Temperature Distribution during Spark Plasma Sintering (SPS)

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Objectives

- Determination of temperature distribution by FEM simulation (FlexPDE®) for typically used FAST tool geometries
- Influence of tool design (die thickness) on temperature distribution during densification by Spark Plasma Sintering for
  - electrically non-conductive material (silicon nitride)
  - electrically conductive material (tungsten carbide)
- Validation of determined FEM data by two point temperature measurements on FAST tool

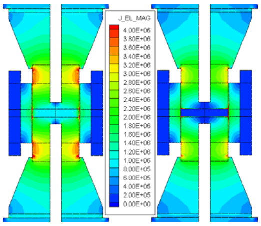

Fig. 1: Current density distribution for silicon nitride (left) and tungsten carbide (right) sample within sample and tool

Methods

Material	Sintering temp. [deg. C]	Isothermal heating [min]	Heating rate [K/min]	Die thickness [mm]	Sample diameter [mm]
Si_3N_4	1750	5	100	10, 15	30, 40, 60
WC	2000	5	100	10, 15	30, 40, 60

Tab. 1: Materials and parameters used for Spark Plasma Sintering

- **Varied parameters during Spark Plasma Sintering, FCT HP D 25/1 (Tab.1):**
  - Tool dimensions
  - Materials (electrically conductive or non-conductive material)
- **Measured parameters:**
  - Temperatures at different positions (Fig.1)
  - Radial distribution of α/β-Si_3N_4 content (XRD)
- **Evaluation and comparison:**
  - Calculated and measured temperature for all tool sizes and die thicknesses
  - Estimation of DT within sample volume on calculated data

Results

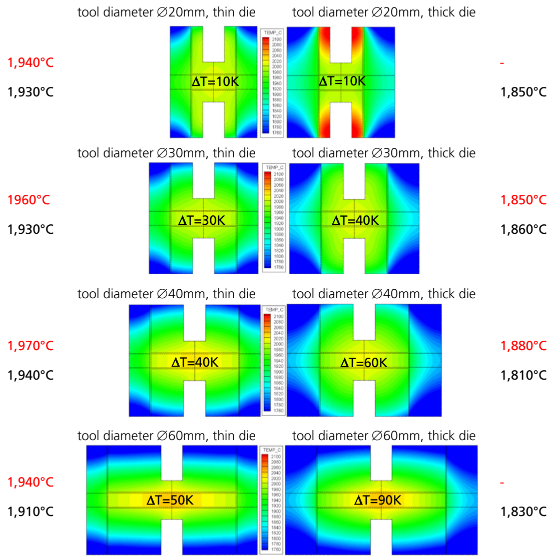

Fig. 2: Temperature distribution during isothermal dwell time at 2,000°C for WC and changing tool diameter and die thickness (measured – red, calculated – black)

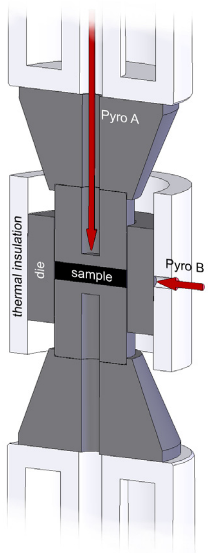

Fig. 3: SPS tool setup

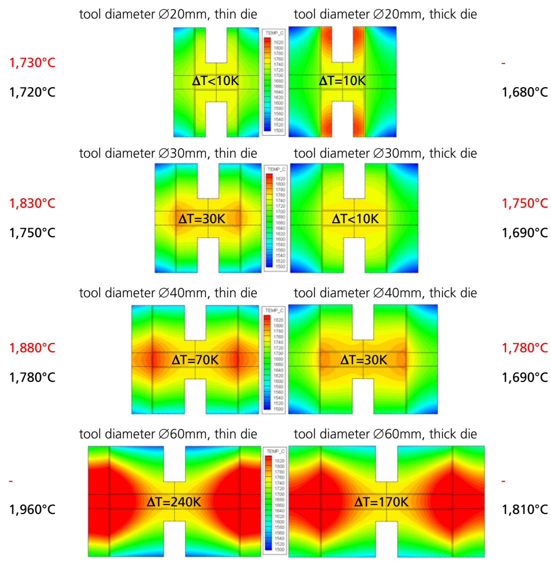

Fig. 4: Temperature distribution during isothermal dwell time at 1,750°C for Si_3N_4 and changing tool diameter and die thickness (measured – red, calculated – black)

Conclusion temperature measurements

- Temperature distribution during densification of
  - a non-conductive material (Si_3N_4): the temperature on the outside of the die is higher than the internal temperature
  - a conductive material (WC): the temperature on the outside of the die is lower than internal temperature
- The (absolute) temperature differences increase for conductive and decrease for non-conductive materials with increasing thickness of the die
- Die thickness influences change of temperature distribtuion and α/β-Si_3N_4 ratio

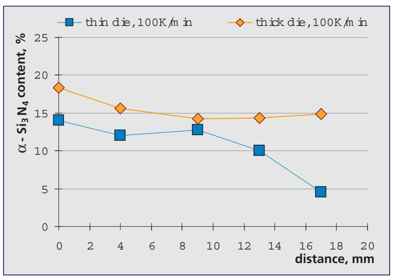

Fig.5: α-Si_3N_4 content as a function of distance from the center (sample diameter 40 mm)

Conclusion FE modelling

- FE modeling
  - is in good agreement with measured temperature data
  - can be used for tool design and determination of temperature distribution before starting experiments
- Possible reasons for temperature mismatch between measured and calculated data
  - FE model is idealized, has perfect geometries and contacts → perfect heat conductivity and heat flow between each part of the tool
  - Temperature dependent properties of FAST sintered materials can not be found (finer grain size → different electrical and thermal properties then traditional sintered samples)
  - Difficulty in exact external pyrometer positioning (movement by sintering shrinkage)
  - Radiation losses at external pyrometer position (not taken into account for FE simulation)

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