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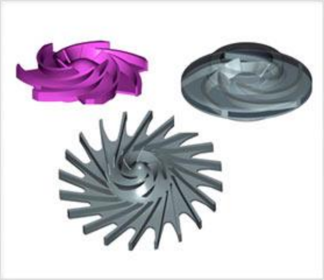


ÉCOLE POLYTECHNIQUE
FÉDÉRALE DE LAUSANNE

Powder Technology

From Landslides and Avalanches to Concrete and Chocolate

Prof. P. Bowen (EPFL), Dr. P. Derlet (PSI)

WEEK 14 – file no. 12

Sintering Mechanisms & New Technologies- (4)

New Technologies – Sintering Methods

Today's Objectives

Last Week

- Standard forming methods…..ceramics and metals
  - Dry Pressing
  - Wet methods – overview - slip casting, injection moulding
  - Limitations …additive manufacturing approach
  - General intro to additive manufacturing
  - Additive manufacturing and sintering combined – SLS

This week …

- Summary of standard sintering methods and procedures
  - Isothermal, controlled rate, two-step, hot press, hot isostatic pressing
  - Solid state, liquid phase, reactive sintering
- New sintering processes, Spark Plasma Sintering, flash sintering, cold sintering…
- Typical questions, Powder Technology – Learning outcomes,
- Exam….

Teaching plan 2018

- Files of lectures and notes to be found on PTG website : http://lmc.epfl.ch/PTG/Teaching

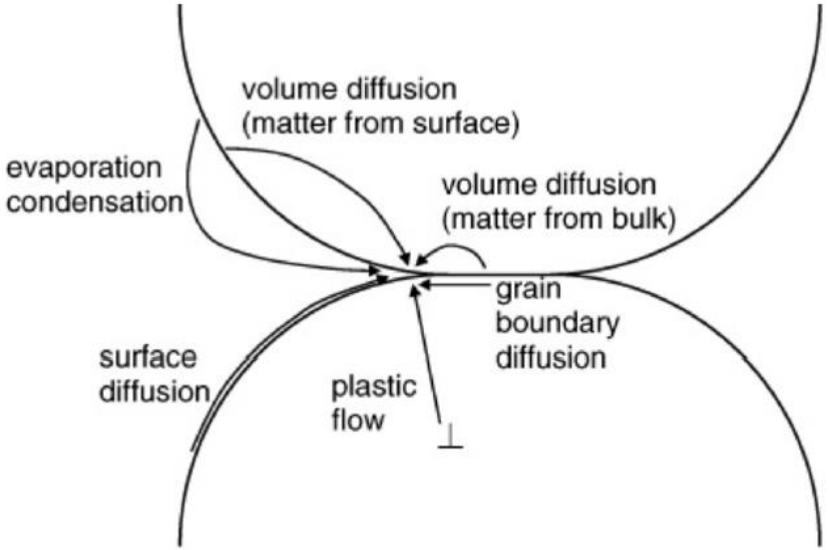
Week- DATE	File. no.		Powder Technology – Wednesday 10.15-13.00 – MXG 110
1- sept 19	1&2	PB	Introduction – example rheology – Yodel - Powder packing and compaction – 1 (i) – (3hrs)
2 – sept 26	2&3	PB MS	Powder packing and compaction – 1(ii), 2- Examples and DEM guest lecturer – (3hrs)
3 – oct 3	4	PD	Powder packing and compaction -3 & 4(i) – (3hrs)
4 – oct 10	4&5	PD PB	Powder packing and compaction - 4 (ii) – (1hr) Particle – Particle Interactions 1 - 2hrs
5 – oct 17	6&7	PB	Particle – Particle Interactions 2 & 3(i) – (3hrs) – Download Hamaker
6 – oct 24	7	PB	Particle – Particle Interactions – 3(ii) YODEL-PB (1hr) ***Exercises*** – Intro to Hamaker & YODEL software & groups project (2hrs)
7 – Oct 30		AKM	***Exercises -*** Hamaker and Yodel Modelling – group projects
8 – nov 7	8	PB PD	***Exercises*** –presentation of interparticle project results (1 hr) Introduction to atomistic scale simulations – (2hrs)
9 – nov -14	9& 11	PD	Compaction, Sintering & Defects in metals at atomistic scale (2hrs) Sintering Mechanisms – 1(i) (1 hr)
10 – nov 21	11	PD	Sintering Mechanisms - 1 (ii) & 2 (3hrs)
11 -nov-28		PD	***Excercises*** -Introduction to Molecular Dynamics Modelling using LAMMPS (3hrs) .
12 - dec 5		PD	***Excercises*** - MD- DEM modelling exercise using LAMMPS –particle packing - Effect of parameters (3 hrs)
13 – dec 12	10	PB	New Technologies -1 Processing – Forming – Shaping (2hrs) & ***Exercises or invited lecture or visit***
14 – dec 19	10	PB	New Technologies-2 – Sintering Methods & ***summary*** **& Exam method**
			PB – Prof. Paul Bowen (EPFL), PD – Dr. Peter Derlet (PSI)
			MS- Dr. Mark Sawley (EPFL), AKM - Aslam Kuhni Mohamed(EPFL)

Standard Sintering Mechanisms

- Driving forces and diffusion theory & mechanisms weeks 9&10 – file 11
- Solid state….

Last Year and TDM*

- liquid phase – (porcelaine, Si_3N_4))
- reactive sintering – Al_2O_3 +TiO_2



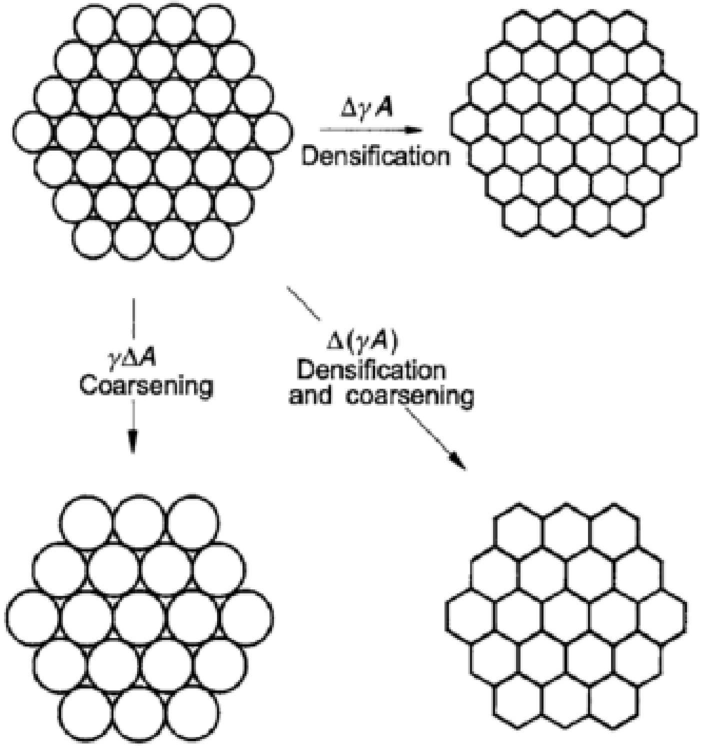


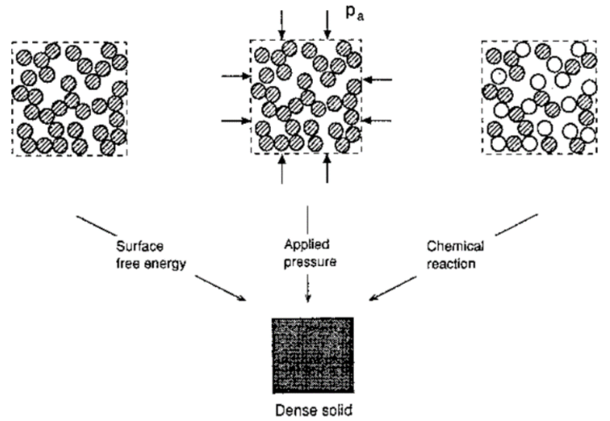
Figure I.5. Basic phenomena occurring during sintering under the driving force for sintering. Δ(γA).

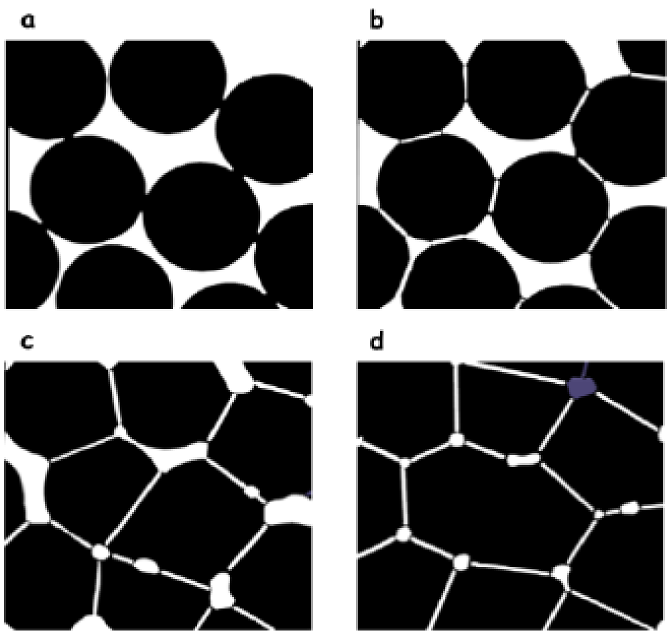
P 315-325

*Les Traité des Matér, Vol. 16 « Les Céramiques », p.315-325 J. Barton, P. Bowen, C. Carry & J.M. Haussonne, PPUR, 2005

Driving Forces & magnitude - Sintering

- Sintering is a process of lowering total free energy of the particle system
- Any sources to promote the lowering of ΔG will be the driving force for sintering
  - The curvature of the particle surfaces (surface energy)
  - An externally applied pressure
  - A chemical reaction





0) Loose powder
1) Initial stage (60-65%)
2) Intermediate stage 65to 90%
3) Final stage 95-99+%

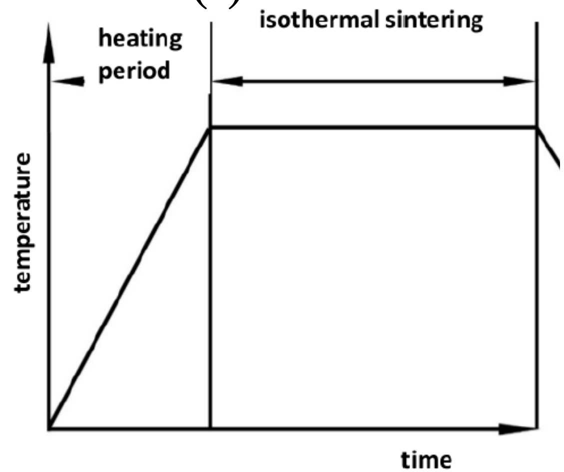
- Surface energy/curvature which gives a localised sintering stress:
  - For 1um particles, the surface energy is about 75 J/mol
- Applied external pressure:
  - For a typical pressure of 30MPa applied in hot pressing, the total energy is 750 J/mol
- Chemical reaction:
  - It is about 20000 J/mol at 1000K if *Keq* is 10.

Video sintering - https://www.youtube.com/watch?v=48Is5ENhkGE

Standard Sintering Methods

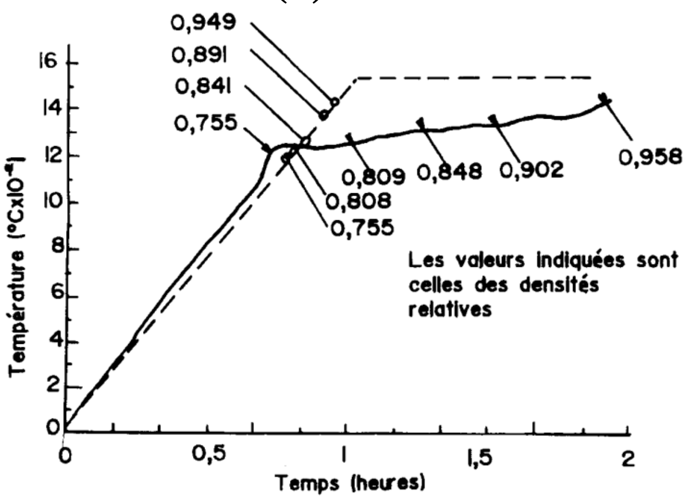
- Isothermal (a)
- Controlled rate (b)
- Two-step (c)
- Hot Pressing (d)
- Hot Isostatic Pressing (e)

(a)

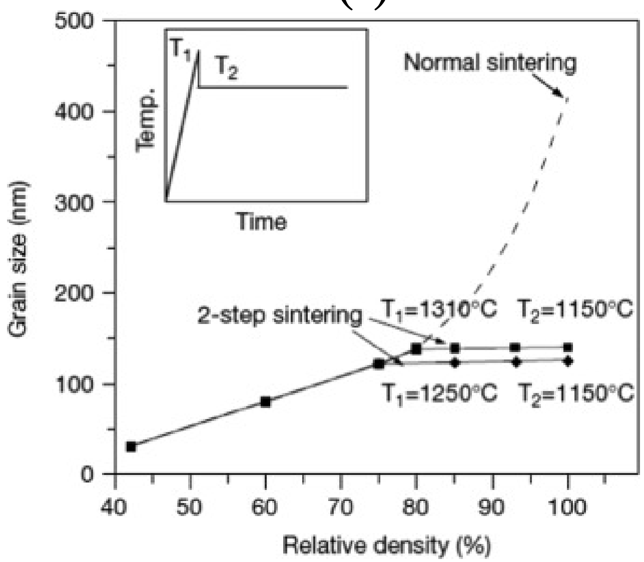


Temperature evolution during a simple sintering cycle

(b)

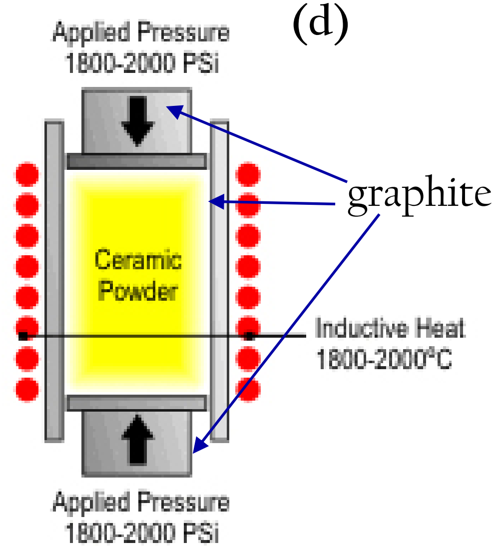


(c)



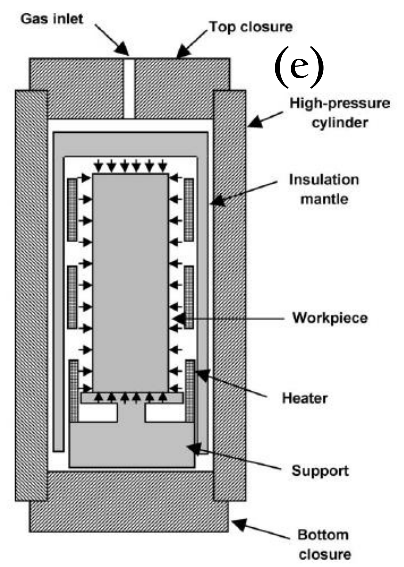
Loh et al. Ceram.Int. 42(2016)12556–12572

(d)



www.dynacer.com/processing/hot-pressing

(e)

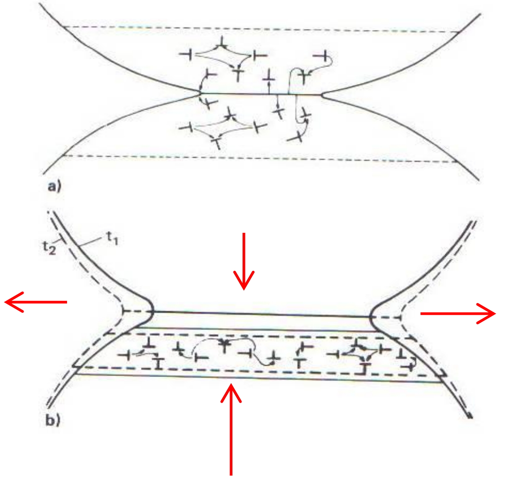


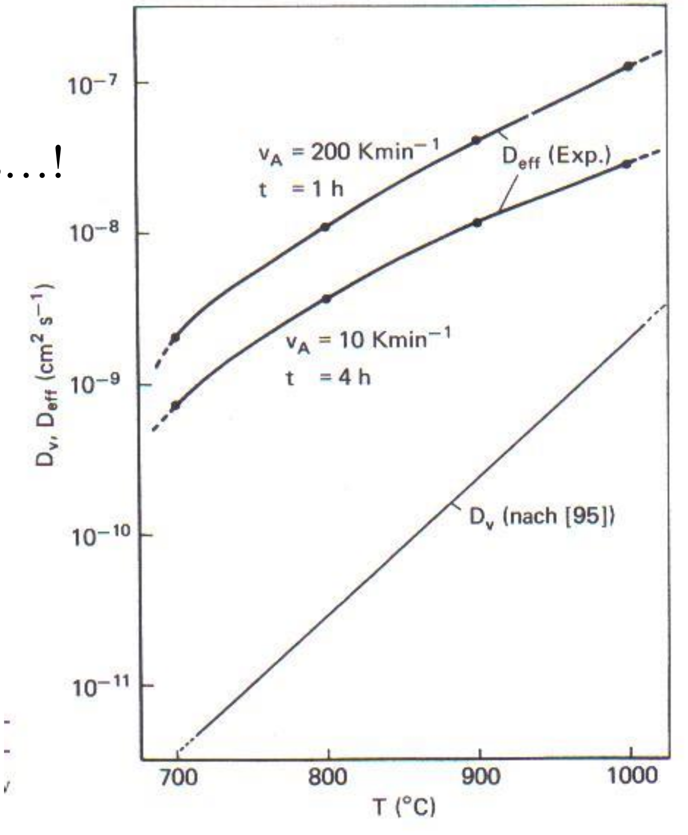
Bocanegra-Bernal, J.Mater.Sci. 39 (2004) 6399 – 6420

Solid State Sintering- real (metal) powder compacts

- Four major mechanisms controlling densification (compaction and sintering) are – rearrangement, plastic deformation, power-law creep and diffusional flow
- Sintering, heat & curvature – continued defect formation – dislocations leading to creep (deformation) – described by a power law - increased diffusion or «flow»
- Enhanced by T and P gradients in Field & pressure assisted sintering -

- From standard solid state sintering equations can calculate effective D.
- In experiment – 2 orders of magnitude greater than predicted from standard sintering theory… .defects…!
- Dislocation movement…climbing – eliminated at grain boundaries!



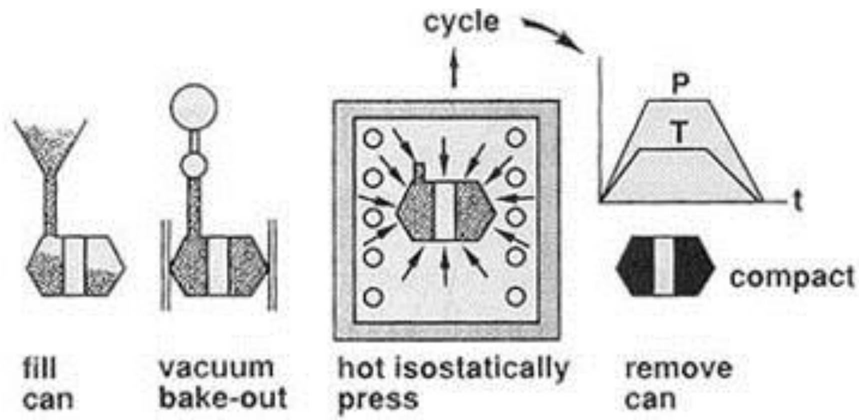


Hot Isostatic Pressing HIP

- HIP is used to reduce the porosity and increase the density of many metal & ceramic materials
- This improves the material's mechanical properties and workability
- Combines high temperatures and high pressures
- Pressures of up to 207 MPa (30,000 psi)
- Temperatures up to 2000°C (3,632°F) ▪
- Typically, an inert gas (Ar or N2)
- Pressure is applied uniformly from all sides (Isostatic Pressure)
- Post –HIP after pressureless
- Can increase mechanical properties of ceramics by factor of 3 and
- wear resistance x 2-3 even…..up to x10! Videos

https://www.youtube.com/watch?v=Pq4ML8_g49M

https://www.youtube.com/watch?v=4oVLlPksMwY



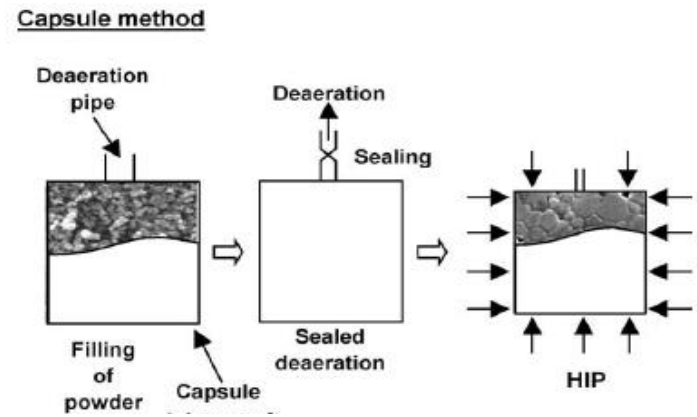
http://www.pvatepla.com/en/products/

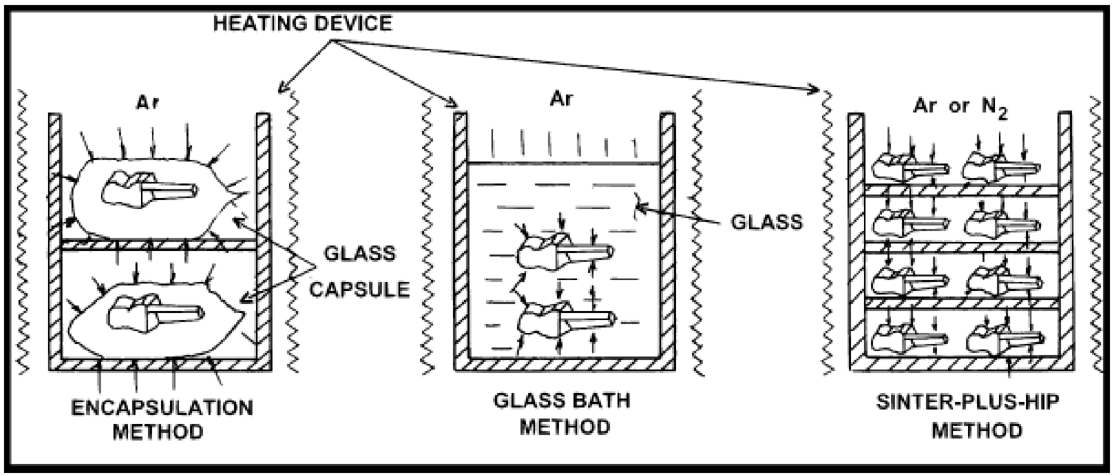
http://epsi-highpressure.com/products/hot-isostatic-presses/

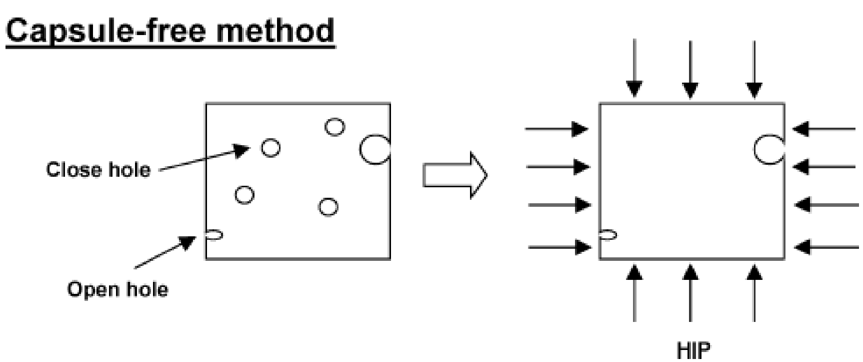
HIP- Encapsulation*

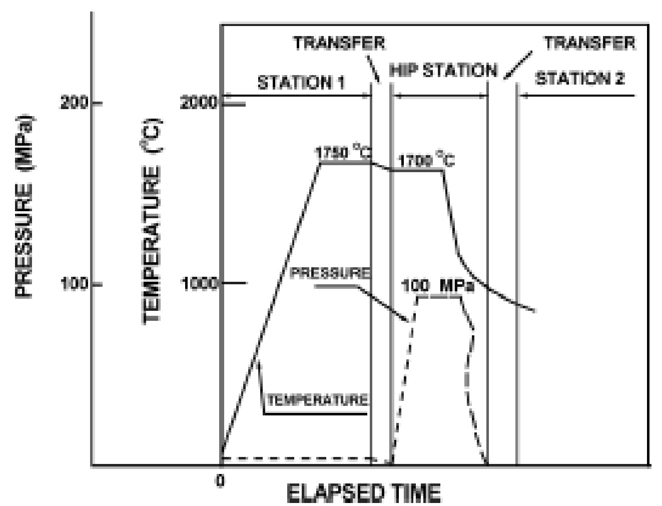
- Material must be relatively strong, gas tight, inert and plastic at applied temperature and pressure
- compatible with the material to be pressed - minimize diffusion reactions
- and readily removable

- Glass (capsule or bath)
- Metals (steel, others…)
- Free (e.g. post-HIP ceramics)









*Bocanegra-Bernal, J.Mater.Sci. 39 (2004) 6399 – 6420

HIP – Improved densities and properties*

- Example - 1
- Sintered Silicon nitride (SSN)
- Improved strength and reliability

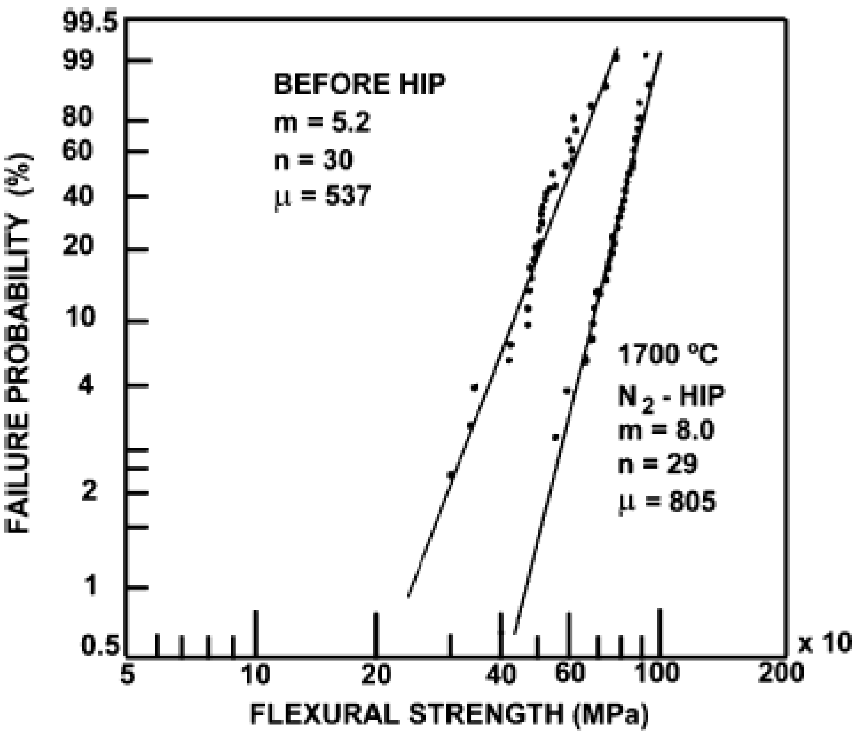


Figure 28 Weibull plot flexural strength for SSN-1 [From ref. 9].

- Example - 2
- Cemeted carbide (WC-Co)
- Improved strength and reliability

Properties	Before HIP	After HIP
Relative density (%)	Nearly 100	Nearly 100
Hardness (HRA)	91.0	91.0
Bending strength (MPa)	2450	2940
Fracture toughness (MPa$\sqrt{m}$)	10.0	10.5

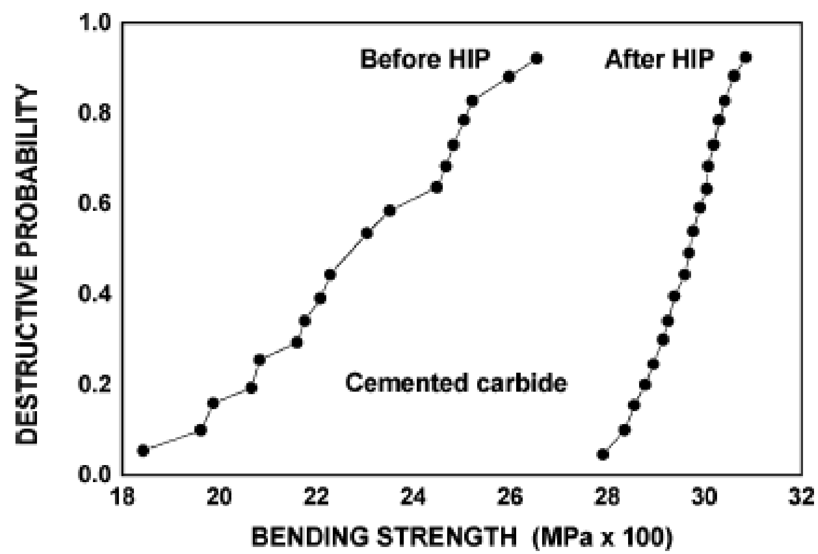


Figure 36 Weibull plot of bending strength before and after HIP treatment of cemented carbide [From ref. 8].

*Bocanegra-Bernal, J.Mater.Sci. 39 (2004) 6399 – 6420

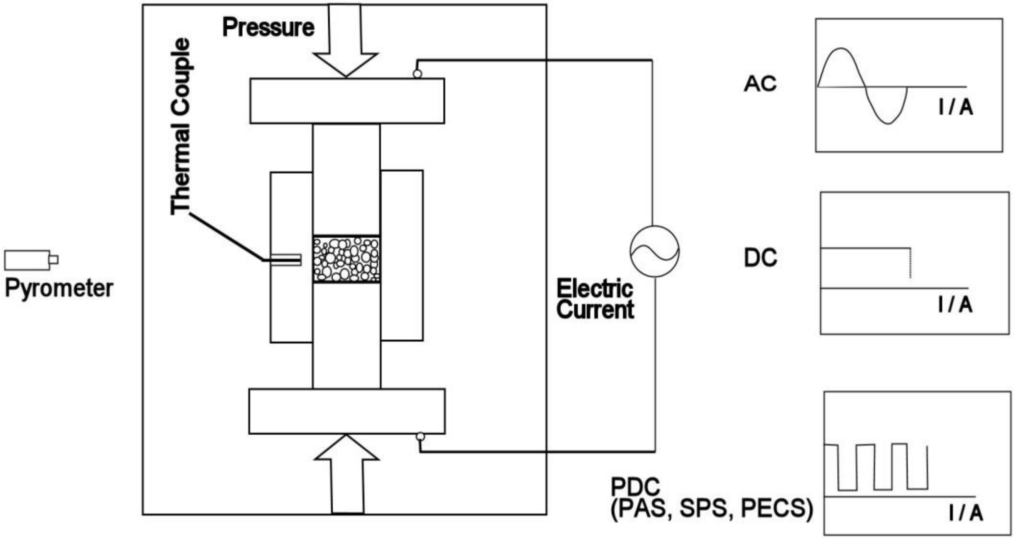
Pressure & Current-Assisted Densification

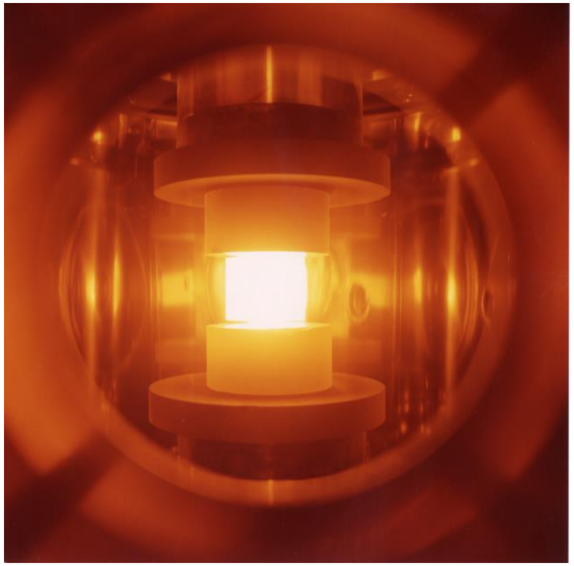
CAPAD: current-activated, pressure-assisted densification
HP: hot pressing
SPS: spark plasma sintering
FAST: field-assisted sintering technique
PECS: pulsed electric-current sintering

- with the aid of high electric currents, it is possible by using CAPAD to consolidate powders to full density much faster and at lower temperatures than by traditional methods such as pressureless sintering and hot pressing (HP).
- offer a platform for producing materials that are extremely difficult, if not impossible, to produce by other methods.
- This is exemplified by the method's recent emergence as a successful production tool for dense, large-sized, nanocrystalline materials.

J.E. Garay Annu. Rev. Mater. Res. 2010. 40:445–68

SPS-Spark Plasma Sintering





1962, Spark Sintering,

https://www.youtube.com/watch?v=UlmUYkG_wW4

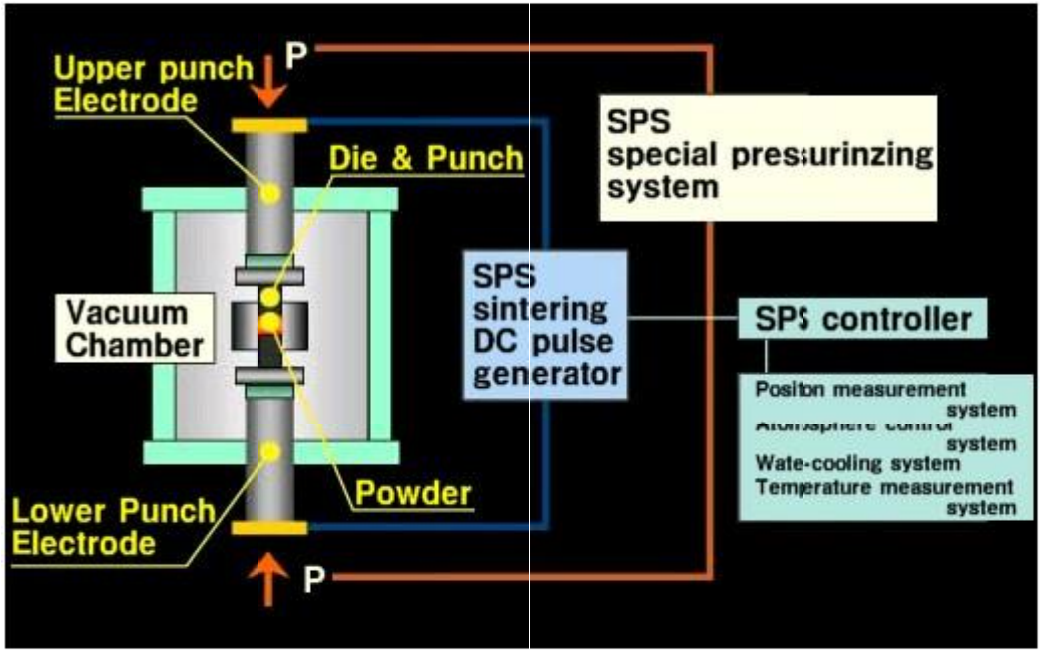
1986, Plasma Assisted Sintering

1990, Spark Plasma Sintering

2001, 4th generation SPS

Pulsed DC current - PDC

Spark Plasma Sintering



Sumitomo Heavy Industries, Ltd.

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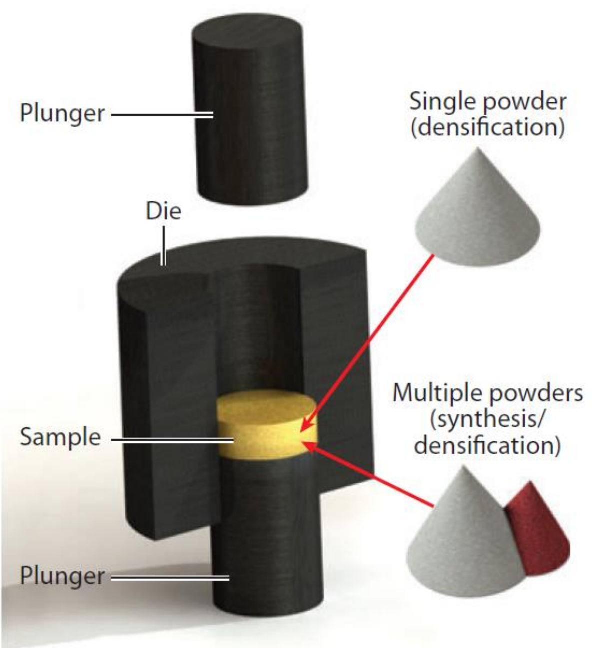
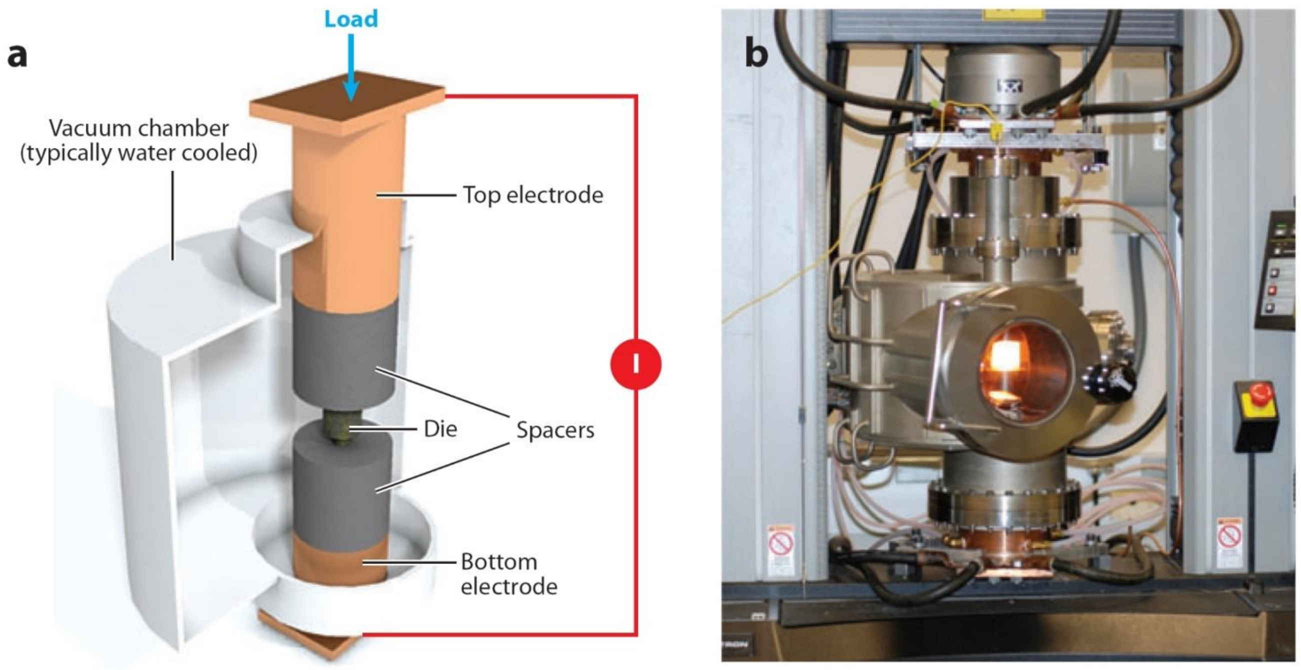
- The existence of plasma and sparking has been one of the most controversial topics in the CAPAD community.
- Hulbert et al. *J. Appl. Phys. 104:033305* recently presented convincing experimental investigations that plasma is indeed absent in CAPAD processing.
- The use of the word plasma in the process name is misleading and perhaps should be discouraged except when refering to commercial trade names. (Garay 2010)

Compaction – SPS*

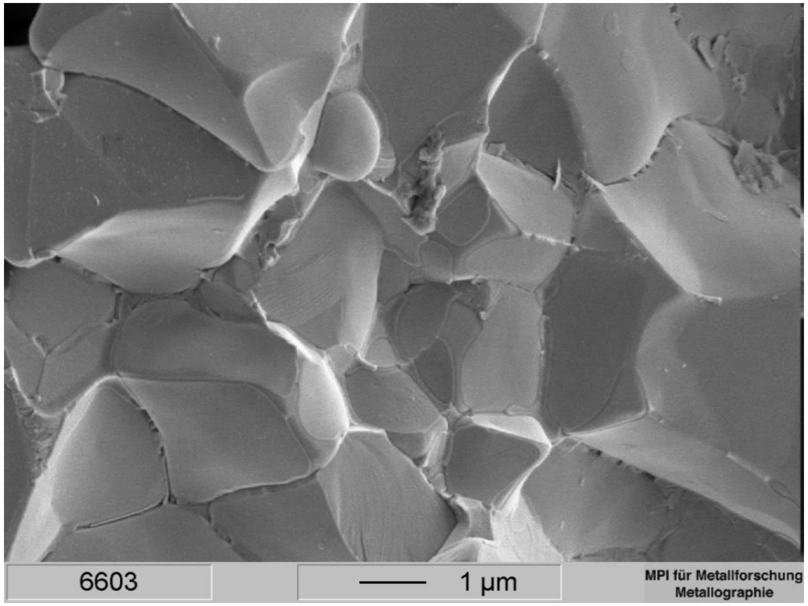
- A schematic of a die (normally graphite….), plungers (e.g. graphite), and powder.
- CAPAD can be used for pure densification or for simultaneous synthesis and densification.



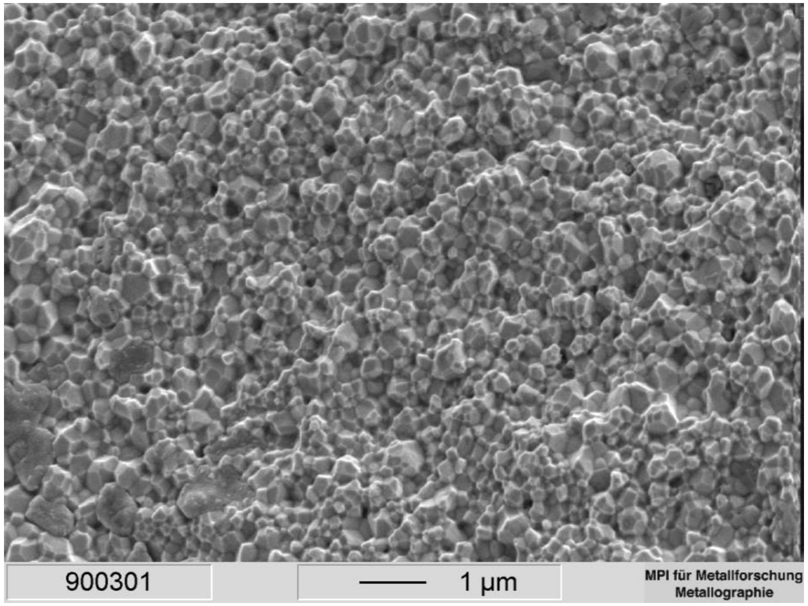


* Garay, J. E., Annu. Rev. Mater. Res. 2010. 40:445–68

Examples of SPS – nanostructured ceramics - PLZT



CS1250^{o}C - 2hr, 98.5%, 7µm



SPS 900^{o}C - 3min, 99.8%, 0.2µm

Future – Transparent polycrystalline alumina – Sintered by SPS !

- PCA samples (Ø 12 mm, thickness 1mm) – Spark Plasma Sintering*
- SPS - Stockholm Univ. Dr. Zhao Zhe

directly on the sheet

RIT of 7.8%

RIT of 57%

Key factors

1. Eliminate pores
2. Small Grain size or Orient grains

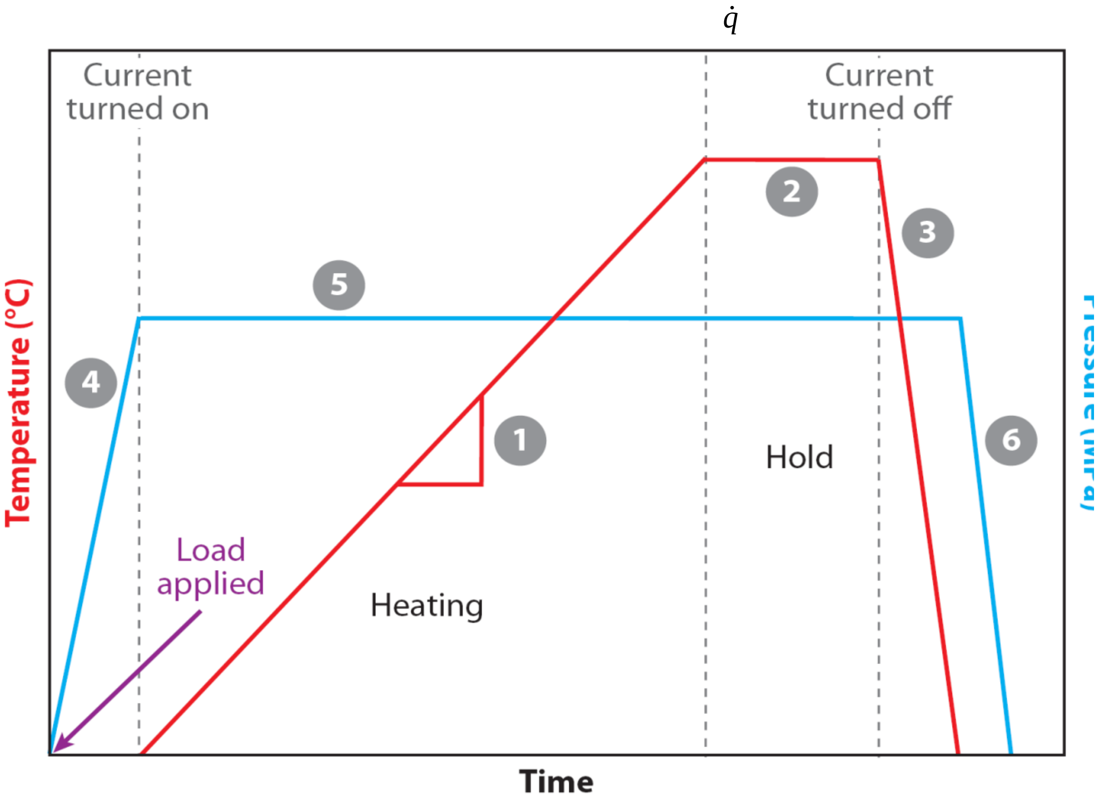
HOW?

1. Better Processing
2. Better Powders

*M. Stuer et al . J.Eur. Ceram Soc. 30 (2010) 1335-1343

SPS/CAPAD - Process Parameters

1. Time, Temperature and Pressure



Powder densification requires mass transfer and is therefore expected to be temperature dependent.
All the heating in CAPAD comes from the flowing current in the form of resistance (Joule) heat with a volumetric heat generation rate, $\dot{q}$, given by

$$\dot{q} = JE$$

where J is the current density and E is the electric field

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Current Density Profiles - SPS

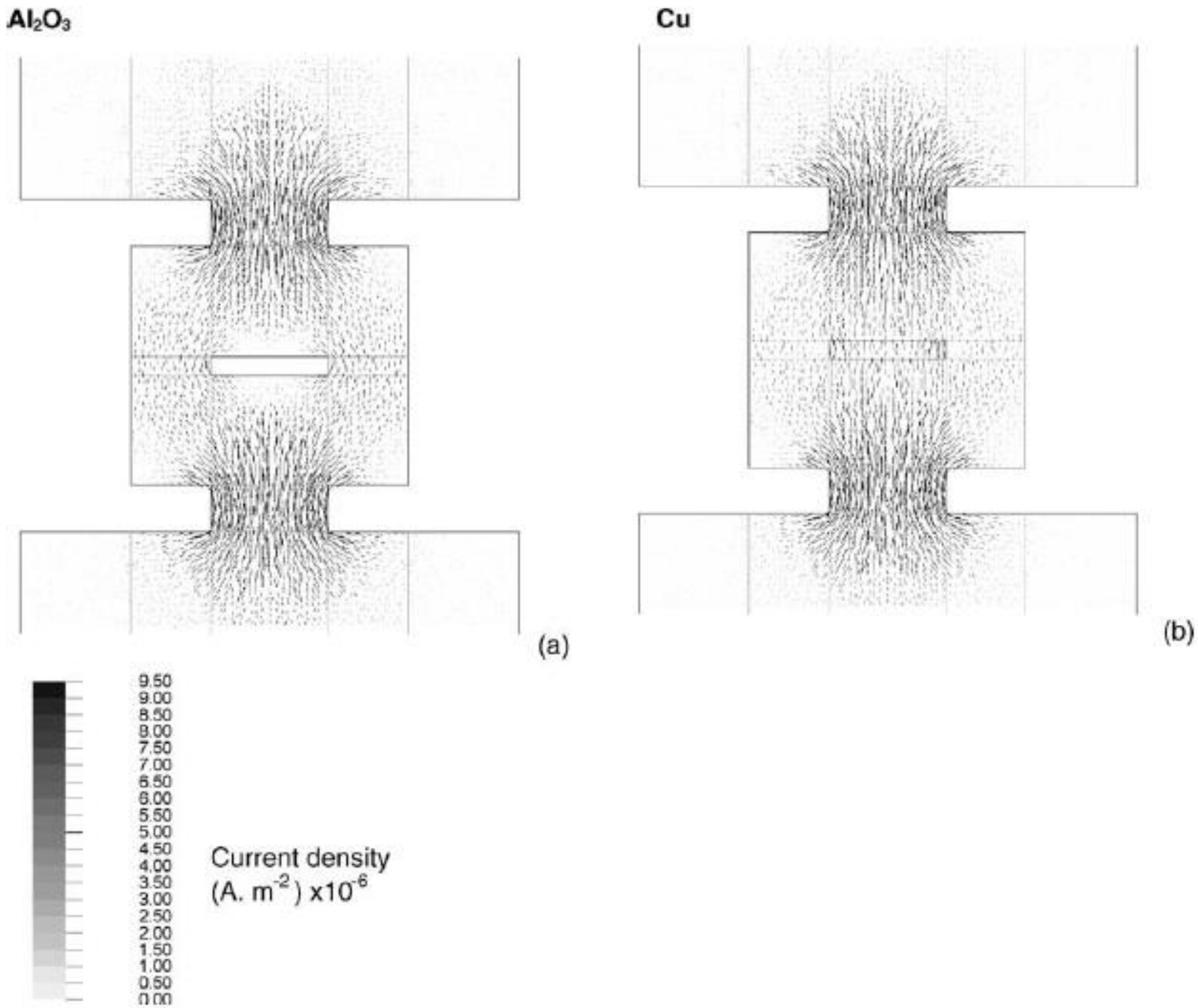
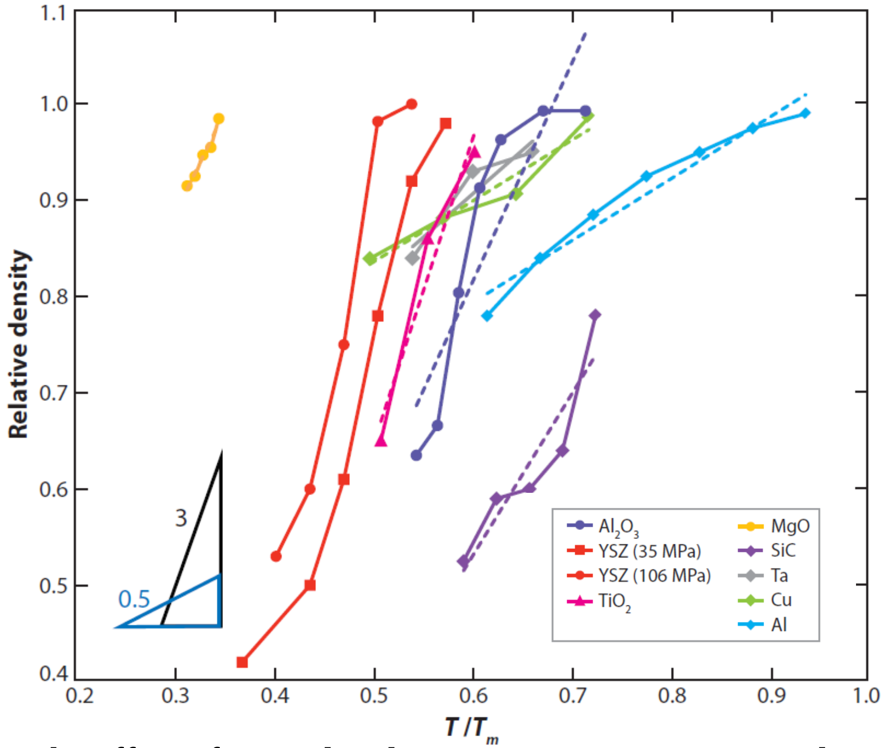


Fig. 3. Current distributions in the SPS die for alumina and copper samples. Applied voltage = 5 V.

U. Anselmi-Tamburini[a,b], S. Gennari[b], J.E. Garay[a], Z.A. Munir[a,*] Mat. Sci.&Eng. A 394 (2005) 139–148

SPS – temperature…sensitivity*

$$\rho = s\left(\frac{T}{T_m}\right) + b$$

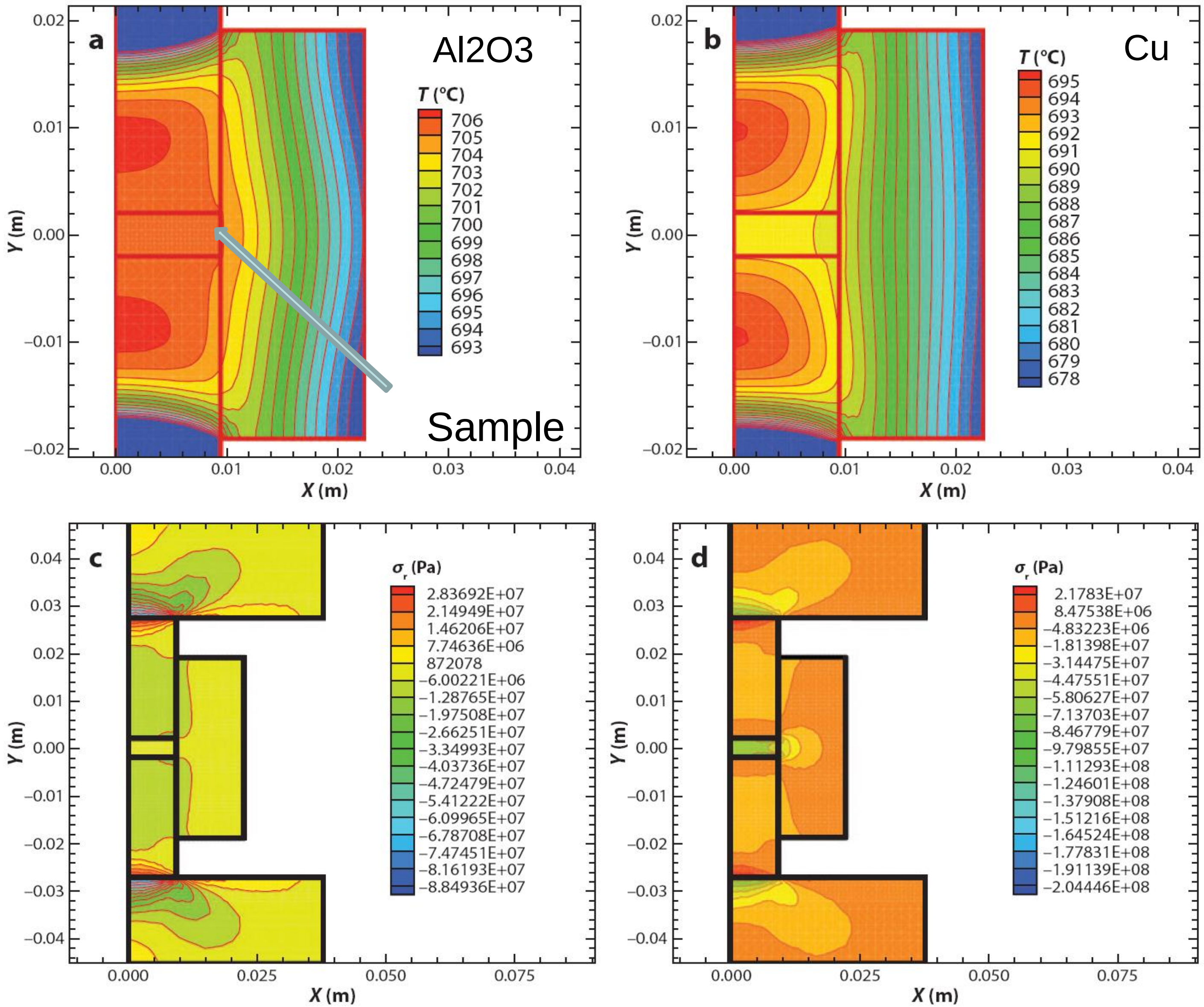


The effect of normalized processing temperature on density for a wide variety of materials

* Garay, J. E., Annu. Rev. Mater. Res. 2010. 40:445–68

- homologous temperature - processing /melting temp (T/Tm)
- ρ is relative density;
- s is the slope = temperature sensitivity;
- b is the intercept on the density axis.
- The function should be sigmoidal (with asymptotes at green and full density)
- The ceramics have similar slopes (~3), as do the metals (~0.5).
- Ceramics, materials with a high degree of ionic bonding fall on the left side of the plot, whereas
- more covalent materials are farther to the right.

Temperature and pressure distribution for Al2O3 and Cu



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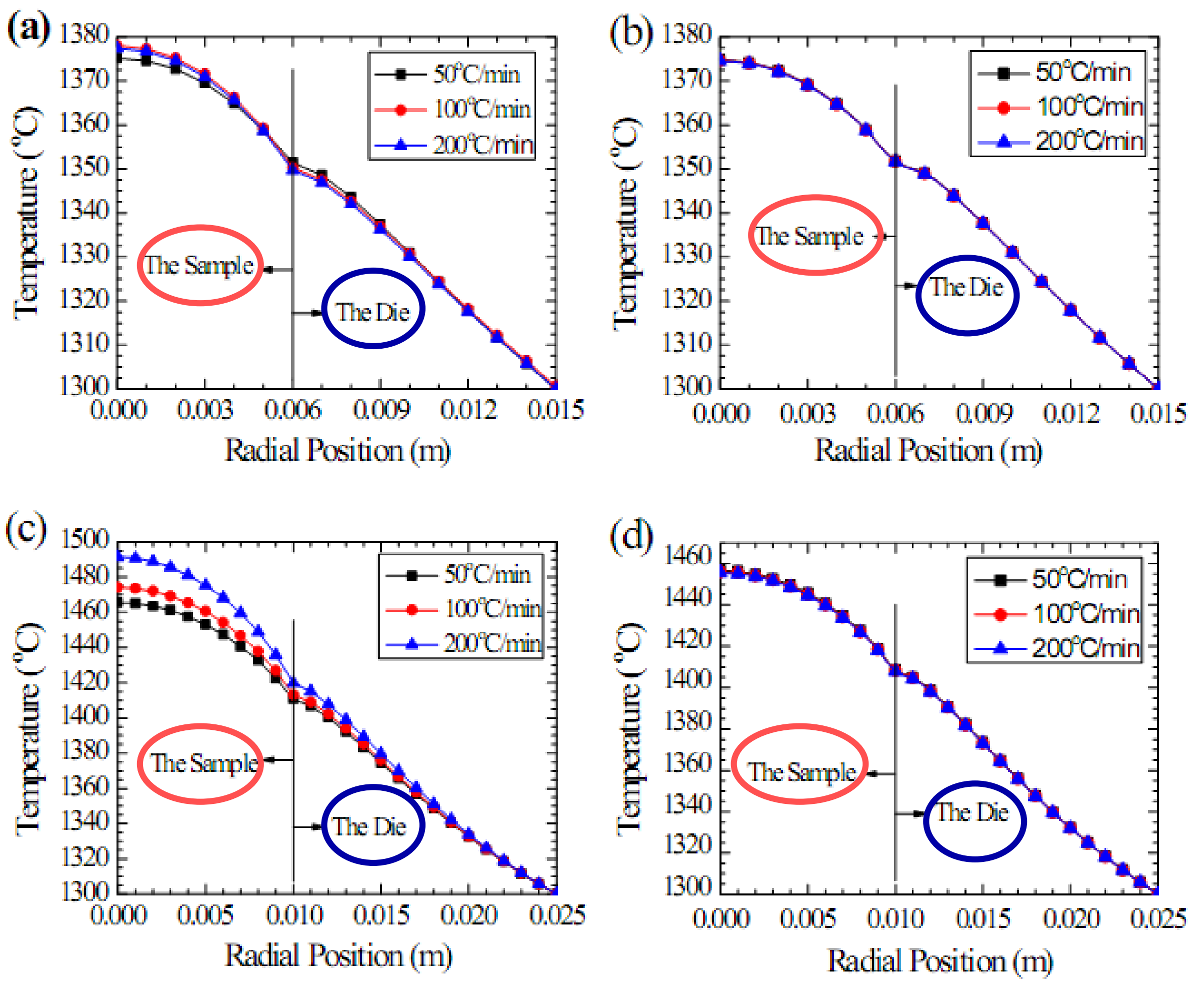
FEM Analysis of Temperature Distribution



The temperature gradient is always there during SPS process

The temperature gradient will be increased for higher heating rate.

The influence of the die size is not linear. – better homogeneity with big dies….for same thickness…mm

Possible densification enhancmenet

- A list of effects (23), categorized into thermal and athermal categories.
- The thermal effects include

(a) high local temperature gradients, which provide conditions for thermal diffusion;

(b) highly non-uniform local temperature distributions, which cause local melting within interparticle contact areas; and

(c) highly non-uniform macroscopic temperature distributions, which create thermal stresses that can cause dislocation creep.

- The athermal factors includes

(a) electroplasticity,

(b) ponderomotive forces,

(c) the electro-magnetic "pinch" effect, and

(d) dielectric breakdown of oxide films, which may cleanse the boundaries.

- Electromigration, or current-enhanced mass transport a well-known effect*,should also be considered in metals.
- The degree to which factors contribute to the CAPAD process is still not known, but investigations are under way…..

[23] Eugene A. Olevsky, Ludo Froyen, J. Am. Ceram. Soc., 92 [S1] S122–S132 (2009)

*J.E. Garay Annu. Rev. Mater. Res. 2010. 40:445–68

THE ELECTROMIGRATION PROCESS

Current flow through a conductor produces two forces to which the individual metal ions in the conductor are exposed:

•The first is an electrostatic force F_{field} *caused by the electric field strength in* the metallic interconnect. Since the positive metal ions are to some extent shielded by the negative electrons in the conductor, this force can be ignored in most cases.

•The second force F_{wind} *is* generated by the momentum transfer between conduction electrons and metal ions in the crystal lattice.

•In a homogeneous crystalline structure, because of the uniform lattice structure of the metal ions, there is hardly any momentum transfer between the conduction electrons and the metal ions.

•Since the metal ions in **grain boundaries** are bonded much more weakly than in a regular crystal lattice, once the electron wind has reached a certain strength, atoms become separated from the grain boundaries and are transported in the direction of the current. This direction is also influenced by the grain boundary itself, because atoms tend to move along grain boundaries.

ISPD '06 Proceedings of the 2006 international symposium on Physical design
ACM New York, NY, USA ©2006 table of contents ISBN:1-59593-299-2 doi>10.1145/1123008.1123017

Enhancement - electromigration

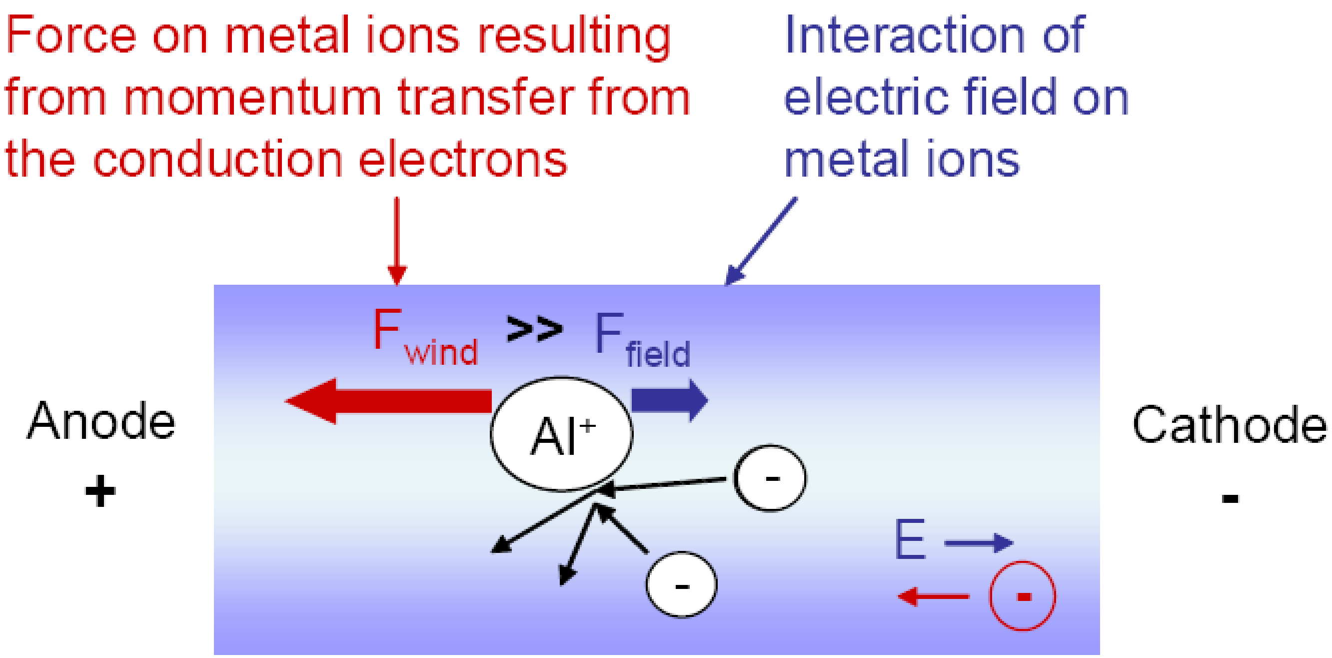


Figure 2. Two forces are acting on metal ions which make up the lattice of the interconnect material. Electromigration is the result of the dominant force, i.e. the momentum transfer from the electrons which move in the applied electric field.

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Enhancement - electromigration

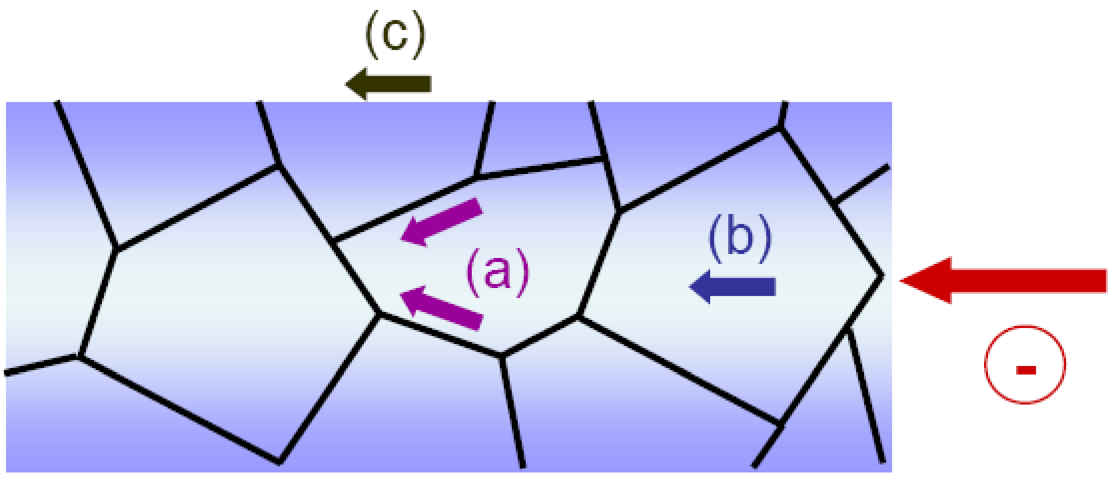

Figure 3. Illustration of various diffusion processes within the lattice of an interconnect: (a) grain boundary diffusion, (b) bulk diffusion, and (c) surface diffusion.

- Diffusion processes caused by electromigration can be divided into grain boundary diffusion, bulk diffusion and surface diffusion.
- In general, grain boundary diffusion is the major migration process in bulk material.

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Enhancment - ponderomotive force - electroplasticity

- In physics, a **ponderomotive force** is a nonlinear force that a charged particle experiences in an inhomogeneous oscillating electromagnetic field.
- The ponderomotive force $\mathbf{F}_p$ is expressed by
  - $$\mathbf{F}_p = -\frac{e^2}{4\,m\,\omega^2}\nabla\mathbf{E}^2$$
- where *e* is the electrical charge of the particle, *m* is the mass, *ω* is the angular frequency of oscillation of the field, and **E** is the amplitude of the electric field (at low enough amplitudes the magnetic field exerts very little force).
- This equation means that a charged particle in an inhomogeneous oscillating field not only oscillates at the frequency of *ω* but also drifts towards the weak field area. (Wikipedia)

Electroplasticity mechanisms:

The influence of the external field appears to be on the migration of vacancies or solute atom-vacancy complexes along grain boundaries to the charged surface

Impact of Thermal Diffusion on Densification During SPS

Eugene A. Olevsky, Ludo Froyen, J. Am. Ceram. Soc., 92 [S1] S122–S132 (2009)

- This paper analyses the influence of thermal diffusion, which is an SPS consolidation enhancement factor of a thermal nature.
- The Ludwig–Soret effect of thermal diffusion causes concentration gradients in two-component systems subjected to a temperature gradient.
- The thermal diffusion-based constitutive mechanism of sintering results from the additional driving force instigated by spatial temperature gradients, which cause vacancy diffusion.
- This mechanism is a commonly omitted addition to the free-surface curvature-driven diffusion (sintering stress) considered in conventional sintering theories.
- The interplay of three mechanisms of material transport during SPS is considered: surface tension- and external stress-driven grain-boundary diffusion, surface tension-and external stress-driven power-law creep, and temperature gradient-driven thermal diffusion.
- It is shown that the effect of thermal diffusion can be significant for ceramic powder systems.
- Besides SPS, the results obtained are applicable to the ample range of powder consolidation techniques, which involve high local temperature gradients.
- The case study fpr SPS conducted on an alumina powder demonstrates the correlation between the modelling and experimental data.

Impact of Thermal Diffusion on Densification During SPS

Eugene A. Olevsky, Ludo Froyen, J. Am. Ceram. Soc., 92 [S1] S122–S132 (2009)

$$\dot{\varepsilon}_x = \bar{\dot{\varepsilon}}^{\mathrm{st}}_{\mathrm{gb}x} + \dot{\varepsilon}_{\mathrm{cr}x} + \bar{\dot{\varepsilon}}^{\mathrm{td}}_{\mathrm{gb}x}$$

$\dot{\varepsilon}_x$ - total shrinkage rate in the x direction

$\bar{\dot{\varepsilon}}^{\mathrm{st}}_{\mathrm{gb}x}$ - macroscopic strain rate corresponding to the surface tension (and external load)-driven grain-boundary diffusion (standard sintering theory)

$\dot{\varepsilon}_{\mathrm{cr}x}$ - x component of the shrinkage rate contributed by power-law creep

$\bar{\dot{\varepsilon}}^{\mathrm{td}}_{\mathrm{gb}x}$ - mesoscopic strain rate corresponding to the temperature gradient driven grain boundary (thermal) diffusion

Grain-boundary diffusion contribution – from surface tension and external load

$$\dot{\bar{\varepsilon}}^{st}_{gbx} = -\frac{9\alpha\delta_{gb}D_{gb}\Omega}{4\pi kTG^4}\frac{\left(\sqrt{\pi}-2\sqrt{\theta}\right)^2}{1-\theta} \times \left\{\left(\sqrt{\pi}-3\sqrt{\theta}\right)\sqrt{\theta} - \frac{2\sqrt{\pi}\theta}{9(1-\theta)^2\left(\sqrt{\pi}-2\sqrt{\theta}\right)}\left(\frac{\bar{\sigma}_x G}{\alpha}\right)\right\}$$

θ	porosity
$\bar{\sigma}_x$	effective (far-field) external stress in the x direction
G	grain size
α	surface tension
δ_{gb}	grain-boundary thickness
Ω	atomic volume
D_{gb}	coefficient of grain-boundary diffusion

Eugene A. Olevsky *J. Am. Ceram. Soc.*, **92** [S1] S122–S132 (2009)

shrinkage rate - power-law creep mechanism

$$\dot{\varepsilon}_{crx} = -\left\{ \frac{\left(\frac{3\theta}{2}\right)^{\frac{m+1}{2}} \left[\frac{3\alpha}{2G}(1-\theta)^2 - \bar{\sigma}_x\right]}{A_0\left(\frac{G}{G_{\text{ref}}}\right)^3 \exp\left(\frac{Q_{cr}}{RT}\right)(1-\theta)^{\frac{m+3}{2}}} \right\}^{\frac{1}{m}}$$

$\dot{\varepsilon}_{crx}$	x component of the shrinkage rate contributed by power-law creep
θ	porosity
$\bar{\sigma}_x$	effective (far-field) external stress in the x direction
G	grain size
Q_{cr}	activation energy for power-law creep
A_0	power-law frequency factor
α	surface tension

Eugene A. Olevsky *J. Am. Ceram. Soc.*, **92** [S1] S122–S132 (2009)

Impact of Thermal Diffusion on Densification During SPS

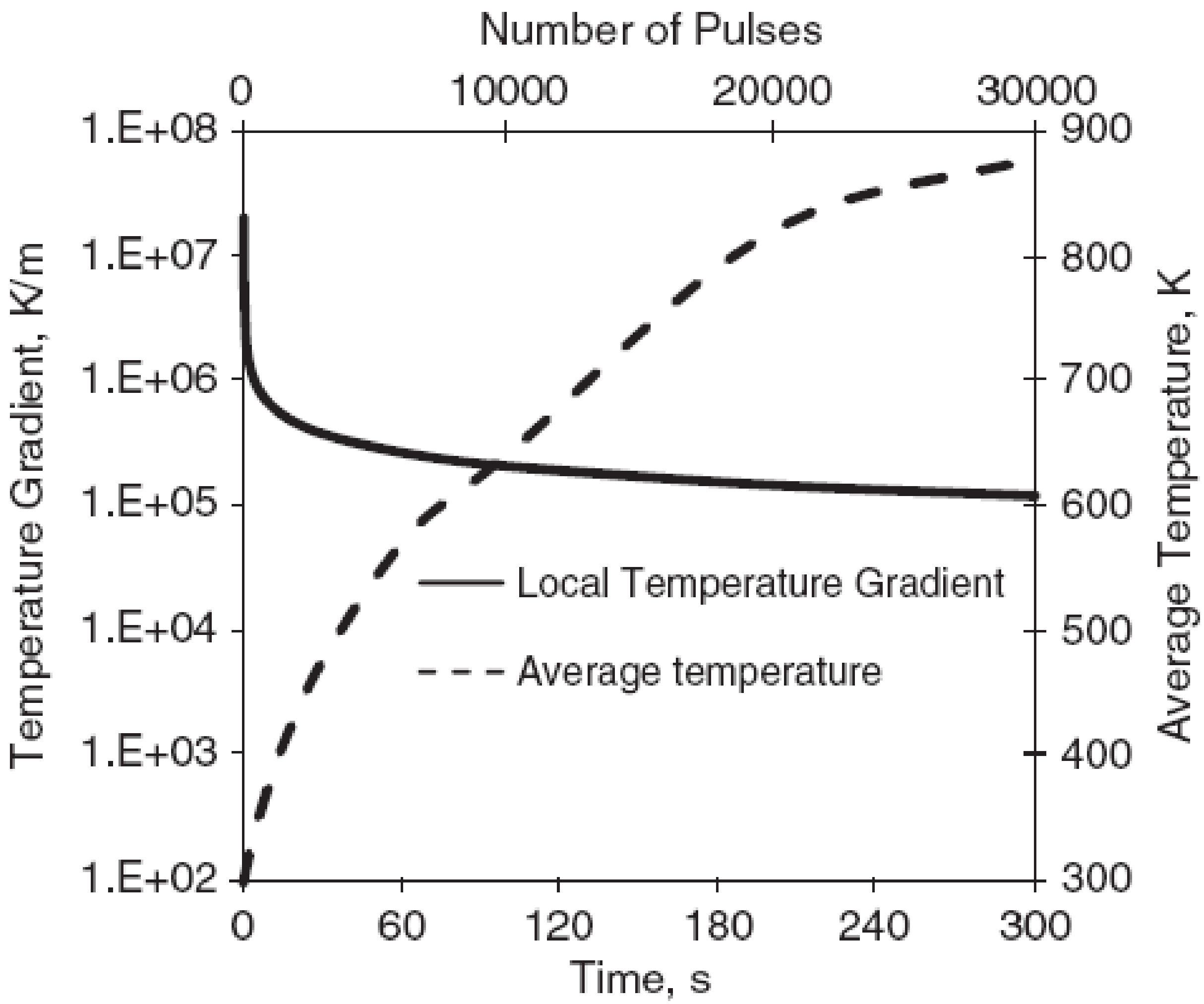
Table I. Material Properties for Alumina Powder

Atomic volume	Ω	4.25×10^{-29} m^3 [95]
Molecular weight	$\hat{A}_{Al_2O_3}$	0.102 kg/mol [95]
Theoretical density	$\rho_{Al_2O_3}$	3980 kg/m^3 [95]
Surface tension	α	1.12 J/m^2 [95]
Grain-boundary diffusion frequency factor	$[\delta_{gb}D_{gb}]_0$	3.00×10^{-3} m^3/s [95]
Activation energy for grain-boundary diffusion	Q_{gb}	477 kJ/mol [95]
Activation energy for power-law creep	Q_{cr}	330 kJ/mol [132]
Power-law creep frequency factor	A_0	2610 MPa s^m [132]
Power-law creep exponent	m	0.333 [132]
Power-law creep reference grain size	G_{ref}	1×10^{-6} m [132]
Electric conductivity (for graphite die)	λ_{efd}	$1.1 \times 10^5 \frac{1}{Ohm \times m}$ [26]
Heat conductivity	λ_{Tfd}	$\frac{6.518 \times 10^7}{8175.85 \times T - 669628.8} \frac{W}{mK}$ [33]
Heat capacity	C_{fd}	$\left(\frac{2.311 \times 10^7 \times T}{249 + T} + 21.6 \times T\right) \frac{J}{m^3 K}$ [33]
Enthalpy of vacancy formation	H_f	129 kJ/mol† [133,134]
Enthalpy of vacancy migration	H_m	367 kJ/mol [113,135]

†Derived for polycrystalline alumina from Fig. 5 and Eq. (7) in Oishi and Kingery.[133]

Eugene A. Olevsky, Ludo Froyen, J. Am. Ceram. Soc., 92 [S1] S122–S132 (2009)

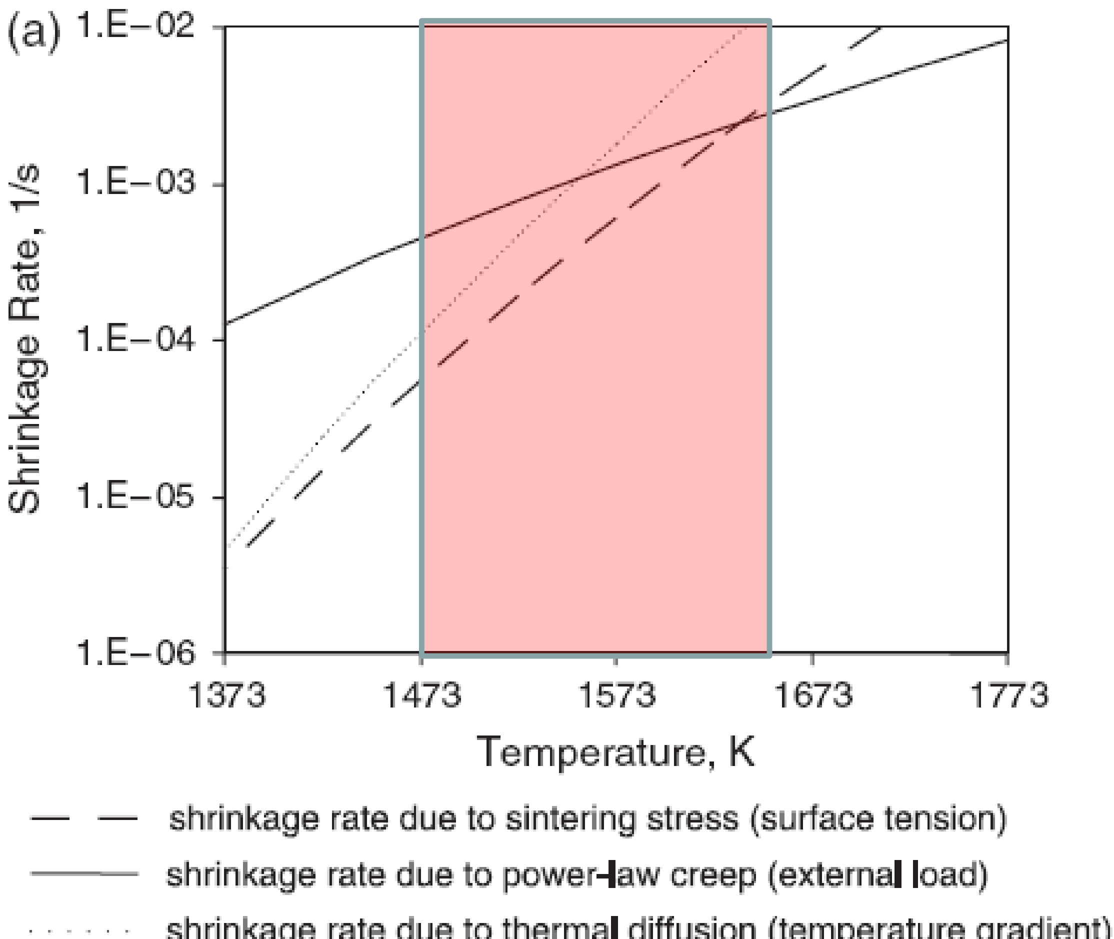
Temperature evolution and the local temperature gradient of an alumina powder specimen subjected to SPS.



Eugene A. Olevsky *J. Am. Ceram. Soc.*, **92** [S1] S122–S132 (2009)

Contribution to shrinkage rate from different mechanisms of mass transport for an alumina powder; G= 0.5 μm;

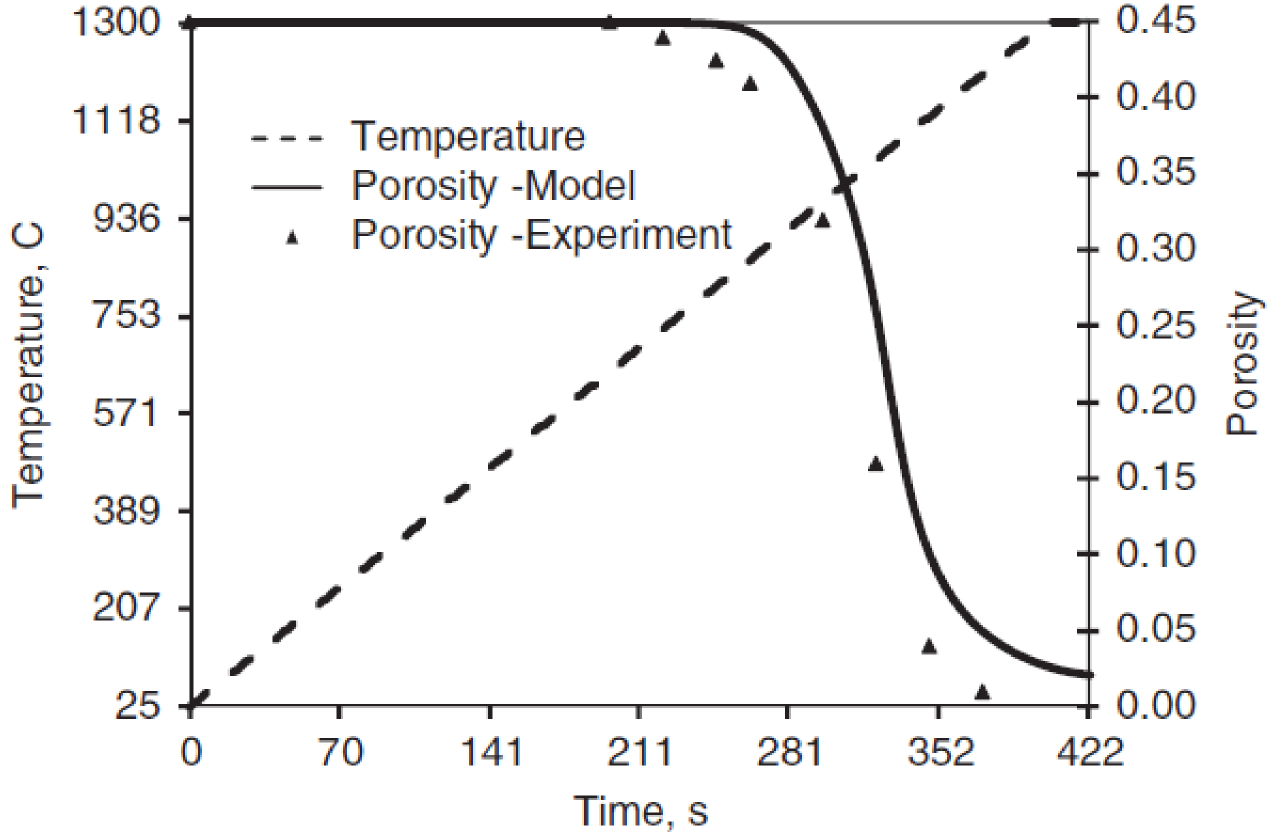
- Results indicate that the effect of thermal diffusion can be significant, especially for small particle sizes.
- Its contribution is considerable not only in the case of free sintering and low pressure-assisted sintering, but it competes with the contribution of external pressure-driven power-law creep within certain temperature ranges.



Eugene A. Olevsky *J. Am. Ceram. Soc.*, **92** [S1] S122–S132 (2009)

Porosity kinetics during SPS of alumina powder.

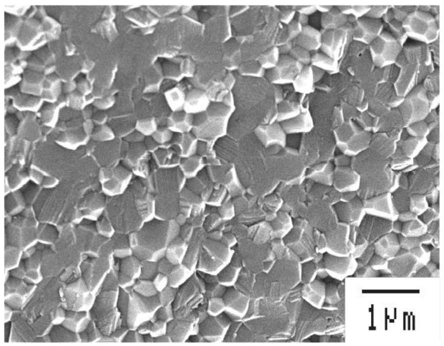
- The thermal diffusion-including model predictions for the shrinkage kinetics are compared with the experiments on SPS of a pure alumina powder and exhibit good qualitative agreement.
- however, the model emphasizes only one factor of a thermal nature—thermal diffusion
- The model developed, thus, should be considered a part of the larger-scale efforts in including various phenomena, listed in this paper….



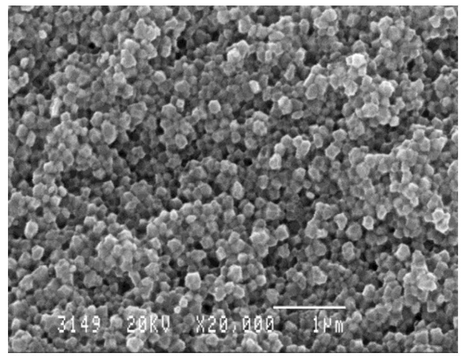
Eugene A. Olevsky *J. Am. Ceram. Soc.*, **92** [S1] S122–S132 (2009)

Transparent Ceramics by SPS

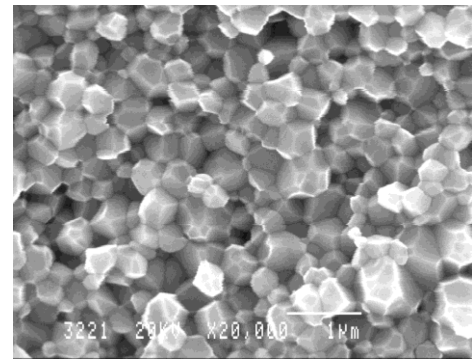
Al_2O_3



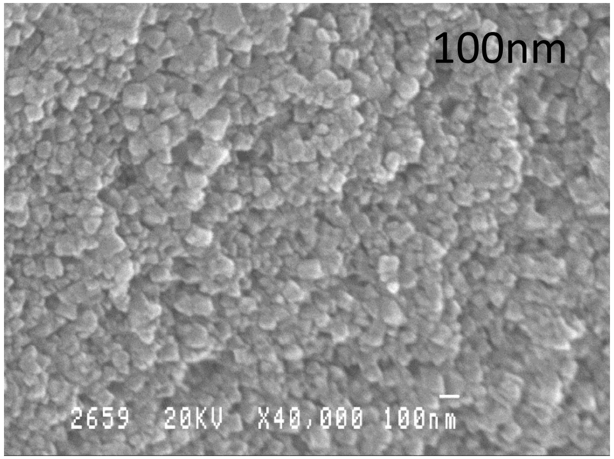
8YSZ

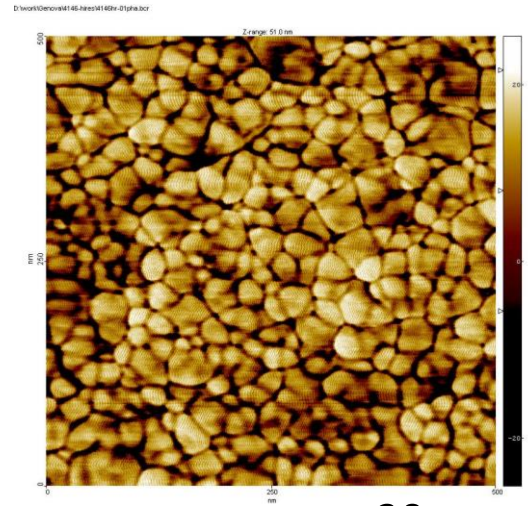


PLZST

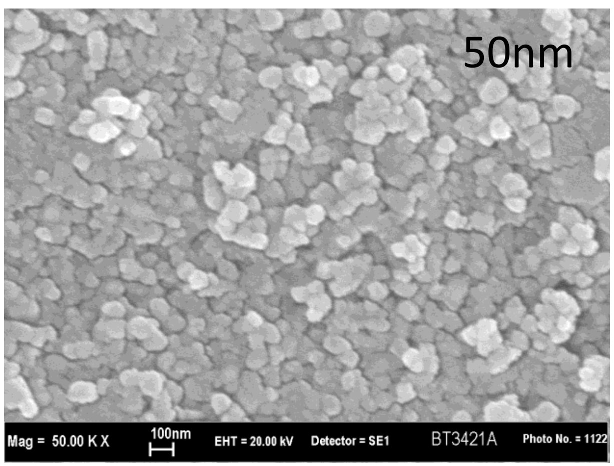


Ferroelectrics_$BaTiO_3$





30nm

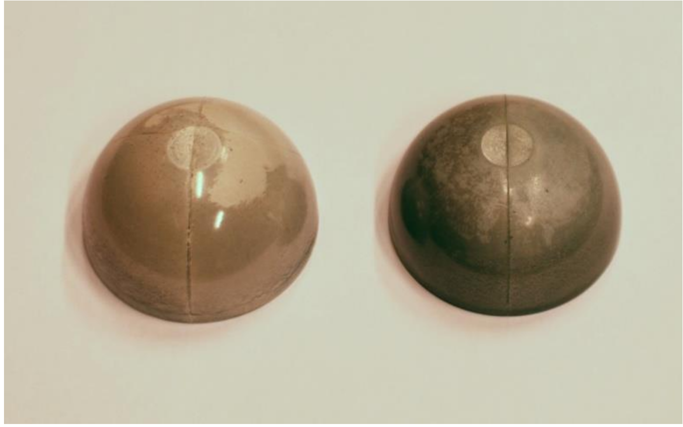


SEM microstructure of samples with grain size of 100nm, 50nm and 30nm. The final microstructure depends on the starting powder.

"Spark plasma sintering of nano-crystalline ceramics", Zhao, Z ; Buscaglia, V ; Bowen, P and Nygren, M , KEY ENGINEERING MATERIALS, Volume: 264-268 Pages: 2297-2300 , 2004

SPS to make complicated shapes

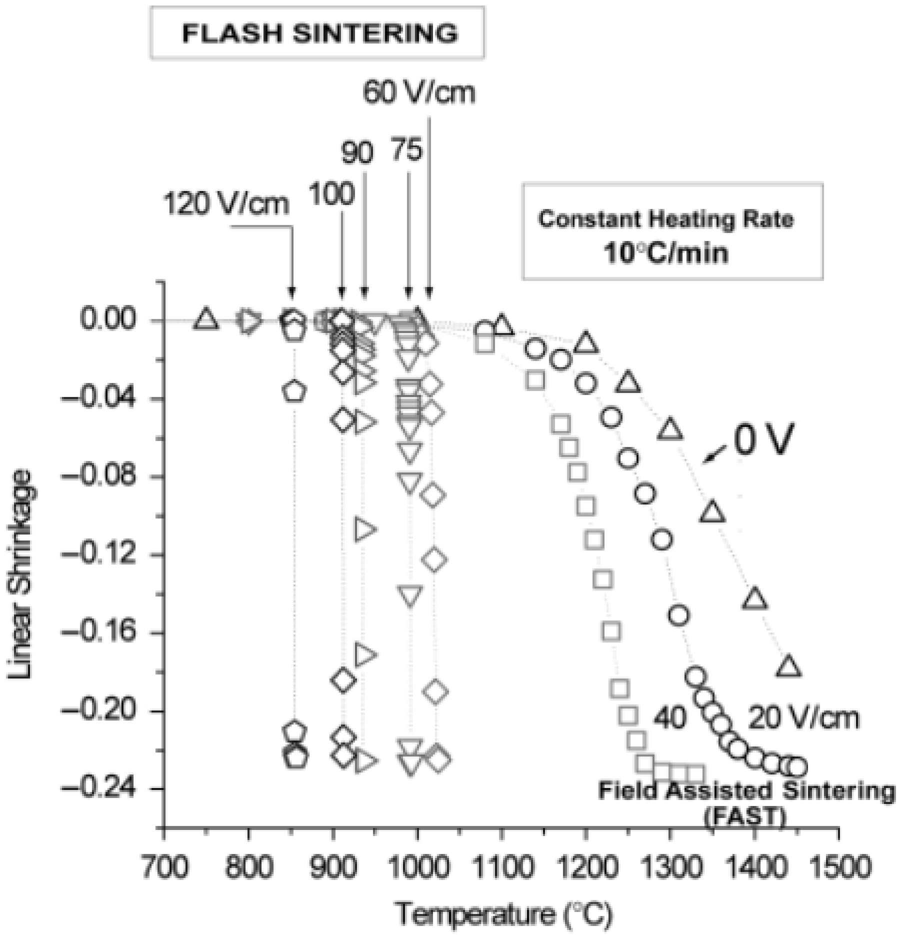
- Silicon-Nitride and Al_2O_3/SiCw

55mm hip joint bowl for medical application

Zhe Zhao, Nanoker report, 2007

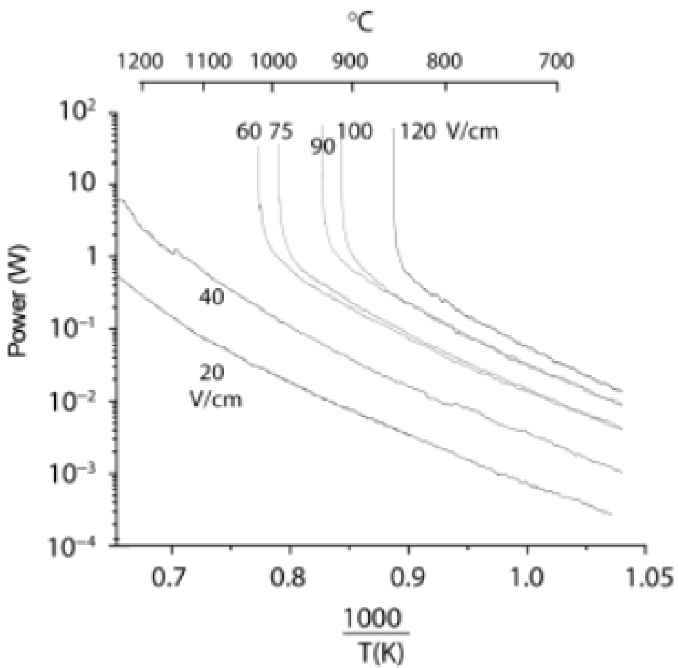
Flash Sintering of TZP

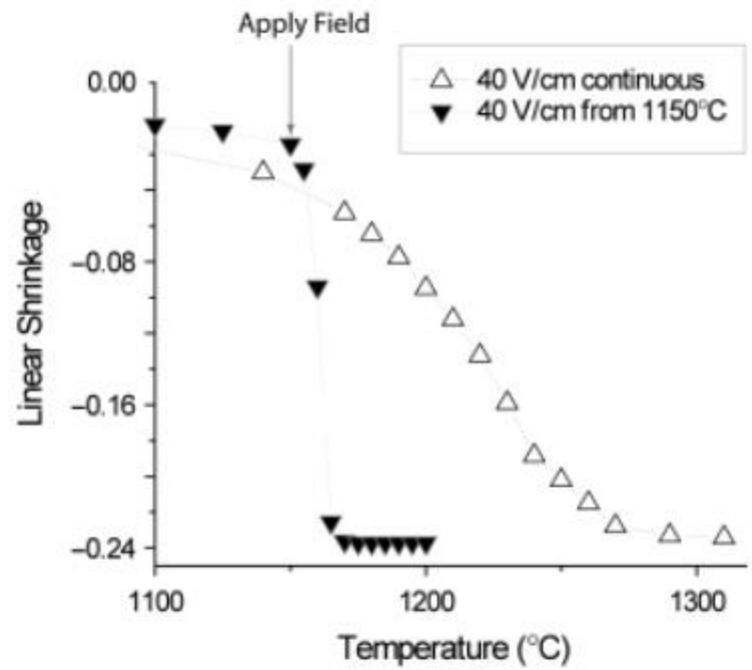


- A threshold electric field intensity is needed for the start of flash sintering
- It's the high local grain boundary temperature leads to flash sintering, which is very different from the conventional Joule heating.
- The local GB temperature is dependent on the relative density.

M. Cologna, B. Rashkova, and R. Raj, "Flash Sintering of Nanograin Zirconia in <5 s at 850°C," J. Am. Ceram. Soc., vol. 93, no. 11, pp. 3556–3559, Nov. 2010.

Flash Sintering of TZP





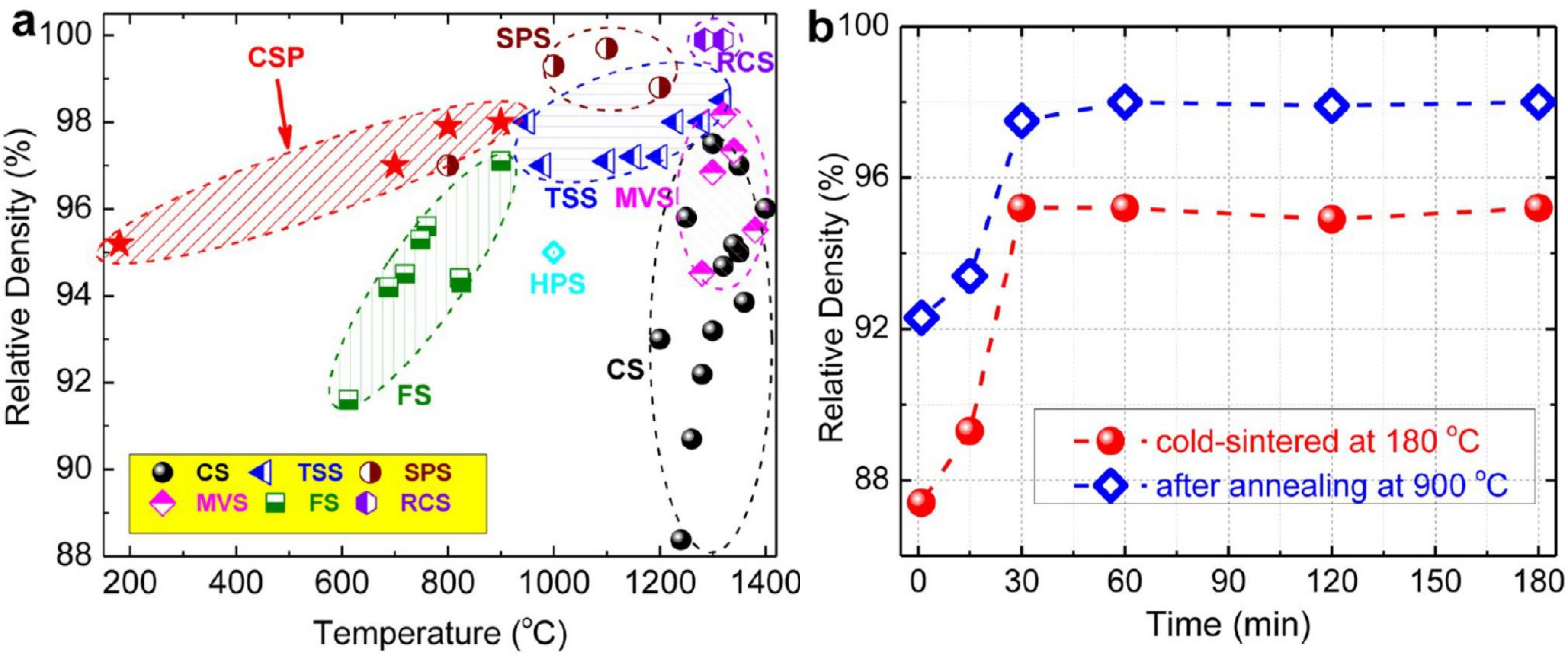
- Power instability is consistent with the onset of flash sintering.
- Instability resulted from the poor neck growth can be of importance
- The "run away" effect mostly contribute the high GB temperature gradient

M. Cologna, B. Rashkova, and R. Raj, "Flash Sintering of Nanograin Zirconia in <5 s at 850°C," J. Am. Ceram. Soc., vol. 93, no. 11, pp. 3556–3559, Nov. 2010.

Flashing Sintering

- Super-Fast processing at relatively lower temperature
- For high resistance material, it will not work that efficiently! For example, high purity alumina.
- The set-up is simply based on conventional sintering furnaces. This is one advantage for industrial interest.
- Not very consistent about the sintering mechanisms.
- Need more development for the future applications.

Cold Sintering Process (CSP)

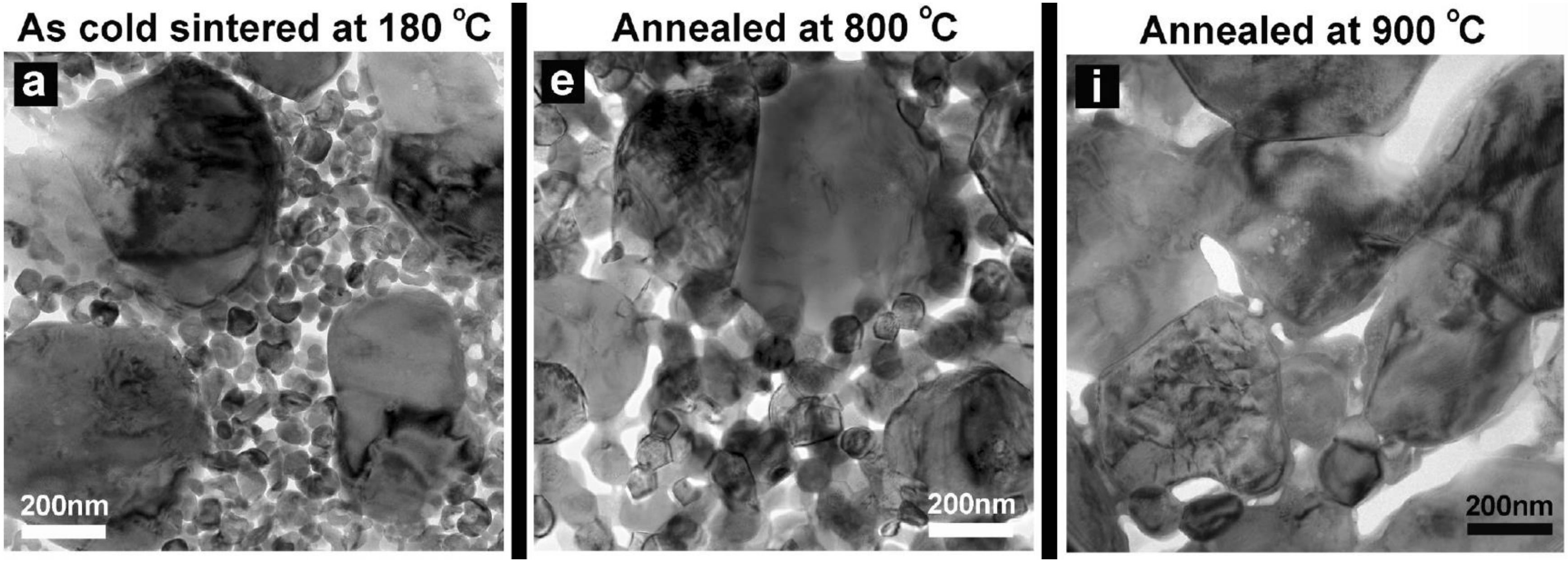


Method : Mixture of submicron and nanoparticles in water – heat - get dissolution of nano-precipitation onto larger particles – Ostwald ripening – same phase….cement precipitates new phase

(a) Summary of relative density-sintering temperature plot for BaTiO3 ceramics in regard to various sintering techniques. CS = conventional sintering; TSS = two-step sintering; RCS = rate-controlled sintering; SPS = spark plasma sintering; MVS = microwave sintering; HPS = high-pressure sintering; FS = flash sintering; CSP = cold sintering process. A theoretical density of 6.02 g cm3 is adopted for BaTiO3 .

(b) Density evolution of cold-sintered and subsequently annealed BaTiO3 ceramics as a function of cold sintering time. *H. Guo, et al, ACS Nano, vol. 10, no. 11, pp. 10606–10614, Nov. 2016.*

Cold Sintering Process (CSP)



- Microstructure very inhomogeneous …not a good ceramic…
- even if 98% dense after annealing at 900°C –
- even if dielectric properties are starting to be interesting
- mechanical properties and lifetimes probably very very low..

H. Guo, A. Baker, J. Guo, and C. A. Randall, "Protocol for Ultralow-Temperature Ceramic Sintering: An Integration of Nanotechnology and the Cold Sintering Process," ACS Nano, vol. 10, no. 11, pp. 10606–10614, Nov. 2016.

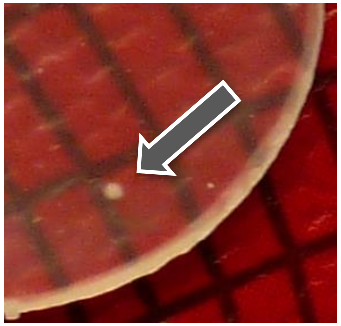
Potentials and Problems of CSP

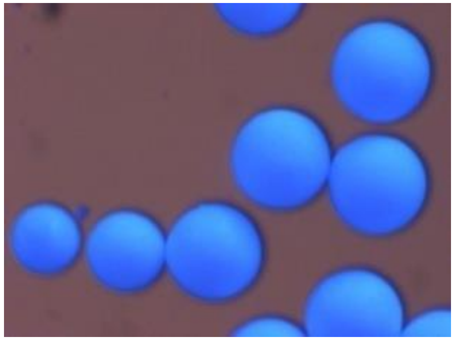
- In total 16 publications (2017)
- 90% of publications come from the same research group led by Prof. Clive Randall!
- No success of obtaining 100% dense body with any reported ceramics, which can be one issue of using the right powder, but more possibly the method itself cant provide good-enough help in crystal packing and chemical bonding between crystal.
- CSP can be good for biomimetic application!
- CSP is still a question mark to be called sintering. If it is true, then cement&concrete and ultra-high pressure compaction of metal are already cold-sintering!

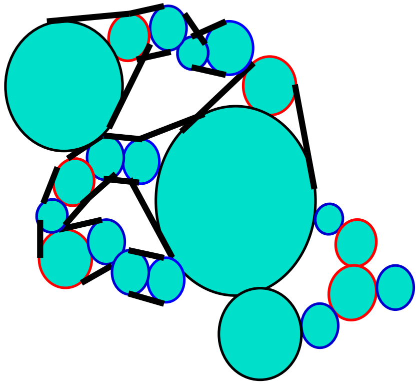
Example

Fundamental Issues in the Processing of Transparent Aluminas : From Interparticle Forces to Dense Transparent Ceramics

Paul Bowen, Michael Stuer



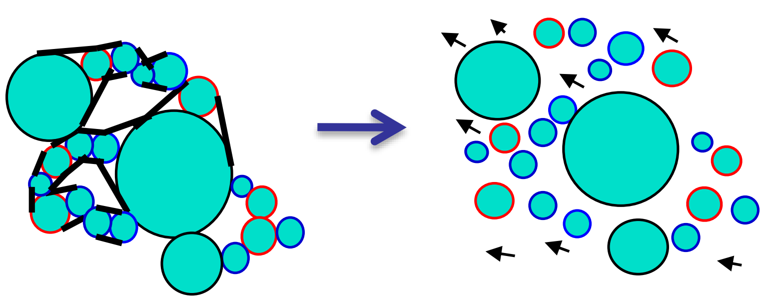




Laboratoire de Technologie des Poudres, Ecole Polytechnique Fédérale de Lausanne, 1015 Lausanne, Switzerland

Plan of Talk

- Introduction – background...
- Ceramic properties – forming methods...why quality powders
- Dispersion & Colloidal stability – Hamaker Programme
- From interparticle forces and particles size distributions to rheological properties
- YODEL – a Yield stress mODEL for concentrated suspensions
- Transparent Polycrystalline Alumina
- Conclusions



Ceramics come in all shapes and sizes

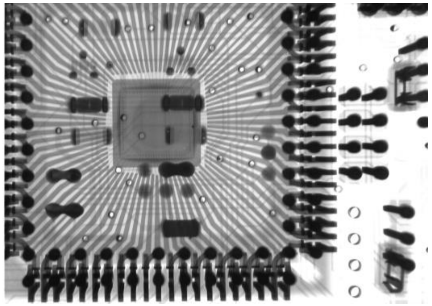
Variety of applications/properties – dictates powder & forming method

Hip joints

Building materials

Automobile – spark plugs

Tableware

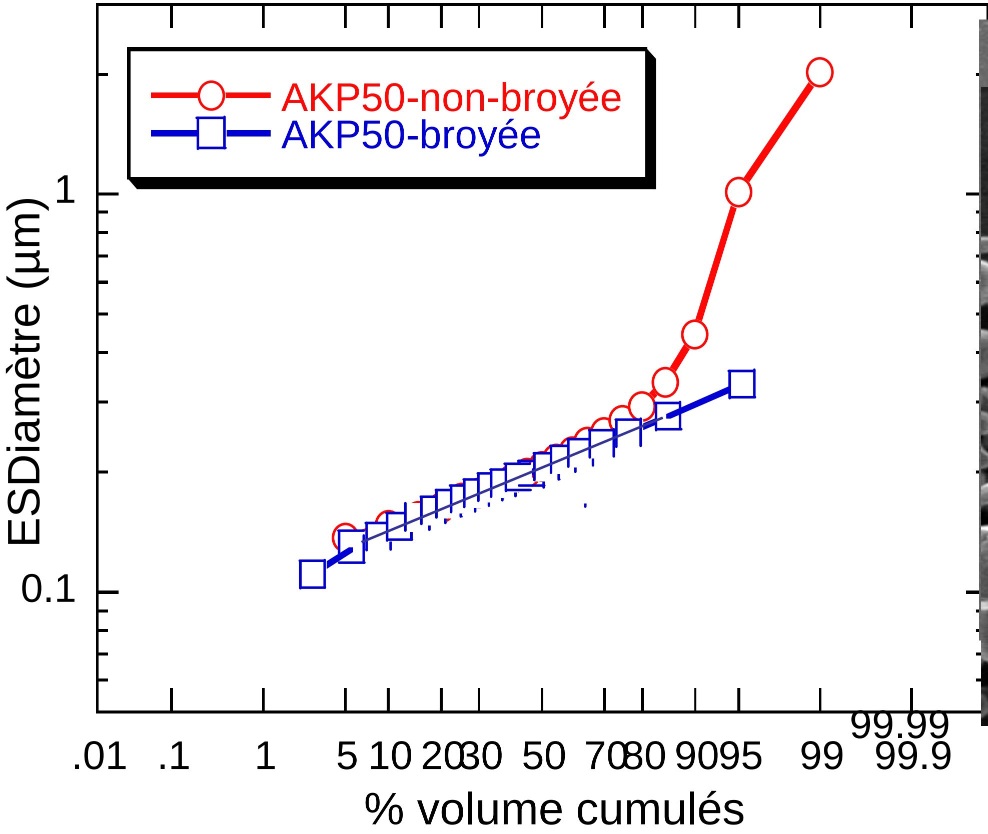


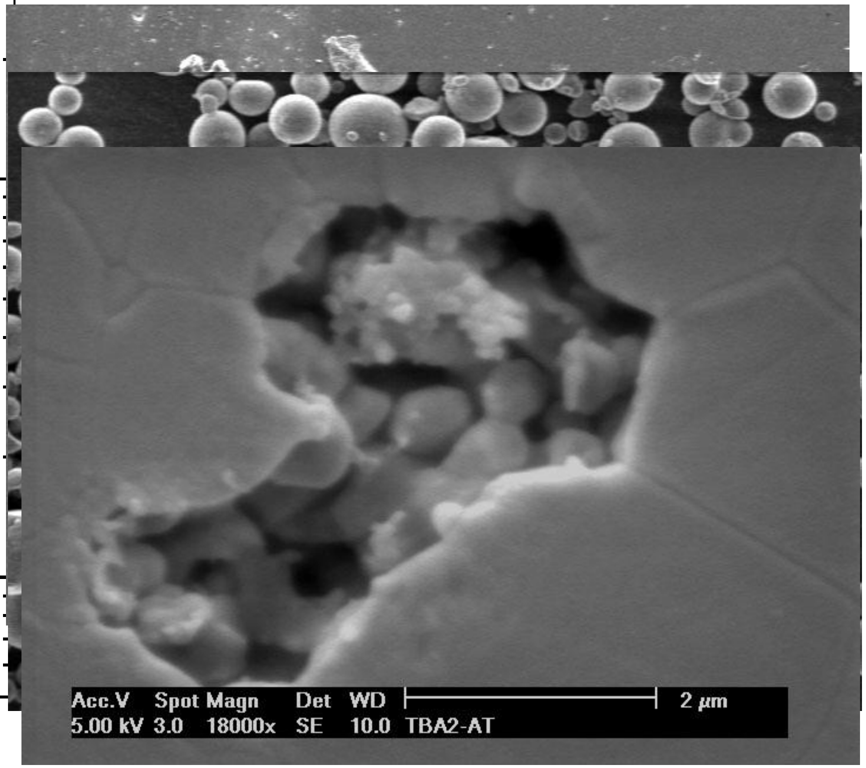
Electronic circuits – the mobile phone – cars…

- **Properties depend on -** microstructures
- Controlled by powder surfaces - grain boundaries after sintering
- Design microstructures – better powders – better processing…?

Why quality powders - Alpha alumina–effect of agglomerates

- Particle size distribution shows small tail of agglomerates – leads to defects in microstructure and low sintered densities (94%)





Powder Influences Microstructure - Properties

Transport properties -

- Electrical, Mechanical, Optical

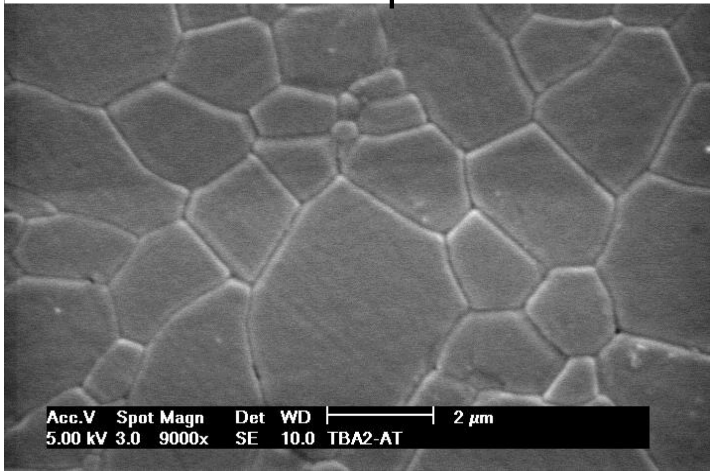
Influenced by - Grain size and

Grain boundary composition

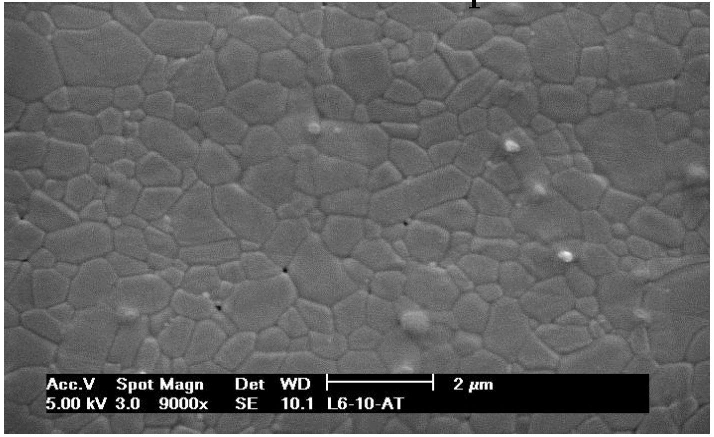
Processing – high quality powder*

- High compact density
- Narrow pore size distribution
- Close pores as late as possible
- Better microstructure
- Understand surfaces and interfaces
- Control microstructure
- Colloidal processing – even dry pressing – need good dispersion for spray drying

As received – slip cast – 94 %



Attrition milled 1hr – slip cast – 99%



*F-S. Shiau, T-T. Fang, T-H Leu, Materials Chemistry and Physics, 57, 33-40 (1998).

ÉCOLE POLYTECHNIQUE
FÉDÉRALE DE LAUSANNE

LTP

A User Friendly Programme for Interparticle Interaction Energy Calculations – Hamaker*.

- Uli Aschauer – Easy to use program - *http://hamaker.epfl.ch*

or

- http://ltp.epfl.ch – Research – Powder Processing – Colloidal Stability

*U. Aschauer, et al. J. Dispersion Science Technology. 32(4), 470 – 479 (2011)

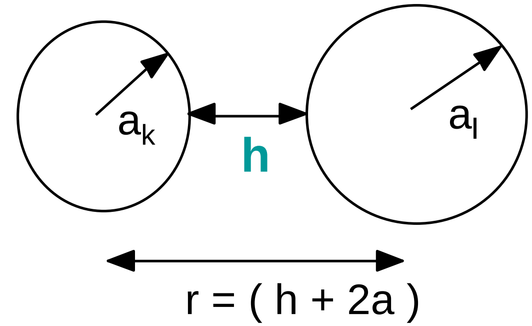
Interparticle Forces - simple to use freeware – Hamaker*

- **Attractive** - dispersion or Van der Waals forces – A(h) – Hamaker constant

$$F_{(a_k,a_l,h)} \cong -A_{(h)} \frac{\bar{a}}{12h^2}$$

Harmonic average radius

$$\bar{a} = \frac{2a_k a_l}{a_k + a_l}$$



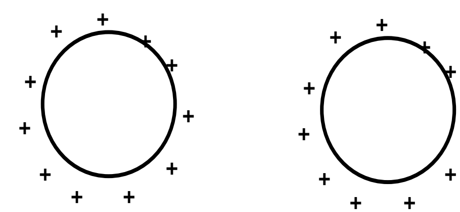
- **Repulsive**

Electrostatic, ion adsorption, dissociation, polyelectrolyte

$$F_{ES} = -2\pi\varepsilon\varepsilon_0 \bar{a}\psi^2 \frac{\kappa e^{-\kappa(h-2L)}}{\left(1+e^{-\kappa(h-2L)}\right)}$$

ψ Electrostatic potential From zeta potential)

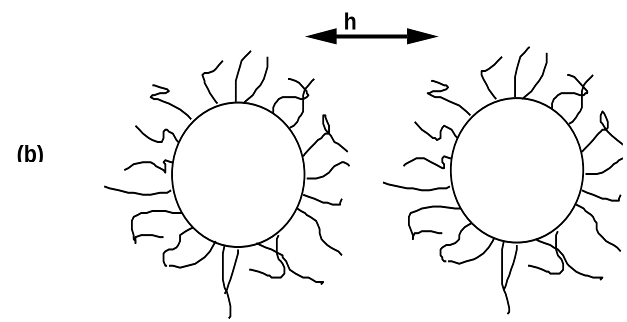
$1/\kappa$ – Electrical double layer thickness



Steric -polymer adsorption – layer thickness

$$F_{ster}\left(a_k,a_l\right) = 2\pi\bar{a}\frac{3}{5}\frac{k_B T}{s^2}\left[\left(\frac{2L}{h}\right)^{5/3} - 1\right]$$

L - Adsorbed layer thickness, s - Spacing of adsorbed molecules
In mushroom configuration – geometry important

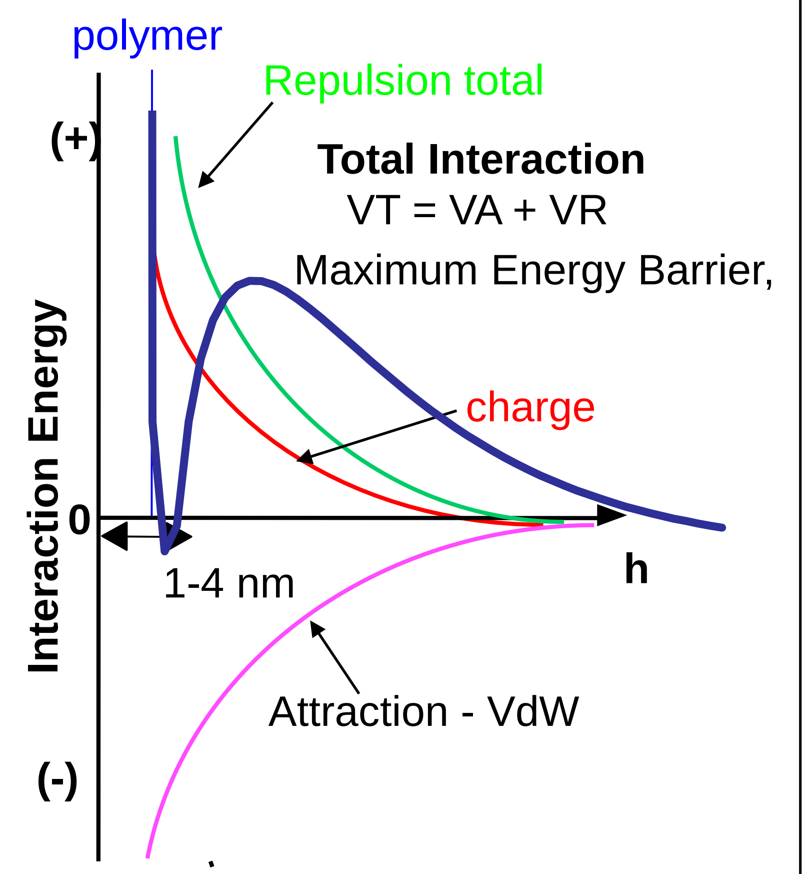


(distance h between particles)

*U. Aschauer, et al J. Dispersion Science Technology, 32(4), 470 – 479 (2011).

Overall Interaction Energy – DLVO*

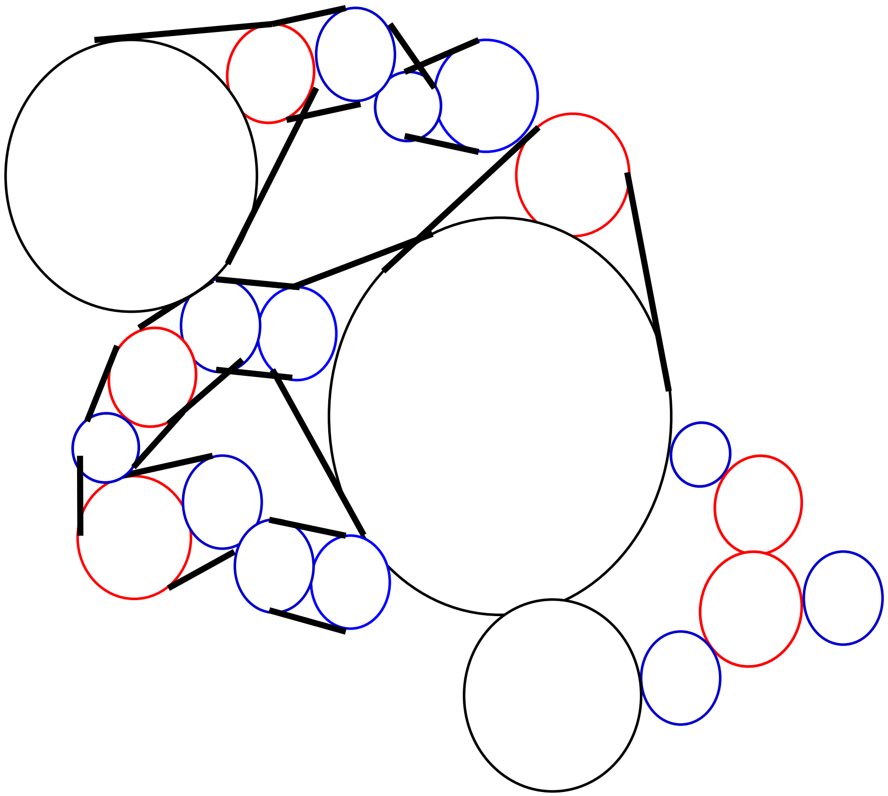
- Net force is algebraic sum of repulsive and attractive forces
- Bergström$ - good qualitative results with alumina & fatty acids
- Not quantitative - used identical spheres - need to use PSD
- Yield stress mODEL (YODEL)# Uses PSD
- Predicts yield point
- Used for cement £ – complex mixture of 4 or more minerals – certain degree of success



$$G_{(\bar{a}_h,h)} \cong F_{Disp} + F_{ES} + F_{Ster}$$

$Bergström, et al J.Am.Ceram.Soc., 75(12) 3305-14 (1992). *Derjaguin & Landau - Vervey & Overbeck
#Flatt&Bowen, J. Am. Ceram. Soc., 89 [4] 1244–1256 (2006), £Houst et al 38 1197–1209 (2008), Perrot et al Cem.&Conc.Res. 42 (2012) 937–944, Palacios et al Mater. de Construcción, 489-513, 62(308), 2012

Taking into account Particle Size Distributions (PSD)



- Suspension may form an attractive network - yield stress
- To flow have to break ”pairs”
- Reduces the effective volume fraction
- To predict - need all the possible pair interactions as a function of zeta potential, adsorbed layer thickness, PSD etc....
- Suzuki & Oshima* statistical model

Total Interaction Force

$$G_{(\bar{a}_h, h)} \cong F_{Disp} + F_{ES} + F_{Ster}$$

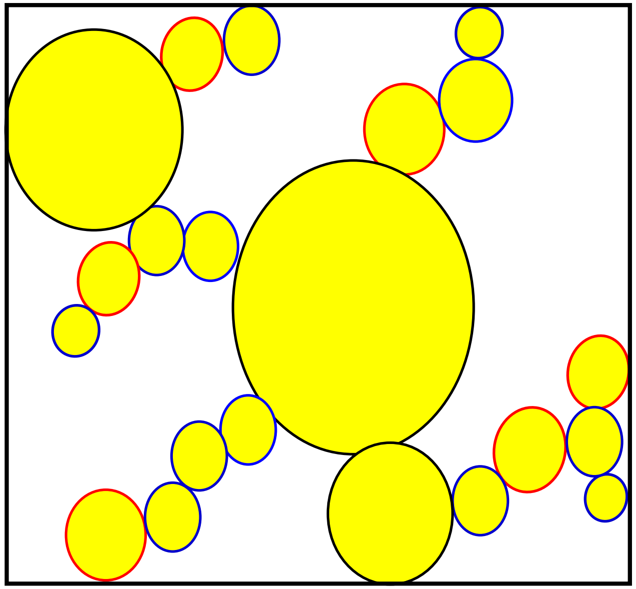
All forces – function of harmonic radius

$$\bar{a} = \frac{2a_1a_2}{a_1 + a_2}$$

(*M. Suzuki, T. Oshima, Estimation of the coordination number in a multicomponent mixture of spheres, *Powder technology*, 1983, 35, pp. 159-166)

YODEL - Effective volume - aggregates

- No. of "bonds" – coordination number from packing models
- Strength of bond from interparticle force calculations
- Certain no. of "bonds" break under a certain shear
- How does effective volume of solids change?



Robert J. Flatt, Paul Bowen, J. Am. Ceram. Soc., 89 [4] 1244–1256 (2006)
Yodel: A Yield Stress Model for Suspensions

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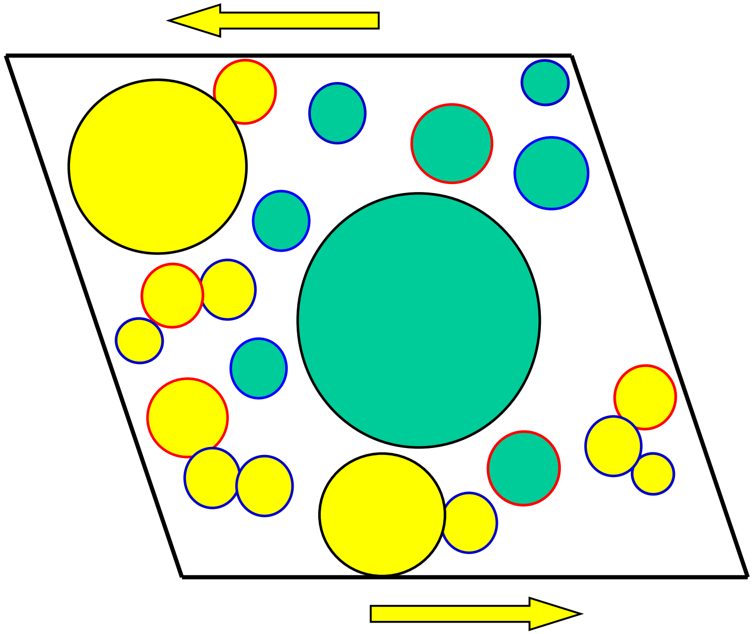
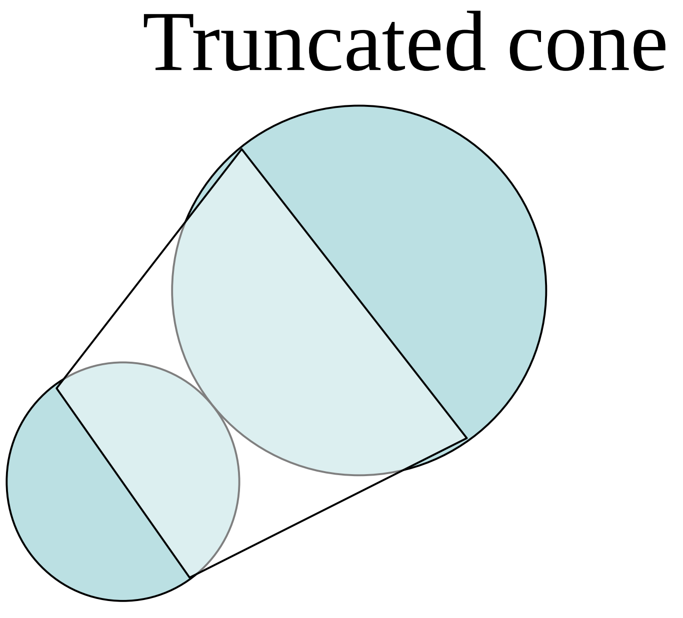
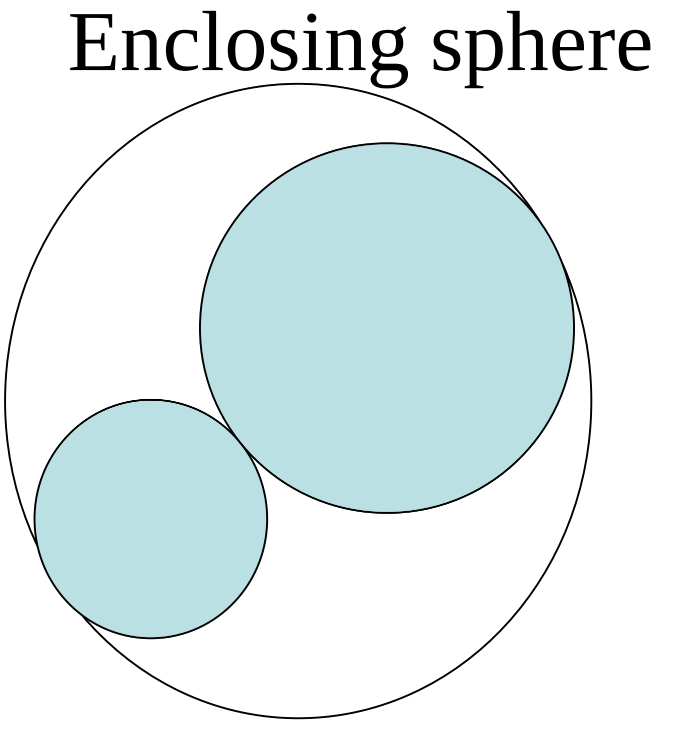


Robert J. Flatt, Paul Bowen, J. Am. Ceram. Soc., 89 [4] 1244–1256 (2006)
Yodel: A Yield Stress Model for Suspensions

YODEL - Increased effective volume - aggregates

- Several geometries looked at – some minor differences but all give same general trends
- Best fit to alumina slurries – Enclosing sphere model





Robert J. Flatt, Paul Bowen, J. Am. Ceram. Soc., 89 [4] 1244–1256 (2006)
Yodel: A Yield Stress Model for Suspensions

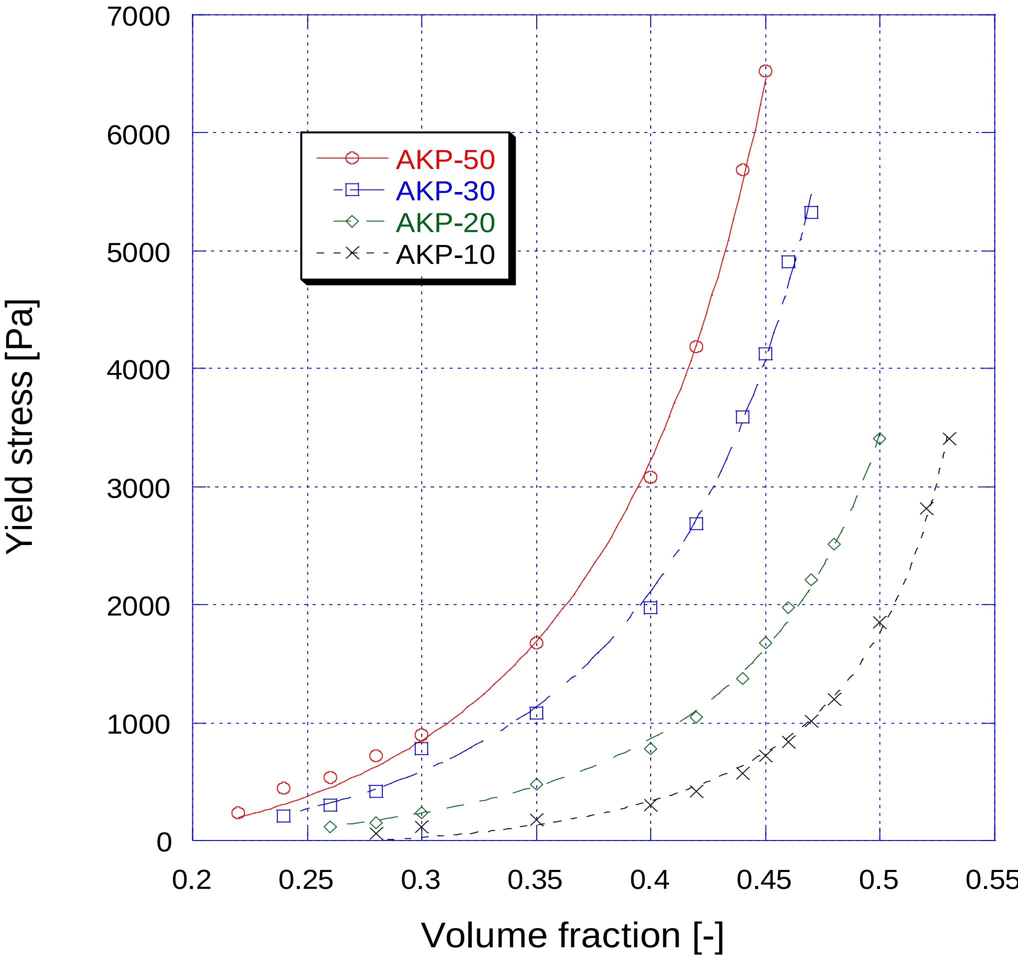
YODEL - Volume fraction functionality

- Model validated with data from attractive network - careful study on alumina slurries near the isoelectric point$
- Yield stress, τ, as a function of volume fraction (ϕ) and maximum packing fraction (ϕ_σ) , percolation threshold ϕ_0

$$\tau = m_1 \frac{\phi(\phi - \phi_0)}{\phi_\sigma^*(\phi_\sigma^* - \phi)}$$

Factor, m_1 includes:

- particle size (a)
- particle size distribution
- interparticle force, G (a,h)
- distance of closest approach, H
- radius of curvature of contact, a*

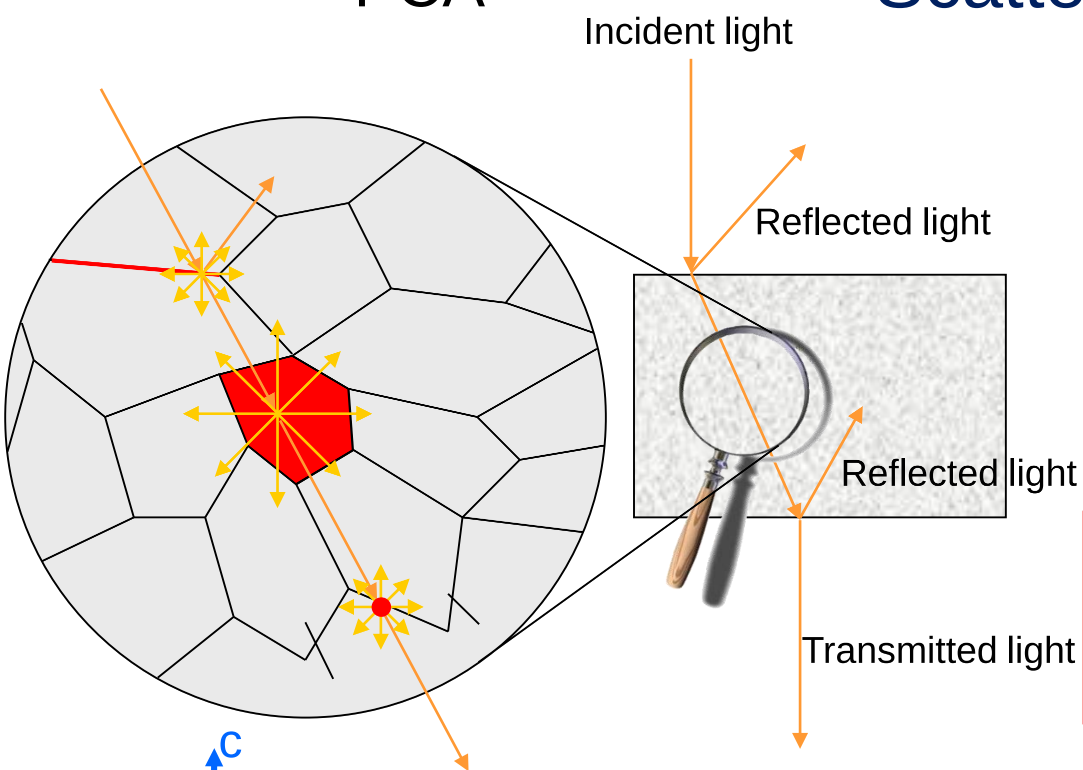


$Zhou, Z., Solomon, M. J., Scales, P., Boger, D. V. - J. Rheol. 43(3) 651-671(1999)

Transparent Polycrystalline Alumina - General Context

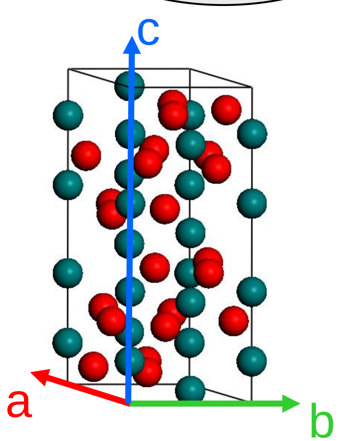
PCA

Scattering sources



- Grain boundaries
- Grains themselves
- Sample surfaces
- Pores / precipitates

Sapphire
Real In line Transmittance
RIT = 86%

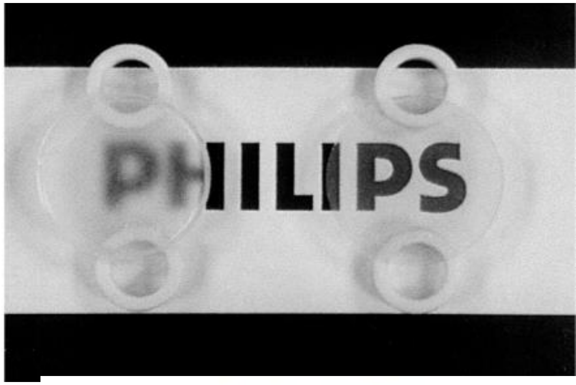


- Hexagonal lattice
  - $n_a = n_b = 1.760$
  - $n_c = 1.768$
- Birefringent

- $\Delta n = [\ 0.0,\ \Delta n_{max} = |n_a - n_c|\ ]$
- $\Delta n_{max} = 0.008$

Transparent Polycrystalline Alumina – applications

- Apetz & van Bruggen *
    - Real–In-line Transmission (RIT)
    - PCA - 50-60% (70%)
    - Krell * “careful” colloidal Processing
- sinter 95% density close porosity – post HIP

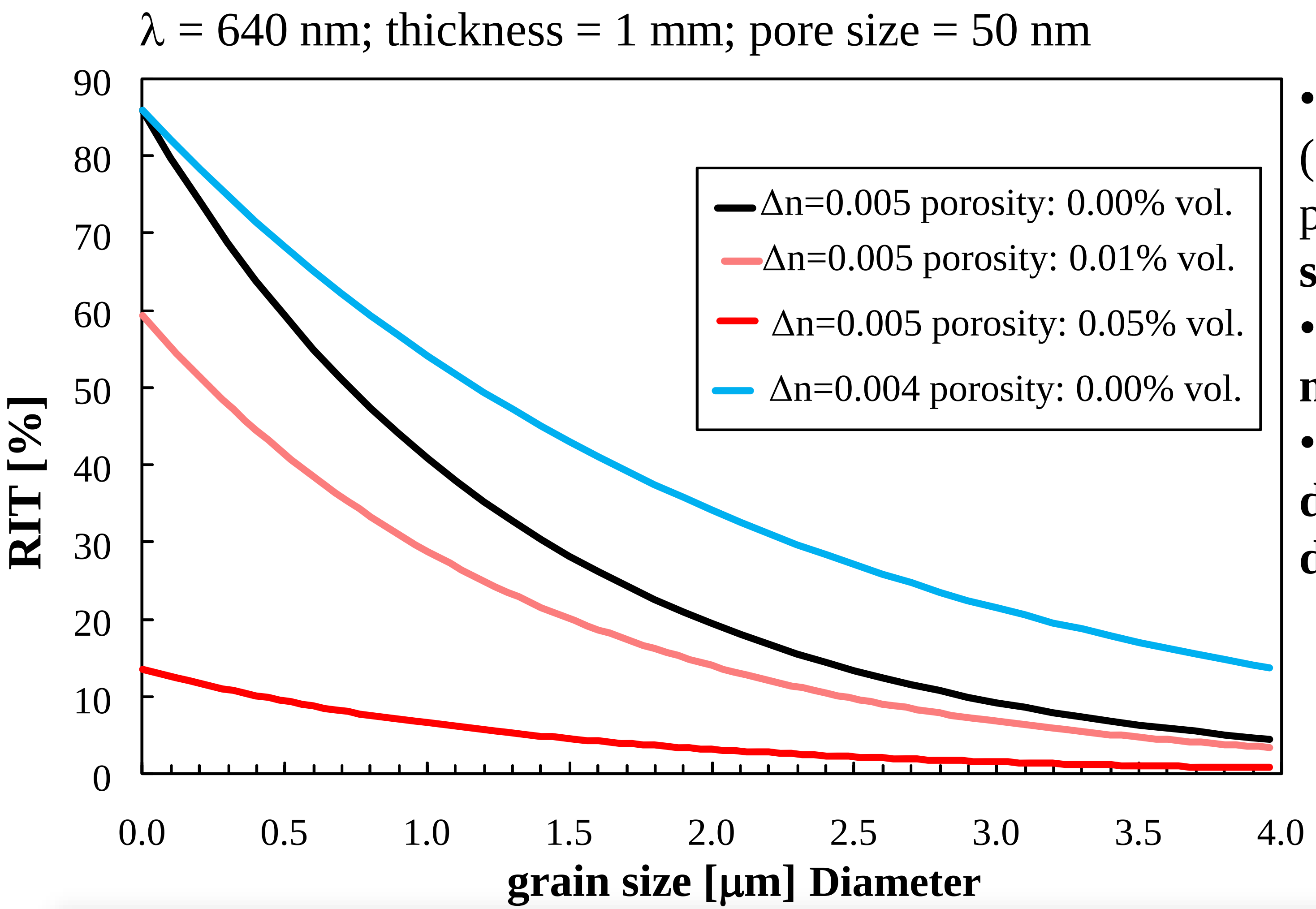


- Swiss Watch industry needs >80%
- Why polycrystalline
    - Easier to shape than sapphire - “hard” work!!

- How can we provide the required microstructures?
    - Reduce grain growth
    - Avoid second phases (dopants) and porosity
    - Can we do it using SPS – can we do it by dry pressing?

**R. Apetz & M.P.B. van Bruggen, J.Am Ceram.Soc. 2003, *A. Krell & J.Klimke J. Am. Ceram. Soc.(2006)*

Transparent PCA - Light Scattering Theory



- <Δn> (alignment) and porosity affect **shape of curve**
- Porosity reduces **maximum RIT**
- **But very difficult to verify density > 99.8%**

To improve the real in-line transmittance (RIT), one needs:

FULL DENSIFICATION + GRAIN ALIGNMENT

AND/OR SMALLER GRAINS

Effects of sintering parameters

Private thesis defense, 2012.

PECS(SPS) parameters for 450 ppm Mg-doped Al_2O_3:

Sample name:	A	B	C	D
Heating rates [°C/min]:	**100**	**233**	**350**	**100**
Sintering T° [°C]:	1300			
Dwell time [min]:	5			
Pressure [MPa]:	100			
Temperature of pressure application [°C]:	**600**	**600**	**600**	**1200**
Total cycle duration [min]:	**15**	**11**	**10**	**15**

Note:

- **Sample D**: sintering pressure applied 1 min before reaching final sintering temperature (during heating ramp)
- **Other samples**: Pressure applied during the whole sintering cycle

SWISS NATIONAL SCIENCE FOUNDATION
FNSNF

*M.Stuer, C. Pecharroman, Z. Zhao, M. Cantoni, P. Bowen " Adv. Funct. Mat.. 22(11) 2303 (2012).

Effects of sintering parameters

Private thesis defense, 2012.
Swiss National Science Foundation

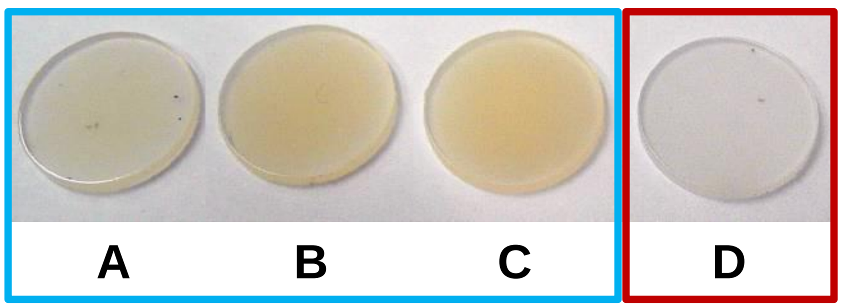
Optical properties after sintering:

Sample name:	A	B	C	D
RIT [%]:	**53.6** ± 0.5	**50.8** ± 0.5	**48.9** ± 0.5	**53.7** ± 0.5
[1]Grain size [µm]:	**0.69** ± 0.02	**0.71** ± 0.02	**0.70** ± 0.02	**0.69** ± 0.02

[1]by line intercept with a correction factor of 1.56

- **Samples A, B and C**: RIT drops slightly with increasing heating rate

After thermal treatment @ 1150° C for 30 minutes:



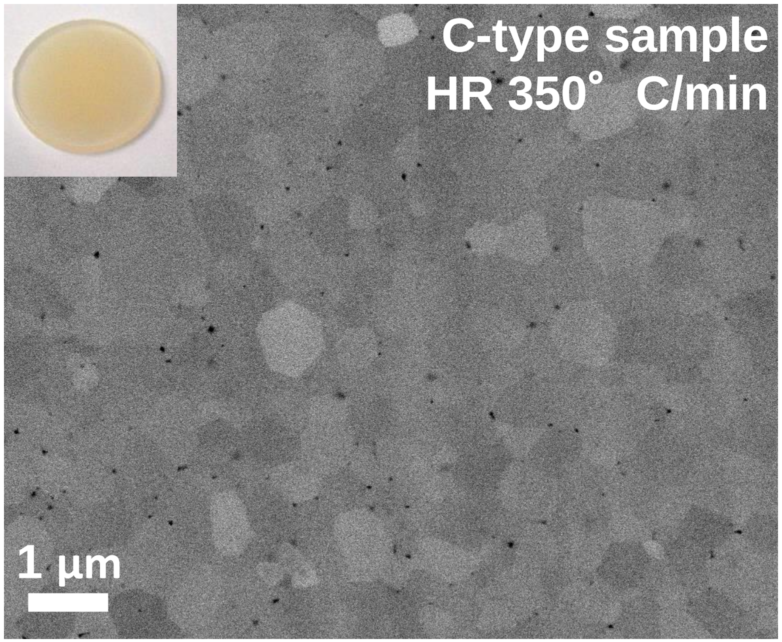
Moment of pressure application is critical if samples exposed to high temperatures!

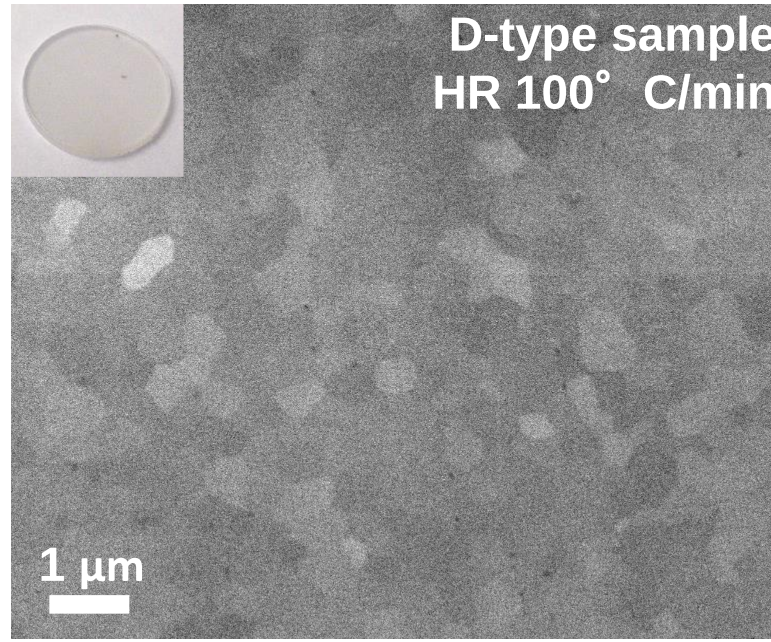
What is the origin of the coloration?

Origins of coloration

Private thesis defense, 2012.

BSE-SEM images after cross-section ion polishing:





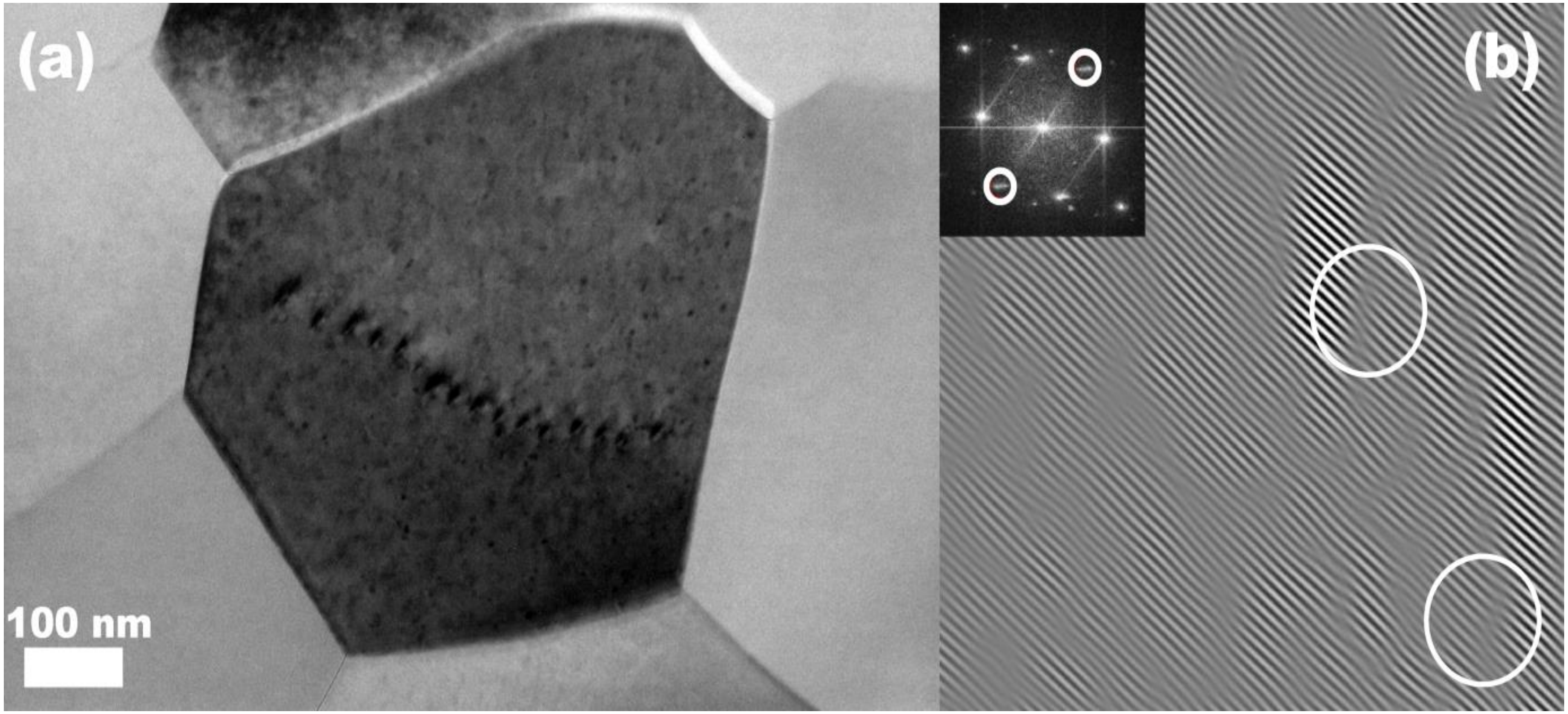
RIT drop and coloration due light scattering by pores formed during thermal treatment:

- For C-type sample porosity appears at triple points
- For D-type sample hardly any porosity can be observed

The pore fraction must be determined and characterized!

Swiss National Science Foundation

Dislocations at high heating rates 350° C/min



TEM image of sample C (cf. Fig. 1) before thermal treatment. The image shows a dislocation wall within the grain, hypothesized to form pores during a subsequent thermal treatment. (b) FFT analysis of a high resolution TEM image showing the presence of defects (dislocations) from irregularities in the periodicity

*M.Stuer, C. Pecharroman, Z. Zhao, M. Cantoni, P. Bowen " Adv. Funct. Mat.. 22(11) 2303 (2012).

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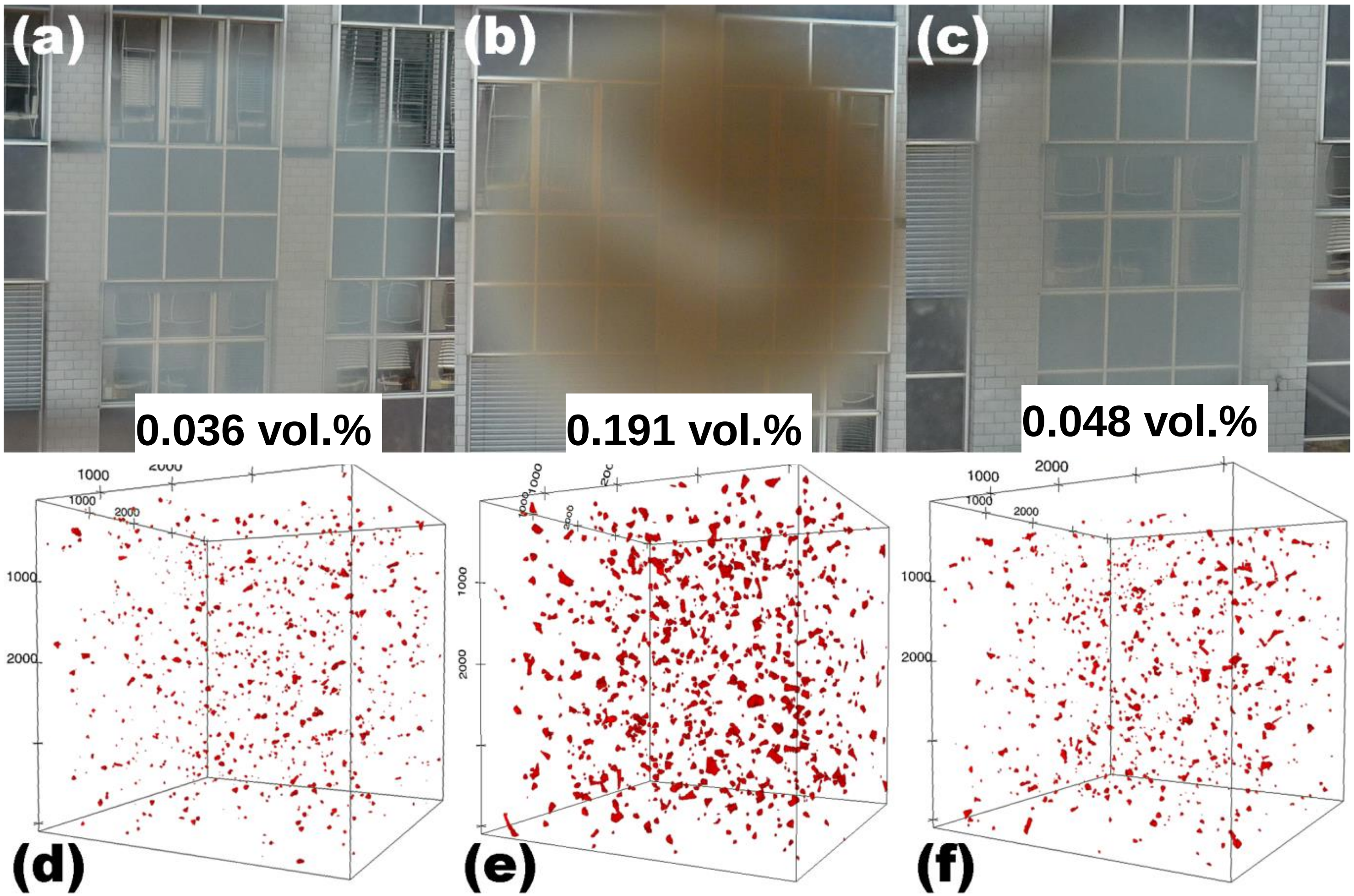
LTP
Private thesis defense, 2012.
FNSNF Swiss National Science Foundation

Private thesis defense, 2012.

Swiss National Science Foundation

FIB-Nanotomography: Link between coloration and porosity

RIT values for 0.8 mm thickness @ 640 nm

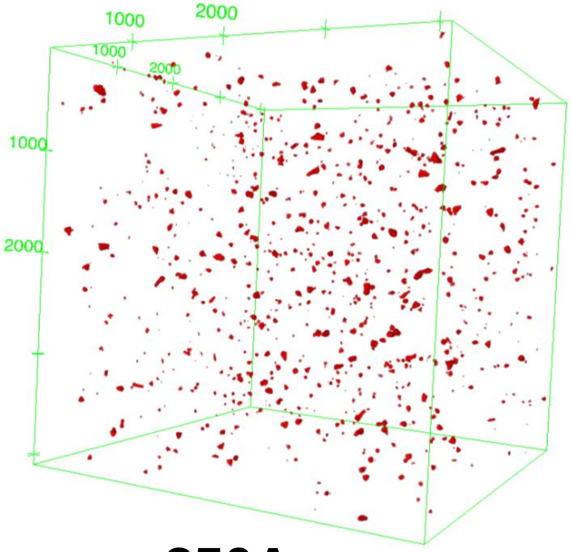


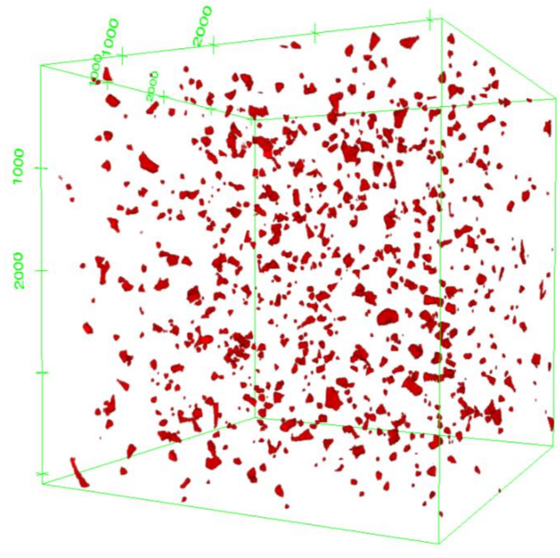
- Coloration is linked with pore **size** and **fraction** increase
- Post-HIP treatment can reduce porosity and coloration (c &f)

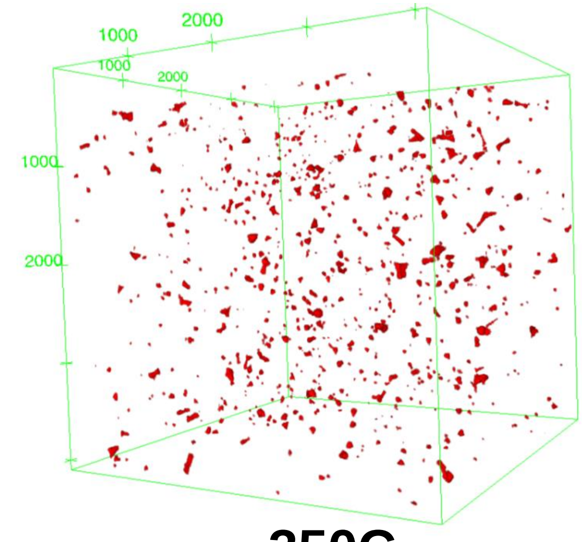
RIT and Porosity - difficult to measure

Pore analysis by 3D-FIB tomography:*

350B







350C

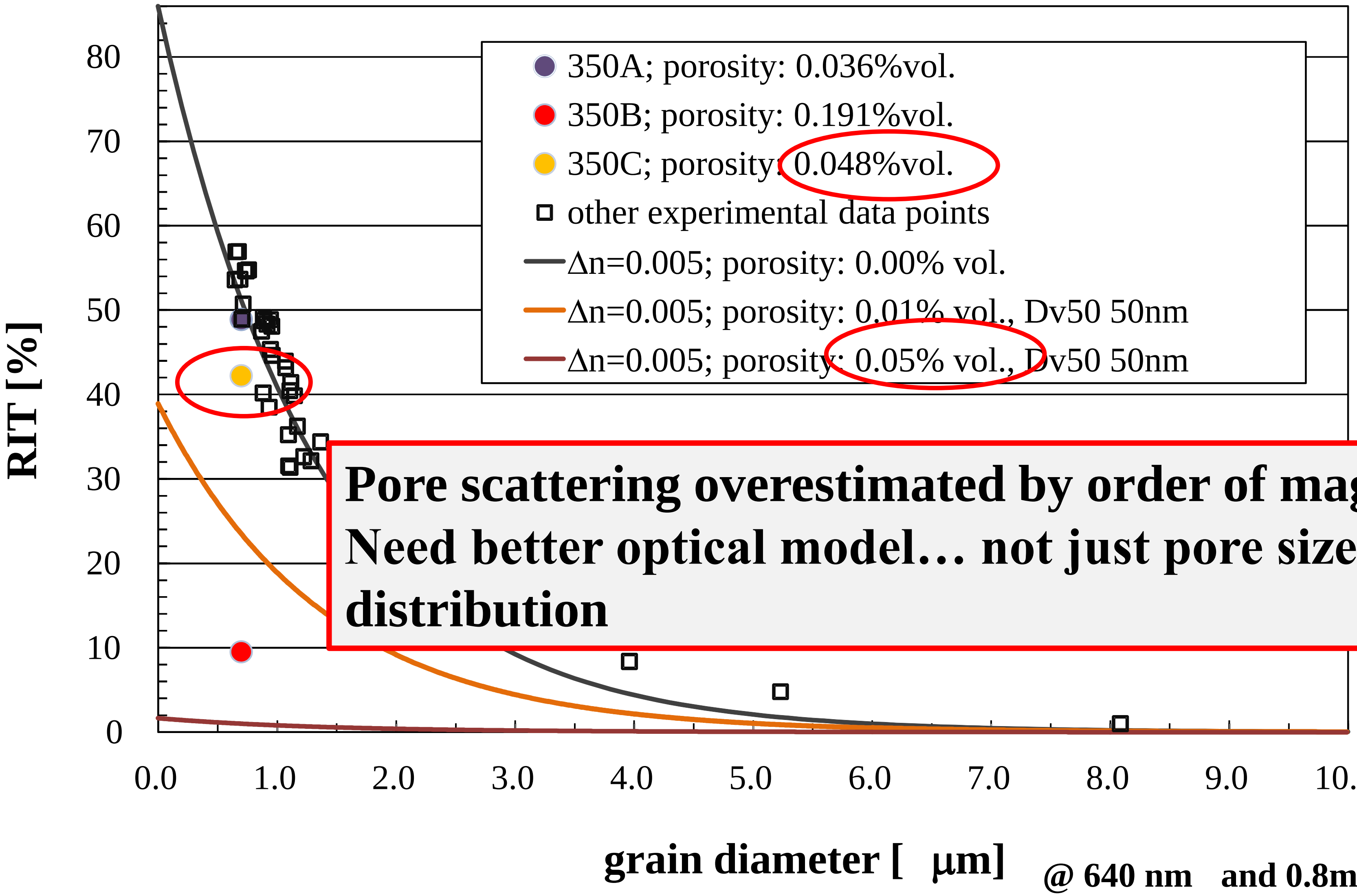
350A

4.11µm x 4.11µm x 4.11µm

Sample name:	350A As sintered	350B After thermal etching	350C Post-HIP
RIT [%]:	**48.9**	**9.5**	**42.2**
Porosity [vol%]:	0.036	0.191	0.048
Dv50,pores [nm]:	51.8	81.9	61.5

*M.Stuer, C. Pecharroman, Z. Zhao, M. Cantoni, P. Bowen " Adv. Funct. Mat.. 22(11) 2303 (2012).

Optical model with scattering from pores*

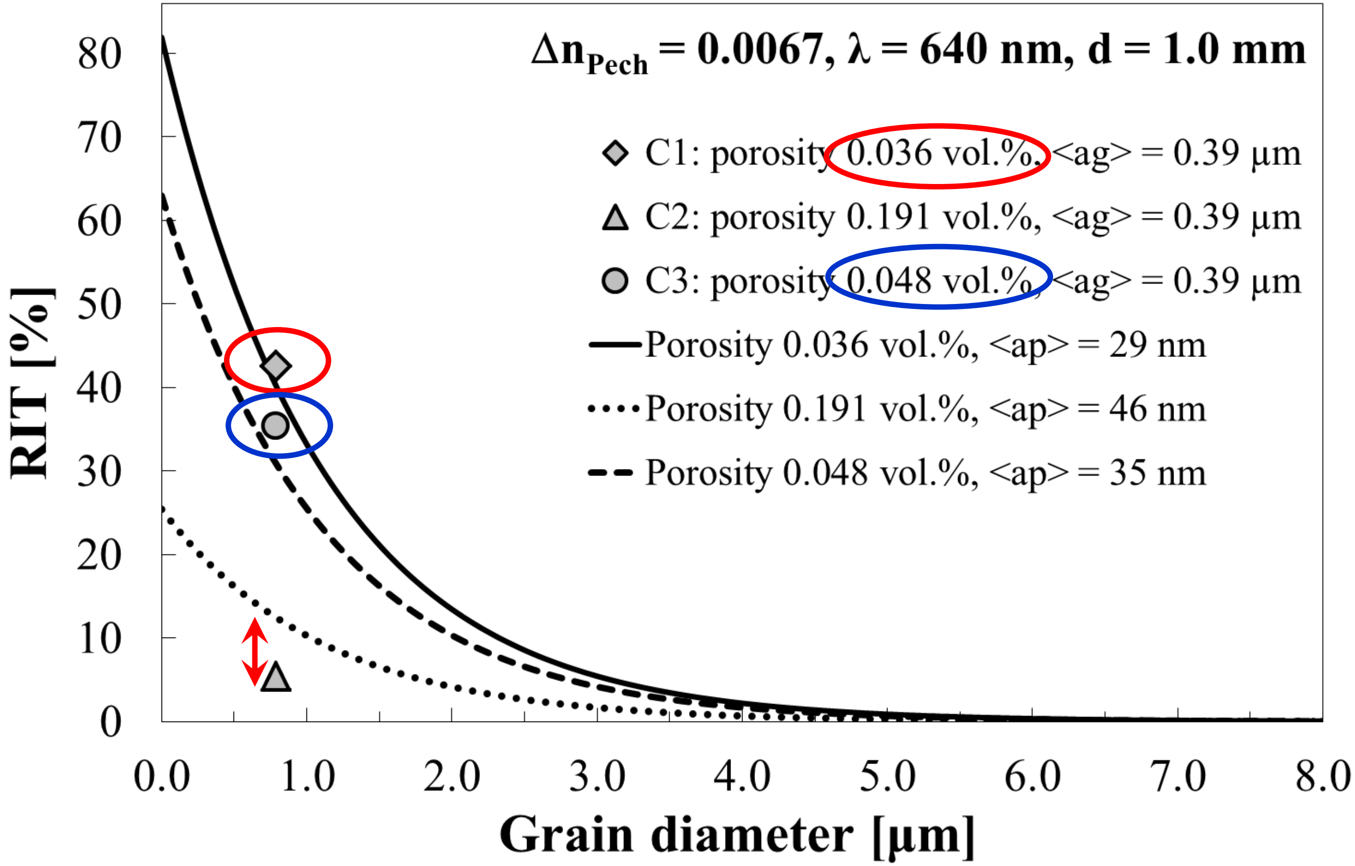


Pore scattering overestimated by order of magnitude
Need better optical model… not just pore size distribution

*R. Apetz , M. P. B. van Bruggen , *J. Am. Ceram. Soc.* ***2003 , 86 , 480 .***
J. G. J. Peelen , R. Metselaar , *J. Appl. Phys.* ***1974 , 45 , 216 .***

The optical model: new description*

Modified model from Pecharromán et al* - fits data well....
Modified characteristic pore and grain sizes with absorption (C2, C3)



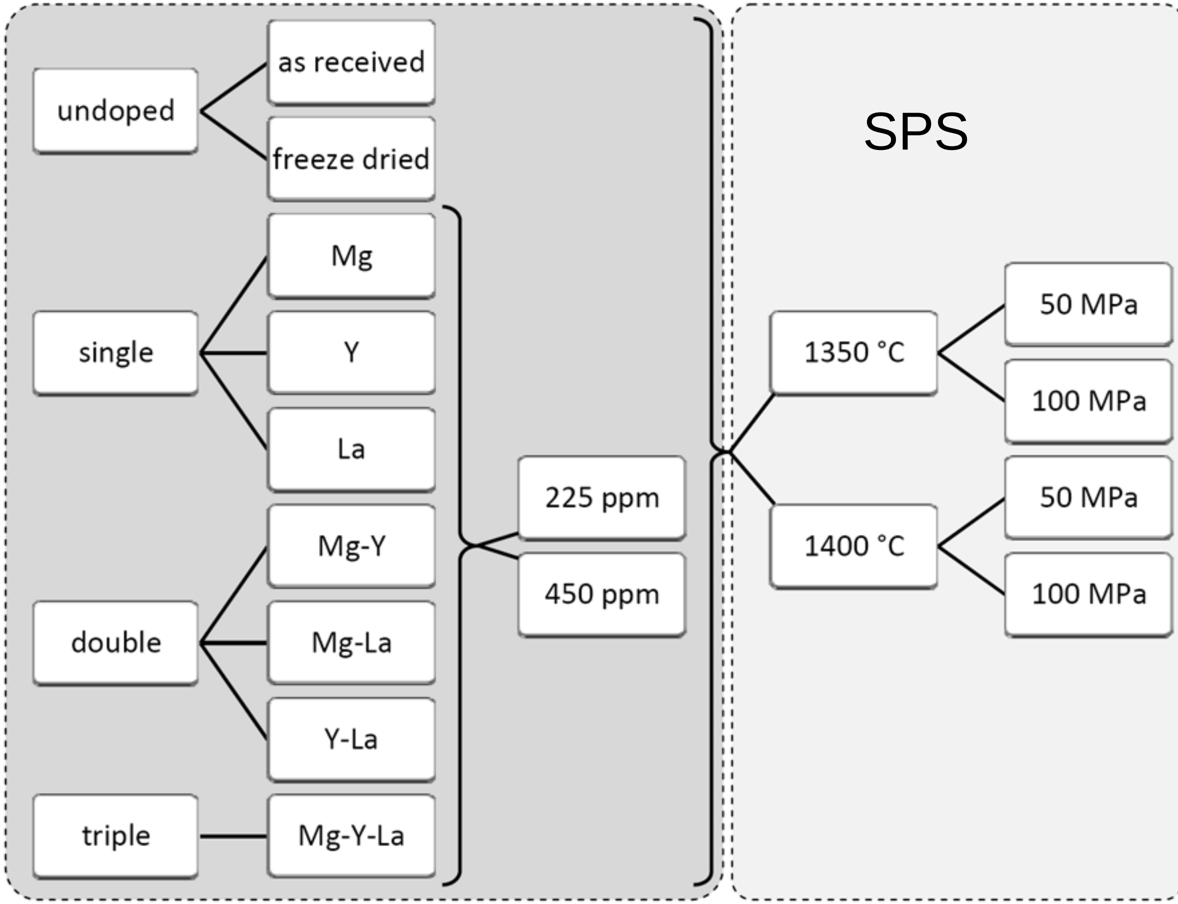
Absorption term measured and required for samples C2 and C3

C2: Rayleigh approximation no longer valid

*C. Pecharroman , et al, *Opt. Express 2009 , 17 , 6899*
*M. Stuer, et al " Adv. Funct. Materials. 22(11) 2303 (2012).

Spark Plasma Sintering – Processing

- Freeze drying & doping – dry pressing in SPS dye (Z. Zhe, Stockholm) – **not granulated** - systematic study of dopant effects:



Powder preparation and doping strategies

Sintering parameters

15 doping strategies

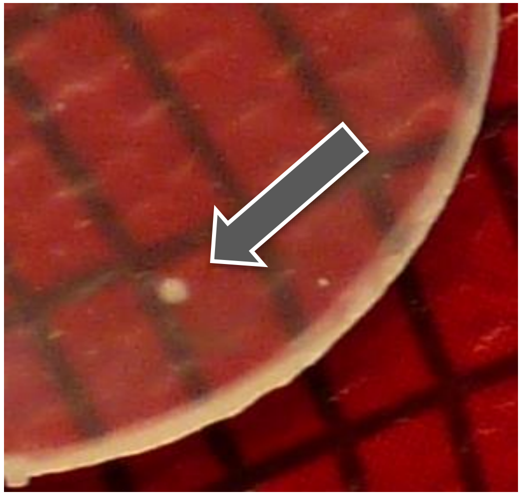
4 sintering parameters

M. Stuer et al.-Transparent polycrystalline alumina using spark plasma sintering: Effect of Mg, Y and La doping **JECS 30 (2010) 1335-1343**

Spark Plasma Sintering

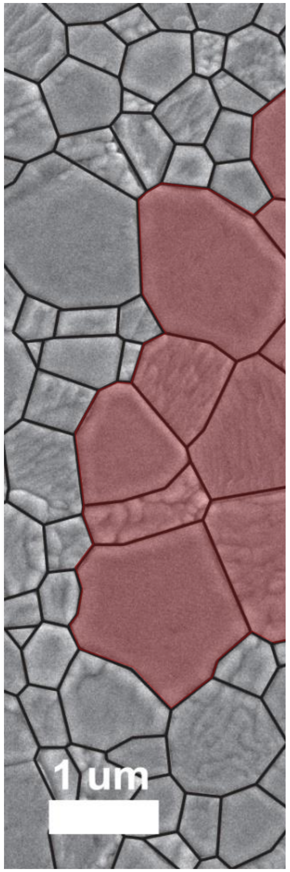
Best results for each dopant and sintering strategy

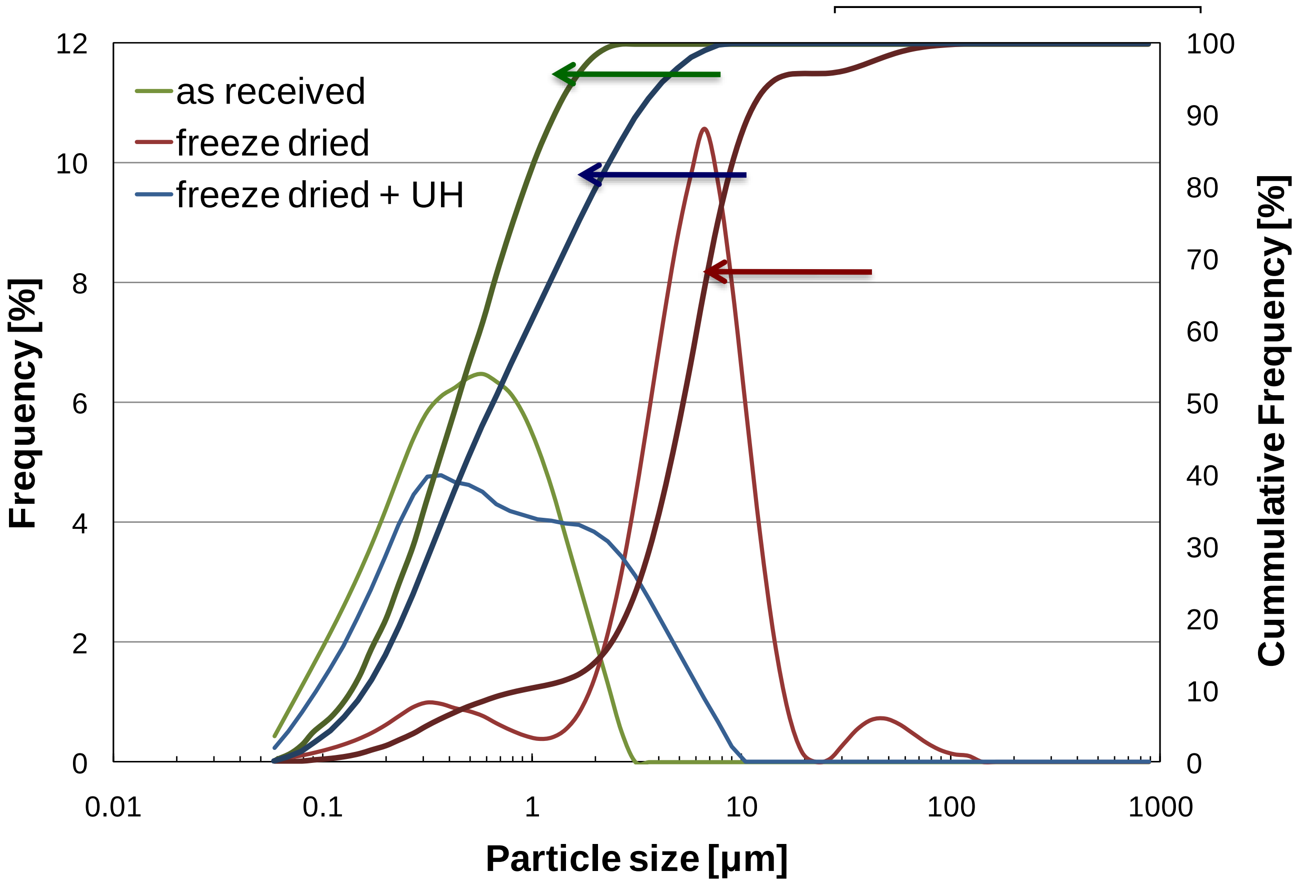
Dopants	225 ppm		450 ppm	
	RIT [%]	Soak temp. [° C]	RIT [%]	Soak temp. [° C]
M00	32.17	1400	52.27	1250
0Y0	55.19	1310	54.71	1350
00L	52.56	1350	50.10	1370
MY0	48.34	1350	54.76	1350
M0L	51.31	1330	54.63	1350
0YL	56.89	1350	49.37	1350
MYL	55.77	1330	56.95	1350



- **Regardless of the doping strategy RIT > 50%** (0.8 mm @ 640 nm)
- **Better than literature for standard SPS** (39% @ 640 nm (0.8 mm))
  - **RIT mainly defect controlled (sintering parameters)**
  - **Improved processing required to get intrinsic dopant contribution?**

SPS - Processing – Limitation – Freeze Drying





Inhomogeneous microstructure from inhomogeneous powder packing – aggregates observed after freeze drying:

Increased grain size distribution ➔ RIT ↓ decreases

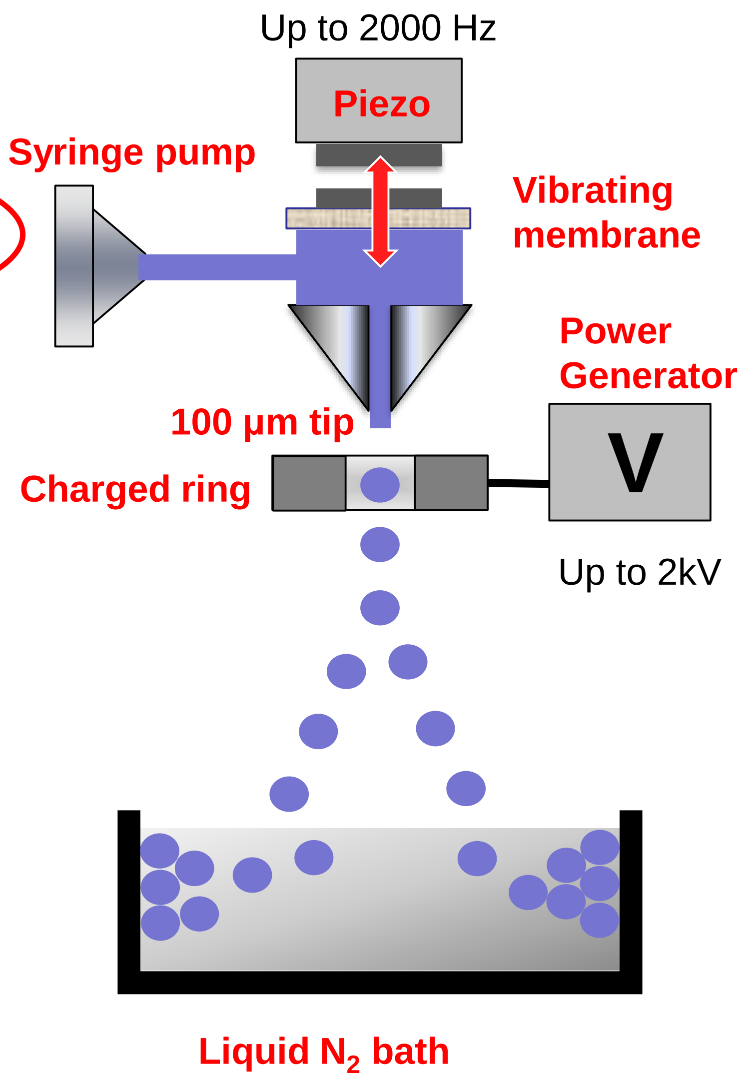
70

Better green bodies – Freeze granulation – dry pressing

Requirements for freeze granulation with "Encapsulator":

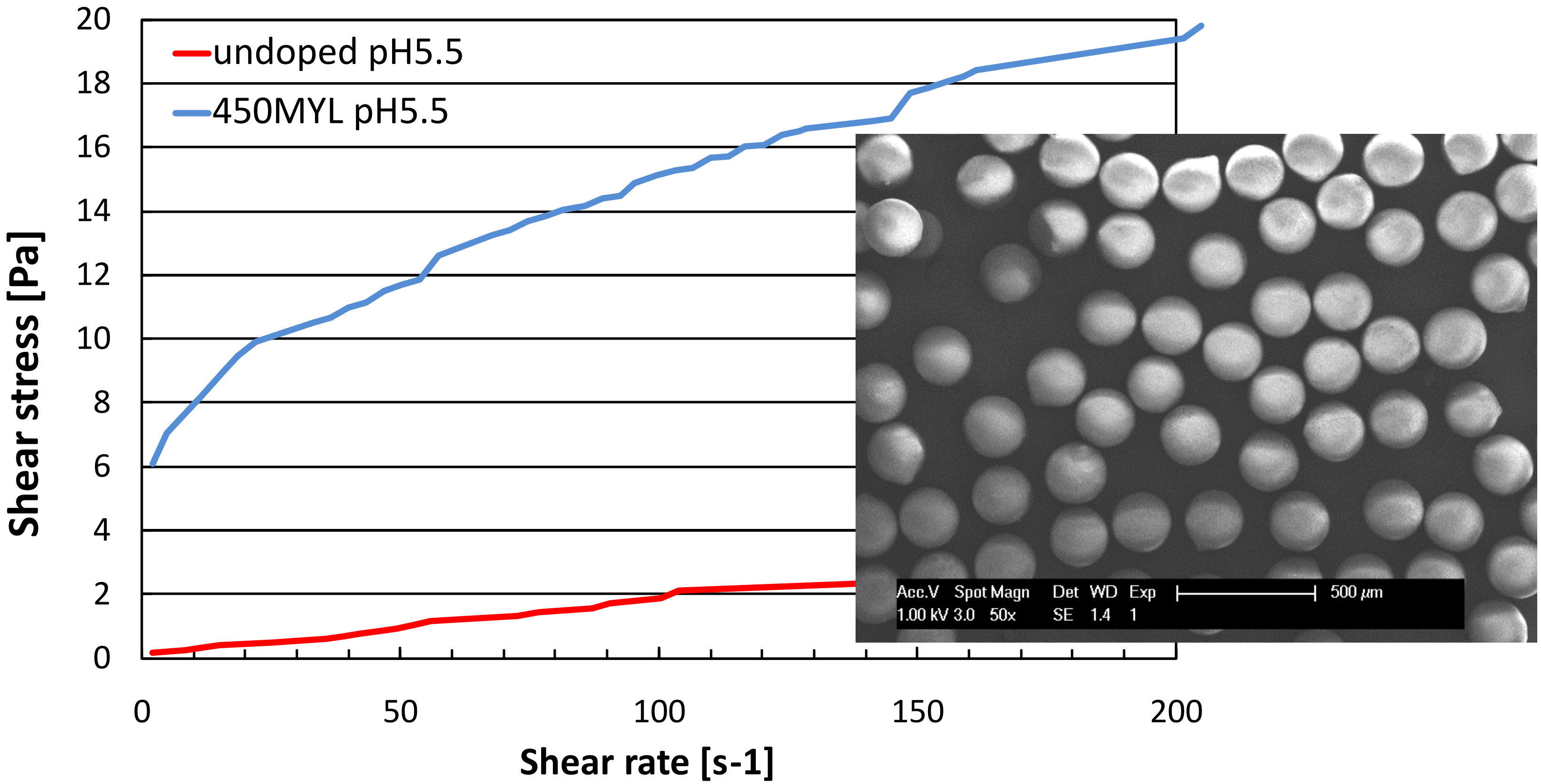
- Low viscosity suspension <0.25 Pa.s @ used flow rate
- Laminar flow (best possible flow speed, just below turbulent flow)
- Homogeneous and stable suspension
- Particle size at least 8x lower than tip size

➢ Final granule size >2x size of tip



Freeze granulation – suspension rheology

Effect of dopant additions 450 ppm @ pH5.5 and 35%vol. solid load



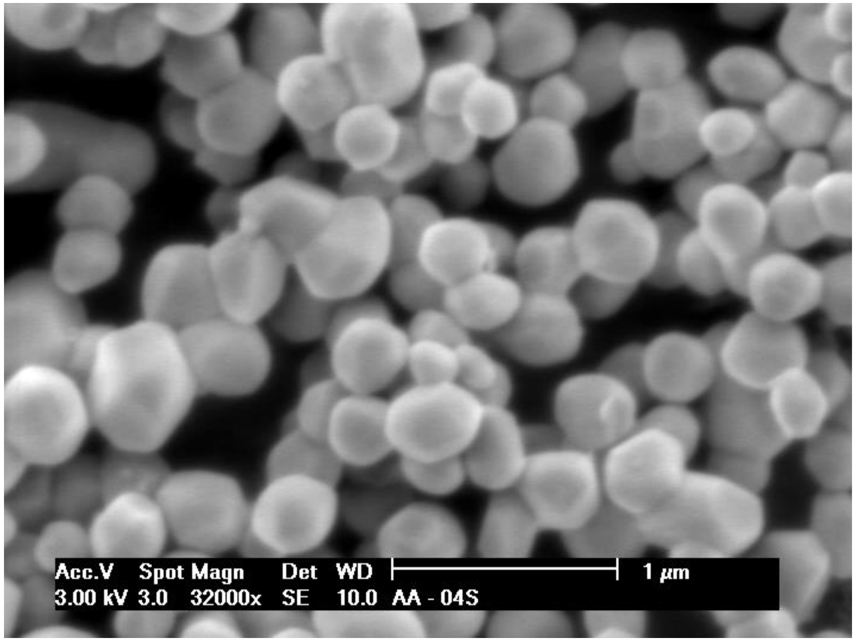
After dopant addition suspension changes behavior:

Newtonian ➔ Shear thinning with yield stress

Hamaker Program*- Interparticle potentials - Dopants

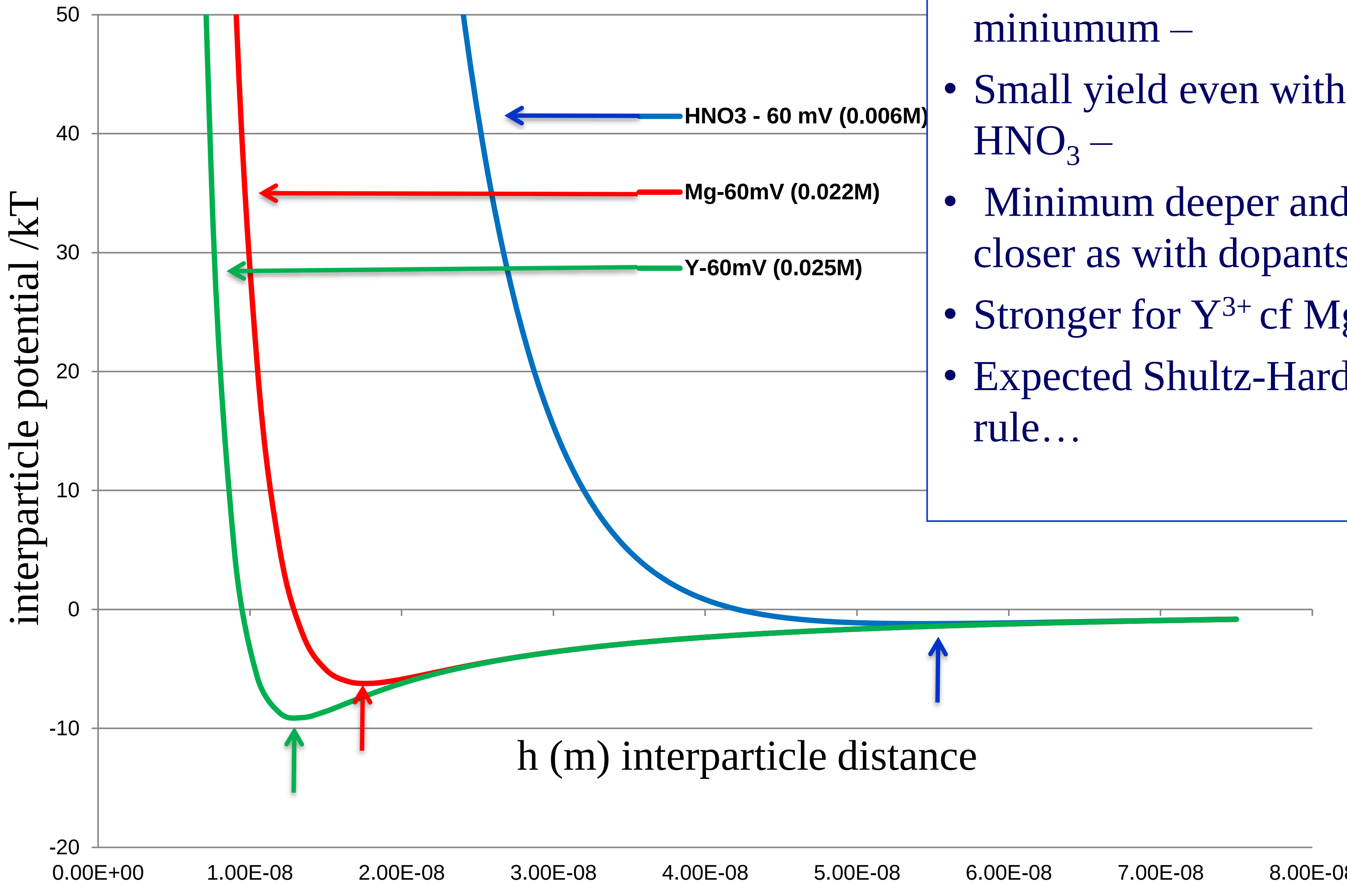
Alumina – effect of dopants 450 ppm – ionic concentration...

- Hamaker constant: $3.67 \cdot 10^{-20}$J
- PSD –
- D_{v10} =200 nm
- D_{v50}=500 nm
- D_{v90} =1600nm,
- pH=4, zeta potential 60 mV
- ionic strength (IS-0.006M)
- Dopants Mg^{2+} ,Y^{3+} 450 ppm
- (IS - 0.022-0.025M)



*U. Aschauer, O. Burgos-Montes, R. Moreno, P. Bowen,
J Dispersion Science Technology. Accepted - In Press (2011)

Interparticle potentials - Alumina doping – Hamaker 2.1



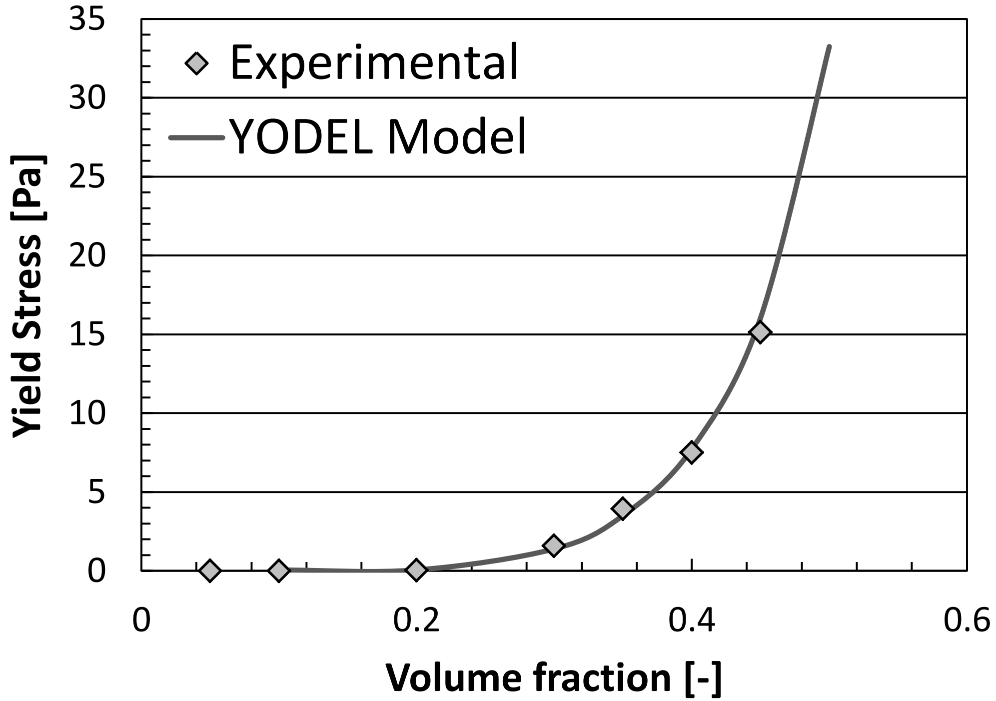
- Always secondary miniumum –
- Small yield even with HNO_3 –
- Minimum deeper and closer as with dopants
- Stronger for Y^{3+} cf Mg^{2+}
- Expected Shultz-Hardy rule…

Yield Stress Model – Yodel

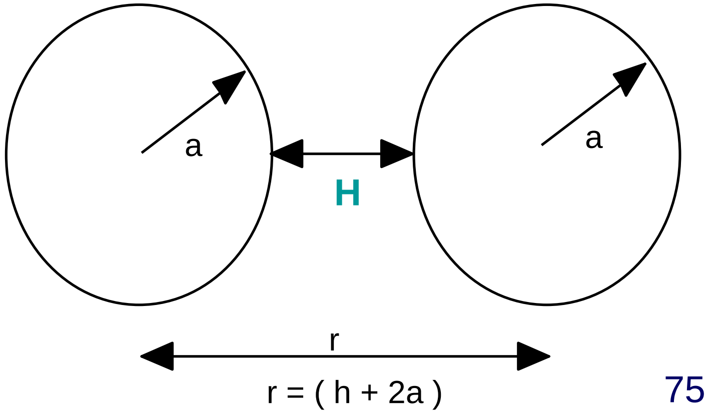
Alumina Slurries for freeze granulation

- YODEL – predicts yield stress for volumes fractions 36% and PSD
- Alumina AA04 - pH=4, zeta 60 mV, zeta plane 2 nm, (no polymer)

YODEL - Volume fraction functionality
- Example Mg doped – vg ....

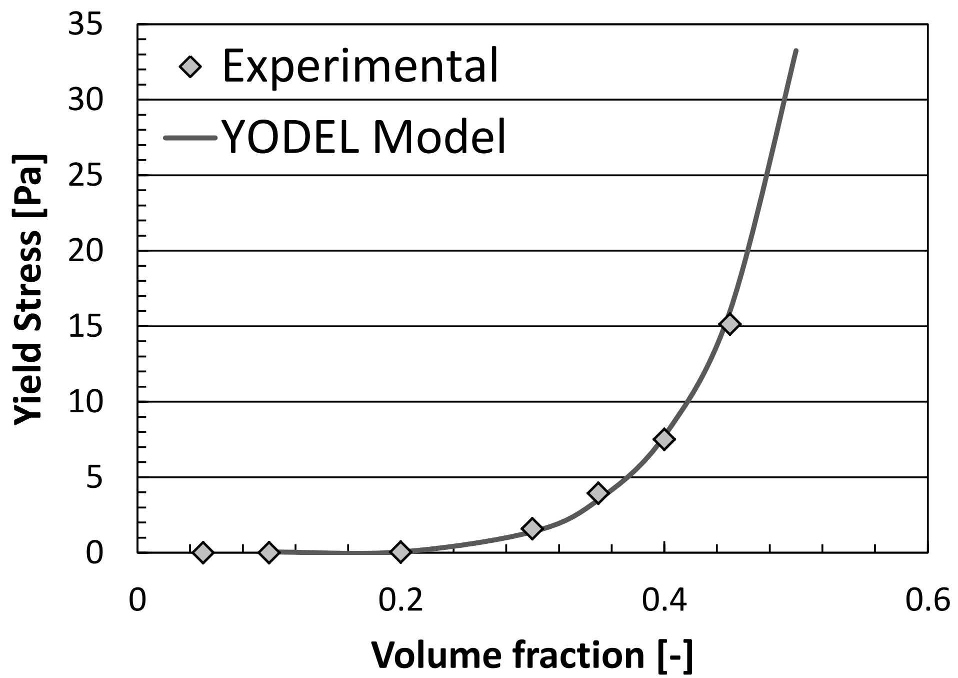


Powder	Measured yield stress (pa)	Predicted yield stress (pa)
Undoped	0.2 ±0.2	0.7
Mg2+	4.6±0.3	5.8
Y3+	5.0±0.3	6.5
La3+	5.0±0.3	6.5



YODEL - Volume fraction functionality*

Yttrium –doped – needed lower percolation threshold



INPUT PARAMETERS:	Mg-doped	Y-doped
Hamaker constant:	3.67E-20	3.67E-20
Minimum separation [nm]:	23.4	16.8
Contact curvature [nm]:	37	37
Percolation threshold [-]:	0.16	0.11
Maximum packing [-]:	0.64	0.64
Yield at ϕ=0.45 [Pa]	15.3	40.7

- **Maximum packing:**
- needs perfectly dispersed suspensions-filter pressing with HNO_3

Parameters difficult to define

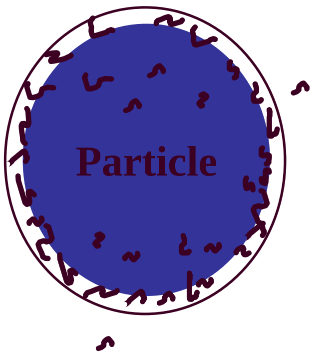
- **Percolation threshold:** function of particle shape - floccs/agglomerates – network structure – from
- sedimentation density – confirmed Y lower percolation threshold

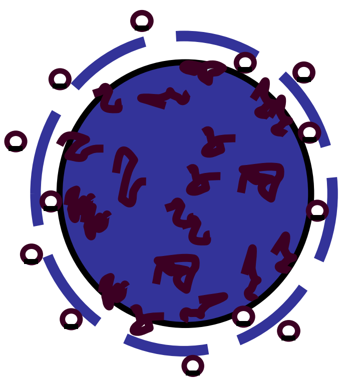
- **Contact curvature**:
- smallest curvature for each particle, average or distribution ? used 37 nm but…..

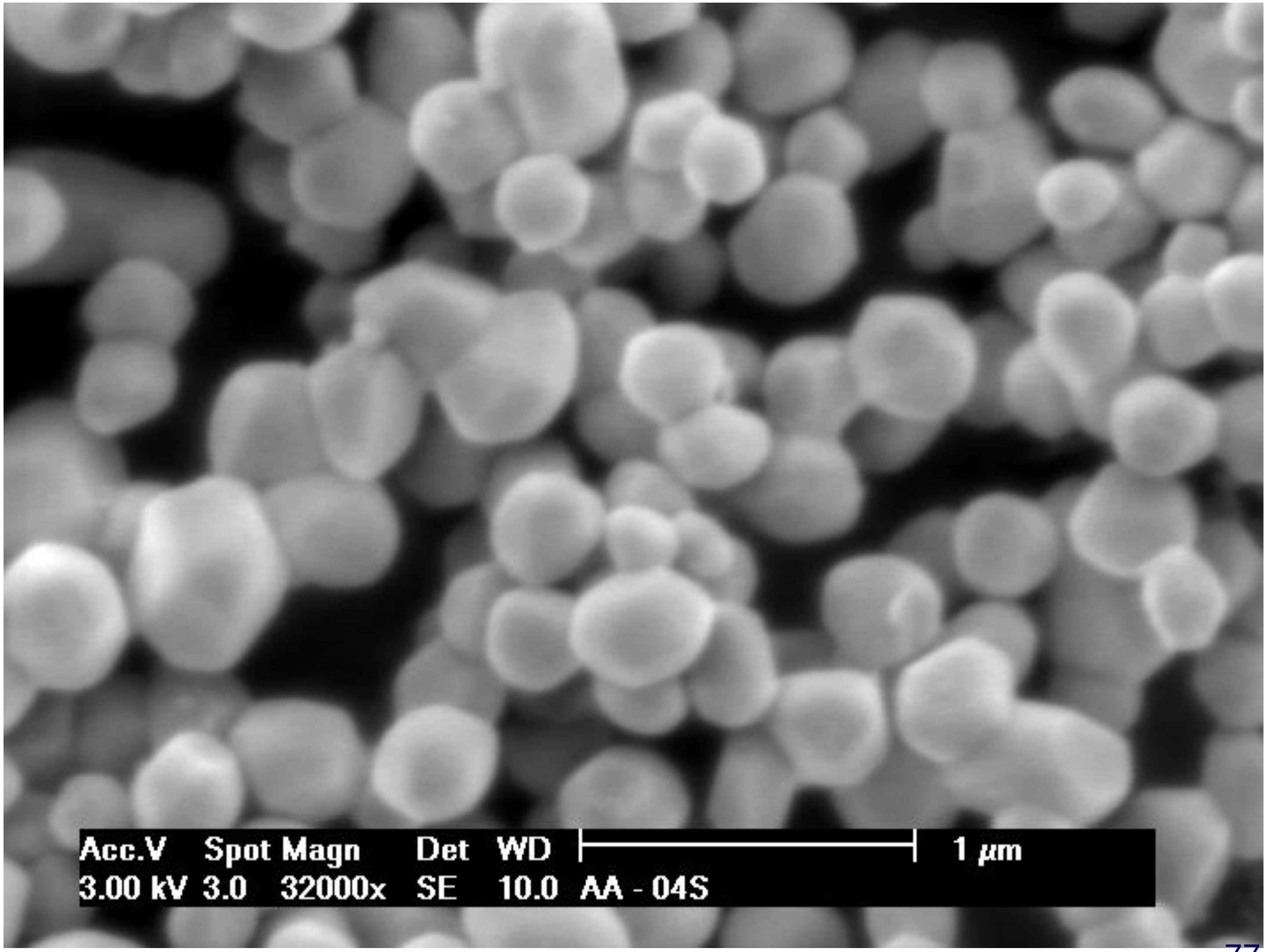
*M. Stuer and P. Bowen- Advances in Applied Ceramics, 111(5/6) 254-261 (2012)

3. Curvature of contact point– Alumina?

- Used 37 nm but....

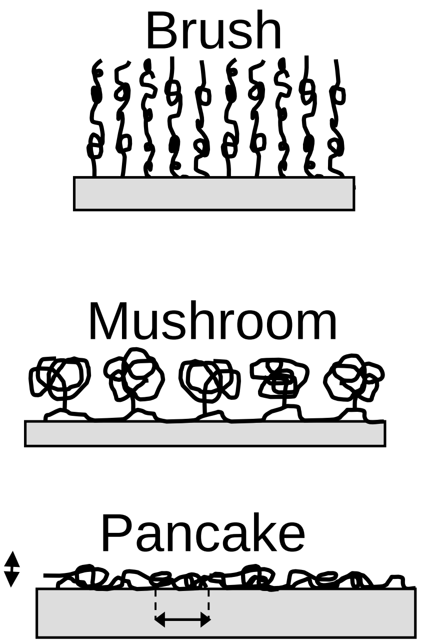


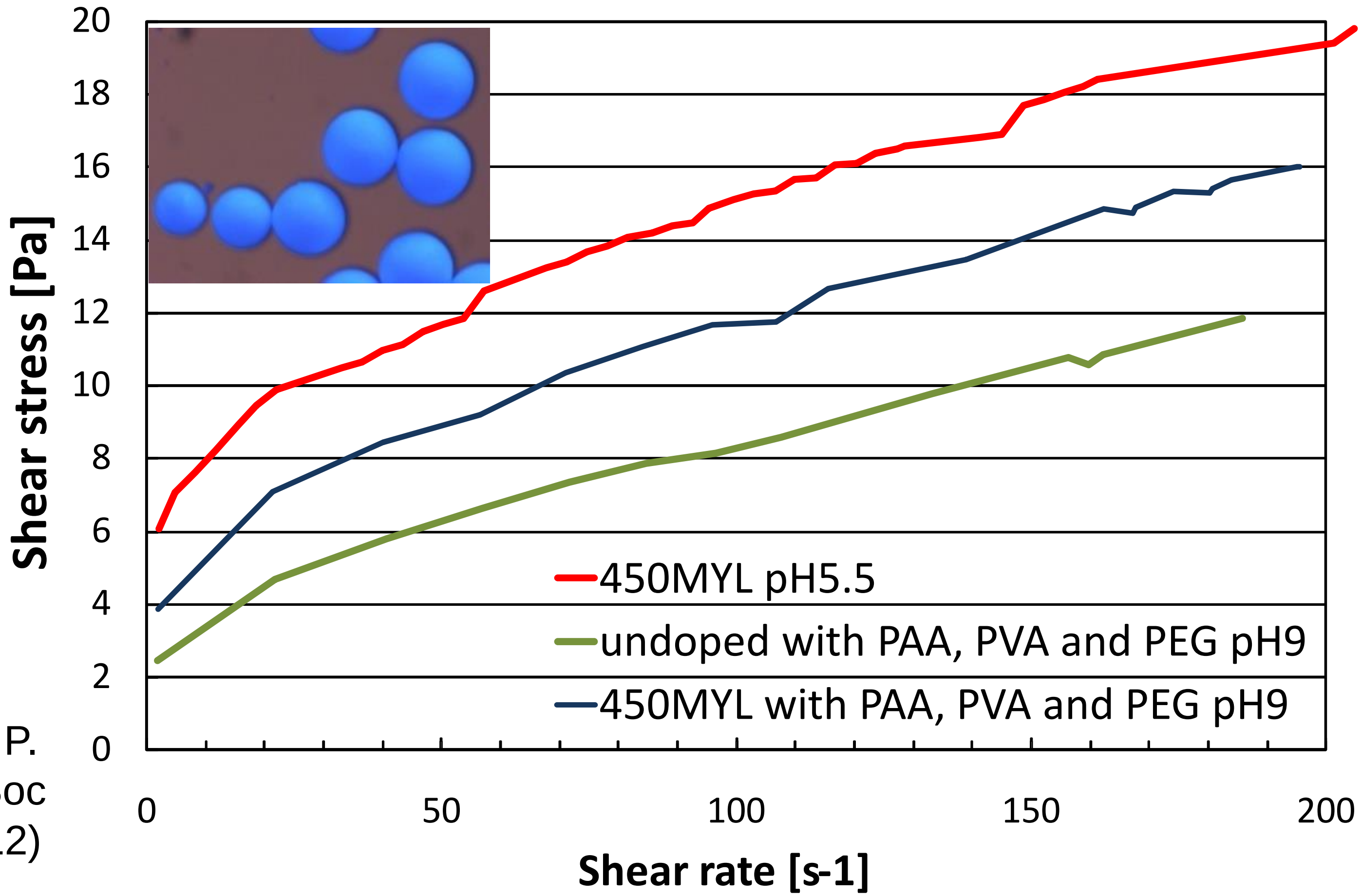




Better green bodies – Freeze granulation*

- pH 5.5 not possible needed - electro-steric barrier – PAA - pH9
- with PAA mol.wt 2000 and 5000 - if brush - 12 and 30 nm steric barrier...rheology improved and granules produced (PVA and PEG for pressing)
- OK but still not good enough – perhaps mushroom or pancake configuration because of complexation with Mg^{2+} and Y^{3+}





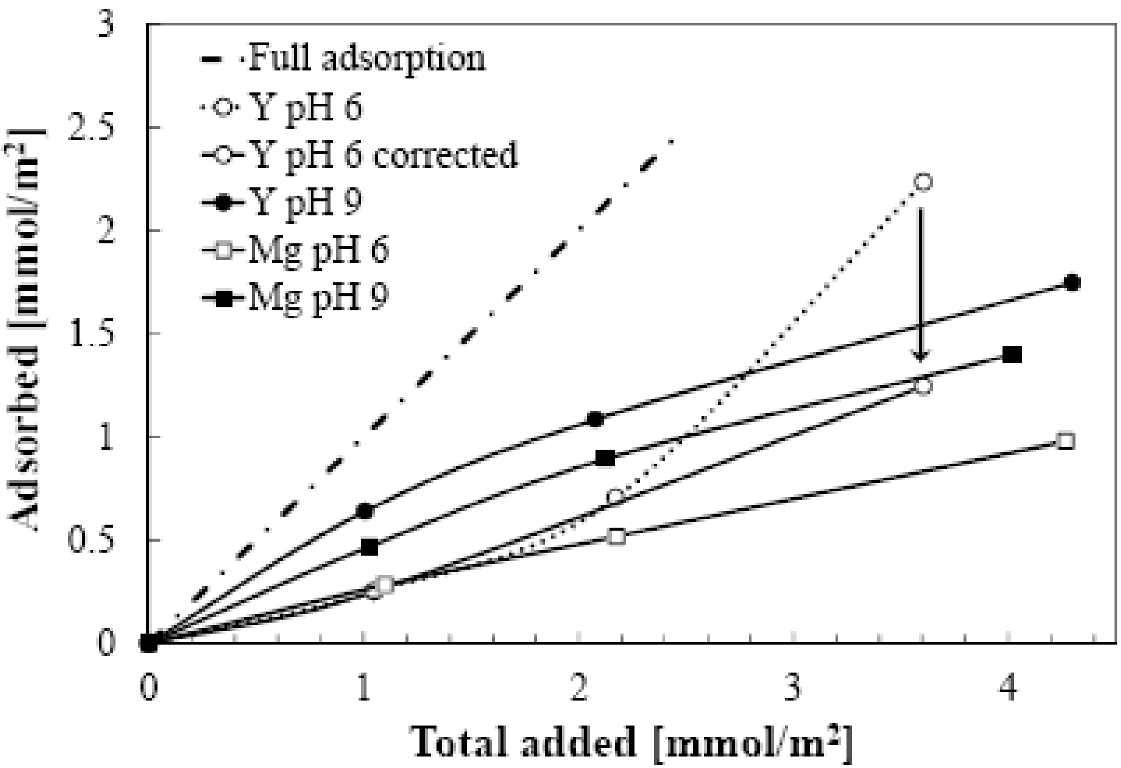
*M. Stuer , Z. Zhe and P. Bowen, J.Eur.Ceram.Soc 32(11) 2899-2908 (2012)

PAA-complexation – Adsorbed Layer Thickness ?

- Complexation with Mg^{2+} and Y^{3+} - two effects*

1 - Reduces the effective ionic strength and thus in secondary minimum distance and depth

2- modifies the adsorption conformation of PAA on the surface



- **>50%** of dopants complexed/adsorbed with PAA
- Used YODEL and Hamaker to compute closest approach, H, by matching with experimental yield stress
- Reduction of ionic concentration more important than steric contribution....

*M. Stuer and P. Bowen , Advanced Engineering materials ,16(6),774-784 (2014)

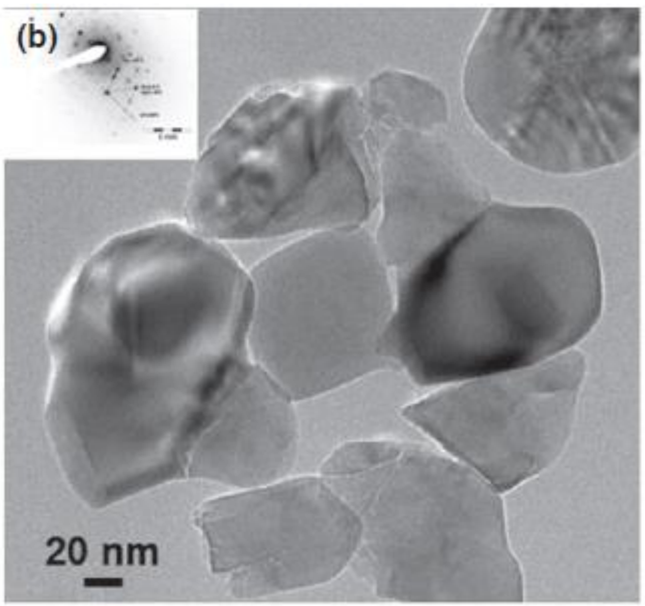
Results – Freeze Granulation - dry pressing*

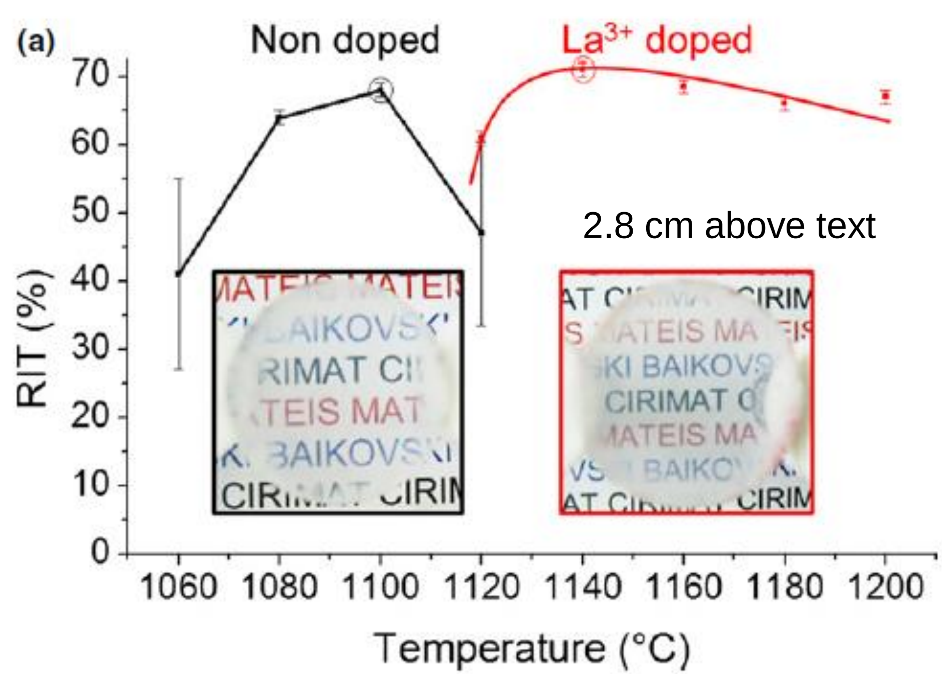
- Green body densities 56%
- SPS > 99.9% dense……
- RITs - 53%* - slightly lower than freeze dried
- Improvements still needed...suspension need higher solids load
- But successful use of "standard" processing for SPS – simpler than slip-casting ....easier and cheaper for industrial application
- Next step apply same process to finer powders, D_{v50}- 130nm – dispersion still challenging.....
- Best result so far 65% RIT at 150 MPa ......>70% soon.
- ....in fact ....

*M. Stuer , Z. Zhe and P. Bowen, J.Eur.Ceram.Soc 32(11) 2899 (2012).

Roussel et al* …reduced ionic concentration – RIT 71%!!

- Baikoswki powder D_{v50} – 130nm, D_{n50} – 80 nm
- Single doped La (480 ppm) using nitrate but……washed out excess dopant ions– reduced ionic concentration – re-dipsered with HNO_3
- Slip casting – green bodies (Vincent Garnier - Lyon)
- RIT 71%...best SPS results so far (complex sintering cycle)





*Roussel et al., J.Am.Ceram.Soc., 1-4, 2013

Conclusions

- Dispersion – not easy to predict or always understand – without calculations
- Agglomerates - poor microstructures or no flow!!
- Hamaker programme - estimate charge and/or steric barrier needed overcome van der Waals attractive forces

YODEL -Yield stress can be predicted - at least semi quantitatively using

- Particles Size Distribution, Maximum Packing Fraction, Percolation Threshold, Hamaker Constant, Distance of closest approach of particles, H,
- Curvature (radius) of contact point between particles a*

Limitations

- Last parameter can be seen as "fitting" parameter - takes into account shape and perhaps other factors not perfectly captured by YODEL
- but yield stress predictions are very coherent once fixed for a given system

Transparent Alumina

- Future work - Try Baikowski D_{n50} 80nm suspension but by granulation…..
- Collaboration with Yves Jorand and Vincent Garnier (Lyon) – master project…
- Soon perhaps dry pressed >70% RIT …application….becoming possible…

Powder Technology – Learning outcomes

- Assess / Evaluate the use of different modelling methods in powder technology
- Model the stability of a colloidal dispersion
- Describe the different sintering methods used in powder technology
- Explain the limitations of classical DLVO theory
- Give an example in detail of the importance of powder characteristics in an everyday example of the application of powder technology
- Discuss powder compaction in detail

Questions Type

1. Describe an additive manufacturing process.
2. What is the difference between stereolithography (SLA) and Digital light processing (DLP)?
3. What are the advantages and disadvantages compared to traditional methods?
4. Describe the Selected laser sintering method (SLS) – what are the key parameters that control the final microstructure?
5. List the different types of sintering methods available, with some limitations and advantages.
6. Explain the SPS and flash sintering processes.
7. What are the additional transport mechanisms in SPS (metal and ceramic) compared to standard isothermal sintering?
8. After sintering of a ceramic material, still 2 % porosity was observed. How can we reduced this porosity without further grain growth.
9. What are the advantages of Field and pressure assisted sintering methods?