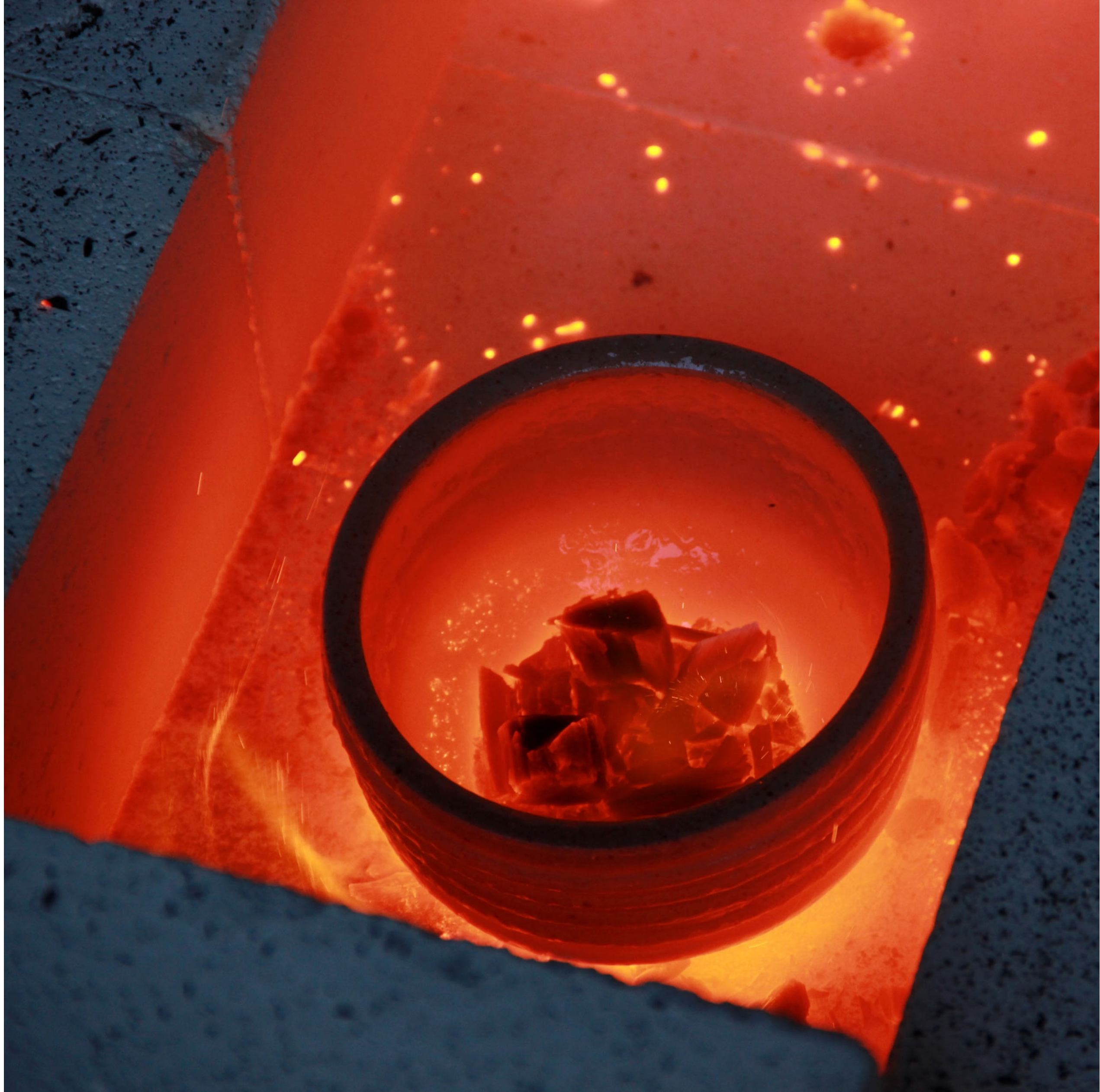
Advanced Ceramic Technologies

Rapid and pressure sintering

M. Stuer



Outline

What's on the
- MENU -
today ?

- General aspects regarding rapid sintering
- Rapid sintering processes
  - Flash sintering
  - Ultrafast high-temperature sintering
- Pressure assisted sintering
  - Spark plasma sintering
  - Stress exponent measurement

Learning objectives:

- Learn about established and emerging rapid sintering techniques
- Understand the mechanistical complexity of selected rapid sintering techniques
- Be able to determine stress exponents in pressure sintering

Rapid sintering

M. Stuer

Conventional and “novel” sintering techniques

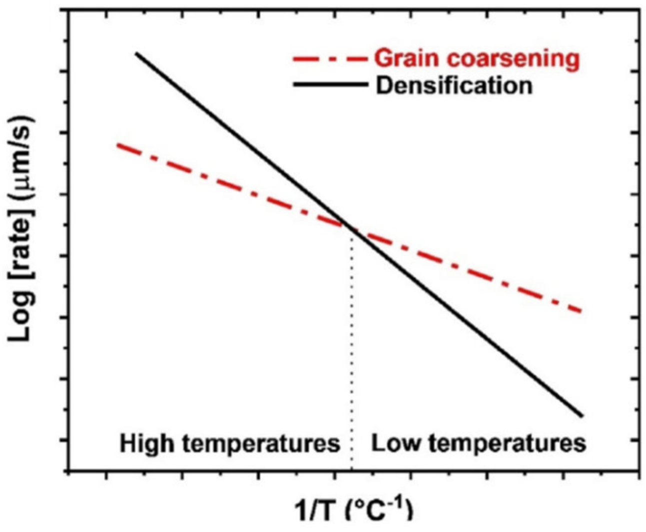
Method	Heading rate [K/min]	Typical sintering time	Grain growth	Special features
Conventional sintering	5-10	Hours	Significant	High energy consumption, long processing times
Spark Plasma Sintering (SPS)	100-1’000	Minutes	Limited	Uses DC (or AC) pulses **and pressure** to accelerate densification
Ultra-fast High-temperature Sintering (UHS)	>1’000	Seconds to Minutes	Minimal	Maintains nanostructures, very high heating rates
Flash sintering	>1’000+	Seconds	Minimal	Extremely high densification rates, maintains nanostructures

- **Fast heating rates** are at the core of rapid sintering techniques

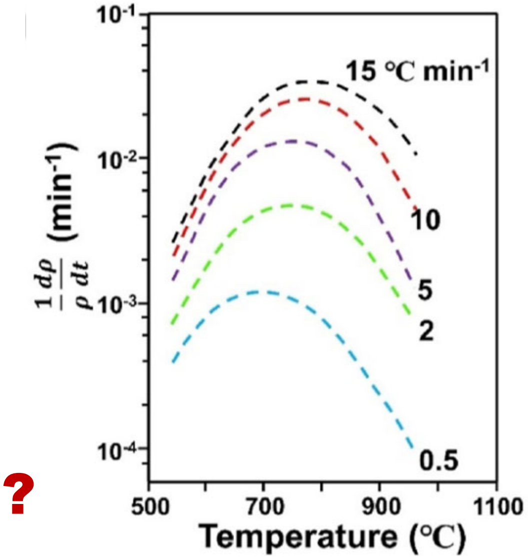
Role of rapid heating in densification

Faster heating:

- Allows quicker activation of densifying mechanisms, reducing the time with only non-densifying grain coarsening mechanisms activated (there are suspected exceptions, see table)



- Increases the peak densification rate during sintering, reducing the sintering times



➔ **Why does fast heating increase sintering rate?**

Activation energies for coarsening (Q_c) and densification (Q_d)

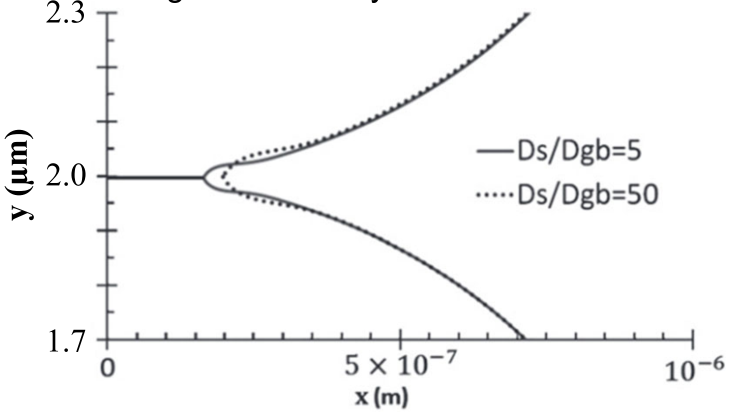
Material	Q_c [kJ.mol^{-1}]	Q_d [kJ.mol^{-1}]
ZnO	20	268-296
Al_2O_3	230-280	580
3YSZ	478-531	615-650
MgO	150-500	360-450

source: 10.1016/j.ceramint.2022.11.091

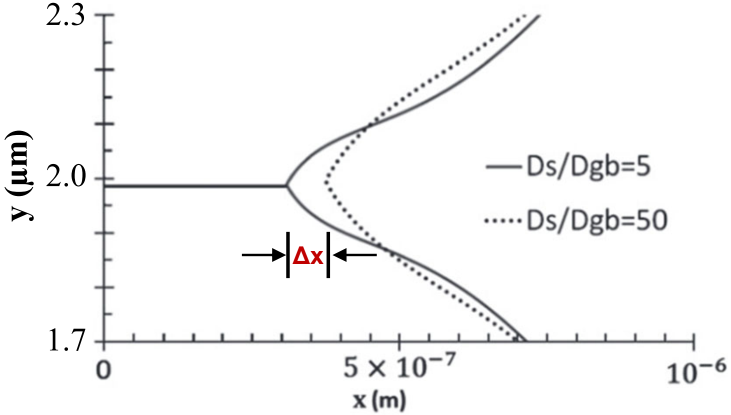
Mechanisms behind increased sintering rate upon fast heating

- With **increasing heating rates**, surface and vapor diffusion do not have enough time to redistribute mass along the pore surface
  ➔ **pore shape increasingly out off equilibrium**
- Following **Laplace**'s, an increase of the pore curvature at the neck (i.e. pore tip) increases the local **vacancy concentration**
- Following **Fick**'s law, the increased vacancy gradient and reduced diffusion length increases the diffusion flux
  ➔ **shrinkage increases as a function of X/a**

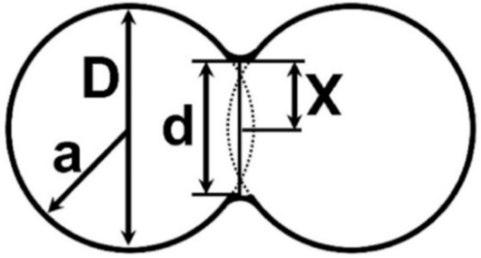
τ_g: time for the neck ***X*** to reach ***a*** by grain-boundary diffusion.

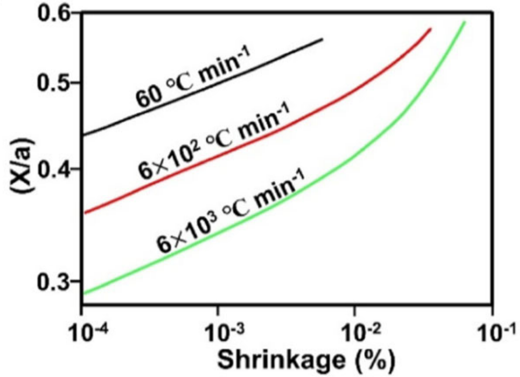


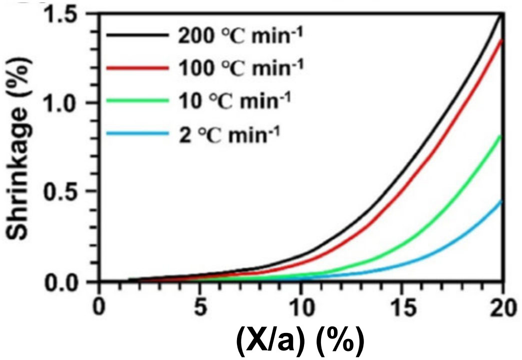
Graph at $t_1 = t/\tau_g = 10^{-7}$



Graph at $t_2 = t/\tau_g = 10^{-5}$







Rapid sintering

M. Stuer

30/04/2025

source: 10.1016/j.ceramint.2022.11.091

Rapid sintering
FLASH SINTERING

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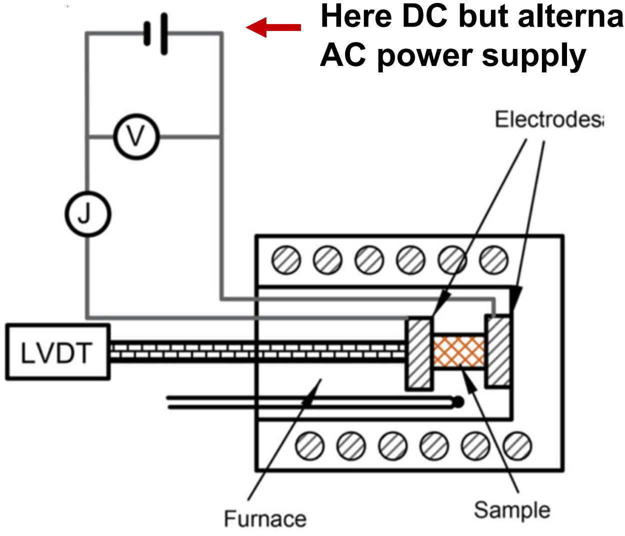
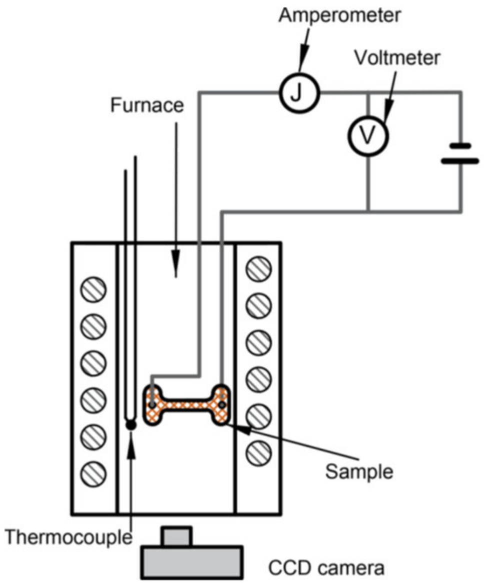
Flash sintering basics

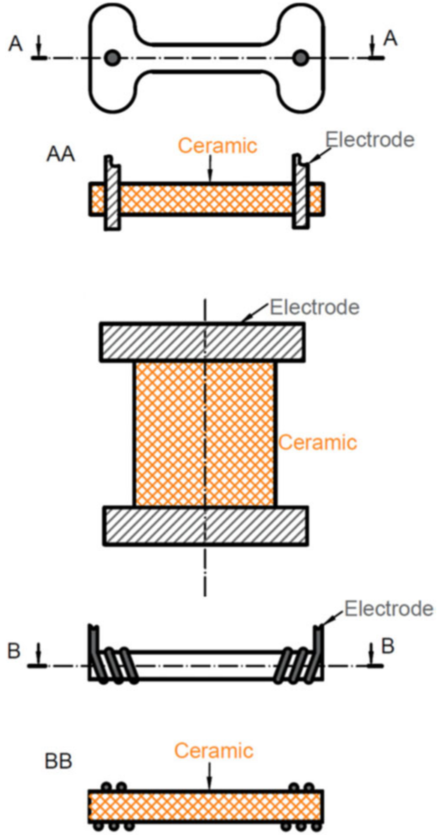
- **Flash sintering:** Rapid densification (seconds timescale) under a specific combination of high electric field (> 60 V/cm) and temperature
- During "flash sintering" three events occur simultaneously:
  - Rapid temperature rise (e.g. thermal runaway) due to Joule heating
  - Material resistivity drop
  - Emission of a light flash ➔ giving the name "Flash sintering"



Here DC but alternatively AC power supply



Typical sample/electrode geometries

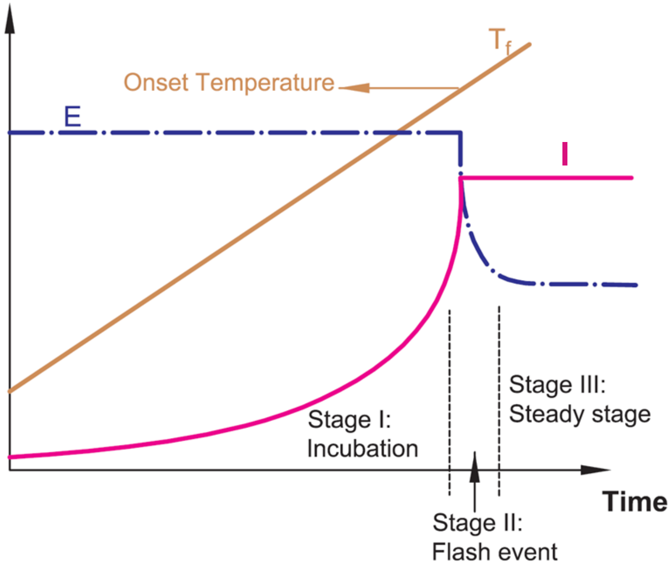


source: 10.1016/j.jeurceramsoc.2018.08.048

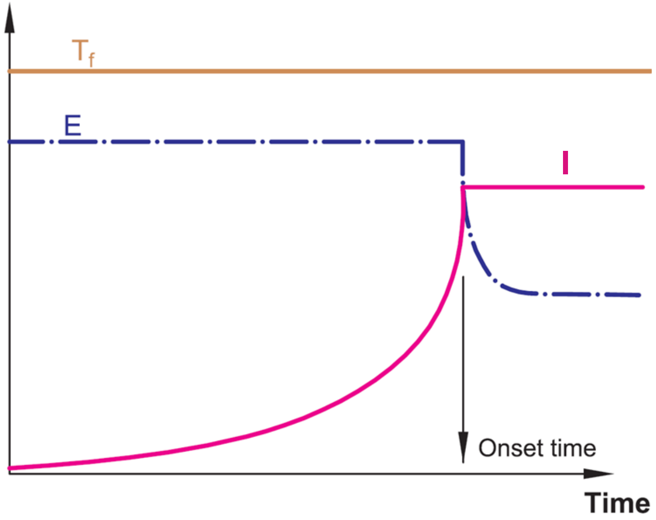
Flash sintering stages and initiation

- **Flash sintering occurs in 3 stages:** Stage I: Incubation,
  Stage II: Flash event
  Stage III: Steady state
- Different electric field (i.e. applied voltage), time and furnace temperature configurations can be used to induce the flash event

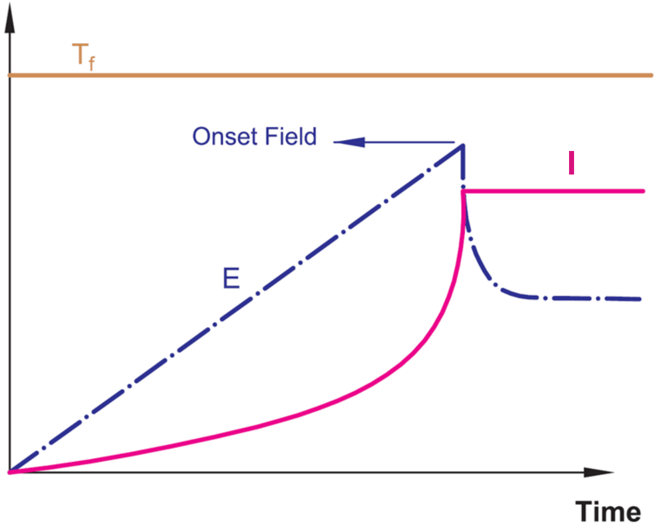
Variable furnace temperature T_f



Variable incubation time



Variable electric field



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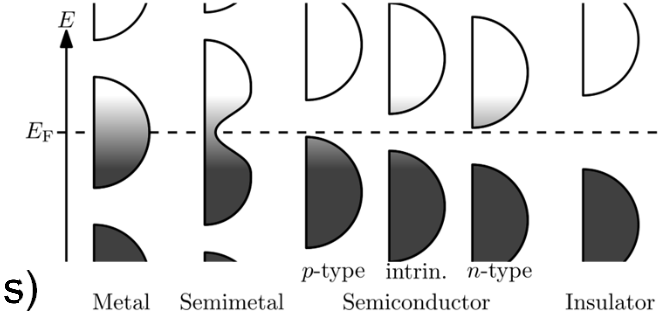
Flash sintering requirements

Flash sintering only occurs for materials with **negative temperature coefficient of resistivity** ➔ **conductivity increases with T**

Metals

- **High population of free electrons**
- Atom vibration increases with temperature (e.g. phonons)
  ➔ Scattering of electrons by phonons increases
- Resistivity of increases with temperature
  ➔ positive temperature coefficient of resistivity

➔ **Not compatible with flash sintering**



Source: Wikipedia

Non-metals

- **No/low population of free electrons**
- Charge carrier concentration increases with temperature
  ➔ Conductivity increases
- Conductivity can be approximated with an Arrhenius-type behavior:

$$\sigma = \sigma_0 \exp\left(\frac{-E_a}{k_B T_s}\right)$$

- E_a: activation energy for conduction
- k_B: Boltzmann constant
- T_s: sample temperature
- σ_0: pre-exponential conductivity constant

- Resistivity: $\rho = \frac{1}{\sigma} = \rho_0 \exp\left(\frac{E_a}{k_B T_s}\right)$

source: 10.1016/j.jeurceramsoc.2018.08.048

Power dissipation involved in flash sintering

Joule heating (e.g. dissipation of electrical power)

- ♦ Electrical power input into sample from power supply

$$W = I * V \overset{V=R*I}{=} I^2 * R = \frac{V^2}{R}$$

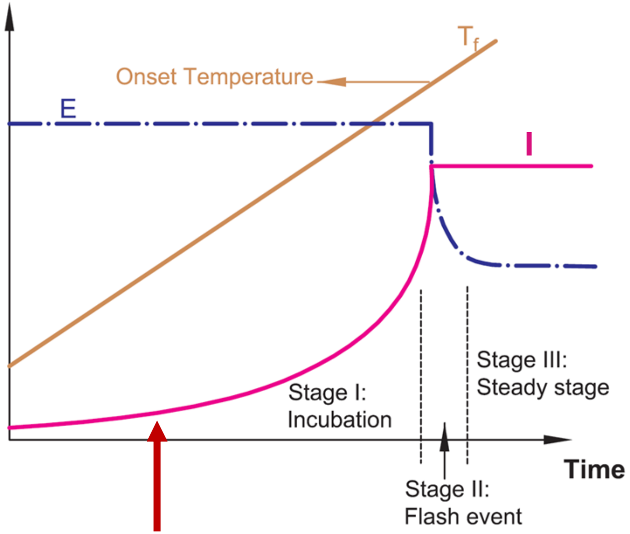
- V: voltage
- I: current
- R: resistance

With: Resistance proportional to resistivity ρ: $R = \rho \frac{d}{A}$

Applied voltage proportional to electric field E: $V = E \cdot d$

- d: sample length
- A: sample cross section area

$$\Rightarrow W_{in} = \frac{V^2}{R} = \frac{E^2 d^2}{\rho_0 \exp\left(\frac{E_a}{k_B T_S}\right)\frac{d}{A}} = \frac{A d E^2}{\rho_0} \exp\left(-\frac{E_a}{k_B T_S}\right)$$



Non-linear current increase due to positive electrical-thermal feedback loop (e.g. negative temperature coefficient of resistivity coupled with Joule heating)

Radiative power loss (e.g. grey body) results in material cooling:

- ♦ Stefan-Boltzmann law:

$$W_{out} = S\sigma\varepsilon\left(T_S^4 - T_f^4\right)$$

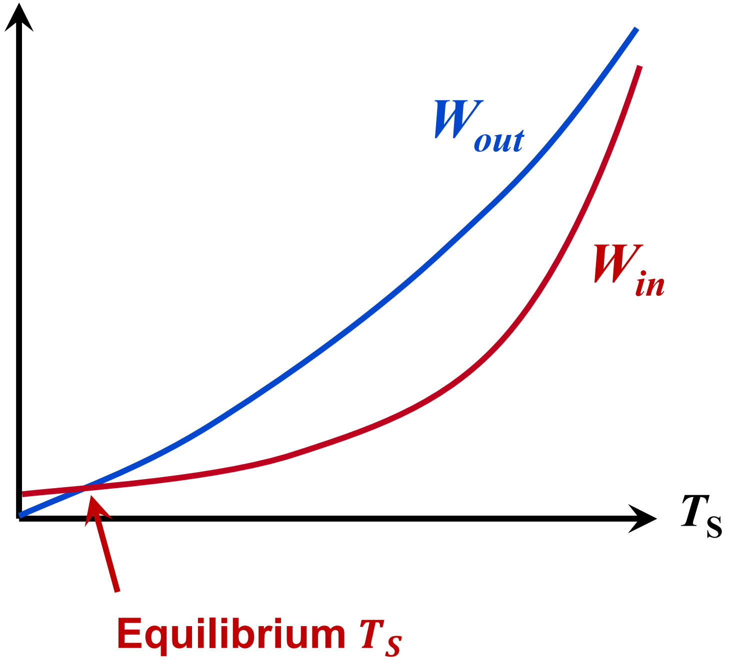
- S: sample surface area
- σ: Stefan-Boltzmann constant
- ε: emissivity
- T_f: furnace temperature

source: 10.1016/j.jeurceramsoc.2018.08.048

Stage I: Power dissipation equilibrium

- W_{in} and W_{out} have different temperature dependences
  - $W_{in} = \frac{AdE^2}{\rho_0} \exp\left(-\frac{E_a}{k_B T_S}\right)$
  - $W_{out} = S\sigma\varepsilon\left(T_S^4 - T_f^4\right)$
- As long as W_{out} exceeds W_{in} as a function of T_s, there is an equilibrium T_s where $W_{in} = W_{out}$
- As $\boldsymbol{T_f}$, $\boldsymbol{E}$, or time increase the equilibrium T_s shifts (because $W_{in}(E, T_s)$ and $W_{out}(T_s, T_f)$ change)

Illustration of equilibrium sample temperature T_S at a given furnace temperature T_f and electric field E



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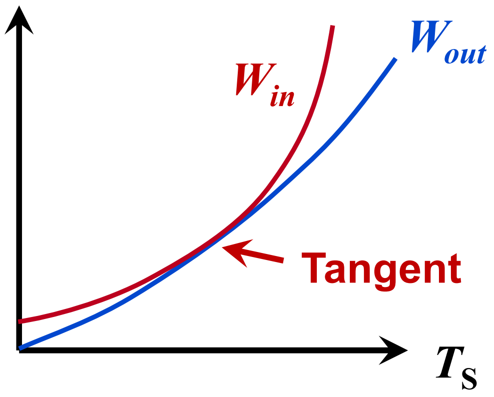
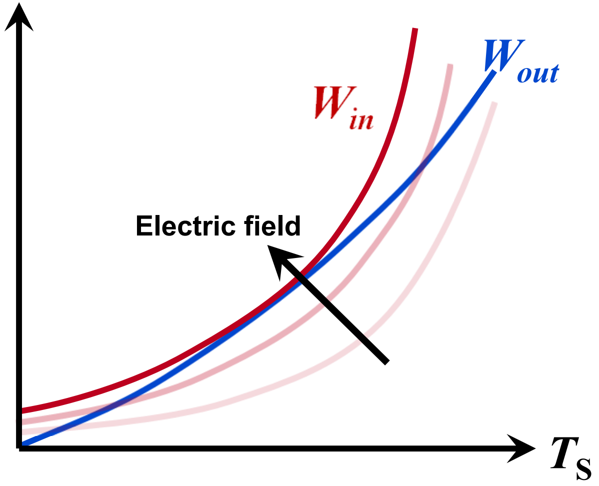
Transition from stage I to stage II



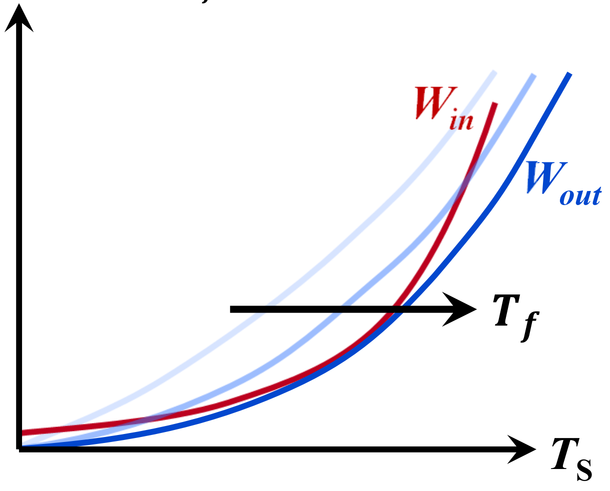
- Thermal runaway occurs when W_{in} and W_{out} meet at a tangent condition with $W_{in} \geq W_{out}$
  - ➔ Occurrence of **flash event**
  - ➔ Transition from Stage I to Stage II
- Influence of T_f and E on transition occurrence:

Assuming E independently adjustable



Increasing E facilitates flash event (transition at lower T_f)

Assuming T_f independently adjustable



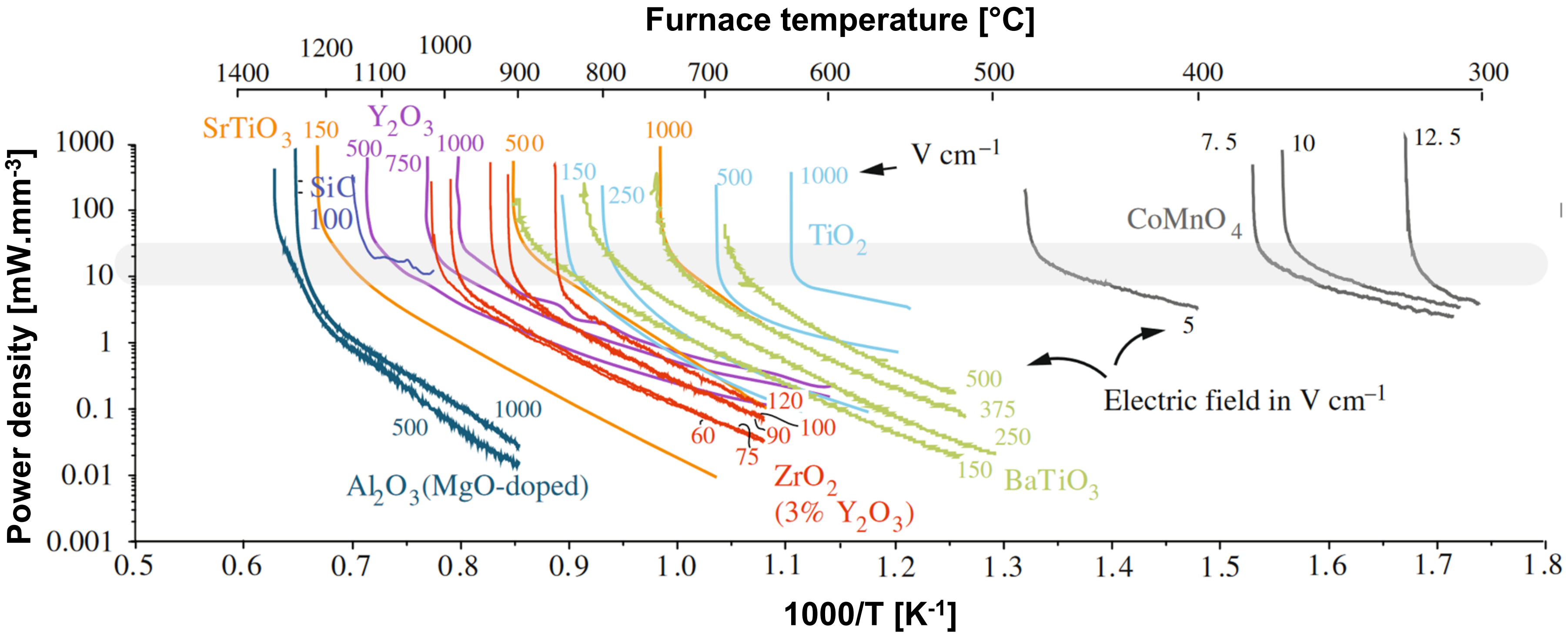
Increasing T_f facilitates flash event (transition at lower E)

Note:
More conductive materials also flash sinter at lower T_f

source: 10.1016/j.jeurceramsoc.2018.08.048

Transition from stage I to stage II

Arrhenius plots of power density for several materials flashed at different values of the applied electric field



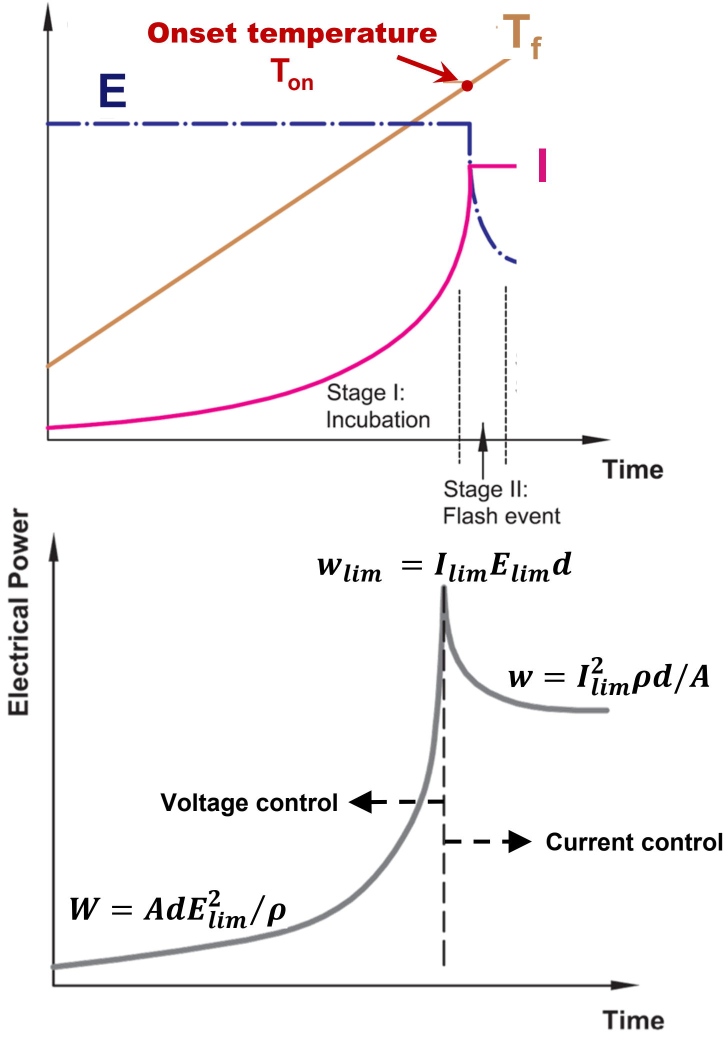
- ♦ **Onset of flash event** appears to occur in a **narrow power density band** irrespective of the applied electric field of oxide ceramic!

source: 10.1557/s43577-020-00011-1

Stage II: Thermal Runaway and Flash Event

At tangency condition ($W_{in} \geq W_{out}$), the positive electrical-thermal feedback loop leads to a **thermal runaway**:

- Rapid resistivity drop, forces the electrical power supply to **move from voltage to current control mode** (e.g. no infinite current source)
  - ➔ Switch from voltage- to current-controlled process
- Sharp **electrical power peak** leads to the emission of a flash light (electroluminescence, thermoluminescence, … ?)
  - ➔ Power supply has internal capacity, this is rapidly released during mode transition such that real electrical power peak $\boldsymbol{w_{lim}}$ should be higher than the theoretical $\boldsymbol{W_{lim} = I_{lim} E_{lim} d}$
- **Material heats very rapidly** (up to 10’000K/min) and sinters



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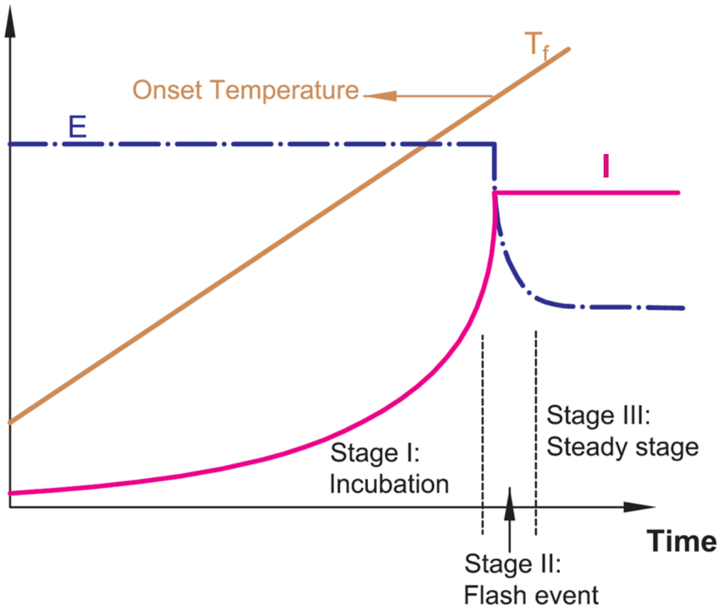
Flash sintering

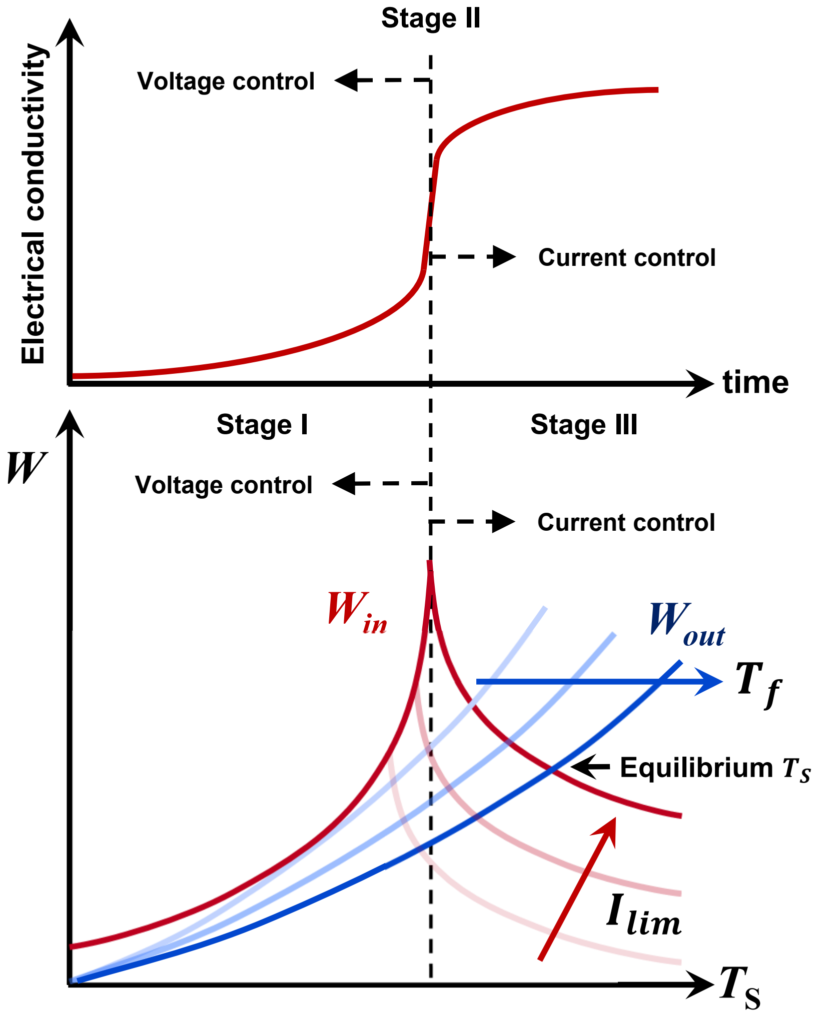
Stage III: Steady-state

- In current control mode

$$W_{in} = \frac{d}{A} I^2 \rho_0 \exp\left(\frac{E_a}{k_B T_S}\right)$$

- Conductivity reaches plateau
- T_s reaches new equilibrium where $W_{in} = W_{out}$





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Why is flash sintering so rapid? Some considerations

No agreement yet on full **mechanistic description** of flash sintering

- Suspected role of **electroplasticity**
  - Electric field application reduces yield for plastic flow at high temperature
  - Following Laplace law, sintering stress σ_{sint} can be approximates as:

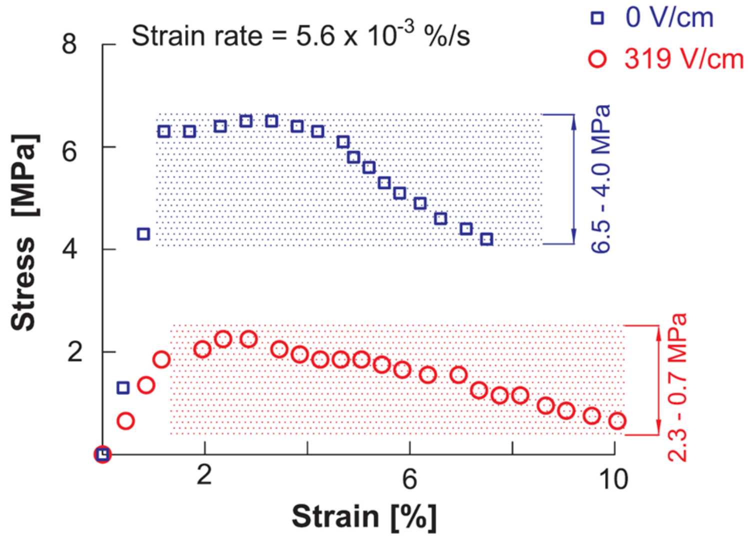
$$\sigma_{sint} = \frac{4\gamma_s}{d_P}$$

- γ_s: surface tension of ceramic
- d_P: particle diameter

  - For typical values γ_s = 0.9 J/m^2 and d_P = 600 nm this gives a sintering stress σ_{sint} = 6 MPa

➔ Electroplasticity is **field driven process** that may explain **enhanced densification** without application of external stress

Stress-strain curve for alumina at 1500°C with strain rate of 5.6.10^{-3} %/s



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Why is flash sintering so rapid? Some considerations

- Suspected role of **thermal gradients**
  - Space charge formation at grain boundaries increases electrical resistivity
  - Constriction at neck (e.g. small cross section) increases current density at grain boundaries
  - Grain boundary formation releases surface enthalpy:

$$P_{gb} = \frac{2\Delta H_s - \Delta H_{gb}}{\delta_{gb}\Delta t}$$

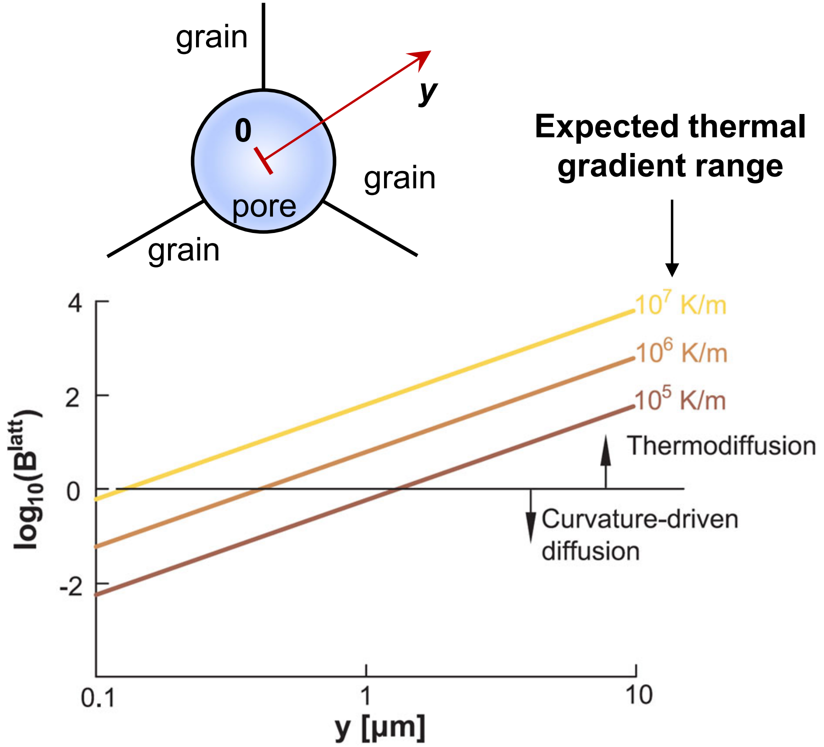
- P_{gb}: power dissipation [W/m^3]
- ΔH_s: surface enthalpy
- ΔH_{gb}: grain boundary enthalpy
- Δt: sintering time

➔ Generate **thermal gradients** between grain core and grain boundary

- Ratio between curvature- and thermal gradient-driven atomic flow towards pore B^{latt}:

$$B^{latt} = \frac{H_m y^2}{2\gamma_s \Omega}\frac{\nabla T}{T}$$

- H_m: vacancy migration enthalpy
- y: distance from pore center of curvature
- Ω: atomic volume
- ∇T: temperature gradient



➔ **Thermal gradients** may explain **enhanced mass flow** for rapid densification

source: 10.1016/j.jeurceramsoc.2018.08.048

Why is flash sintering so rapid? Some considerations

- Suspected role of **Frenkel pair defects**
  - Electric field promotes formation of lattice disorder

$$M_M^x \rightarrow M_i^{\cdot\cdot} + V_M'' \text{ (for cation with valency of 2)}$$
$$O_O^x \rightarrow O_i'' + V_O^{\cdot\cdot}$$

  - Frenkel pair defects can be ionized to generate electronic disorder and discharge lattice defects

$$M_i^{\cdot\cdot} \leftrightarrow M_i^{\cdot} + h^{\cdot} \leftrightarrow M_i + 2h^{\cdot}$$
$$O_i'' \leftrightarrow O_i' + e' \leftrightarrow O_i + 2e'$$
$$V_O^{\cdot\cdot} \leftrightarrow V_O^{\cdot} + h^{\cdot} \leftrightarrow V_O + 2h^{\cdot}$$
$$V_M'' \leftrightarrow V_M' + e' \leftrightarrow V_M + 2e'$$

- **May explain**
  - Enhanced sintering due to increased mass carrier concentration
    - ➔ Interstitial defects move towards pores
    - ➔ Vacancy defects move towards grain boundaries
  - Increase of electronic conductivity due to free electrons and holes
  - Flash emission due to recombination of electronic defects

➔ **Thermal gradients** may explain **enhanced mass flow** for rapid densification

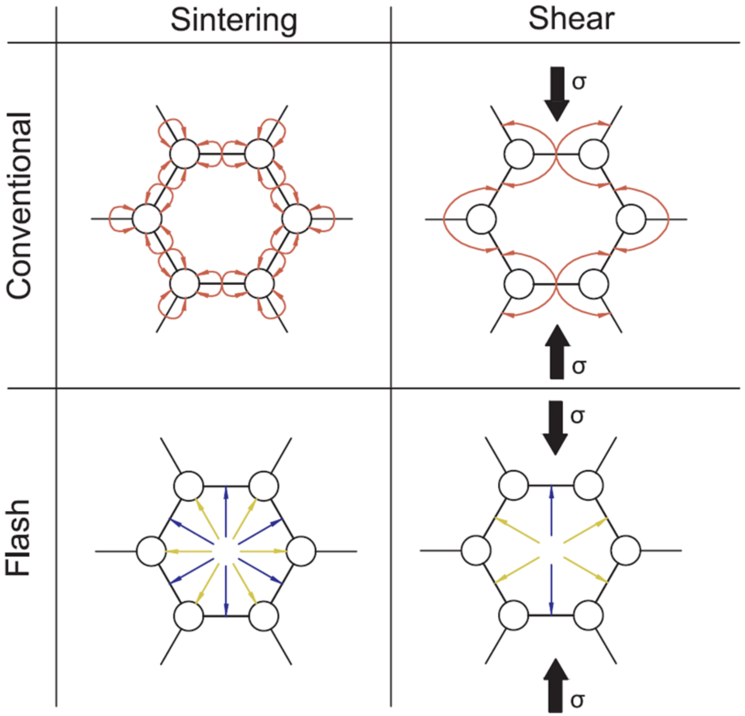


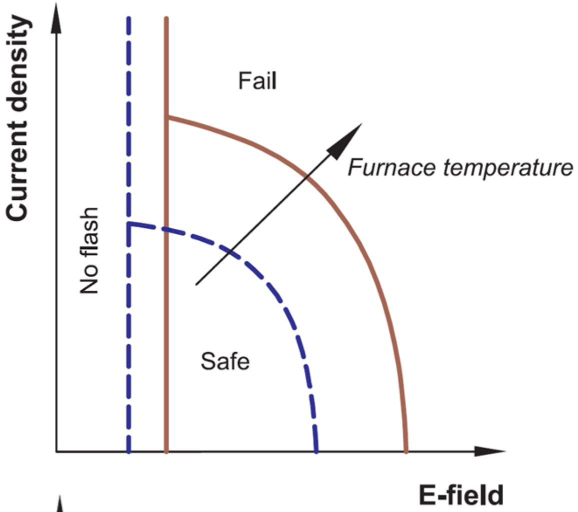
Illustration of suggested mass flow changes between conventional and flash sintering (with and without stress)

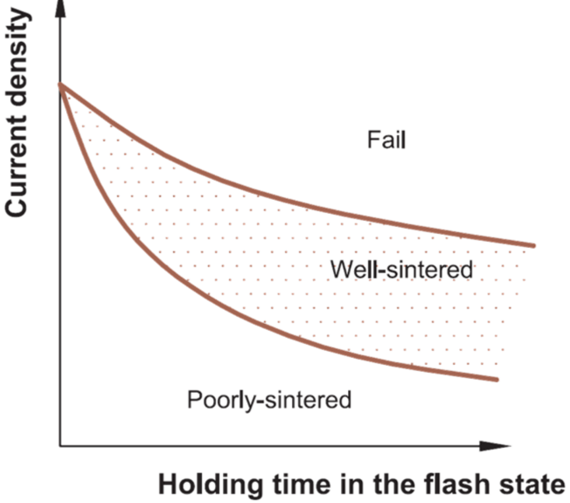
- **Orange:** atom trajectory
- **Lime:** interstitial trajectory
- **Blue:** vacancy trajectory

source: 10.1016/j.jeurceramsoc.2018.08.048

Drawbacks of flash sintering

- **Local inhomogeneities** can lead to formation of preferential current paths
  ➔ causes **hotspots** and local melting
- **Inhomogeneities** can be
  - Already **present in green body**
  - **Form** during flash sintering (e.g. surface-core temperature gradients)
- Hotspot failure occurs above **critical current density** $J = I/A$ and **electric field** E which can be shifted to higher values with increasing T_f
- Risk can be reduced by
  - **Decreasing** negative temperature coefficient of resistivity
    ➔ reduces conductivity increase rate
  - Controlling the thermal runaway by **stepwise increase** of I_{lim}
  - Optimizing the **dwell time** in the flash state





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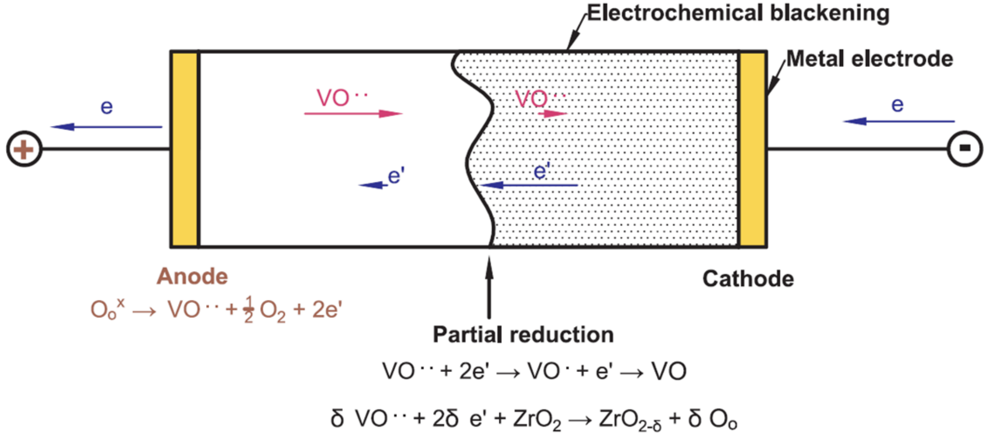
Electrochemical reactions

- When using DC or low frequency AC flash sintering, **local electrochemical reactions** occur at electrodes
- In O^{2-} conductors (e.g. yttrium stabilized zirconia (oxide))
  - **Anode** (electron removal):

$$O_O^x \Rightarrow V_O^{\cdot\cdot} + 1/2\, O_{2(g)} + 2e'$$

  - **Cathode** (electron injection):
    Inverse reaction not fast enough
    ➔ $V_O^{\cdot\cdot}$ species trap electrons leading to **partial oxide reduction** (e.g. blackening)

$$\delta V_O^{\cdot\cdot} + ZrO_2 + 2\delta e' \Rightarrow ZrO_{2-\delta} + \delta O^x$$

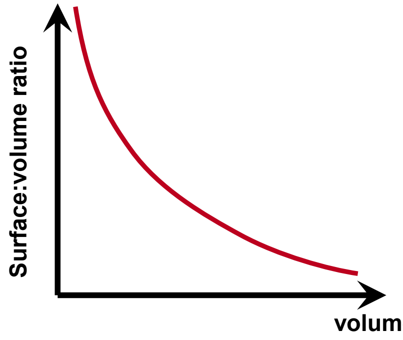


- **Partially reduced zirconia** turns black and has an **enhanced conductivity** (n-type semi-conductor)
  - This reaction can **contribute to the flash event onset**
  - Causes **local temperature differences** between the anode and cathode
- **Cathodic reaction front moves towards the anode**

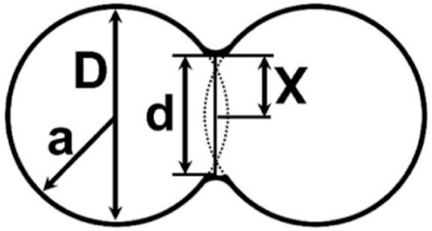
source: 10.1016/j.jeurceramsoc.2018.08.048

Final remarks on flash sintering

- **Surface to volume** ratio affects balance between W_{in} and W_{out}
  ➔ reducing sample size increases onset temperature
- **Pre-sintering** or external pressure application is
  - **Favorable** when **volume conductivity** is dominating
    ➔ **reduces onset temperature** for the thermal runaway (e.g. favoring resistivity reducing neck growth)
  - **Unfavorable** when **surface conductivity** is dominating (e.g. alumina)
    ➔ **increases onset temperature** for thermal runaway (e.g. reducing free surface area by neck growth and densification)
- Point defect formation in **reducing atmospheres** (e.g. vacuum, forming gas, …) may **promote flash** event
- Flash event **without substantial densification** possible **if surface conduction** is **dominant** in the material to sinter
  ➔ **Increase of applied electric field** necessary to force densification and power dissipation during flash and stage III



Higher surface to volume ratio increases radiative cooling proportion



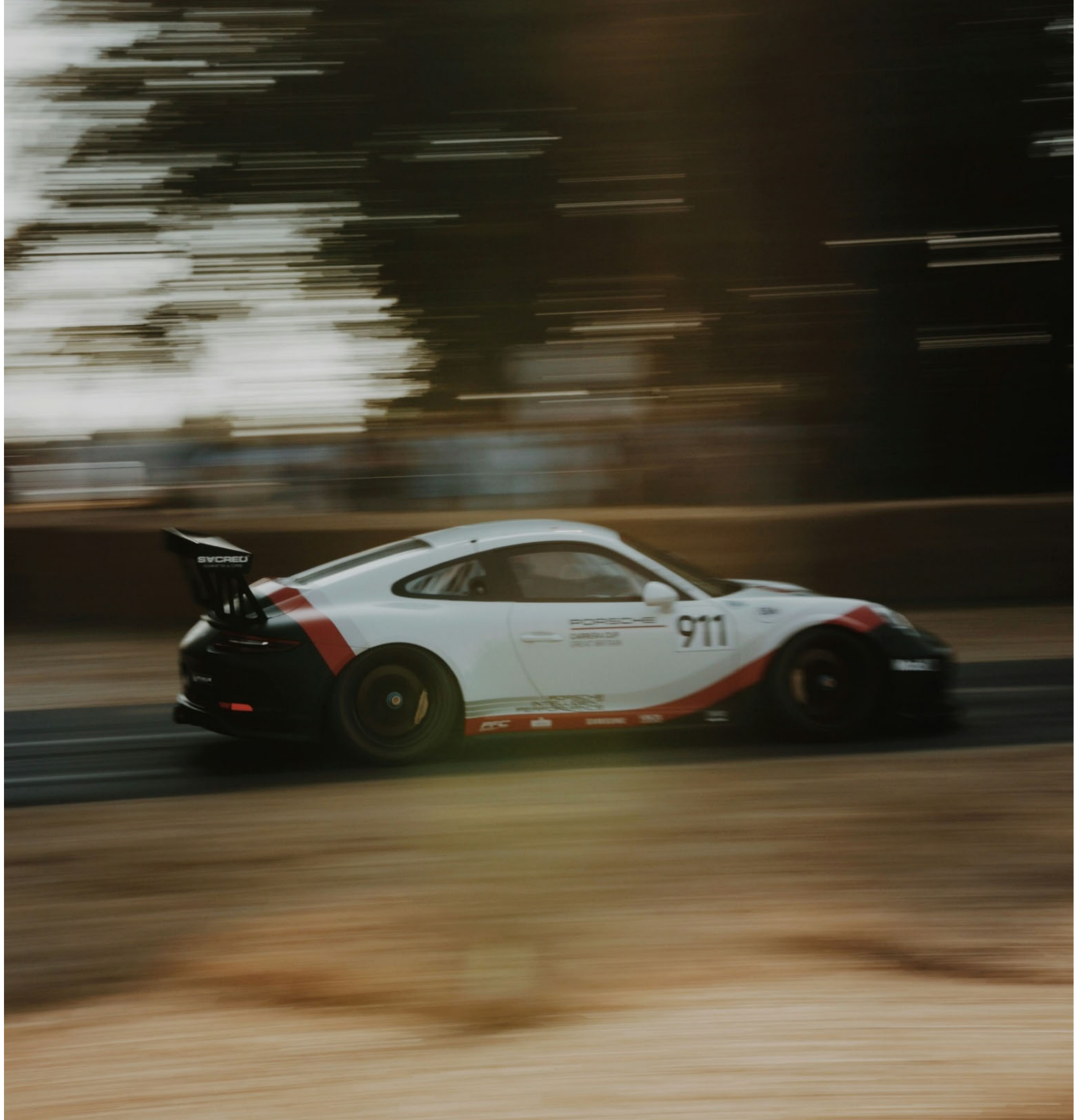
Neck formation reduces contact resistance in volume conduction and increases resistance in surface conduction

source: 10.1016/j.jeurceramsoc.2018.08.048

Rapid sintering

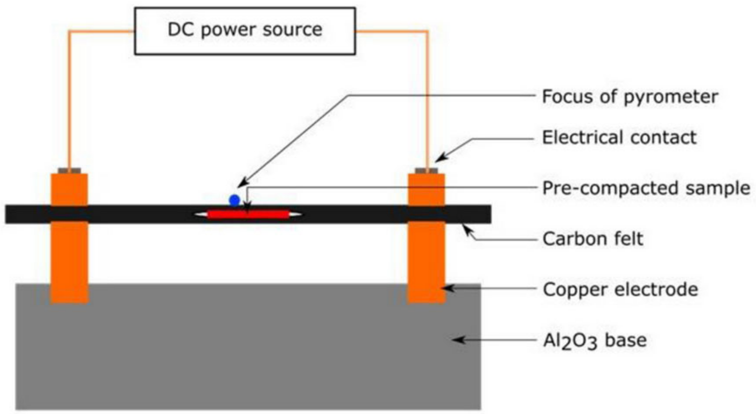
ULTRAFAST HIGH-TEMPERATURE SINTERING (UHS)

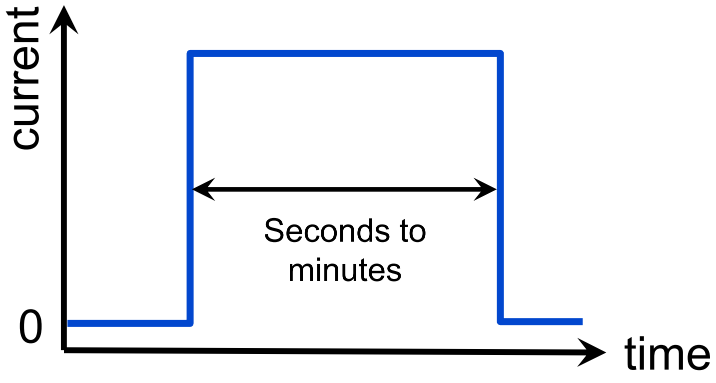
M. Stuer



Ultrafast high-temperature sintering (UHS)

- **Sample** is placed into a **graphite felt** placed in vacuum or argon atmosphere
- Current is applied to the felt instantaneously leading to **very fast heating rates by Joule heating**
- Temperature is controlled by the applied current and **depends on a given felt geometry** (and oxidation state)
- Pyrometers and/or thermocouples are used to follow the temperature but can be unreliable
- For a given setup, **calibration curves** are recommended
  - ➔ Current scan to determine limit current needed to melt metals with defined melting temperatures



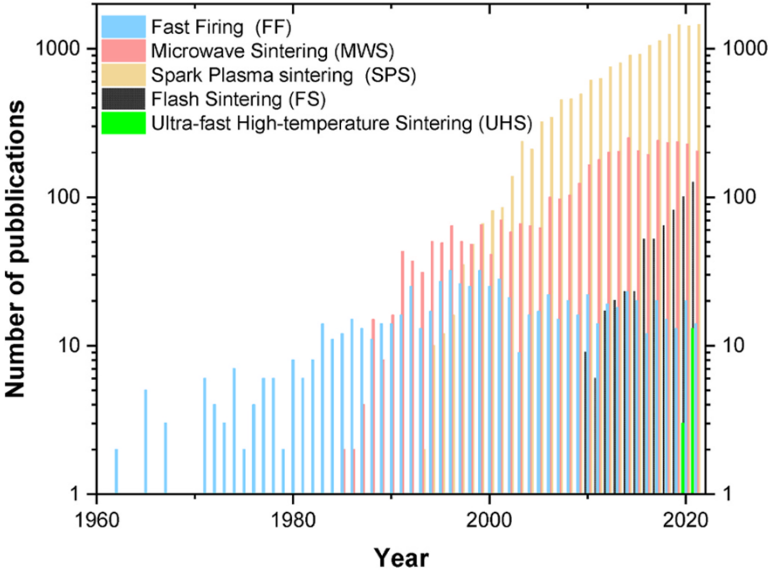


source: 10.1016/j.actamat.2022.117918

Ultrafast high-temperature sintering (UHS)

Why UHS leads to fast sintering?

- UHS remaining a new technique, there is still **a lot of debate**
- Reduced **neck size and radius** lead to faster diffusion rates (stronger gradients)
- **Temperature gradients** may also play a role (see flash sintering)
- **Surface impurities** may lead to appearance of **transient liquid phases** with rapid particle rearrangement (e.g. capillary forces)
- **Limited change of particle/neck curvature** occurring during rapid heating may lead to **plastic flow** at high temperatures to accordingly preserved **high sintering stresses**



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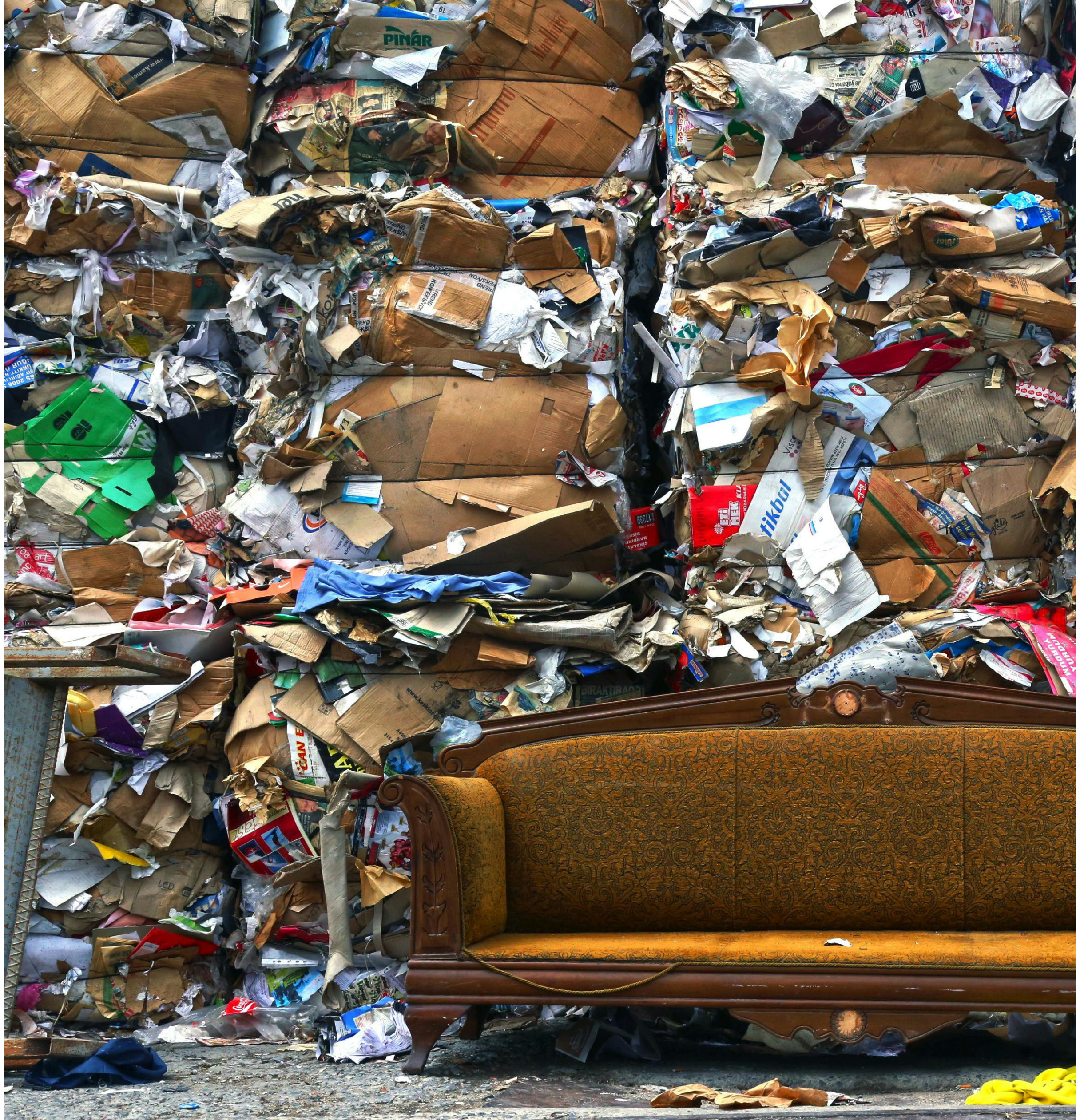
UHS sintering

Pressure assisted (rapid) sintering

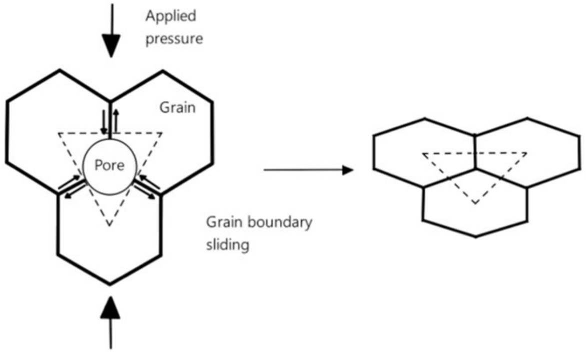


M. Stuer

Typical pressure assisted sintering basics

- Application of **external pressure** (uniaxial or isostatic) **improves densification kinetics** by:
  - Promoting **particle rearrangement** and **plastic deformation**
    ➔ increased particle contact and packing efficiency
  - **Counteracting** internal **pore gas pressure**

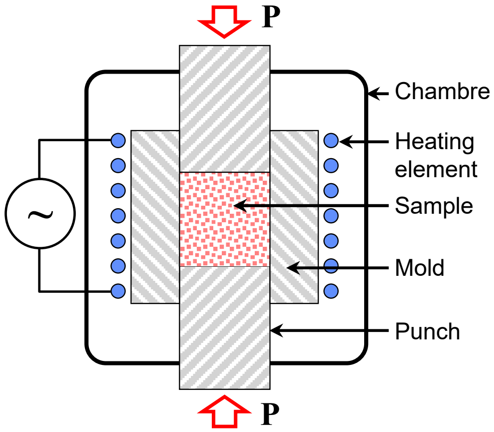
$$P_{dens} = \frac{2\gamma_{SV}}{r_{pore}} - P_{pore,gas} + \mathbf{P_{ext}}$$



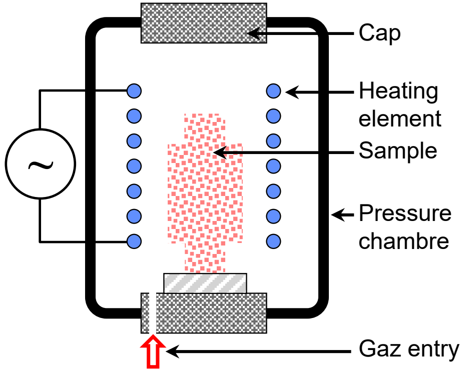
- Pressure assisted sintering allows
  - Reaching full densification **faster**, before significant grain growth has time to occur
  - Reaching full densification **at lower temperatures**, reducing the risk of grain growth (and complexion transitions)
- Typical pressure-assisted sintering techniques

1. **Hot pressing**	20-40 MPa of uniaxial pressure
2. **Hot isostatic pressing**	20-300 MPa of isostatic pressure
3. **Spark plasma sintering**	20-250 MPa of uniaxial pressure
4. **Ultra-high pressure sintering**	up to 10 GPa in dedicated press designs

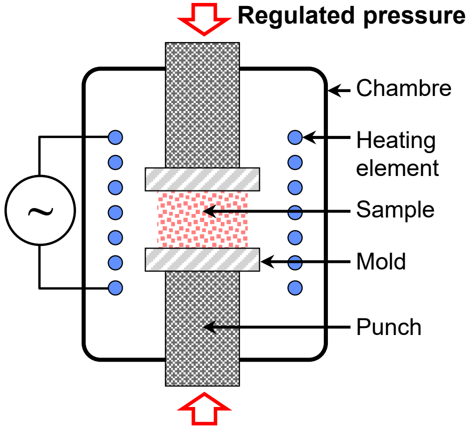
Typical pressure assisted sintering techniques



Hot pressing (HP)



Hot isostatic pressing (HIP)



Sinter forging

Advantages:

- ♦ Reduction of sintering temperatures between 100 and 500°C
- ♦ Improved densification with reduced (residual) defect risks ➔ Improves mechanical properties of ceramic
- ♦ Microstructure refinement ➔ Improved reliability and stability of ceramic properties

- ♦ X-Y dimensions controlled by mold

Limitations:

- ♦ High costs
- ♦ Carbon contamination
- ♦ Rather simple shapes
- ♦ Part-by-part process

Advantages:

- ♦ Compatible with complex shapes
- ♦ Batch sintering possible

Limitations:

- ♦ High costs
- ♦ Requires pre-sintering up to pore closure

Advantages:

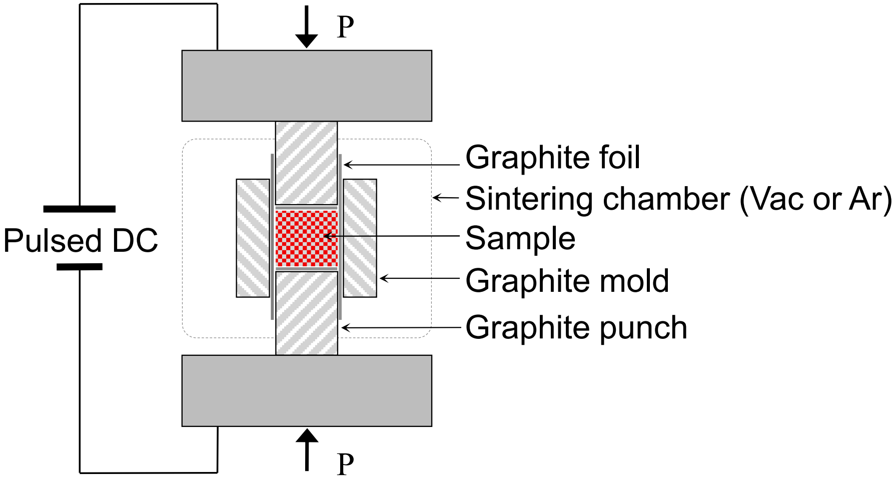
- ♦ Maximizes plastic deformation

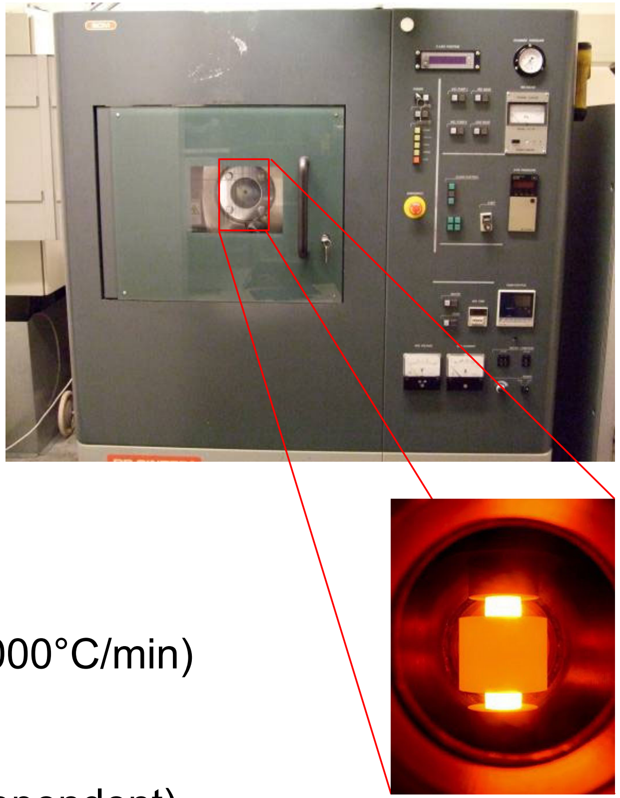
Limitations:

- ♦ High costs
- ♦ No shape control ➔ machining!
- ♦ Part-by-part process

Pressure sintering

Spark Plasma Sintering (SPS | FAST | PECS)





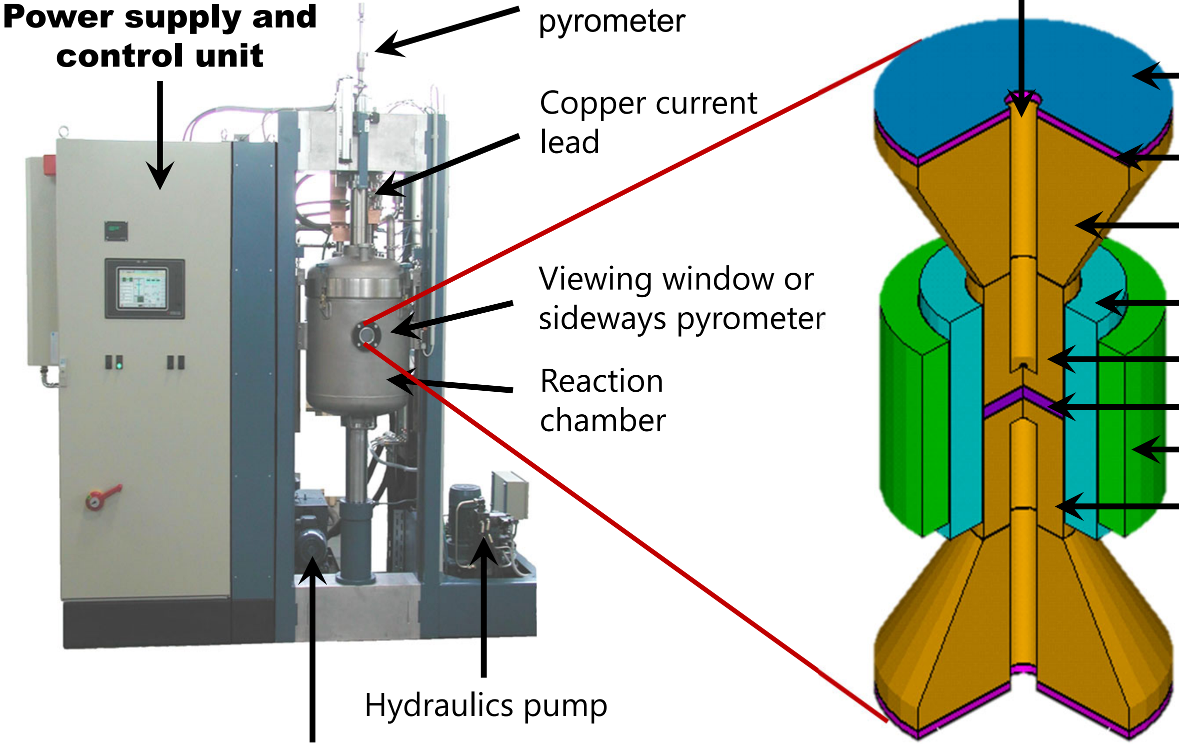
♦ Characteristics:

- Heating ramps typically between 50-500°C/min (up to 1000°C/min)

$$W = I * V = I^2 * R = \frac{V^2}{R}$$

- Maximum sintering temperature 2500°C (tool material dependent)
- Pressure: 5-200 MPa (higher possible with special molds)
- Atmosphere: Vacuum or Argon (N_2 avoided due to risk of cyanide compound formations!)

Spark Plasma Sintering: Furnace and tooling details



Power supply and control unit

Central pyrometer

Copper current lead

Viewing window or sideways pyrometer

Reaction chamber

Hydraulics pump

Vacuum pump

Central pyrometer shaft

Piston

Graphite paper

Conical spacer

Die

Top punch

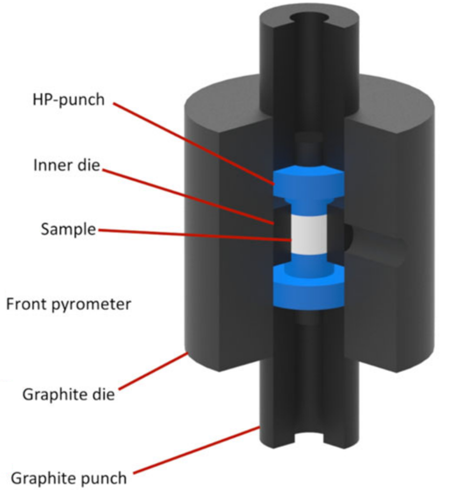
Powder/sample

Thermal insulation

Bottom punch

Die variation for high-pressure SPS

(research)



source: 10.1016/j.oceram.2023.100433

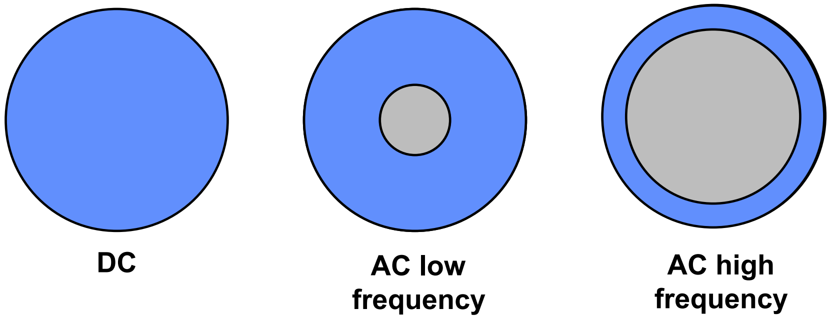
Spark Plasma Sintering: General aspects

- Maximum temperature/pressure depend on **tool materials**

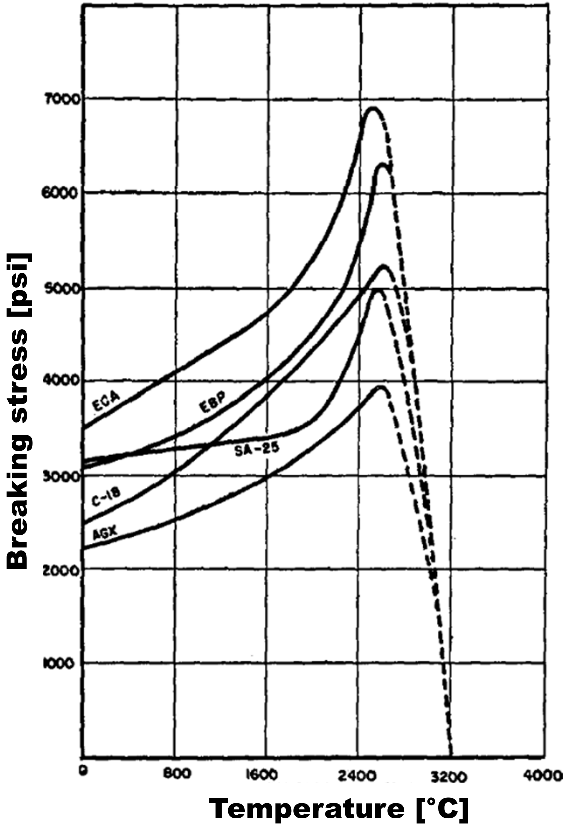
Steels:	~400°C	400 MPa
Ti-Zr-Mo (TZM) alloy	~1000°C	200 MPa
Graphite	<2500°C	150 MPa

- **Graphite tools** commonly used
  ➔ Dwell at 600°C with minimum pressure common to **homogenize temperature** before pressure appl.
- SPS in **vacuum promotes densification** but may lead to formation of **volatile phases** (e.g. suboxides)
- SPS is typically done in DC ➔ current flow homogeneity

Current distribution in conductor cross section



Temperature dependence of breaking stress for different graphite qualities

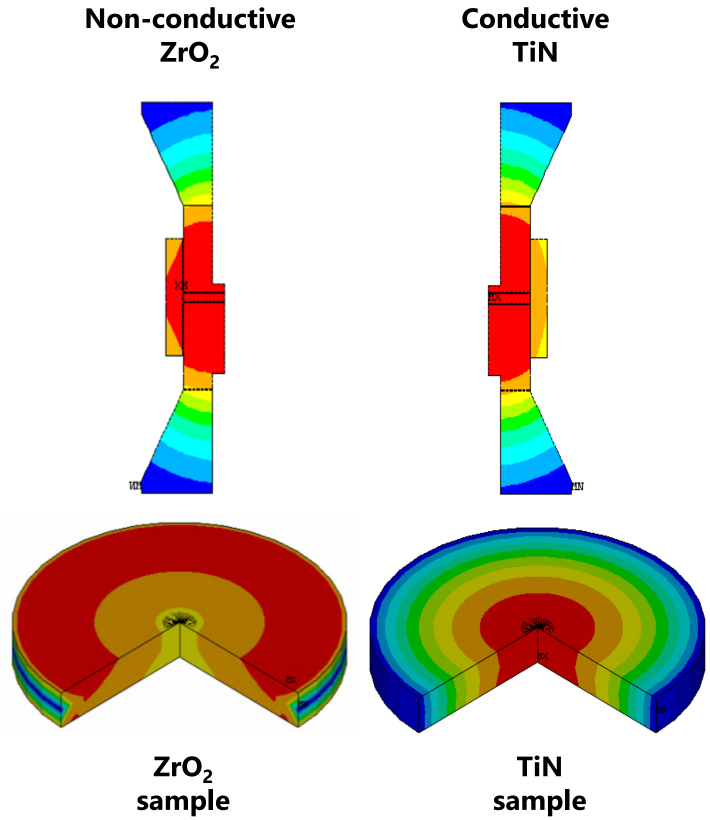


source: 10.1063/1.1700013

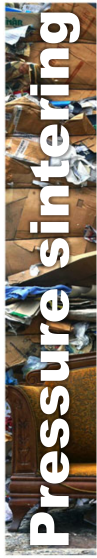
Spark Plasma Sintering: Current flow

- Current density and **temperature distribution** can be complex leading to **local variations in densification**
- **Sample conductivity** determines thermal distribution
  - **Insulating powders** (oxide ceramics)
    - Current flows mainly through the die
      ➔ Inwards heating (hot die <> cold sample)
    - Central pyrometer will measure the coldest temperature
  - **Conductive powders** (carbide/nitride ceramics)
    - Current directly flows through the powder sample
      ➔ Direct sample heating (cold die <> hot sample)
    - Central pyrometer measures more accurately

Temperature distribution with conductive and non-conductive samples



Stress exponent measurement



- **Sintering model** of HP and SPS is given by

$$\frac{1}{\rho}\frac{d\rho}{dt} = -\dot{\varepsilon}_z = \frac{HD(T)\Phi(\rho)^n\sigma_z^n}{G^m k_B T}$$

- ρ: density
- $\dot{\varepsilon}_z$: normalized densification rate
- H: constant (combines all pre-exp. constants related to creep deformation)
- σ: stress
- n: stress exponent
- G: grain size
- m: grain size exponent
- $D(T)$: diffusion coefficient
- $\Phi(\rho)$: stress intensification factor

- n and m indicate **sensitivity of densification on stress and grain size**, respectively
  - $n = 1$ $m = 2$ ➔ Nabarro-Herring creep: Diffusion of vacancies through the grains (lattice/bulk diffusion)
  - $n = 1$ $m = 3$ ➔ Coble creep: Diffusion of vacancies along grain boundaries (grain boundary diffusion)
  - $n = 1 - 2$ $m = 1$ ➔ Diffusion accommodated grain boundary sliding
  - $n = 2 - 5$ $m = 0 - 1$ ➔ Dislocation accommodated grain boundary sliding → more common in nanocrystalline ceramics (example: fine grained ceramics under flash sintering $n = 2.5$, $m = 0.5$)
- More generally, n **governs degree of non-linearity** of sintering model
  - $n = 1$: **linear** mechanisms, grain boundary and lattice **diffusion**
  - $n > 1$: **non-linear** mechanisms, **dislocation** based mechanisms or grain boundary **sliding**
- Knowledge of **stress exponent** provides important sintering insights

source: 10.1016/j.mechmat.2023.104664

Stress exponent measurement

- Different methods exist to measure the stress exponent, accuracies depend on assumptions regarding $\Phi(\rho)$ (generally unknown) and G
  - **Logarithmic linear regression** of applied stress
    Assumes theoretical function for relative density dependence of $\Phi(\rho)$
    ➔ May influence n determination
  - **Creep tests** on fully sintered dense specimens to determine n and temperature behavior
    Assumes constant grain size knowing that creep is strongly influenced by grain size
    ➔ High temperatures (e.g. ceramic sintering) alter grain size
  - **Porous stage methods**:
    - **Stepwise pressure** single test
    - **Constant porosity** multiple test

Illustration of stepwise pressure method

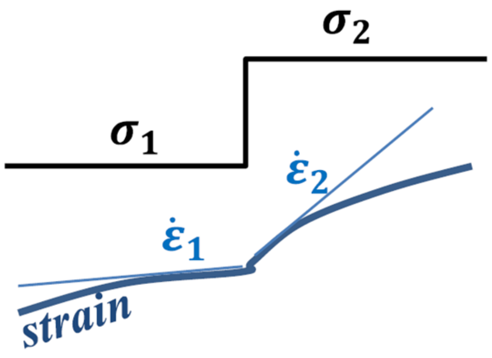
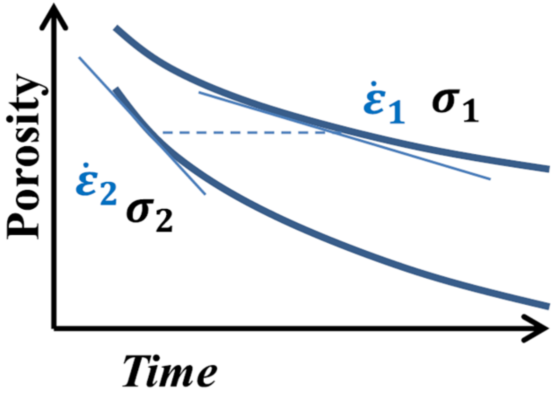


Illustration of constant porosity method



source: 10.1016/j.mechmat.2023.104664

Stress exponent measurement: Stepwise pressure method

- Tests done in **porous regime** and temperature where $G^m \approx constant$ holds due to **low grain growth kinetics**
- Combined with **isothermal conditions** $\frac{HD(T)}{G^m k_B T} = constant$
- Effect of $\Phi(\rho)$ (unknown) suppressed by performing measurements at **nearly constant ρ**
  - ➔ Pressure jump should be fast to minimize relative density variation
- Assuming radial displacement close to zero **in SPS** neglecting relative density variation at pressure step (e.g. ideal step function)

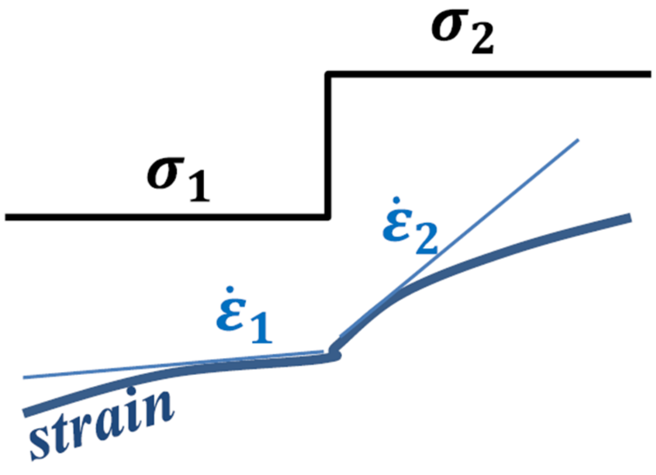
$$n = \frac{\ln\left(\frac{\dot{\varepsilon}_1}{\dot{\varepsilon}_2}\right)}{\ln\left(\frac{\sigma_1 - \sigma_{sint}}{\sigma_2 - \sigma_{sint}}\right)}$$

- $\dot{\varepsilon}_{1,2}$: axial strain(/densification) rate
- $\sigma_{1,2}$: axial stress
- σ_{sint}: sintering stress

with: $\sigma_{sint} = \frac{3\gamma_s}{r}(1-\theta)^2$

- γ_s: ceramic surface tension (J.m^{-2})
- r: particle radius (m)
- θ: porosity

Illustration of stepwise pressure method



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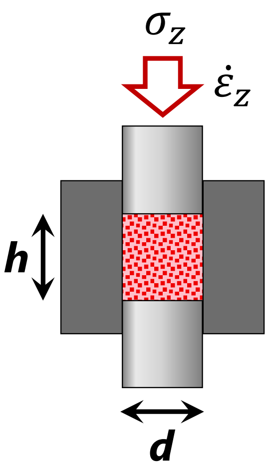
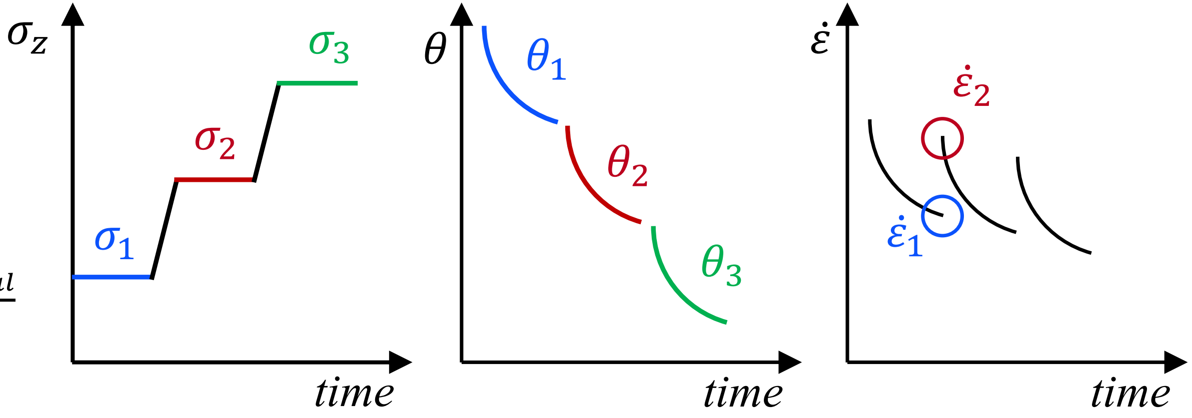
Stress exponent measurement: Stepwise pressure method



$$\dot{\varepsilon}_z = \frac{\dot{h}}{h}$$

$$\sigma_z = \frac{F}{\pi(d/2)^2}$$

$$\theta = 1 - (1 - \theta_{final})\frac{h_{final}}{h}$$



- σ_{sint} inversely proportional to r
  ➔ might be neglected for large particle sizes to simplify the stress exponent equation

$$n = \frac{\ln\left(\frac{\dot{\varepsilon}_1}{\dot{\varepsilon}_2}\right)}{\ln\left(\frac{\sigma_1}{\sigma_2}\right)}$$

- σ_{sint} increases stress exponent and should be considered for (sub-)micron ceramics
- Correction of porosity jump during pressure step can lead to calculation instabilities

source: 10.1016/j.mechmat.2023.104664

The end

What was on the
- MENU -
today ?

- **Summary**
  - Types de frittages
  - Forces motrices pour le frittage
  - Mécanismes de transport (de matière) pendant le frittage
  - Stades de frittage en phase solide selon le modèle de Coble
  - Aperçu d’une sélection de moyens de contrôle du développement microstructural

- **Questions**