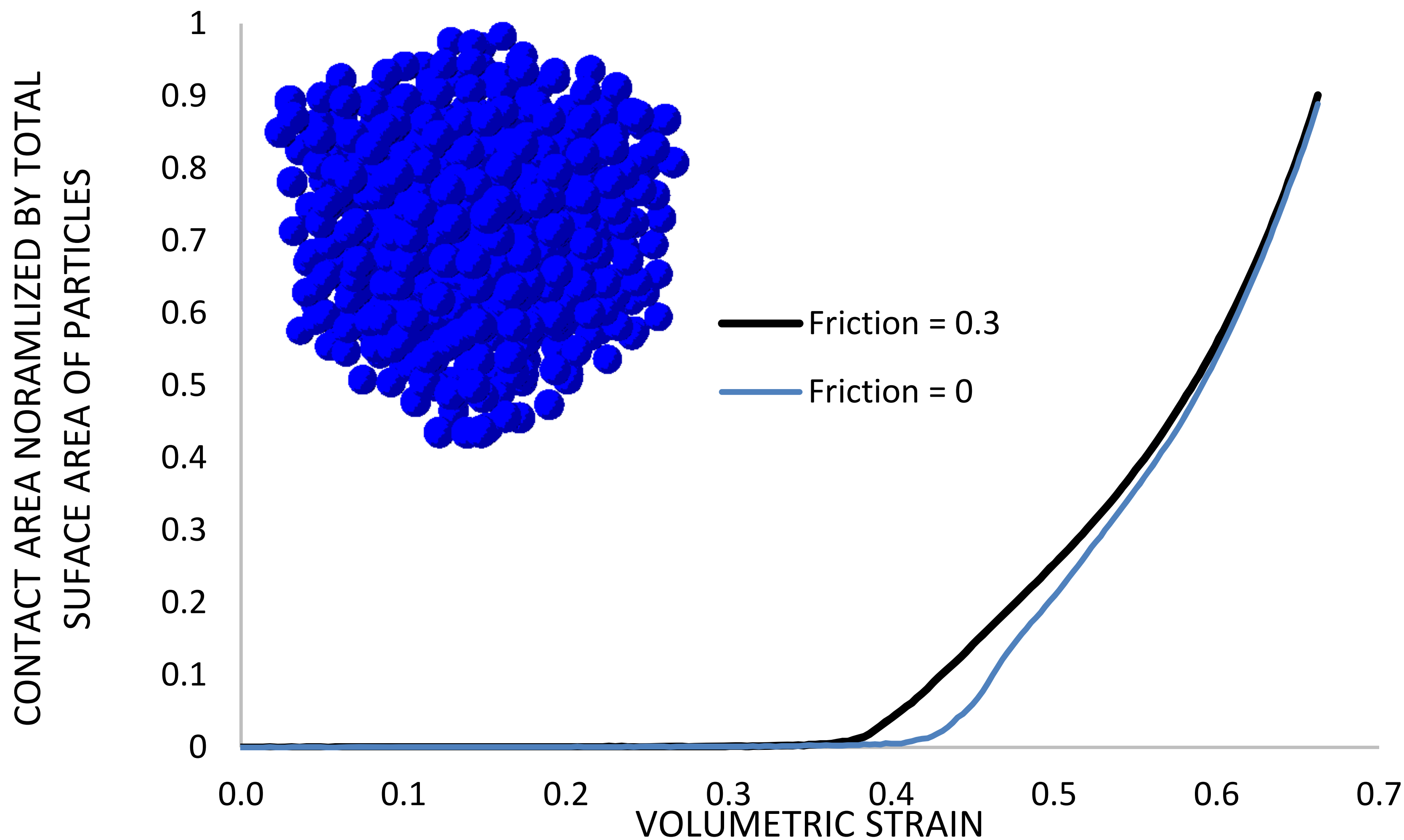
- **Cold Isostatic Press simulation to generate starting microstructure**
  - **450 um box**
  - **Particle D = 45 um**
  - **Initial packing density = 0.32**
  - **612 particles**
- **Thermal-electric simulation to determine effective conductivity and local gradients**







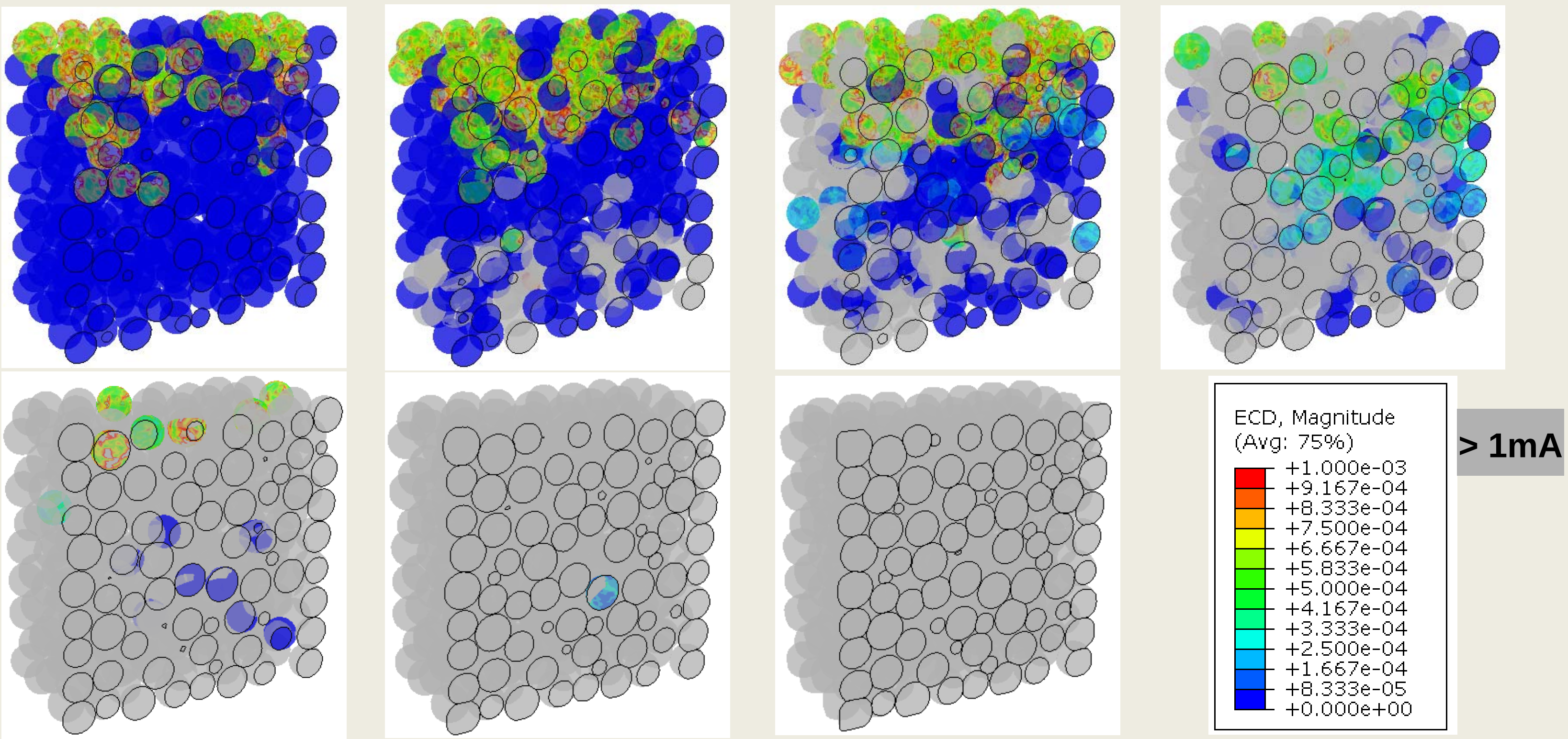
CIP simulation results – evolution of contact area



Thermal-electric model results

Evolution of conduction paths – 50% transparency, 1mA contour limit

0 MPa, vf = 0.44 | 0.017 MPa, vf = 0.45 | 0.024 MPa, vf = 0.47 | 0.03 MPa, vf = 0.51



3.6 Mpa, vf = 0.54 | 35 Mpa, vf = 0.63 | 75 MPa, vf = 0.71

Huge increase in conductivity with small evolution of microstructure

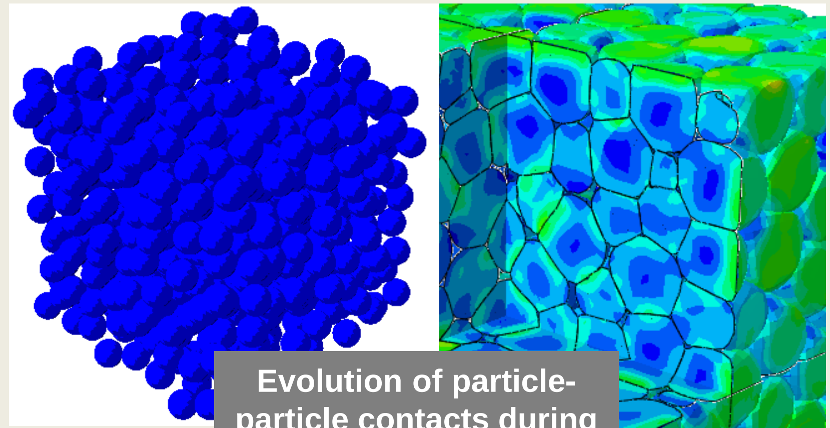
Conclusions

- "Flash" sintering phenomena demonstrated in metallic powders
- Field plays a strong role in sintering and diffusion kinetics
- Rapid and permanent microstructure change during "flash"
- Field strength plays a strong role
- Joule heating contributes but, alone, cannot account for flash phenomena and enhanced sintering kinetics
  - Also cannot explain effect of field strength on flash temperature

- Questions...
  - Oxide/dielectric breakdown?
  - Space charge depletion layers between particles?
  - Electromigration?
  - …?

Ongoing and future work

- Quantification of activation energy for “flash” sintering of A5083
- Modeling
  - Micromechanical to understand current pathways and local fields
  - Continuum scale modeling of sintering including field enhanced kinetics
- Experimental facilities being upgraded
  - 1200^{o}C vacuum furnace (4” tube)
  - 30kV power supply
  - *In situ* heating stage for SEM



Evolution of particle-particle contacts during cold isostatic pressing

Acknowledgements

Dr. Anit Giri

Jim Catalano

Dennis Miller

Frank Kellogg

Clara Hofmeister

Dr. Tim Walter

Dr. Scott Walck

Thank you!