Workshop on Field Assisted Sintering Techniques

Spark Plasma Sintering: Homogenization of the compact temperature field for non conductive materials

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Overview

- ❑ Introduction
- ❑ Heating = Inner Joule Effect in the shaping tools
  Localisation of the main heat sources
- ❑ Steady-state lumped elements thermal model
- ❑ Searching for an optimal temperature field
- ❑ Influencing parameters
- ❑ Conclusions

Introduction

- SPS = Constrained sintering technique

Compaction and heating at the same time

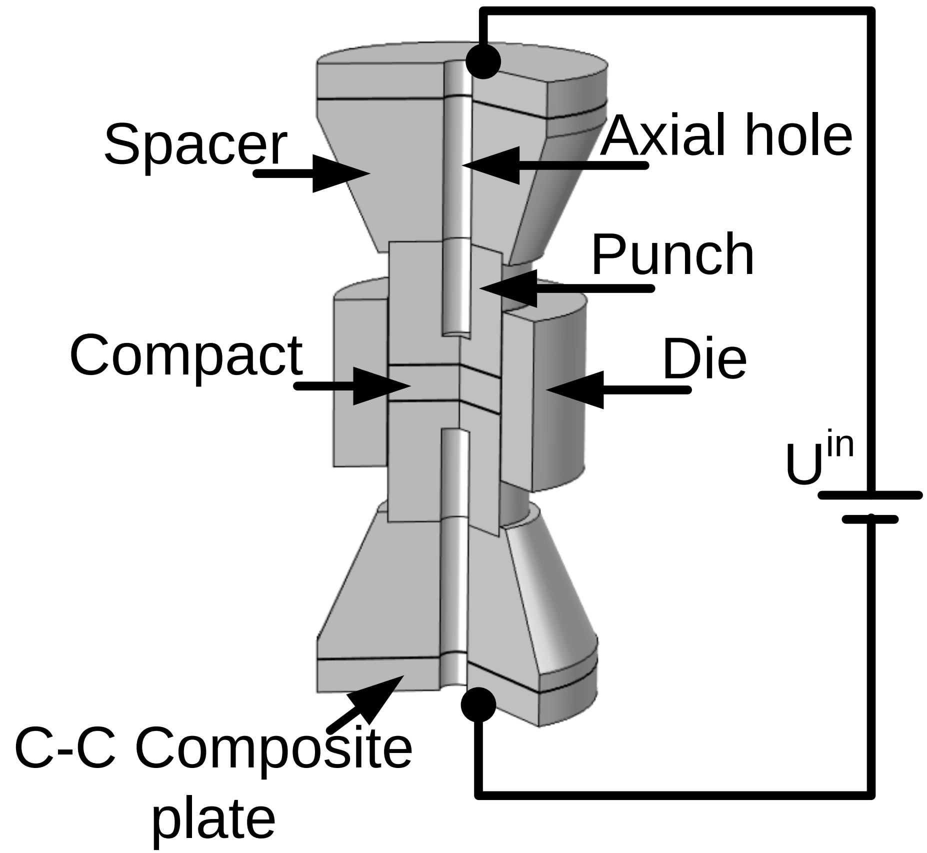
- Simple principle :

Inner heating of the shaping tools (and compact) by Joule Effect

➔ Shaping tools have to be electrically conductive: graphite

- Many advantages :

Rapid sintering cycle, high density compact, limited grain coarsening, sintering of materials not easily obtainable by conventional sintering techniques, new materials, …



Introduction

- Main drawback :
  Heterogeneity of the compact temperature field during temperature dwell

  ➔ Heterogeneity of the final microstructure
- Reason : Bad understanding and empirical knowledge of the temperature field and its influencing parameters.
- Objective :

  Obtain the more homogeneous temperature field possible in the compact

  ➔ Minimizing the maximal temperature difference in the compact $\Delta T_{max} = T_{max} - T_{min}$
- Optimization tool : Finite Element Method
- Compact = Electrically non conductive (Alumina Al_2O_3)

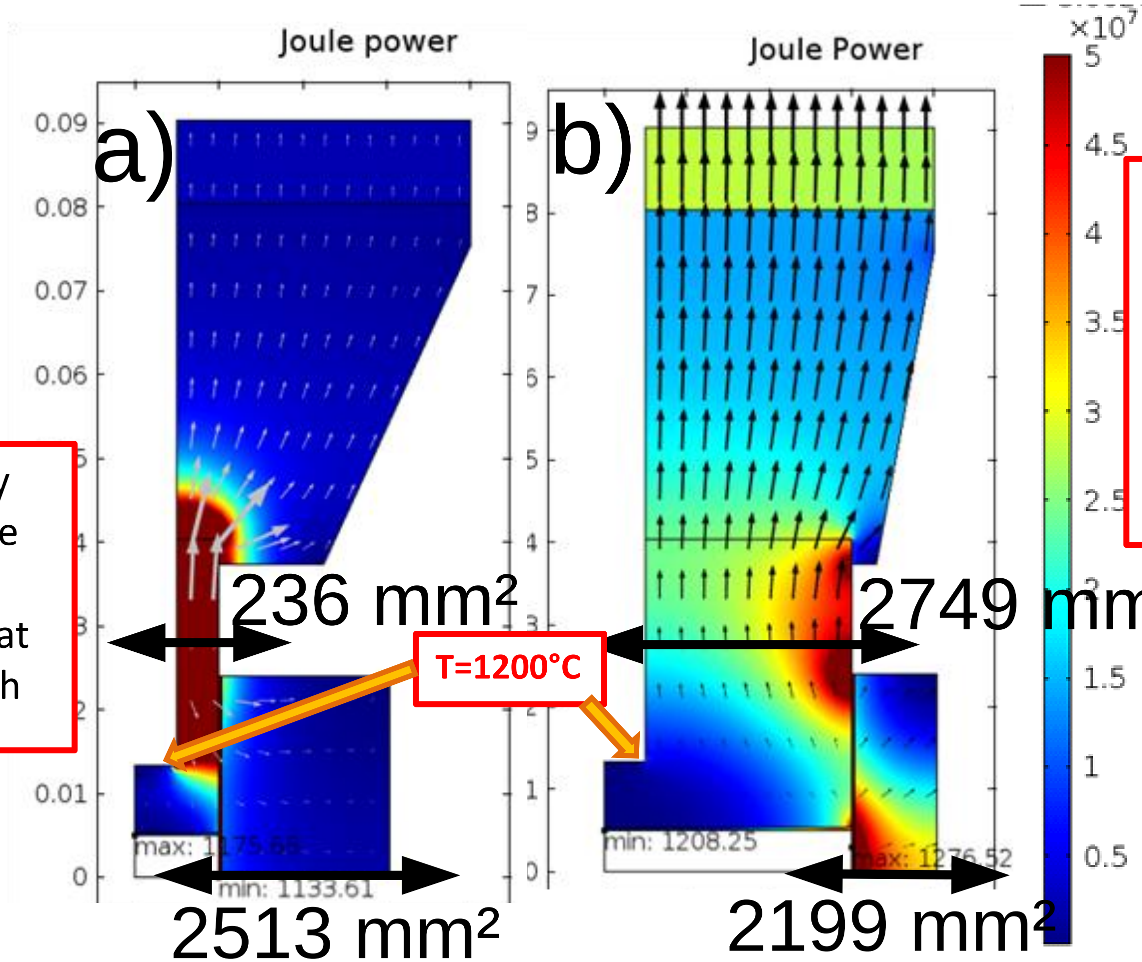
Heating = Inner Joule effect

$$p_j = \rho j^2$$

Dense alumina, T_{dwell} = 1200 °C

a) Small compact diameter (20 mm)
Thick die (20 mm)

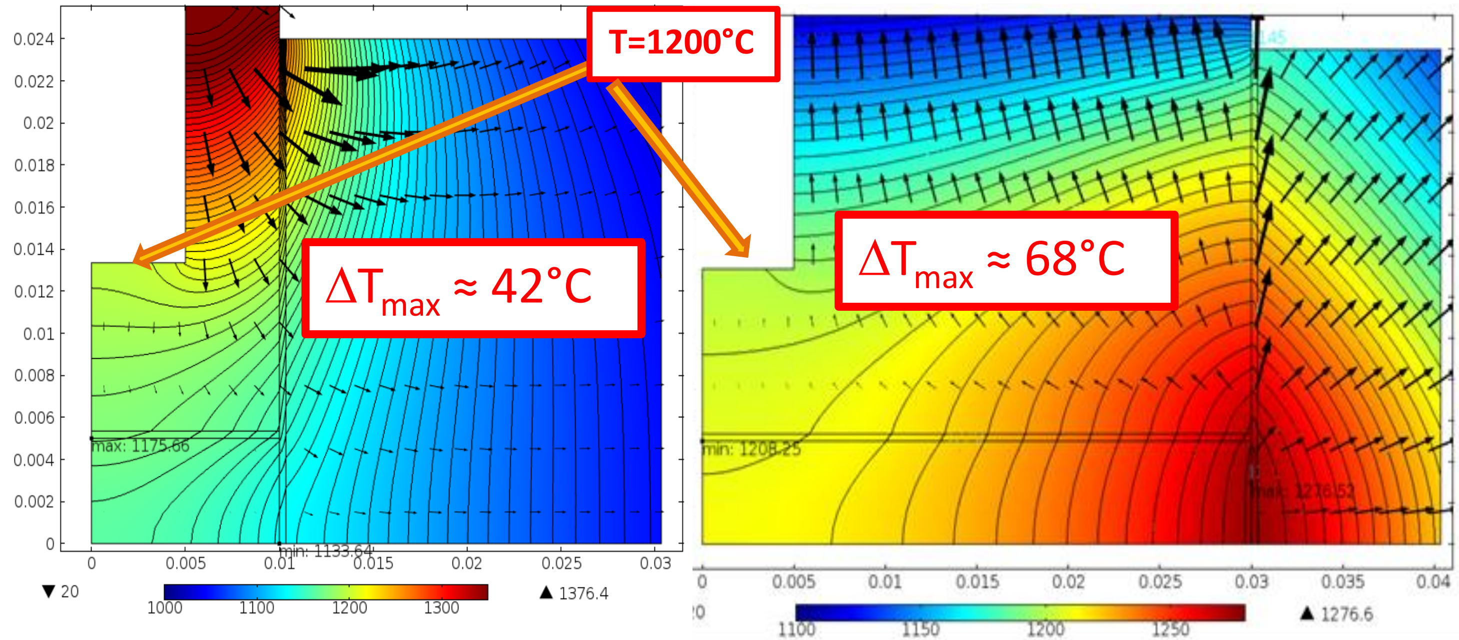
b) Large compact diameter (60 mm)
Thin die (10 mm)



Joule power is very concentrated in the punch.
➔ Only 1 main heat source in the punch

Joule power is more diffuse but still more concentrated in the punch and in the die
➔ A 2nd main heat source appears in the die.

Heating: Consequences of the relative importance of the two main heat sources



The die dissipates more heat (by radiation) than it produces.

➔Heat flows from the punch to the die

➔The compact is everywhere colder than the dwell temperature

The die produces more heat than it dissipates by radiation.

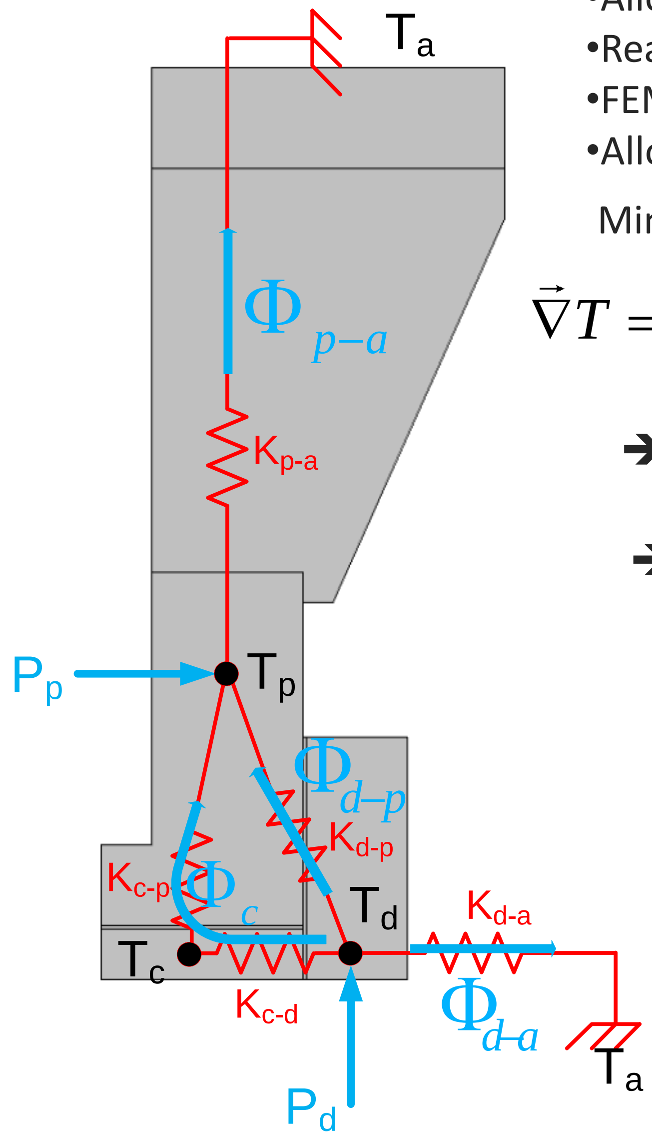
➔The excess of heat flows from the die to the punch.

➔The compact is everywhere hotter than the dwell temperature.

$$\vec{\nabla} T = -\frac{\vec{\phi}}{\lambda}$$

Inversion of the heat flow
➔ inversion of the direction of temperature gradient

Lumped elements thermal model



Remarks :

- Allowed because of the concentration of the heat sources.
- Reality is more complex.
- FEM is closer to the reality.
- Allows the understanding of the main heat fluxes

Minimizing the temperature gradient $\vec{\nabla} T$ in the compact

$\vec{\nabla} T = -\frac{\vec{\phi}}{\lambda}$ ➔ Minimizing the heat flux $\vec{\phi}$ in the compact

➔ Minimizing the heat flow Φ_c through the compact

➔ Minimizing the total heat flow $\Phi^t_{d-p} = \Phi_{d-p} + \Phi_c$ between the die and the compact.

$\Phi^t_{d-p} = 0$ allowed if perfect balance between local heat production and local dissipation:

$$P_p = \Phi_{p-a} \quad \text{and} \quad P_d = \Phi_{d-a}$$

Searching for an optimal temperature field: Steps

- Fixing the sintering conditions :
  - Material (electrical and thermal characteristics)
  - Geometry of tools
  - Compact diameter
  - Compact height
  - Dwell temperature (controlled at the bottom of the axial hole)

- Geometric parameter on which to play ?

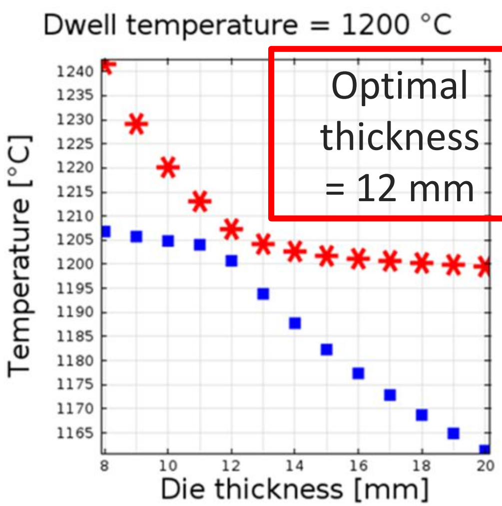
  On the die thickness

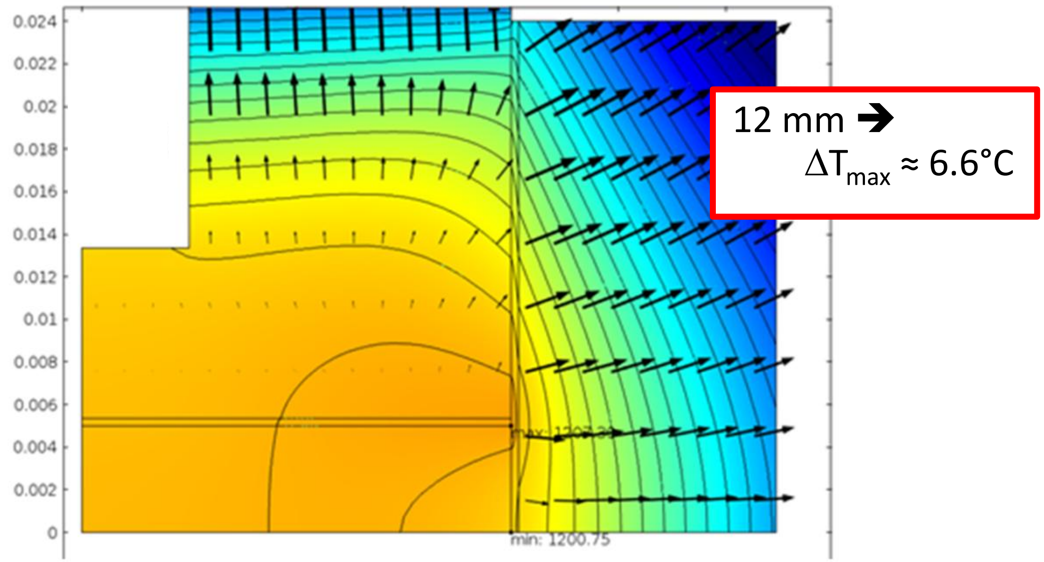
- Simulating the temperature field for a range of die thicknesses (each mm)

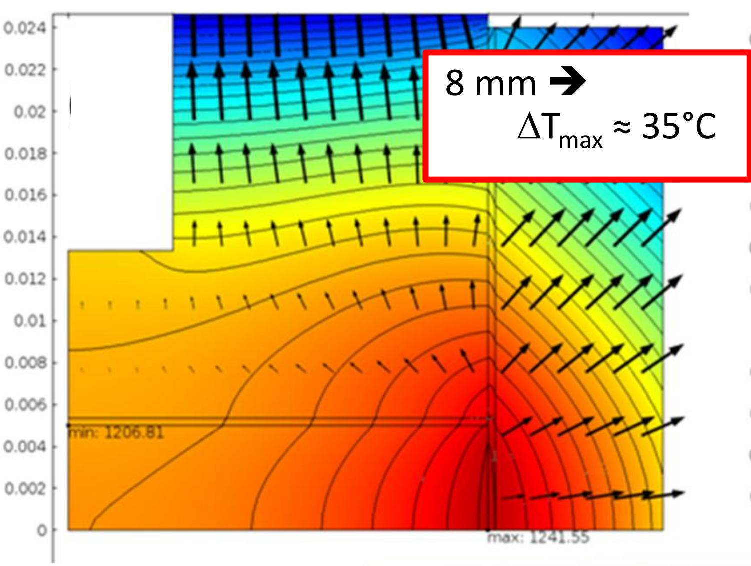
- Postprocessing and determining the optimal die thickness.

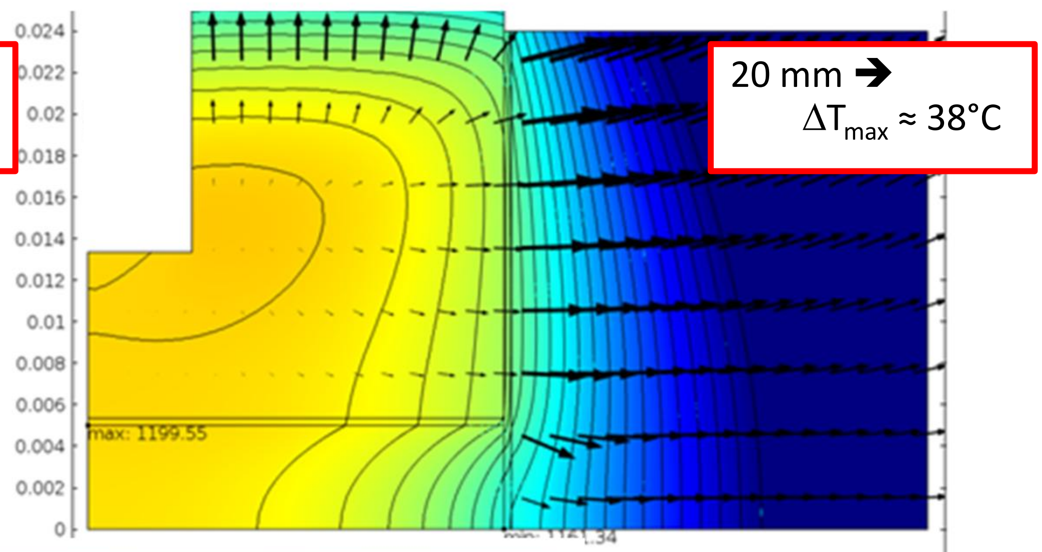
Searching for an optimal temperature field: exemple

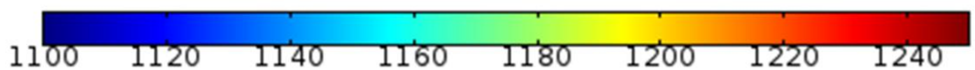
- Dense alumina, 1200°C dwell, compact diameter 40 mm, compact height 10 mm





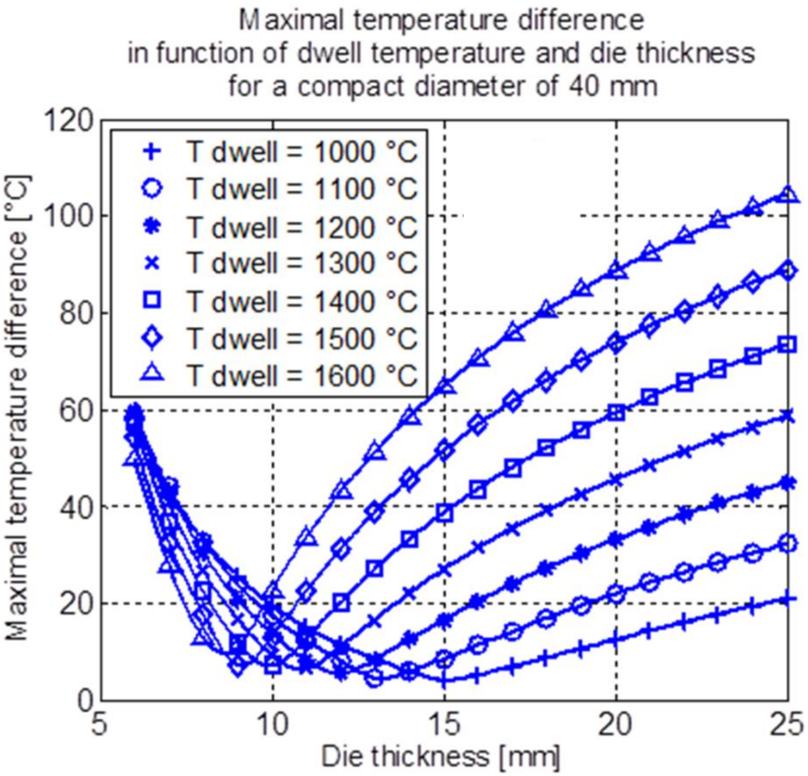


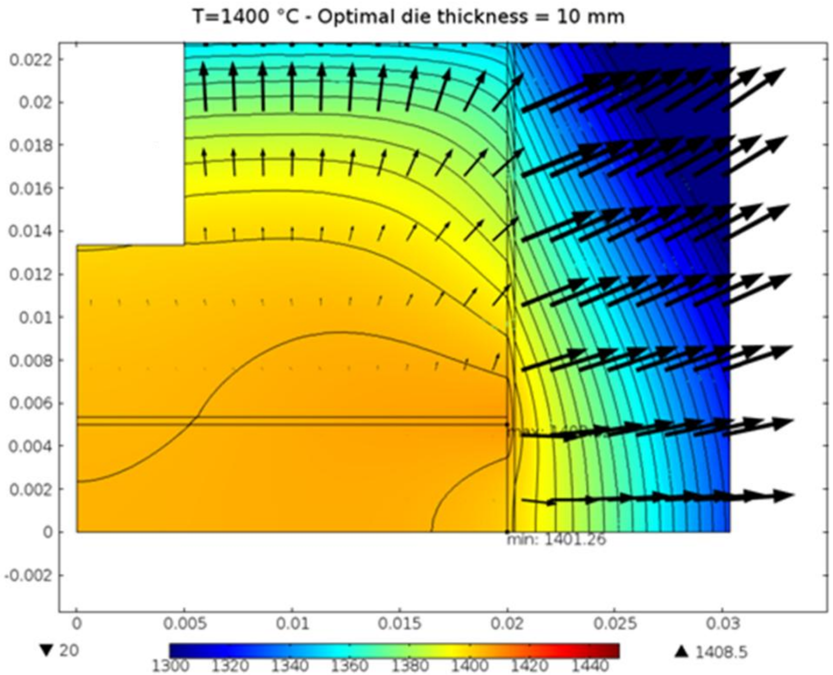




Influencing parameters: Dwell temperature

- Same material (dense Alumina), same compact dimensions (40 mm diameter)
- T_{dwell}: 1000°C → 1600°C





Increasing of T_{dwell} →
decreasing of the optimal die thickness

Why does the optimal die thickness change with T_{dwell} ?

- Dependance of the material characteristics with the temperature: not significatively

- Thermal Radiation: Yes

$$\Phi_{d-a} = S\varepsilon\sigma\left(T_d^4 - T_a^4\right)$$

$$\Phi_{d-a} = K_{d-a}\left(T_d - T_a\right) \qquad K_{d-a} \cong \propto T_d^3$$

$$\begin{aligned} T_d^4 - T_a^4 &= \left(T_d^2 - T_a^2\right)\left(T_d^2 + T_a^2\right) \\ &= \left(T_d - T_a\right)\left(T_d + T_a\right)\left(T_d^2 + T_a^2\right) \\ &= \left(T_d - T_a\right)\left(T_d^3 + T_d^2 T_a + T_d T_a^2 + T_a^3\right) \\ &= \mathcal{O}\left(T_d^3\right) \quad \left(T_d - T_a\right) \end{aligned}$$

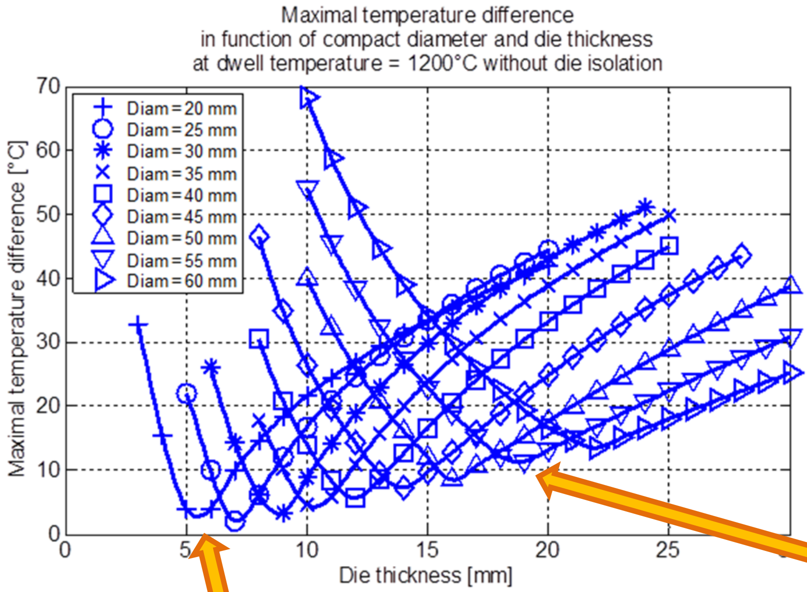
$$T_m \cong 4 \text{ à } 6\, T_a$$

T [°C]	1000	1200	1400	1600
T [K]	1273	1473	1673	1873
$K/K_{(1000°C)}$	1.00	1.49	2.12	2.91

The radiative conductance is significantly altered with the dwell temperature
➔ The balance between heat production and heat dissipation in the die is significantly altered with the dwell temperature

Influencing parameters: Compact diameter

- Al_2O_3, compact height 10 mm, T=1200°C
- Range of compact diameter 20 → 60 mm



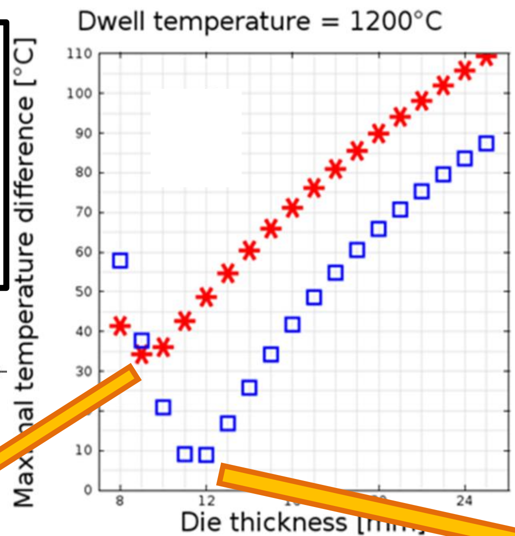
➔Optimal die thickness increase with compact diameter.

Small compact diameter
➔ optimal thickness out of the range of die thicknesses allowing mechanical reliability

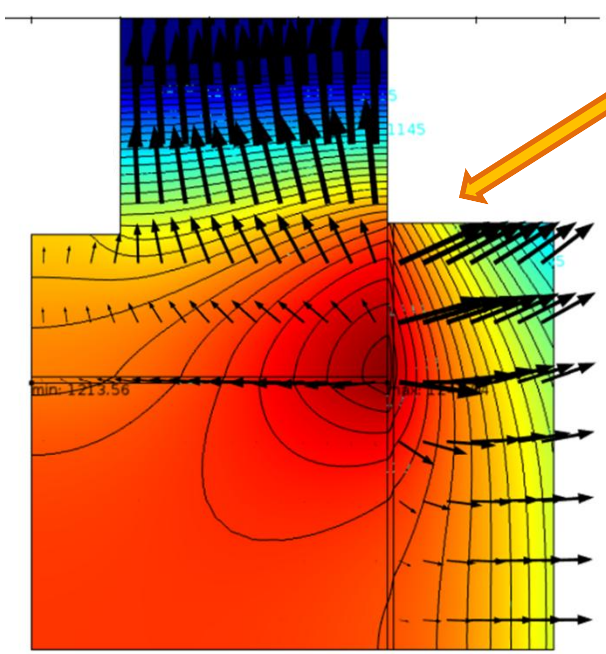
Influencing parameters: Compact height

- Dense Al_2O_3, compact diameter 40 mm, T=1200°C
- Compact height 30 mm

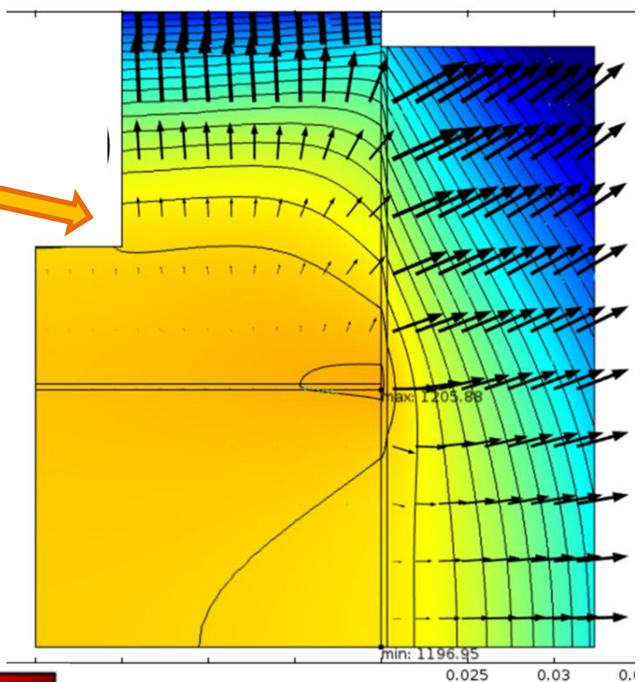
Standard die height (48mm)
➔Hot point appears
$\Delta T_{max} \approx 35°C$
at optimal die thickness = 9 mm

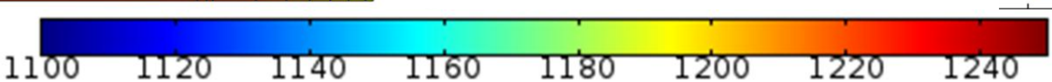


Increased die height (70 mm)
➔$\Delta T_{max} \approx 9°C$
at optimal die thickness = 12 mm



Thick compact ➔
Increased die height to avoid a hot point

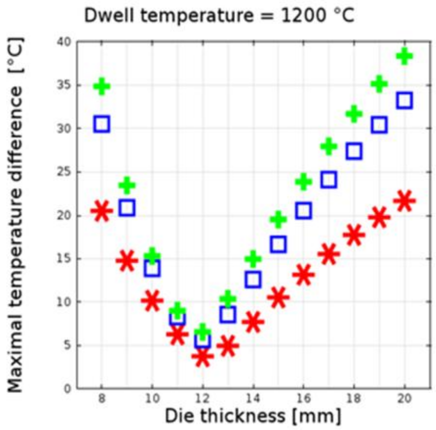


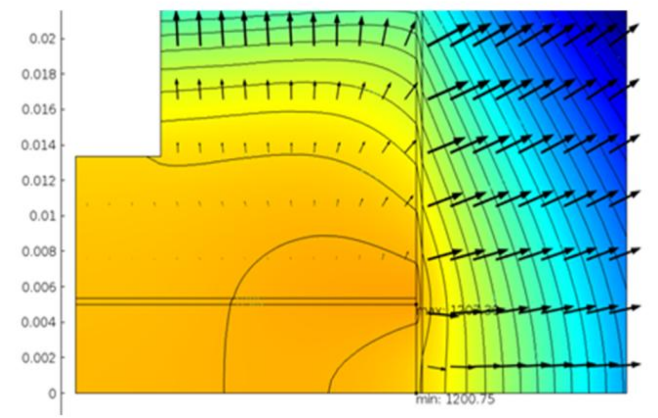


Influencing parameters: Compact thermal conductivity

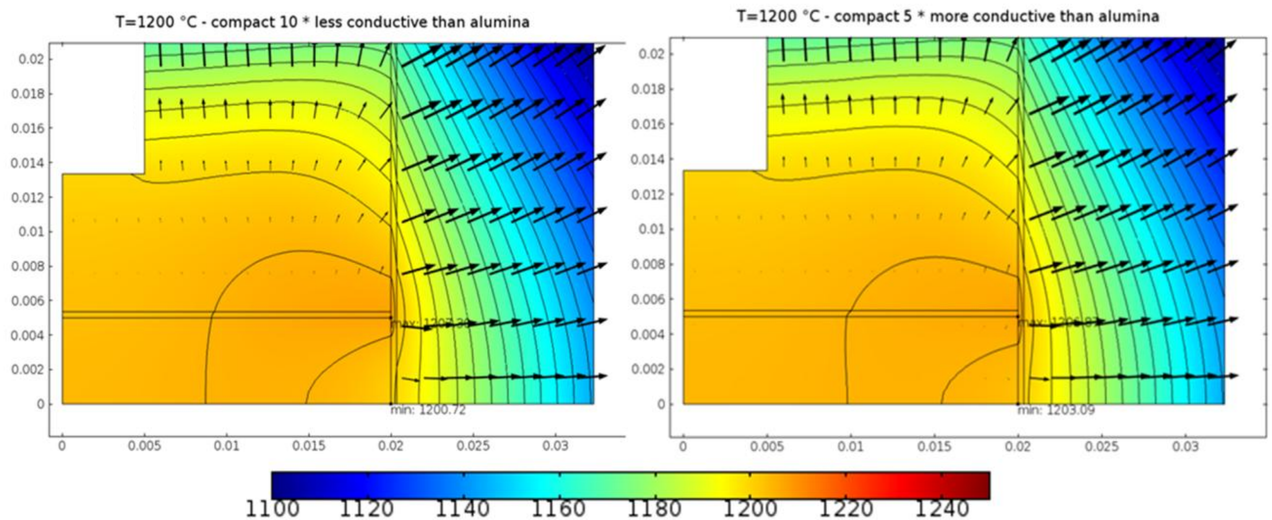
- Compact diameter 40 mm, compact height 10 mm, T_{dwell}=1200°C
- Compact thermal conductivity:
  - **Same as dense alumina**
  - **10 times less conductive**
  - **5 times more conductive**

The thermal conductivity of the compact does not influence the optimal die thickness value.



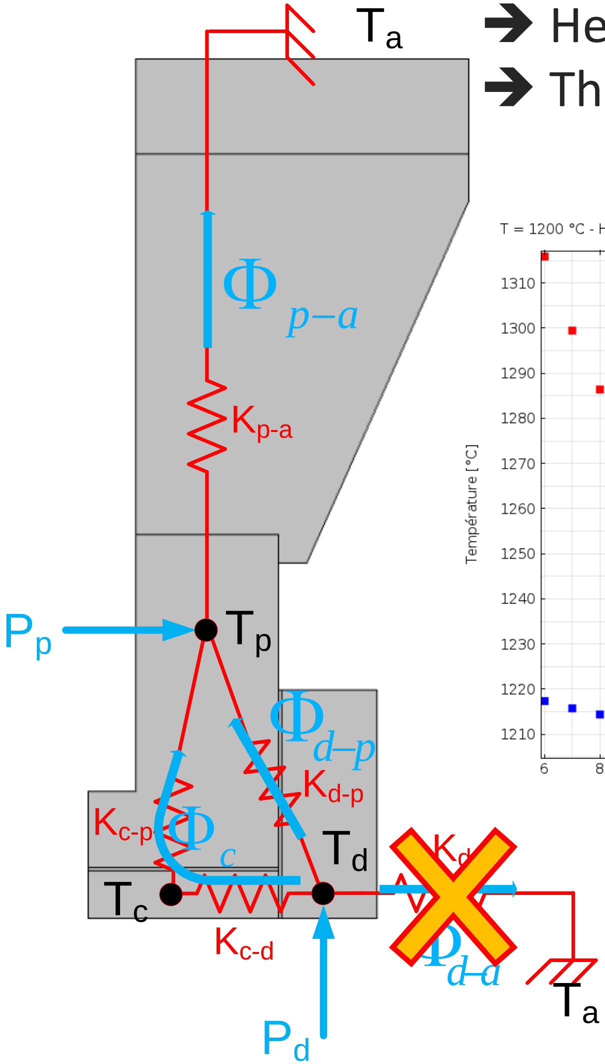


Because in case of perfect balance between local heat production and local dissipation, no heat flows through the compact

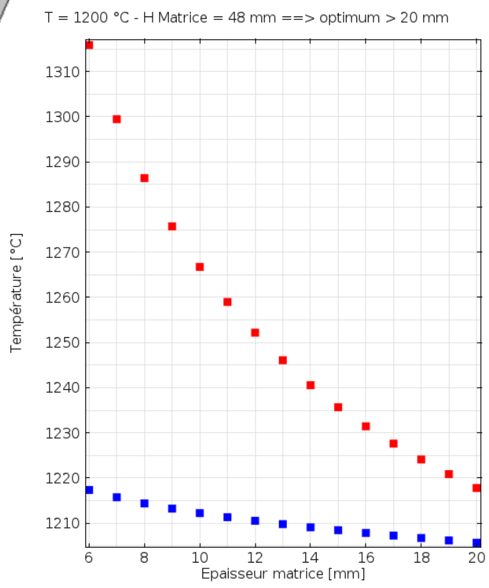


Influencing parameters : Graphite felt die isolation

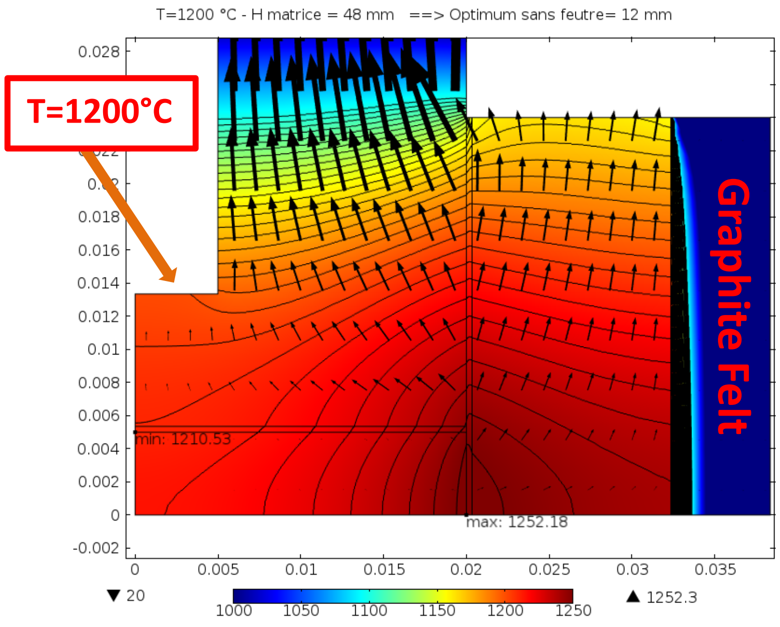
- Use of die isolation to decrease thermal dissipation by radiation.



- Heat produced in the die must dissipate through the punch
  ➔ Heat flows through the compact from the die to the punch
  ➔ Thermal gradient in the compact

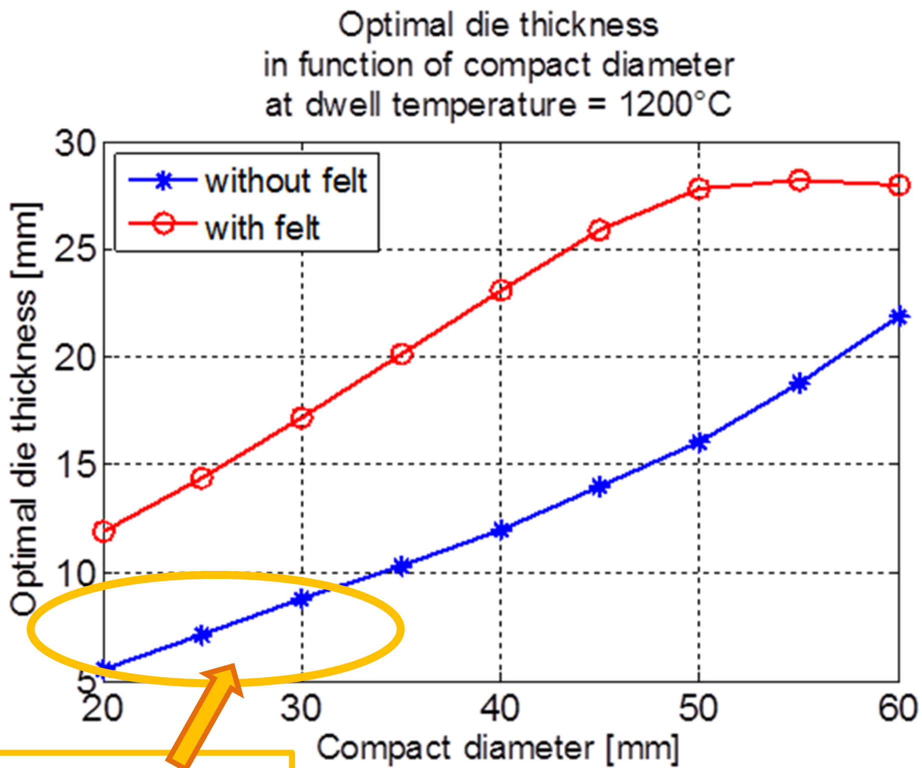


- 1200 °C, compact diameter 40 mm
  ➔ Optimal thickness without felt = 12 mm
  $\Delta T_{max} \approx 6.6$°C
- Same thickness with felt
  ➔ $\Delta T_{max} \approx 42$°C



Influencing parameters

Graphite Felt die isolation: It can be useful



Non reliable dies

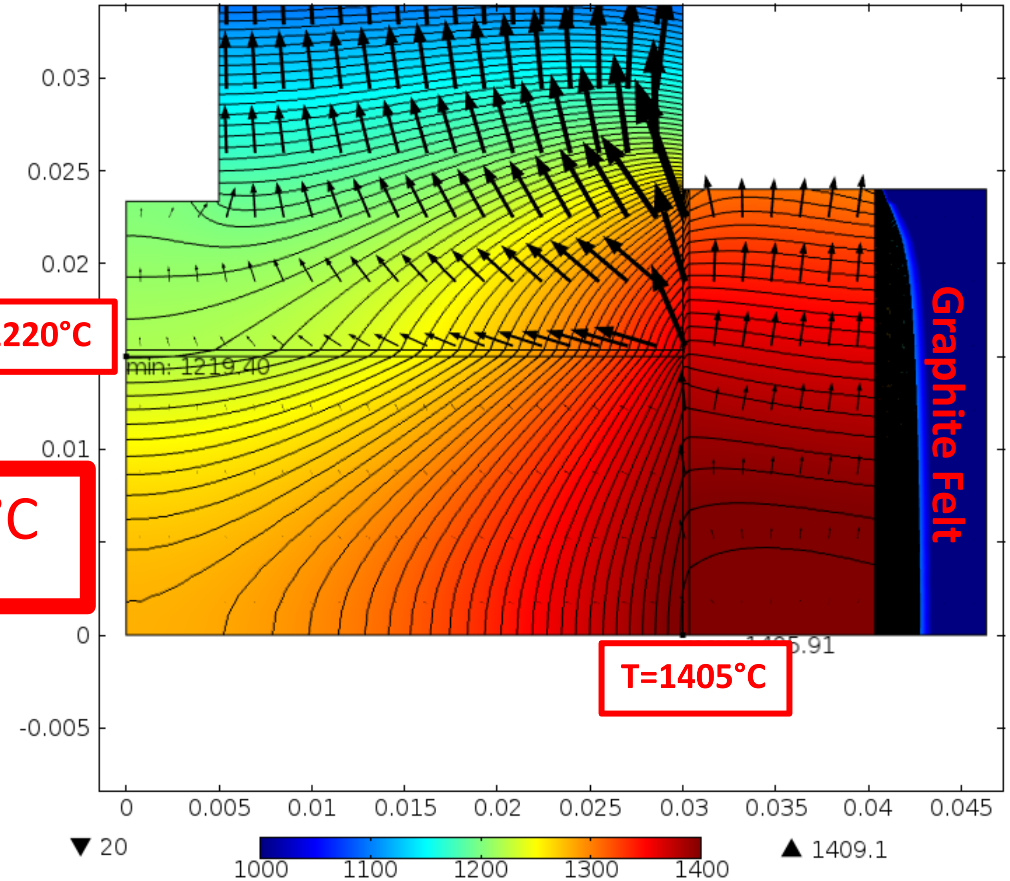
Use of graphite felt is interesting in case of small compact diameters because it allows to reach an optimal die thickness which gives a mechanically reliable die

Influencing parameters: worst case

- ❑ 1200°C, dense alumina
- ❑ Large compact diameter (60mm)
- ❑ Thin die (10mm)
- ❑ Thick compact (30mm)
- ❑ Standard die height (48mm)
- ❑ Graphite felt die isolation (6mm)

It could still be worse if the compact was less thermally conductive than dense alumina

➔ $\Delta T_{max} \approx 185°C$



Conclusions :

- The thermal gradient in the compact can be largelly decreased for non conductive materials
- Importance of understanding heat production, heat flows and the temperature field
- Optimization of temperature field = searching a balance.
- The balance = avoiding heat transfer through the compact.
- Tool for optimization : FEM
- After optimization : $\Delta T_{max} < 10$ °C.
- Necessary to machine graphite shaping tools when changing sintering conditions or compact dimensions
- Outlook: Experimental observation of the FEM results

  Based on the principle that the final microstructure depends on the dwell temperature (→ Guillaume Jean)

Thank you for your attention

Accepted paper:
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