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Application of electric current assisted sintering techniques for advanced processing of energy materials

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Abstract

At Forschungszentrum Jülich, the Institute of Energy and Climate Research (IEK-1: Materials Synthesis and Processing) has long-term expertise in the field of electric current assisted sintering (ECAS) techniques. IEK-1 operates a broad spectrum of related equipment including Field Assisted Sintering Technology/Spark Plasma Sintering (FAST/SPS), Hybrid FAST/SPS with additional heater, Ultra-fast high temperature sintering (UHS), Flash SPS, Flash Sintering (FS) and Sinter Forging (SF). Current research topics - ranging from fundamental to applied research - are discussed on selected examples.

Introduction

Forschungszentrum Jülich GmbH, member of the German Helmholtz Association (HGF), conducts cutting-edge research to solve global challenges in the areas of health, energy and environment as well as information technology. Combined with the two key competences physics and supercomputing, research in Jülich has a long-term, basic and interdisciplinary character, but also deals with the know-how transfer of specific technological applications to industry. In the energy and environment sector, Institute of Energy and Climate Research (IEK) covers all related topics to bring German "Energiewende" to success. In this context, the subdivision IEK-1: Materials Synthesis and Processing (Director: Prof. Dr. Olivier Guillon) provides a worldwide unique portfolio of materials synthesis and processing technologies for envisaged applications in the field of all-solid-state batteries, gas separation membranes, solid oxide fuel and electrolyser cells and high-temperature materials e.g. for thermal barrier coatings. In all cases, advanced processing of ceramic and metal powders plays a key role and requires fundamental know-how with respect to powder synthesis, powder handling, powder characterization, shaping technologies and sintering. It is a well-known fact that high performing materials are often quite sensitive to processing conditions and show an inherent risk of abnormal grain growth, evaporation, chemical reaction or decomposition especially if sintered at high temperatures with long dwell times. Electric current assisted sintering (ECAS) techniques are highly promising to face this challenge due their potential of significantly enhancing and accelerating sintering kinetics compared to conventional sintering by providing an electric field, respectively an electric current, as additional processing parameter. There are three basic principles how this additional degree of freedom can be advantageously used for fast sintering with high heating rates and short dwell times:

1) Indirect heating of a non-conductive powder in a conductive tool: In field assisted sintering technique/spark plasma sintering (FAST/SPS) devices, a rigid, electrically conductive tool (e.g. made of graphite) is heated by the Joule effect (= resistance heating) and the heating power is effectively transferred to the powder by thermal conduction based on the tight contact between powder and sintering tool, which can be further improved by a contact layer (e.g. graphite sheet) [1]. Recently, a novel technology based on the same principle has been introduced by Wang et al. [2]. Ultra-fast high temperature sintering (UHS) with heating rates up to 10^4 K/min and temperatures up to 3000°C has been successfully demonstrated by placing a powder compact between two flexible, conductive felts (e.g. carbon felts). Then, these felts are rapidly heated by the Joule effect enabling densification of powder compacts within less than 1 min. **Figure 1a** shows custom made UHS set-up of IEK-1 in operation. It enabled to sinter a $SrTiO_3$ powder compact to almost complete density using a dwell time of 10 s (**Figure 1b**). It is obvious, that understanding the underlying mechanisms and their effective use is a hot topic in the sintering community due to the large potential of increasing the overall efficiency of sintering.

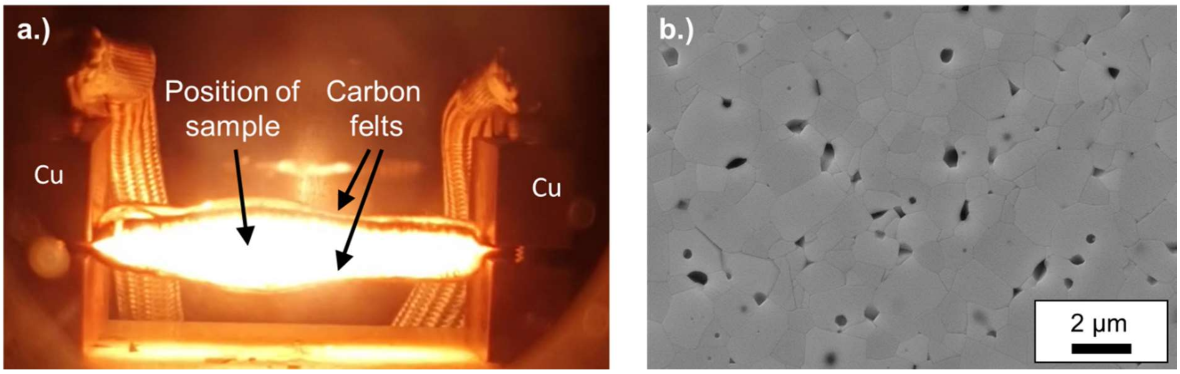

Figure 1: a.) Custom made ultra-rapid high temperature sintering (UHS) device at IEK-1 (sample size 8 mm, Cu = copper electrodes) **b.)** Microstructure of $SrTiO_3$ densified by UHS with a dwell time of 10 s in vacuum, density 96 %.

2) Heating of an at ambient conditions non-conductive powder to an onset temperature, where the material becomes conductive and current starts to flow through the sample: In 2010, this specific kind of sintering has been introduced as "Flash Sintering (FS)" [**3**]. When achieving the onset temperature, the powder is directly heated mainly by the Joule effect, but additional mechanisms of energy dissipation within the sample like formation of defects are under discussion [**4,5**]. In voltage-to-current controlled FS experiments densification is a very dynamic process and takes place in seconds [**6**]. Alternatively, current-rate controlled FS [**7,8**] as well as power-rate controlled FS [**9**] enable a better control of densification and microstructure formation. A similar densification mechanism can be also triggered in a hybrid FAST/SPS device. In the case of "Flash SPS", powder compact is heated by an external heater to onset temperature and then the fast heating is done by a DC current pulse with defined length and limitation of maximum power enabling densification within seconds [**10,11**]. Manière et al. report heating rates in the range of 10^4 – 10^6 K/min, but control of the complete sintering cycle remains challenging [**11**].

3) Direct heating of a conductive powder: Here, the current flows through the sample from the beginning of the experiment and Joule effect is the main heating mechanism. An advantage to established sintering technologies is the potential of achieving increased energy efficiency when heating power is directly dissipated by the sample. The principle of "Flash SPS" can be also applied to conductive powders [**11,12**]. In this case, external heater is not necessarily required. As promising alternative, ultra-fast densification of conductive powders with heating rates up to 10^6 K/min and full densification within milliseconds has been demonstrated by sudden release of energy stored in a capacitor via the sample [**13,14**]. This technology has been introduced as electro-sinter-forging (ESF) or electro-discharge-sintering (EDS).

In all cases, the densification can be significantly improved by superposition of an external load. In ideal case, the load promotes the further acceleration of the sintering kinetics and therefore, to further decrease sintering temperature and dwell time. It is obvious, that electrical conductivity of powder and tool material decides about the main heating mechanism. If e.g. applying graphite as tool material, metals, alloys and ceramics with electrical conductivity higher than graphite like TiC, TiN, Ti(C,N), WC, TiB_2, ZrB_2 and MAX-phases can be directly heated, while most oxides (Al_2O_3, ZrO_2, YSZ, MgO, CeO_2, GDC,…) and specific carbides and nitrides (BN, Si_3N_4, SiC, B_4C) just enable indirect heating. In the case of using a non-conductive die (e.g. boron nitride die) or completely omitting the die it is possible to force the current through the sample, which is attractive with respect to energy efficiency but might have limitations with respect to homogeneous heating and densification as well as keeping the shape. **Table 1** summarizes general advantages and challenges of ECAS techniques.

Table 1: Advantages and challenges of ECAS techniques (CAPEX = capital expenditure, OPEX = operational expenditure).

Advantages	Challenges
• High heating rates and optional external pressure enable clear reduction of sintering temperature and processing time compared to conventional sintering • High heating rates may limit or inhibit grain growth, therefore fine grained microstructures down to nano meter scale become obtainable • Chemical reactions, decomposition and evaporation can be reduced or avoided	• Limitations with respect to sintering of complex shaped parts • Limitations with respect to fully automated production of large batches, application requires careful calculation of CAPEX/OPEX. • Choice of suitable tool material and reliable control of interfacial reactions between sample and tool material, respectively contact layer.

• Highly promising for materials with high activation energy for sintering like refractory materials (e.g. borides, carbides, nitrides, refractory metals) • Highly promising for composite materials combining phases with large difference of properties (e.g. diamond reinforced steels) • Promising with respect to energy efficiency especially if the heating power is directly dissipated by the sample	• Reliable adaption of the proportional-integral-derivative (PID) control of the FAST/SPS device to the tool material. • Reliable control of the fast and dynamic nature of ECAS techniques due to being prerequisite for homogeneous densification • Reliable measurement of the sample temperature and reliable indication of temperature gradients in the sample. Therefore, modelling of temperature distribution is recommended.

To take full advantage of ECAS techniques, the challenges must be solved properly. At Forschungszentrum Jülich´s institute IEK-1, a broad and long-term expertise in the field of ECAS techniques exist [1,15]. Hereinafter, the experimental equipment available at IEK-1 including Field Assisted Sintering Technology/Spark Plasma Sintering (FAST/SPS), Hybrid FAST/SPS with additional heater, Ultra-fast high temperature sintering (UHS), Flash SPS, Flash Sintering (FS) and Sinter Forging (SF) is introduced and current research topics are discussed on selected examples.

ECAS equipment available at IEK-1

At IEK-1, a broad spectrum of ECAS techniques is available. **Table 2** summarizes the main technical details of our devices and introduces related applications.

Table 2: ECAS techniques available at IEK-1 and current applications.

Equipment, manufacturer	Operation mode	Operation parameters	Example of applications at IEK-1
HP-D 5 FCT Systeme GmbH, Rauenstein, Germany	FAST/SPS	Pulsed DC current Max. power 37 kW Max. voltage 8V Max. temperature 2,200°C Load 2 – 50 kN Max. sample size ≤ 30 mm Vacuum 0.5 mbar Ar, Ar/2.9% H_2, N_2, technical air (up to 600°C)	Influence of thermal insulation [16] High pressure FAST/SPS of battery materials [17,18] Atmosphere control in a FAST/SPS device [19] Cold sintering of ZnO [20-22] Sintering of NASICON battery materials [23]

			Sintering of refractory materials ZrB_2 [24] Smart W alloys [25,26]
H-HP-D 25 SD/FL/MoSi Hybrid system FCT Systeme GmbH, Rauenstein, Germany	FAST/SPS	Pulsed DC current Max. power 60 kW Max. voltage 8V Max. temperature 2,200°C Load 10 - 250 kN Max. sample size ≤ 100 mm Vacuum 0.5 mbar Ar, Ar/2.9% H_2, N_2, technical air (up to 600°C)	Scaling up of FAST/SPS technology [15] Joining of metallic components for PEM electrolyzers Net-shape manufacturing of ceramic and metallic parts in a graphite powder bed
	Hybrid FAST/SPS	Operation FAST/SPS with additional heater a.) Induction coil b.) $MoSi_2$ furnace Max. heating power 80 kW	Scaling up of FAST/SPS technology [15]
	Flash SPS	Loading of the sample and adjusting the heating power in the control unit, rapid heating of the sample with this heating power, no control of the heating rate, optional preheating of non-conductive powders to achieve the onset temperature	Preliminary experiments with NdFeB materials, no preheating required
	FS	Preheating with induction coil or $MoSi_2$ furnace, power source of FAST/SPS unit turned off, heating by external AC and DC power source: Max. current 25 A Max. voltage 1,000 V AC frequency 10 Hz – 80 kHz Sample size ≤ 40 mm	Scaling up of Flash Sintering technology with pellet shaped samples [15] Optimization of technique with YSZ and $SrTiO_3$ as model materials

DSP 515 Dr. Fritsch, Fellbach, Germany	FAST/SPS	Constant DC current Max. power 170 kW Max. voltage 8V Max. temperature 2,200°C Load 47 - 555 kN Sample size ≤ 180 mm Vacuum 20 mbar Ar, Ar/2.9% H_2, N_2 Debinding unit (cooling trap)	Scaling up of FAST/SPS technology [15] Joining of parts for PEM electrolyzers Debinding and sintering of tape casted ceramic and metal powders Recycling of metals via powder metallurgical route
Ultra-fast high temperature sintering device Custom made	UHS	Heating of two carbon felts clamped between opposite copper blocks by AC or DC current flow External AC and DC power source: Max. current 25 A Max. voltage 1,000 V AC frequency 10 Hz – 80 kHz Sample size ≤ 8 mm Vacuum 0.5 mbar Ar, Ar/2.9 % H_2	Preliminary experiments with $SrTiO_3$ and battery materials
Flash Sintering device Custom made	FS	Vertical furnace Max. temperature 1,200°C Constant DC current Max. current 2 A Max. voltage 300 V Dog bone shaped samples with gage dimensions 15x3x2 mm^3, contact via Pt wires/Pt paste Vacuum 0.5 mbar Ar, Ar/2.9% H_2	Flash sintering of GDC [8,27,28] Fundamental flash sintering studies on YSZ, $SrTiO_3$ and NiO/YSZ
Sinter Forging device Custom made	Sinter experiments in the presence of an electric field and uniaxial pressure	Vertical furnace Max. temperature 1,300°C AC and DC current Max. current 20 A Max. voltage 1,000 V	Investigation of sintering parameters (viscosities, viscous Poisson´s ratio, sintering stress) under

	FS	Pellet samples ≤ 10 mm Loading dilatometry equipment consisting of electromechanical testing system (Instron, Norwood, USA) and two optical laser scanners Contact by Pt electrodes on both sample faces Air atmosphere	electric field on the example of YDC [29-31] Flash sintering of $BiFeO_3$ [32] Flash sintering of 3YSZ, influence of sample size on onset temperature [33]

Densification of powders via FAST/SPS requires a conductive tool, which is placed between the water cooled electrodes. Depending on the heating power and the tool geometry, heating rates up to 1,000 K/min are possible, but in usual praxis heating rates range between 50 – 200 K/min. In most cases, graphite is applied as tool material, which enables sintering temperatures up to 2,200°C but restricts the maximum load with standard graphite qualities to 50 – 100 MPa. Alternative tool materials enable significantly higher pressures, but limitations with respect to the maximum operation temperature must be carefully considered. At IEK-1, there exists experience with application of metal tools made of hot work steel (e.g. W-360, Böhler, Germany) or molybdenum based alloys (e.g. TZM, Plansee, Austria), which can be used for pressures up to 400 MPa [17,18,22]. In general, metal tools are prone to creep and recrystallization. If exceeding a critical operation temperature, significant decrease of strength and ductility results. Therefore, it seems to be reasonable to limit the maximum operation temperature of steel tools to 600°C and for TZM tools to 1,100°C, but further studies are required to prove these values. For realizing high pressures and high temperatures, electrically conductive carbides and nitrides and composites thereof are investigated as tool materials [34]. In this case, inherent brittleness and complex manufacturing requires handling with care. Usually, a conductive layer (e.g. graphite or metal foil) is placed between tool and sample aiming on to reduce the contact resistance at the interface. The interlayer helps to avoid sticking of the sample on the tool walls, but on the other hand enhances the risk of interfacial reactions and impurity uptake. Another important thing to consider is the sintering atmosphere. Usually, FAST/SPS is conducted in mild vacuum, Argon or Nitrogen to protect the tools from severe oxidation, but when handled with care other atmospheres like strongly reducing Argon/H_2 or even air are possible [19].

Examples of applying ECAS technologies at IEK-1

A general overview about manifold application of ECAS techniques at IEK-1 can be found in a recent review paper [15]. Some of the current research topics are shortly introduced below.

Cold sintering of ZnO and battery materials in a FAST/SPS device: In the last years, cold sintering gained a lot of interest in the sintering community [35]. The application of water based or other liquid sintering aids in combination with high pressure enables almost full densification of oxide ceramics at temperatures below 600°C. Conducting cold sintering in a FAST/SPS device is attractive due to a better process control compared to non-instrumented presses with respect to atmosphere control, liquid evaporation and data logging. FAST/SPS with steel tools was extensively used to study the mechanisms of cold sintering process and to optimize the main processing parameters on the example of ZnO [20-22]. Almost full densification of ZnO (> 99 %) while keeping the grain size below 200 nm was achieved at 250°C, 300 MPa and 20 min dwell time when 3.2 wt. % water was added as sintering aid (**Figure 2**) [22]. Currently, there is a lot of interest to apply cold sintering for the densification of all-solid-state battery materials. At IEK-1, cold sintering is used for the processing of composite electrodes based on $LiMn_2O_4$ (LMO) aiming on to avoid interfacial reactions with the electrolyte (e.g. $Li_{1.5}Al_{0.5}Ti_{1.5}(PO_4)_3$, LATP), which take place at temperatures above 600°C [36].

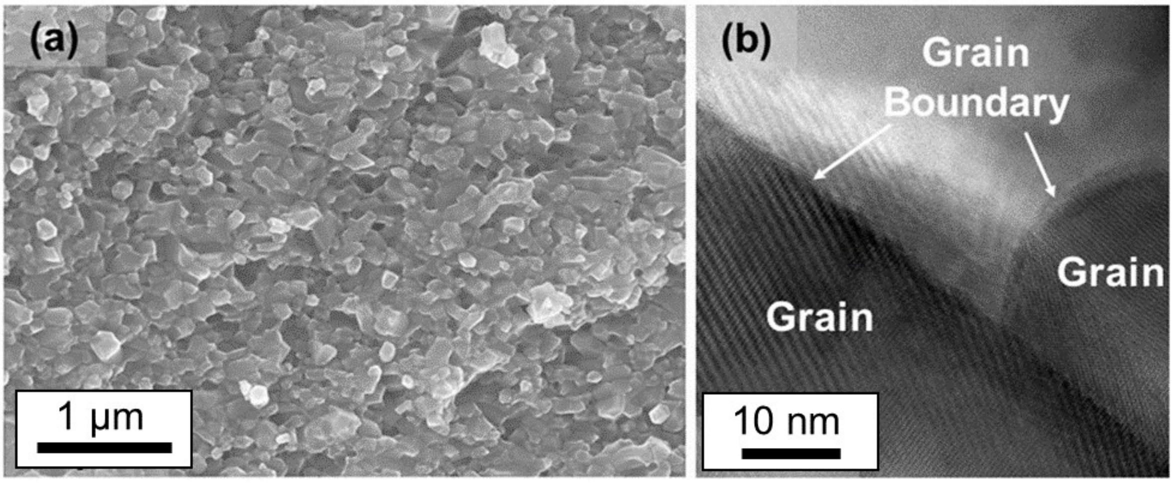

Figure 2: ZnO with a density > 99% and grain size below 200 nm cold sintered at 250°C and 300 MPa using pure water as sintering aid a.) Overview b.) Detail [22].

High-pressure FAST/SPS of battery materials: An alternative to improve densification in a FAST/SPS device at moderate temperatures is a further increase of the applied pressure. Recently, a composite cathode for an all-solid-state lithium battery consisting of $LiCoO_2$ as electrochemically active material and $Li_7La_3Zr_2O_{12}$ (LLZ) as ionic conducting phase in a ratio of 50:50 (wt. %) was successfully sintered in the temperature range of 675 – 750°C to a density > 95 % by applying high pressure up to 440 MPa in Mo-based TZM tools [**17,18**]. The dwell time at sintering temperature was 10 min. **Figure 3** shows the microstructure of the LCO/LLZ-composite cathode sintered at 750°C. Furthermore, sintering was conducted in Ar atmosphere to diminish Li evaporation. The result demonstrates the potential of high pressure FAST/SPS for almost full densification of battery materials at moderate temperatures, but residual amorphous phases on grain boundaries deteriorated electrochemical performance and cycling stability. Crystallization of this layer by post-annealing at 1050°C improved electrochemical performance. Developing strategies to reliably avoid formation of amorphous phases at the interfaces without the need of additional thermal treatments and applying this technology for other cathode materials like $Li[Ni_{1-x-y}Co_xMn_y]O_2$ (NMC), $Li[Ni_{1-x-y}Al_xMn_y]O_2$ (NCA), and $Li_2NiMn_3O_8$ (NMO), which are even more sensitive to interfacial reactions, are part of our ongoing work.

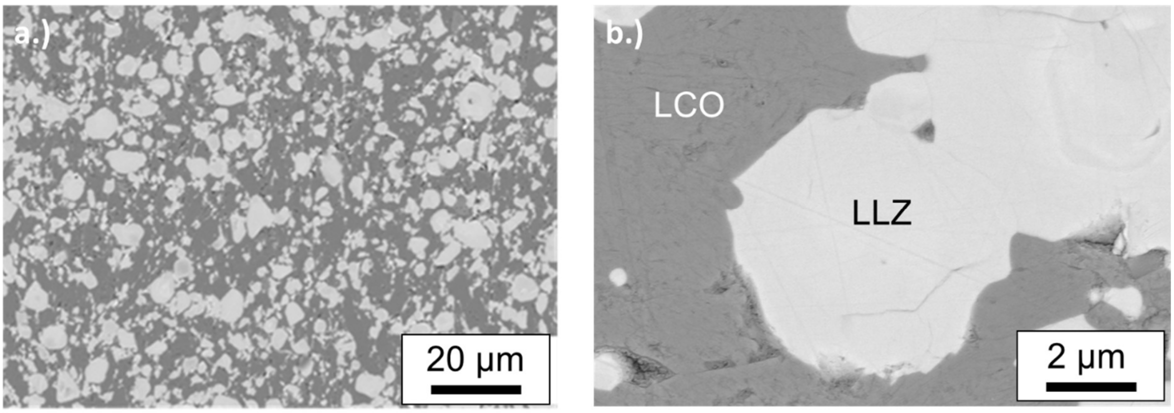

Figure 3: Microstructure of the mixed cathode of an all-solid-state battery processed by high-pressure FAST/SPS [**18**] **a.)** Overview **b.)** Detail (density 95%, 750°C, 440 MPa, LCO = $LiCoO_2$, LLZ = $Li_7La_3Zr_2O_{12}$).

Atmosphere control in a FAST/SPS device: Recently, we demonstrated that an atmosphere change in the FAST/SPS device from reducing to oxidizing conditions during cooling can be helpful to sinter materials sensitive to chemical expansion like Gadolinium doped ceria (GDC) [19]. Atmosphere control in a FAST/SPS is also expected to be advantageous for debinding and sintering of parts, which still contain organic binders (e.g. tape cast layers).

From FAST/SPS to Flash Sintering (FS): Contrary to Flash SPS, which can be done in a conventional FAST/SPS device, FS is conducted with much higher electric fields up to several 100 V/cm and moderate current densities of a few A/cm^2. In a tight cooperation with University of Boulder, USA, detailed studies on FS of gadolinium doped ceria (GDC) were conducted on dog bone shaped samples [8,27,28]. Results of these studies are the basis to transfer FS from dog bone shaped samples to pellets in our hybrid FAST/SPS device H-HP-D 25 SD/FL/MoSi. Our research hypothesis is that current rate controlled FS and optimized contact to the electrodes are the keys to reliably control FS and avoid hot spot formation even at larger sample dimensions. Related experiments are currently done with 8YSZ and $SrTiO_3$. **Figure 4** shows the potential of current rate controlled FS achieved with GDC on lab scale [8].

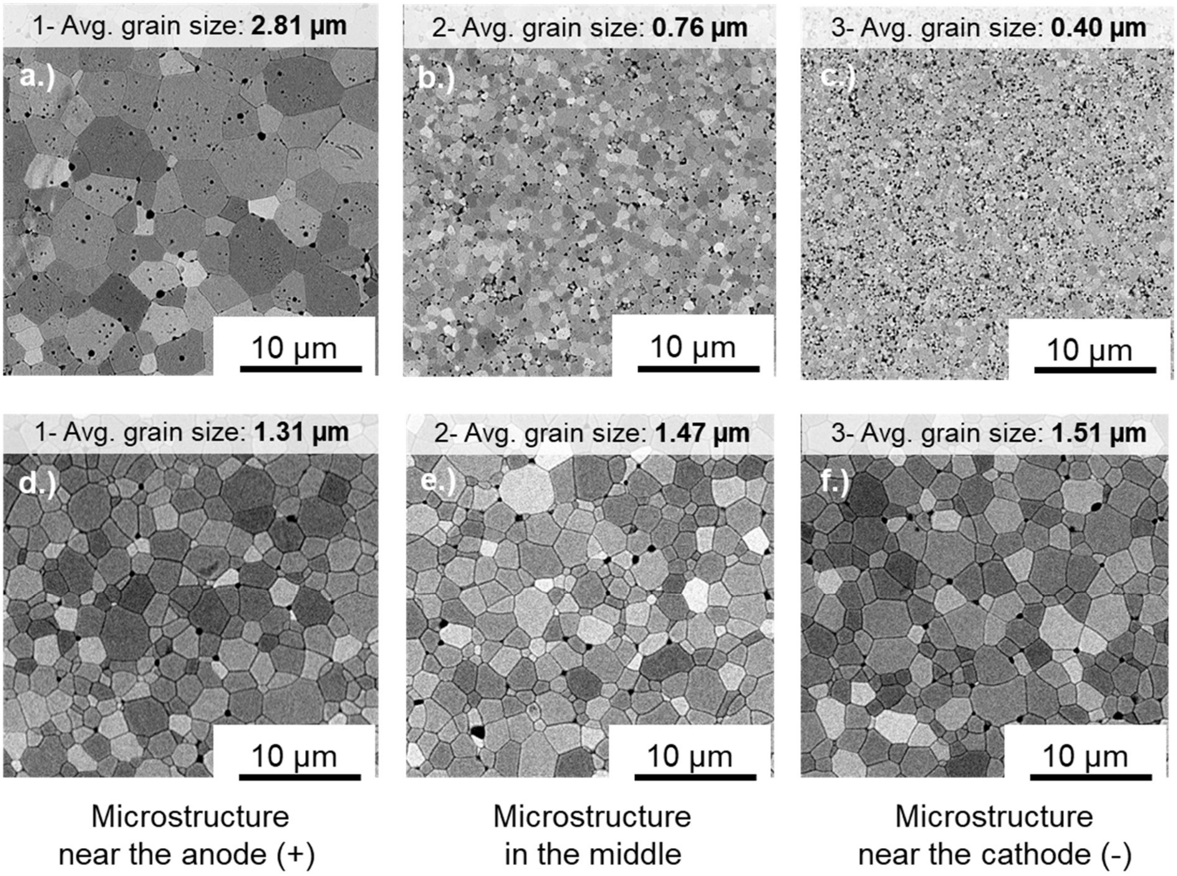

Figure 4: FS of gadolinium-doped ceria (GDC) at a furnace temperature of 680°C in air, sample length 15 mm **a.-c.)** Voltage-to-current control, DC electric field 150 V/cm, maximum current density 200 mA/mm^2 **d.-f.)** Current rate control, constant current rate 50 mA/min, maximum current density 200 mA/mm^2 [28].

Study of sintering behaviour in the presence of electric field: Experimental results on GDC reveal that even under conventional FAST/SPS conditions, electric field can cause polarity induced grain growth [37]. Our custom made sinter forging device enables to study the influence of electric fields on sintering parameters like uniaxial viscosity, uniaxial sintering stress, Poisson´s ratio as well as microstructure evolution (grain growth) in detail. Cao et al. did a related study on sintering yttria doped ceria pellets without and with AC electric fields, while reliably excluding the effect of Joule heating [29-31]. She demonstrated a clear modification of densification behaviour and change of sintering parameters in the presence of moderate electric fields (below the flash regime). Amongst others, ease of grain boundary sliding in the presence of AC electric field was found.

Conclusions

The Institute of Energy and Climate Research (IEK-1: Materials Synthesis and Processing) of Forschungszentrum Jülich GmbH operates a unique platform of electric current assisted sintering (ECAS) devices including conventional Field Assisted Sintering Technology/Spark Plasma Sintering (FAST/SPS), Hybrid FAST/SPS, Ultra-fast high temperature sintering (UHS), Flash SPS, Flash Sintering (FS), and Sinter Forging (SF). In general, all of these techniques are attractive for densification of ceramic or metal powders, which are difficult to sinter due to low sintering activity, limited chemical stability or sensitivity to interfacial reactions and evaporation. Furthermore, these techniques are known for their efficient generation of heat. Direct heat transfer enables high heating rates in combination with reduced sintering temperatures and dwell times. Amongst others, this combination is attractive to decrease overall energy consumption and to reduce grain growth to a minimum. Nevertheless, reliable control of these often quite dynamic sintering processes, which take place far from thermodynamic equilibrium, is challenging and our strategies to face these challenges are shortly introduced on examples of current research. In addition, mesoscale and macroscopic modelling is a valuable additional tool to better understand the underlying sintering mechanisms. For measuring the required input parameters such as uniaxial viscosities, sintering stresses, and viscous Poisson´s ratio, we operate a custom-made instrumented sinter-forging device, which enables to study the influence of electric fields on these parameters.

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