Spark Plasma Sintering: from controlling the microstructures to the development of complex shapes

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Highlights

- Controlled microstructure and tailored properties
- Reliable Finite Element Modeling of the Process
- Complex Shapes

Pulsed Electric Current Sintering (PECS) techniques have known a huge development over the last two decades. In particular, Spark Plasma Sintering (SPS) is an extremely powerful technique to sinter all classes of powders (metallic, ceramic) as well as composites. It consists in applying simultaneously a load and a high intensity pulsed direct current on tools containing powder to sinter. The very fast temperature increase is driven by the Joule's effect and the grain growth is almost suppressed. We will try to illustrate via several examples the potentialities of this technology in terms of control of microstructures, tailoring the properties of composites materials end developing complex shapes.

i) Ceramics exhibit interesting mechanical and thermo-mechanical properties (hardness, stiffness, wear resistance,...) but are penalized by their low toughness (K_{Ic}). However, some zirconia based ceramics (ZrO_2) were described as "ceramic steel" because, while retaining the usual properties of ceramics, they exhibit exceptionally high toughness which paved the way for many applications, particularly in the field of aeronautics and biomaterials. Spark Plasma Sintering (SPS) allows to densify materials, at lower temperature and with shortest sintering time, leading to nanoscale microstructures non-obtainable by natural sintering or hot pressing methods. This technique is thus well suited to produce nanostructured zirconia ceramics particularly interesting for biomedical applications (hip and dental implants), energy storage (SOFC…) and aeronautics (thermal barriers…). Recently, using SPS, we have densified 3Y-ZrO_2 ceramics which exhibit grain size around 200 nm and high mechanical properties (fracture strength σ_f = 692 MPa and toughness K_{Ic} = 10.3 MPa.m$^{1/2}$) [1].

ii) In electronics and telecommunications sectors the demand of new functional ceramics has recently known a drastic increase. To meet it, ferroelectric materials with high non-linear permittivity under electric field are considered for their potential use as ceramic capacitors, tunable filters or supercapacitors. In this context, elaboration of multimaterials mixing at different scale a ferroelectric phase ($Ba_{1-x}Sr_xTiO_3$ with x = 0 to 1) and low losses dielectric material (SiO_2, Al_2O_3, MgO, TiO_2) leads to composites with adjustable permittivity values and tenability [2, 3].

iii) Recently, the modeling of Spark Plasma Sintering by finite element method has known drastic development. Coupling three main physics, Electric Thermal and Mechanic (ETM), it allows now to predict the evolutions of temperature, grain size and porosity during the process. The electrical and thermal parts of the ETM model are used to calibrate the contact (thermal and electrical) resistances and to calculate the temperature at any point of the SPS tool and column. Ex-situ measurements of contact resistances were also performed in different conditions to compare the results of the calibrations [4, 5].

Creep parameters are identified on dense and porous materials, and sintering models (Olevsky and Abouaf) are used to predict the densification of the powders to be sintered. Last, a grain growth law coupled with the densification model may also be considered. Finally, this type of modeling enables the definition of the optimized SPS parameters and tool geometry in order to minimize the porosity and microstructure gradients in a complex shape part.

This will be illustrated by two examples.

- In the first one a fully coupled numerical computation model on TiAl 48-2-2 commercial powder densification will be presented. The experimental relative density map will be presented and the correlation with the numerical model will be analyzed, both on simple and complex shape specimens. The evolution of the microstructure during creep test but also after densification of a complex part will be presented and correlation with numerically assessed temperature field will be discussed.
- The second part will be devoted to Ti-6Al-4V alloy. On simple shape, sintering cycles below or above T_β allow to obtained, in repeatable manner, fully dense samples with controlled and homogeneous microstructures. Fully dense dog-bone specimens have been obtained and their mechanical behavior have shown high values and very homogeneous of ultimate tensile strength.

iv) Finally, Electro Thermal and Mechanical modeling of the process by Finite Element allows the definition of the optimized SPS parameters, tool geometry in order to minimize the porosity and microstructure gradients in a complex shape part and to improve the productivity limitations. Different approaches will be detailed.

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Keywords
Ceramics, alloys and composites; Tailored microstructure and properties; Finite Element Modeling; Complex Shapes