

FIELD ASSISTED MATERIAL ENGINEERING (FAME)

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In order to further improve the energy saving of Spark Plasma Sintering we have developed a very rapid sintering technique called Flash SPS (FSPS) with heating rates in the order of 10^4 - 10^5 °C/minute[1]. Unlike the Flash Sintering based on high voltage ($\approx 100V$), FSPS is based on low voltage ($\approx 10V$) and it can be up-scaled to samples volumes of several tens of cubic centimetres. Flash SPS allows densification of metallic conductors like ZrB_2 and HfB_2 , under a discharge time as short as 20-30 seconds. FSPS of semiconductors like silicon carbide and boron carbide was also demonstrated. Highly customized and versatile equipment with ultrafast responsive controls and programmable bipolar power supplies (up to 20 kHz, 1 MA, 500V) has been built. The developed methodology has been applied to produce FSPSed samples even larger than 6 cm in diameter of ultra refractory materials. Understanding the intrinsic electrical field role in the triangle properties-microstructure-processing remains one our primary scientific goal and the main open question. We tried to give some answers by approaching the problem at different length scales (see figure 1) by developing dedicated equipment/controls, simulations (FEM and ab-initio), thermo-kinetic analysis, in situ observations and accurate temperature measurements/calibrations.

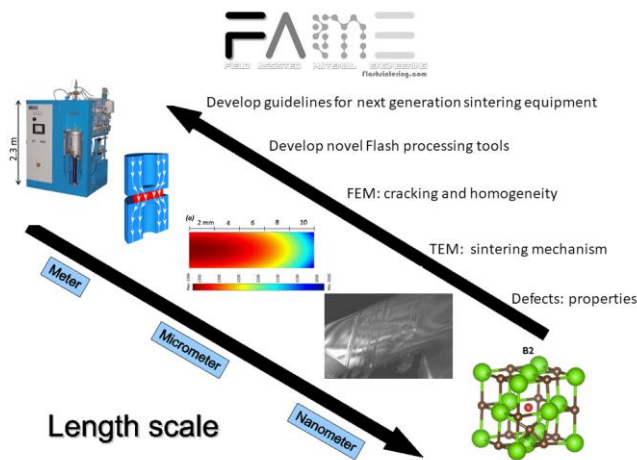


Figure 1 – Proposed Multiscale approach

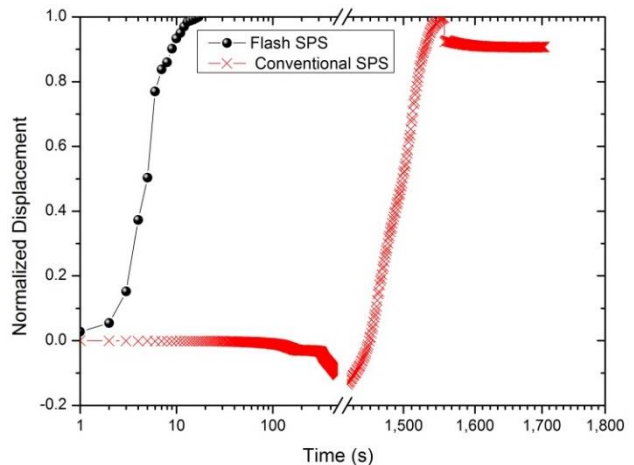


Figure 2 – Comparison of normalized displacement (SiC, Φ 20mm) as function of time for Flash and Conventional SPS

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HYBRID SINTERING – THE BENEFICIAL COMBINATION OF SINTERING PRINCIPLES

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Key Words: FAST, SPS, Hybrid, Hintersing

„Hybrid Sintering“ can be defined as a beneficial combination of different sintering principles, e.g. direct and indirect heating, uniaxial and gas pressure, high and low electrical fields (Figure 1). In the technological context this can be completed by debinding, decoupled pre-heating and cooling as well as rate controlled sintering optionally. Hybrid sintering systems enable the development and production of all new material solutions.

FCT Sintering Technology	ACTIVE SINTERING PRINCIPLES			
	HEAT	GAS PRESSURE	MECH. PRESSURE	ELECTR. FIELD
PRESSURELESS	●			
GAS PRESSURE (SINTER) - HIP	●	●		
HOT PRESSING	●		●	
FAST/SPS	○		●	●
Hybrid Sintering	●	●	●	●
FAST/Hybrid (FAST/Flash)	●		●	●

Fig. 1 – Combination of active sintering principles forming different classical and hybrid sintering technologies

“FAST/Hybrid” is a real trendsetter in this field, complementing the conventional hot pressing by an additional pulsed current direct heating of the powder compact (“FAST/SPS”). It is well established that this combination (Figure 2) allows a further improvement of heating rates and thermal homogeneity compared to conventional hot pressing, but even superior to the characteristic advantages of the FAST/SPS technique. This is demonstrated by several examples of the industrial application of FAST/Hybrid, illustrating the development and optimization of innovative materials (Figure 3) for high-end applications in the field of e.g. power engineering, electric mobility or aerospace as well as other future-oriented fields.

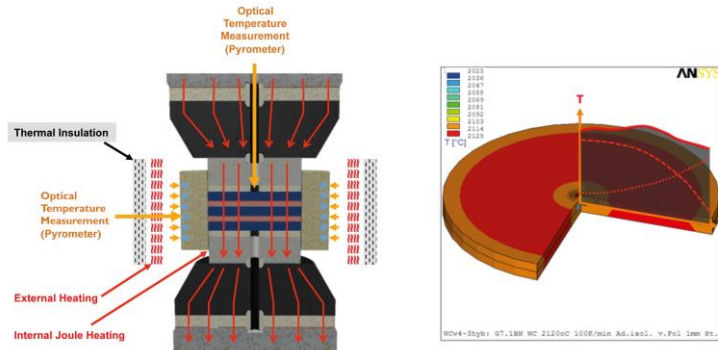


Figure 2 – Principle and thermal gradients of FAST/Hybrid, the combination of classical hot pressing using an external heater, and internal Joule heating of FAST/SPS

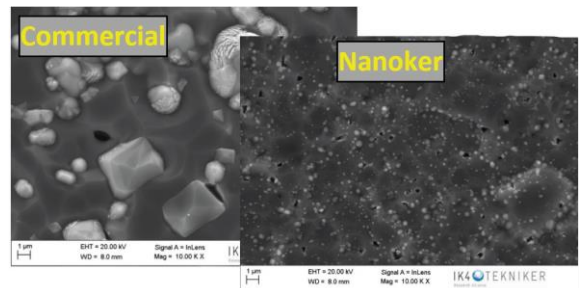


Figure 3 – Microstructure of a high-quality AZO target compared to commercial grade

EXPLOITATION OF INDUSTRIAL APPLICATION OF FLASH TO SINTER CERAMICS

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Key Words: FLASH, Ceramics, Industry application, Sustainability.

FLASH is an electric field-assisted sintering technique recently proposed to densify materials in a more sustainable, energy reductive and cost-effective way than conventional sintering (CS). FLASH sintering promotes the densification of materials by using a combination of temperature and electric field. The use of electric field allows a decrease in the sintering temperature, and as important as well, in the sintering cycle duration. The advantages of FLASH, when compared with other field-assisted techniques, are: low investment, no need for specific atmosphere and dies, and specimen shape versatility.

Consequently, there is an industrial interest on the implementation of sustainable alternative sintering techniques, as FLASH. However practical questions arise as, among many: is this reduction, in addition to the power source consumption, enough to make FLASH energetically favorable from an industrial processing point of view? Most of the authors argue yes, but comparative studies with industrial CS are yet missing. In this work, and as a result of collaborations with two Portuguese ceramic companies, we present a systematic study on the energetic savings of FLASH sintering when applied to two different industrial ceramic products processing: (i) Rapox brown, a manganese-doped alumina used in insulating and wear resistance applications, and (ii) floor tiles used in construction.

An experimental approximation was considered, in which the FLASH temperature for each material was studied. As a mean to calculate possible energy savings, the FLASH thermal cycle was extrapolated and compared with CS cycle. The electric power necessary to FLASH sinter each material was calculated (normalized to 1 kg of material), and a global energy consumption study was performed. Considering the FLASH temperature of 1000 °C for Rapox brown, the reduction in the cycle time is huge, and can be seen in fig. 1.

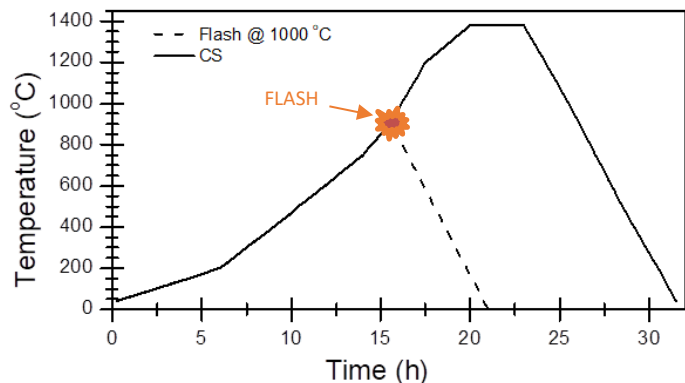


Figure 1 – Temperature vs time comparison of FLASH and conventional sintered Rapox-brown

FIELDS MATTER: A LARGE-SCALE GERMAN PRIORITY PROGRAM (SPP 1959)

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Key Words: electric field, magnetic field, inorganic materials, synthesis, sintering, deformation

The emerging field of temperature dependent electro-chemo-mechanics of solids offers the opportunity to gain novel, energy-efficient, environmentally-friendly, less costly synthesis and processing routes for inorganic materials¹. Evidence for the interactions between external electromagnetic fields, diffusion and deformation mechanisms have been gathered over the years, a global yet detailed understanding of the interactions between electromagnetic fields and solid-state matter transport is far from being reached.

The DFG Priority Program “Manipulation of matter controlled by Electric and magnetic field: Towards novel synthesis and processing routes of inorganic materials” aims to provide a rational, comprehensive knowledge based on experimental evidence and complementary numerical simulations for intentionally using electromagnetic energy to manipulate matter in metals, intermetallics, oxide and non-oxide ceramics as bulk, thin or thick films. As shown in Figure 1, research focuses on the following areas:

- Synthesis and phase formation by solid-state reactions and diffusive phase transformations
- Densification of particle-based materials and microstructure coarsening (pore elimination and grain growth)
- Mechanical deformation (plasticity and creep)

In all these processes, defects such as single or clustered point defects, dislocation networks, interfaces between two reacting solids or crystalline phases (grain boundaries) play a key role. Therefore, a fundamental question to address is: What are the interplays between electric / magnetic fields and matter transport, defect formation, structure and mobility? These properties will determine the response of the whole material and its processing ability.

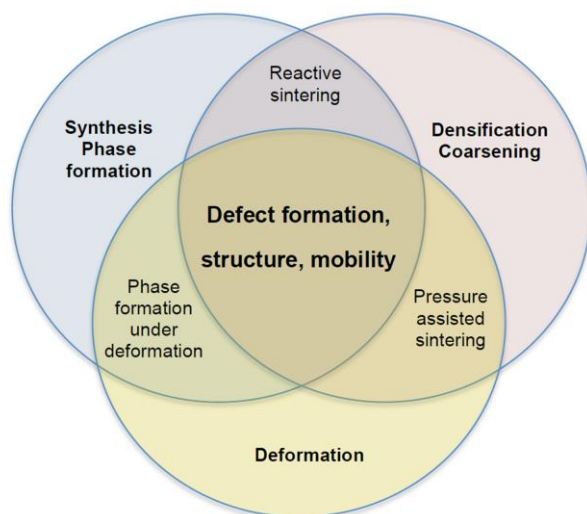


Figure 2 – Scientific focus of SPP 1959
“Fields Matter”

¹O Guillon, C Elsässer, O Gutfleisch, J Janek, S Korte-Kerzel, D Raabe, C. Volkert, *Manipulation of matter by electric and magnetic fields: Toward novel synthesis and processing routes of inorganic materials*, Materials Today (2018)

FIELD ASSISTED PROCESSING OF 3D PRINTED CERAMICS

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Keywords: microwave processing, flash sintering, advanced ceramics, 3D printing, additive manufacturing

Advanced ceramic products for highly demanding applications in electronics, energy, healthcare and defence sectors require densification/sintering, a high temperature process (~1000–2000°C) that in industry can take days. The amount of energy needed, and CO₂ emitted, is therefore very significant. Conventional processing of these functional devices/components are often plagued by interfacial issues, unwanted grain growth and limitations of co-firing dissimilar materials. Thus, rapid and efficient sintering methods such as SPS, Microwave Assisted Sintering (MAS) and Flash Sintering (FS) are continuously being developed. These approaches referred as Field Assisted Sintering Techniques (FAST) use an external field that was demonstrated to have a positive effect on densification. For example, the FS method, for reasons that are far from fully understood, has yielded full densification in very short periods (5 s) at very low furnace temperatures (850°C) for zirconia, and at a surprisingly low temperature of 325°C for Co₂MnO₄ spinel ceramics. The associated time and energy advantage is estimated to be staggering, as well as the ability to tailor the microstructure. In this talk, we will have a closer look at MAS and FS methods— one a well-established and the other a newly emerging densification method. The MAS method can be suitable for the processing of various simple and complex shaped engineering components, the early use of FS method was restricted to dog-bone shaped ceramic specimens – that are both difficult to make and do not have much industrial applicability. However, the recent developments have demonstrated that FS can also be used to sinter different sample shapes. We investigated the feasibility of sintering of 3D printed ultra-low loss 5G microwave dielectrics, YSZ/ZTA biomedical components using MAS and FS methods along with measurements of shrinkage and thermal mapping. This talk will review these developments on FS along with the operative mechanisms in comparison with MAS.

INFLUENCE OF 3YSZ SAMPLE HEIGHT AT THE ONSET OF FLASH SINTERING

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Key Words: Zirconia, Electric Current Assisted Sintering, Field Assisted Sintering.

Flash sintering has drawn attention at the science community because of its capable of densifying materials in few seconds, with low energy consumption, and low furnace temperatures [1]. In spite of these advantages, there are still some difficulties to apply flash sintering at an industry level. These difficulties are mainly regarded about the dimensions of the material which would be sintered. Bigger ceramic bodies have a tendency to form hotspots that decrease its mechanical properties. In this work, we study the influence of the height of 3YSZ cylindrical samples at the onset temperature of flash sintering. It was observed that samples with greater height have a tendency to start densifying earlier than the one with lower height; it means that the onset of flash sintering for bigger samples happens sooner than smaller ones, Figure 1. Illustrate that behavior. This phenomenon can be attributed at a voltage drop at the resistance at the contact between electrode and sample, since the voltage drop is equal for samples with same diameter, but the voltage applied is lower for samples with smaller height (in order to keep the same electric field). The experimental procedure was taken for five different sample height (2, 4, 6, 8 and 10 mm) of a cylindrical shape with 6 mm diameter. All the samples were flash sintered at a tubular furnace setup proposed before [2]. We used 90 V/cm as maximum electric field at AC mode (sinusoidal waveform with 1000 Hz frequency), and 100 mA/mm² as maximum current density for all the different samples height (the voltage and current values were expressed in RMS basis). No conductive paste (such as Pt paste) was used between the electrodes and the samples. The bulk density of the samples was measured by the Archimedes method and a strong correlation between the sample height and the bulk density was observed. Higher samples presented a lower density, it could be explained for two subjects: (1) because of the onset temperature differences that could lead to a minor maximum temperature during the flash event when compared with the lower samples; and (2) higher samples have more open surface area which allows more energy lost at radiation process. The Pt conductive paste is usually expensive and the dependency of conductive paste to minimize this electrode/sample electric resistance is a challenge that must be overcome for flash sintering be applied at industry level.

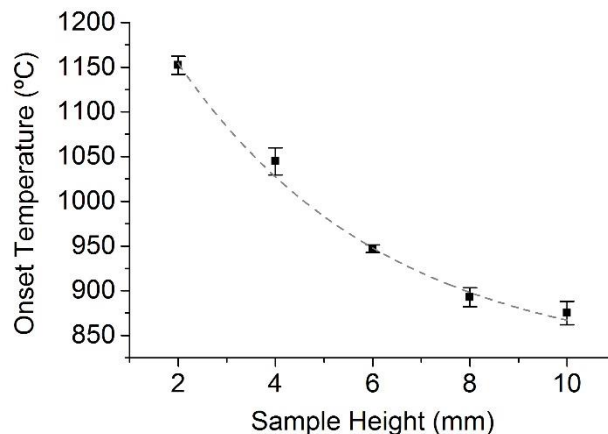


Figure 1 – Onset flash temperature for different sample height, 90 Vcm⁻¹ AC 1000 Hz electric field was applied in each case.

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PHOTOLUMINESCENCE IN SPS-PROCESSED TRANSPARENT Ce:YAG CERAMICS

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Key Words: Transparent ceramics, Ce:YAG, Spark plasma sintering, Photoluminescence.

Ceramic phosphors display great promise for the realization of high-power lighting devices. Cerium-doped yttrium aluminum garnet (Ce:YAG) is commonly used as a phosphor in white light emitting diodes. Therefore, it was chosen as a case study to investigate photoluminescence of transparent ceramic phosphors fabricated by spark plasma sintering (SPS). In the present work, 0.5 at.% Ce:YAG nano-powder was synthesized by a co-precipitation method and subsequently consolidated by SPS into highly transparent ceramic samples. The effect of varying sintering parameters (temperature and pressure) and post-sintering treatments (hot isostatic pressing and air atmosphere thermal treatment) on optical properties was investigated. Correlations between in-line transmittance, photoluminescence (PL) and residual porosity characteristics (pore size and volume fraction) were established. It was also found that PL emission intensity and external quantum efficiency were significantly affected by intentionally created surface roughness.

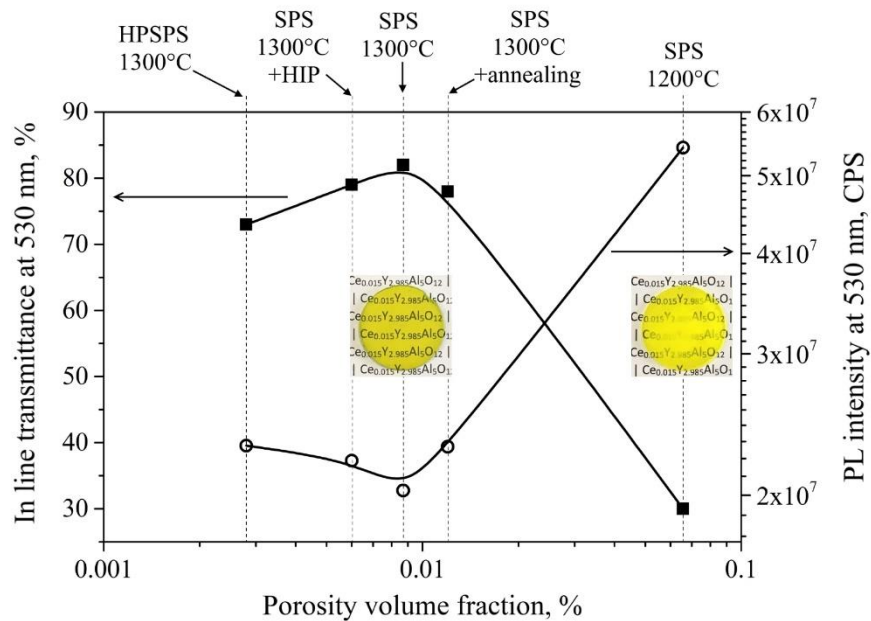


Figure 1- In-line transmittance and PL intensity of 0.5% Ce:YAG as a function of residual porosity volume fraction, inserts of the sintered samples.

IN SITU MEASUREMENTS OF PARTIAL DISCHARGE PATTERNS ON POROUS YSZ PELLETS PRESSED BETWEEN PLANAR PLATINUM ELECTRODES USED FOR FLASH SINTERING

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Key Words: partial discharges, YSZ, electrodes, contact resistance, flash sintering.

Flash sintering of prismatic or cylindrical samples placed between planar electrodes allows processing larger volumes of material, compared to dogbone-shaped samples. The contact surface with platinum electrodes are usually coated with Pt paste in this setup, in order to improve the galvanic contact with the sample, and obtain a better current homogeneity. This coating is however too expensive for industrial applications whenever large surfaces are involved. Nevertheless, a homogeneous contact resistance remains in all cases essential to control the uniformity of the electrical current through the sample, hence the importance of an accurate characterization of the quality of the contact between electrodes and sample in the non-coated case.

We propose to analyse the Partial Discharges (PDs) patterns obtained by Phase Resolved Partial Discharge (PRPD) method as an in situ measurement technique to study this problem. The setup consists in placing a ceramic sample in a furnace in the same configuration as flash sintering experiments but at a temperature lower than the onset temperature of the material. In this work, porous cylindrical samples of stabilised zirconia (average dimensions of 9 mm in diameter and 5 mm in height) are studied between 200°C and 400°C. A 50 Hz AC voltage is therefore applied to the sample and increased gradually up to 1.7 kV. The sample is connected in parallel with a 1 nF coupling capacitor in series with a quadrupole used to separate the high frequency current of the partial discharge signals from the AC current at 50 Hz.

At a given amplitude of the applied voltage, we record for 60 s the patterns of partial discharges, their number and their phase with respect to the applied voltage. We compare the partial discharge signals to the net AC current at the power frequency measured simultaneously with a current probe at the primary winding of the low noise transformer.

At a given temperature, we notice that well-defined patterns of low amplitude partial discharges appear when the applied voltage is increased above 300 V. At much higher voltages, partial discharges of larger amplitudes initiate and grow rapidly as well as the net 50 Hz current which increases non-linearly resulting from the temperature rise of the sample and its associated resistivity drop (similarly to what happens in (pre-)flash sintering conditions). Cross examination of the sample surface by optical microscopy shows physical traces related to the occurrence of partial discharges. For comparison, a YSZ sample coated with Pt paste is also analysed in the same conditions. In that case, at the same temperature and for applied voltages up to 1 kV, partial discharge patterns are almost inexistent.

These results show that the partial discharge measurement is an efficient technique that can be used in situ prior to flash sintering experiments in order to assess the quality of the contact between the electrodes and the sample when no Pt paste is used.

This study is supported by ERDF and the Walloon Region, in the frame of IMAWA-FLASHSINT research project (program 2014-2020).

A NOVEL SYSTEM FOR QUENCHING DURING FLASH SINTERING

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Key Words: Flash sintering, Electric-field-assisted sintering, Quenching, sintering mechanism.

The study of the ruling mass transfer mechanisms in Flash Sintering (FS) has gotten a lot of attention in recent years. Some mechanisms have been suggested, e.g. nucleation due to movement of charged defects, Joule heating runaway and chemical reaction propagation^[1]. In order to further study the phenomena which occur during the different FS stages and shed light on the ruling mass transfer mechanisms, a novel simple system was developed. In this system (Figure 1), FS is done on a ceramic green body in a vertical tubular furnace using vacuum to hold the sample inside the furnace. This configuration enables dropping the sample into a glass of distilled water (or other suitable coolant) at any time, thus quenching the sample and "freezing" the microstructure of the material under FS conditions. The quenched sample can be taken to further investigation of the microstructure, such as SEM analysis. The system design and initial results of FS and quenching of gadolinium-cerium oxide samples will be presented.

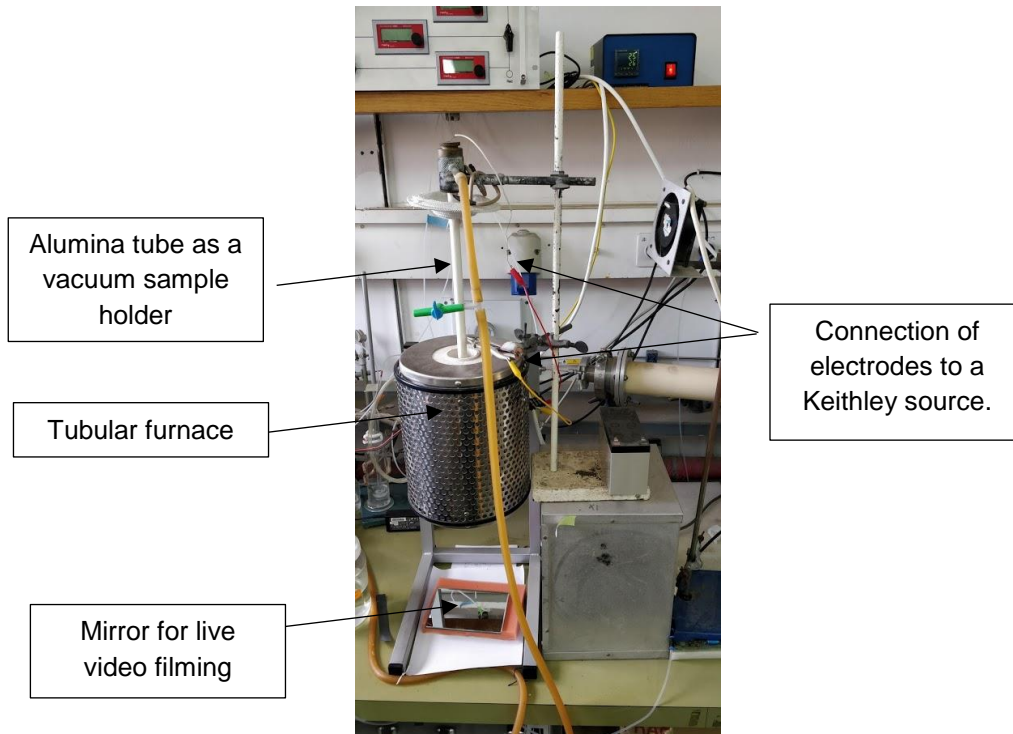


Figure 3 – The flash and quenching system

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IN-SITU ELECTRON MICROSCOPY STUDIES OF ELECTRIC FIELD ASSISTED SINTERING OF OXIDE CERAMICS

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Key Words: Sintering, Flash sintering, ZnO, Electrical induced behavior, Ceramics

A wide range of studies shows a dramatic effect of applied electric fields or currents on the sintering behavior of oxide ceramic powders. However, the mechanisms accounting for the so-called flash sintering remain elusive despite the wide application potential. Using in-situ scanning and transmission electron microscopy, we aim to gain insight into the atomic origins of sintering behavior, as well as of the high conductivity states that occur in conjunction with flash events during field-assisted sintering.

We investigate the sintering dynamics of ZnO green bodies with a density between 50% and 70% and ZnO thin films with and without electric fields and under different oxidizing and reducing gas pressures. Specifically, we use a specially designed SEM heating stage to study the evolution of microstructure and morphology, including grain/void morphology, segregation, and precipitation, both with and without applied fields and with and without gas pressures up to 2 mbar. The in-situ TEM sintering studies, also under controlled electric field and gas pressure, allow us to detect chemical segregation and valence changes (using EDX and EELS) near the sintering boundaries. By gaining access to structural and chemical information down to the atomic scale, we hope to determine how the electric field causes flash sintering.

Several conventional sintering experiments (no field, only heating) have been performed which confirm that sintering can be instigated and observed in the TEM. Furthermore, experiments have been performed with additional applied voltage / current to cause the flash sintering effect. Initial tests indicate that it is difficult to reach sufficiently high current densities to cause flash sintering in free standing green body specimens without causing phase changes due to Joule heating induced temperature increase. Therefore, we have started to grow thin layers of ZnO on Al₂O₃ and YSZ substrates which are both good thermal conductors thereby reducing the Joule heating. Since both substrates are poor electrical conductors, the applied current passes mainly through the ZnO layer. Furthermore, the thin film geometry allows more uniform current densities in the ZnO and facilitates the alignment of grain boundaries for high resolution TEM investigation. Results of studies on the dynamics of grain boundaries during sintering with and without applied current (Figure 1) will be discussed.

Support through the DFG Priority Program (SPP 1959) "Manipulation of matter controlled by electric and magnetic fields: Towards novel synthesis and processing routes of inorganic materials" is gratefully acknowledged.

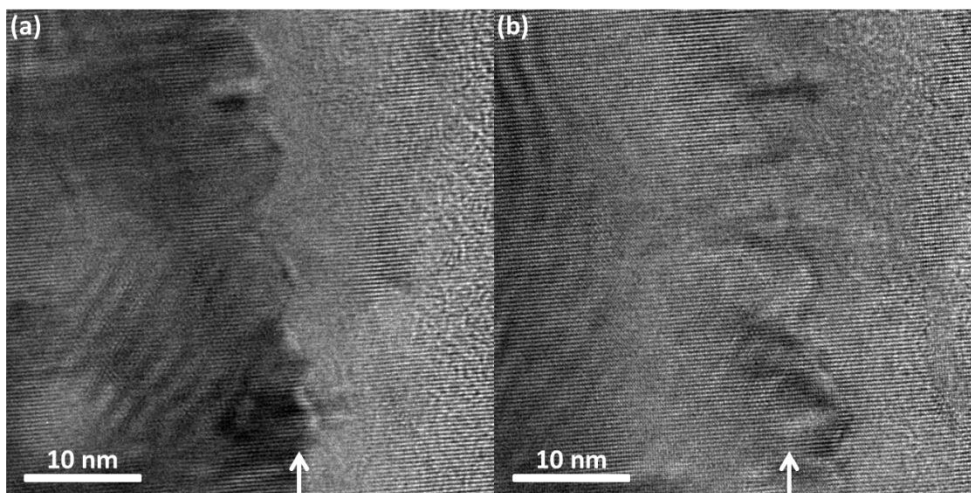


Figure 4 – Grain boundary migration in a ZnO film under an applied electric current and without additional heating. Images from an in-situ video show that the faceted grain boundary moves from its initial position (a) (marked by arrow) a distance of approximately 4 nm in 160 s (b).

TENSILE STRENGTH OF MATERIALS OBTAINED BY ELECTRIC PULSE CONSOLIDATION OF POWDERS

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Key Words: electric pulse consolidation, strength of the material, small specimens, Brazilian test, bending of a thin disk on a ring support.

A wide range of products of modern technology, obtained by electropulse consolidation of powders, has dimensions that do not exceed 10-15 mm in diameter, and their thickness varies from 1 to 10 mm. Standard mechanical testing methods for such sample sizes are often not applicable.

The article investigates the possibility of determining the resistance to fracture of materials obtained from powders by electric pulse consolidation method using the test method for bending thin disks on an annular support and testing short cylinders according to the "Brazilian test". A numerical simulation of the process of bending a thin disk on an annular support and compression of a short cylinder according to the "Brazilian test" scheme has been carried out.

Techniques are verified by testing small-sized thin disks and short cylinders made of gray cast iron and graphite. It is revealed a difference in the nature of the destruction of cast iron samples (plastic failure) and samples of graphite (brittle fracture). The good correspondence of the characters of destruction of graphite samples and samples produced from powders by the electric pulse consolidation is shown. The results of comparative studies of the tensile strength of compacts of nano and ultrafine alumina powders with a spherical shape of particles obtained by the SPS method are presented.

The influence of various additives and manufacturing technology of materials on the resistance of the material to brittle fracture is revealed. The effect of the thickness of the cylinders on the strength of aluminum oxide Al_2O_3 is investigated.

The possibility of determining the tensile strength of materials obtained by consolidating from powders using the electric pulse method by bending small-sized thin disks on an annular support, and short cylinders using the "Brazilian test" scheme is demonstrated.

MICROSTRUCTURE EVOLUTION DURING HIGH-PRESSURE SPARK PLASMA SINTERING (HPSPS) OF TRANSPARENT ALUMINA

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Key Words: Alumina; High-pressure spark plasma sintering; Microstructure; Transparent ceramics.

Applying high-pressure during spark plasma sintering (so-called HPSPS) enables rapid densification of a ceramic powder at relatively lower temperatures, limiting grain growth and allowing fabrication of fine-grained highly transparent ceramics. The present work focuses on the fabrication of fine submicron transparent alumina from untreated commercial powder by HPSPS and the microstructure evolution during consolidation under high applied pressure. The sintering was conducted at relatively low temperatures (1000-1100°C) under high pressure varied from 250 to 800 MPa. We review unique sintering phenomena such as stress-enhanced grain growth and de-sintering, which are related to creep taking place during the final stage of pressure-assisted densification. In addition, optical and mechanical properties of obtained samples are discussed.

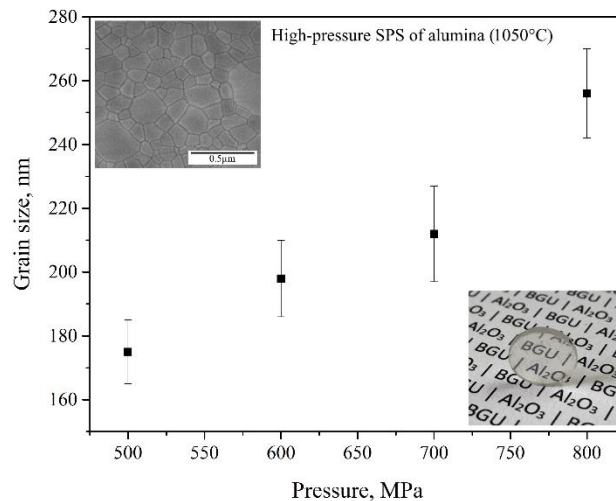


Figure 5 – Grain growth with pressure following HPSPS at 1050°C with no dwell time.

IMPACT OF AN EXTERNAL ELECTRIC FIELD ON GRAIN GROWTH IN OXIDES: COMPARISON OF FLASH SINTERED SAMPLES TO FIELD ASSISTED GRAIN GROWTH

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Key Words: Grain growth, flash sintering, Oxides, external electric field, defects.

In the last years ample effort was done to investigate the effect of electric fields on matter. We investigated the effect of an external electric field on the oxide ceramic model system strontium titanate. More precisely, we observed that a non-contacting external electric field has an impact on the defect distribution and the grain growth. Oxygen vacancies are migrating towards the negative electrode yielding a higher oxygen vacancy concentration compared to the positive electrode. As a result, faster grain growth was observed on the negative electrode. Recent thermodynamic defect calculations revealed the mechanism for this relationship [1]: A high oxygen vacancy concentration results in less space charge and, as such, in less segregation of cationic defects. As less segregation requires less diffusion for grain boundary migration, faster grain growth occurs.

We extended these findings to flash sintering of doped strontium titanate. TEM imaging and EDS analysis were used to investigate the microstructure and to map the dopant segregation at the grain boundaries. Observing different dopant species (acceptors and donors) gives insight on flash sintering for different defect concentration and types with different segregation properties. In addition, field assisted microstructure evolution experiments with titania (no current, insulating electrodes) allow to apply the gained knowledge to different material systems with different defect chemistry.

[1] Work of Jana P Parras and Roger A. de Souza

PATTERN FORMATION DURING CURRENT SINTERING (SIMULATION)

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Key Words: Current sintering, pattern formation, reaction-diffusion model, Onsager network

Current sintering is becoming increasingly important for creating nanostructured materials from powders. Joule heating inside the sample enables the rapid densification necessary to preserve the nanostructure. However, the presence of electric fields and currents activates additional effects, such as inhomogeneous grain-growth and coarsening, thermoelectric effects, as well as dopant migration. In return, these effects influence the transport properties of the material during sintering which constitute feedback mechanisms that, ultimately, can lead to the formation of patterns.

The Winterer group observed such pattern formation in Al-doped ZnO. The material is prepared with an Al-concentration above the solubility limit which leads to precipitation when the material is sintered. Only during current sintering distinct layers of different porosity and precipitate concentration form. Because of the complex intertwining of transport processes, we study two complementary aspects. On one hand, we investigate the influence of precipitates on Joule heating. Our results show that reduced conductivity due to precipitates yields patterns perpendicular to the electric current (cf. Figure 1a). On the other hand, we study migration of anions and cations in a reaction-diffusion model. Here, we propose as feedback mechanism a precipitate-dependent ion mobility. This, too, leads to patterns (cf. Figure 1b) which are qualitatively different from the Joule heating case. Both models have in common that random fluctuations trigger the emergence of the patterns.

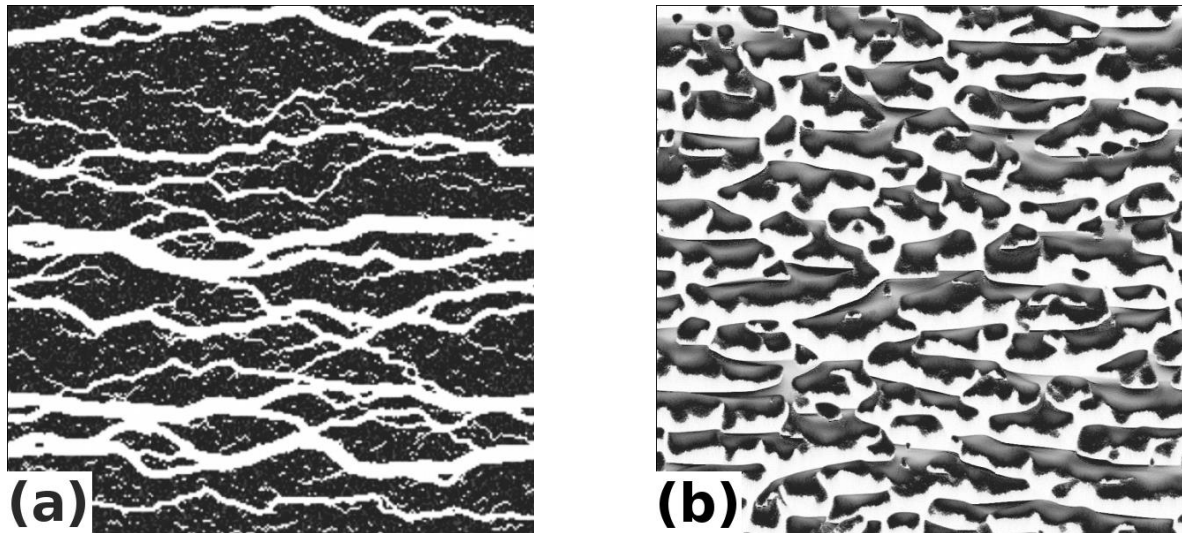


Figure 1 – Precipitation patterns obtained from (a) Joule heating (local conductivity is color-coded) and (b) precipitate-dependent ion mobility (precipitate concentration is color-coded).

MICROSTRUCTURAL EVOLUTION OF 3YSZ FLASH SINTERED WITH CURRENT RAMP CONTROL

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Key Words: Flash Sintering, Ytria Stabilized Zirconia, Field Assisted Sintering.

Ceramics sintered by Flash Sintering (FS), sometimes have heterogeneity in their microstructure. Among the possible causes of this problem, the formation of hotspots is probably the principal issue observed in flash sintering. The hotspots are formed due to the heterogeneity of the resistance of current passing in the green sample. In this way, in the flash phenomenon, the current finds preferential paths, carrying on non-uniform thermal runaway. In this work, we observed that one way of reversing this problem was gradually increasing the electric current density at FS. For this, 3YSZ was shaped in cylindrical form (5 mm height and 6 mm diameter) and flash sintered, at a tubular setup proposed before¹, under the application of an AC electric field of 120 V/cm (RMS basis) applied from the beginning of the furnace heating. Three different electric current density ramps were studied: 0.012, 0.024 and 0.048 A.s⁻¹, named as Z1, Z2 and Z3 respectively, until they reached the maximum value of 100 mA.mm⁻² (the moment the power supply was turned off). For comparison purposes, conventional FS (Z0) was performed using the same electrical parameters. The electric source, in this case, remained on after reaching 100 mA.mm⁻² for 142 s (time calculated to reach the same total energy supplied by the electrical source of samples Z1). After sintering, the apparent densities of the samples were measured according to the Archimedes principle. For analysis of the microstructure, the samples were cut radially and three regions of each sample were observed in SEM: center, right and left surfaces. The grain size distribution was made for each region using ImageJ software. The apparent density of samples sintered by FS was 94 % (Z0) and samples with electrical current ramp were 93 %, 92 % and 87 % for Z1, Z2 and Z3, respectively. The apparent density is proportional to the total energy supplied to the sample (energy provided by the power supply and thermal energy provided by the furnace). Thus, the density of Z0 was expected to be close to the density of Z1. As samples Z2 and Z3 had more abrupt ramps, the energy supplied was lower, which resulted in lower apparent density. The grain size distribution indicates that the mean grain size between the three different regions did not present statistically significant differences (ANOVA test) for the same sample. Figure 1 shows the microstructure of sample Z1 in three different regions. It was suggested that the gradual increase of the electric current during the FS prevented the formation of hotspots and consequently resulted in homogeneous microstructures. The Z0 sample presented a statistically significant difference in grain sizes between the surfaces and the center. In FS the heating is very fast, which causes heterogeneity in the microstructure due to thermal retention in the center of the sample, while in the surfaces the temperature is lower, due to energy losses by radiation. When the electric current increases gradually the heating ramp will be smoother, so there is no significant heat retention in the center of the sample². Thus, the gradual rise of the current during flash sintering provides a better homogeneity of grain size along the center of the same sample. However, for the experiments studied here, the densification of sintered samples with the current ramp was lower than that of the sintered sample in FS. Therefore, for better densification results, an even longer ramp could be the solution.

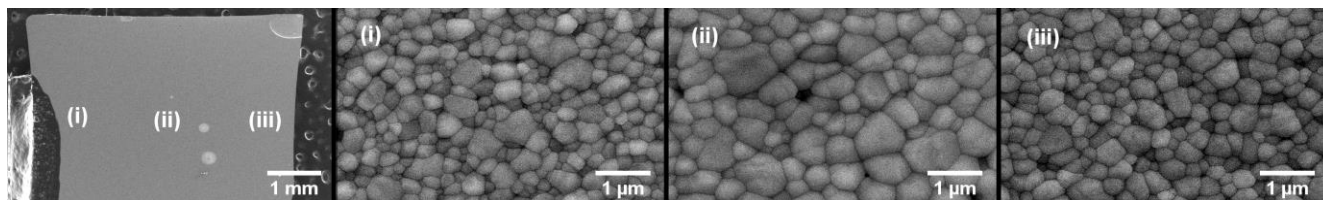


Figure 6 – Overview of the Z1 sample and micrographs of (i) left surface, (ii) center, and (iii) right surface.

¹CAMPOS, J. V., LAVAGNINI, I. R., SOUSA, R. V., FERREIRA, J. A., PALLONE, E. M. J. A. Development of an instrumented and automated flash sintering setup for enhanced process monitoring and parameter control. *Journal of the European Ceramic Society*, Oxford, v. 39, n. 2-3, p. 531-538, 2019.

²M K KUMAR, P. YADAV, D., LEBRUN, J., RAJ, R. Flash sintering with current rate: A different approach. *Journal of the American Ceramic Society*, v. 102, n. 2, p. 823-835, 2019.

INFLUENCE OF THE CONFORMATION METHOD ON FLASH SINTERING OF ZNO CERAMICS

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Key Words: ZnO, Flash Sintering, Field Assisted Sintering.

Flash Sintering has been shown to be a promising alternative in obtaining high-density ceramics with grain growth control. However, some conditions are still under development. Regarding the dimension of the ceramic, there is a limitation related to obtaining a homogeneous material in large specimens, in order to avoid the formation of preferential current flow paths. Depending on the temperature required for the flash event to occur, some electrode materials have a high cost, or controlled atmosphere operations are required. Typically, the electrodes used in flash sintering consist of platinum, however, in some cases, other materials may be a cheaper suitable alternative, such as stainless steel^[1] or nickel-chromium alloys. Also, the use of different compositions in the electrode influences the conductivity of the material, which affects the onset of thermal runaway^[2]. In this regard, different conformation techniques were studied in order to improve the homogeneity of the sintered ZnO ceramic body. Additionally, the feasibility of the use of Inconel (nickel-chromium) electrodes in the replacement of platinum electrodes was evaluated. Then, ZnO specimens were conformed (cylindrical shape - 6 mm diameter and 5 mm thickness) by uniaxial pressing under 140MPa and 300 MPa, isostatic pressing under 200 MPa, and slip casting. All experiments were conducted isothermally at 800 °C in an adapted tube furnace^[3], with an applied field of 60 V/cm and 200mA/mm² as maximum current density, using either platinum or Inconel electrode. Figure 1 shows the varying in time incubation with respect to the conformation method used. The distinct incubation times are justified by the difference in the pore distribution, which affects the electrical resistivity of the samples. Also, according to Figure 1, it can be seen that in addition to presenting a higher electrical resistivity, which increased the incubation time, the samples conformed by slip casting are also characterized by the high formation of hotspots, which can be observed by the appearance of several spikes during the occurrence of the flash event. Figure 2 shows, for the samples conformed by uniaxial pressing (140 MPa), the difference between incubation times in relation to the material used for the electrode. It can be seen that, in this case, the use of Inconel increased the incubation time. Microstructural and further analyzes are being conducted.

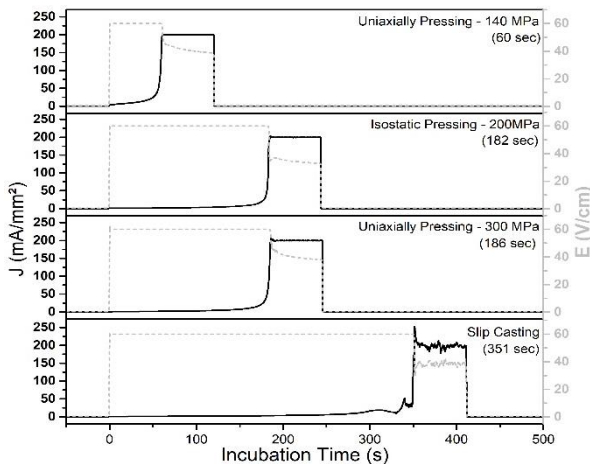


Figure 1 - Variation in incubation time (s) according to the conformation method used, for Pt electrodes.

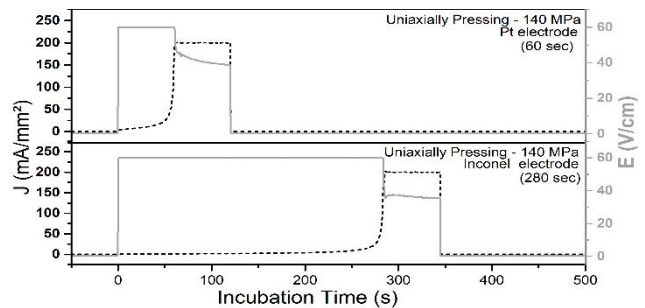


Figure 2 - Variation in incubation time (s) for Pt and Inconel electrode, using 140 MPa uniaxial

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[3] CAMPOS, J. V. et al. Development of an instrumented and automated flash sintering setup for enhanced process monitoring and parameter control. *Journal of the European Ceramic Society, Oxford*, v. 39, n. 2-3, p. 531-538, 2019.

DC ELECTRIC FIELD ASSISTED 3YSZ CERAMIC SUPERPLASTIC DEFORMATION

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Key Words: DC electric field; 3YSZ; grain boundary sliding; superplasticity; accommodation mechanism.

3YSZ ceramic superplasticity has been investigated for many years, and the primary mechanism for superplastic deformation is grain boundary sliding. However, the accommodation mechanism for grain boundary sliding is controversial. In this paper, the 3YSZ ceramic was deformed in tension at different constant stress conditions. Superplastic deformation of 3YSZ ceramic was observed with the assistance of DC electric field. It indicated that at the high tensile stress condition, dislocation motion may play a role in the accommodation mechanism for grain boundary sliding.

FIELD-INDUCED MASS TRANSPORT PHENOMENA IN FLASH SINTERED HIGH TEMPERATURE CERAMICS EXPLORED BY IN SITU SEM AND TEM

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Key Words: flash sintering, in-situ SEM, in-situ TEM, mechanical behavior, defects

Flash sintering has attracted significant attention lately as its remarkable rapid densification process at low sintering temperature leads to the retention of fine grains and enhanced dielectric properties. However, the underlying mechanism of flash sintering and mechanical behaviors of flash-sintered ceramics remain poorly understood. Here, we report the microstructure of flash-sintered yttria-stabilized zirconia (YSZ) and TiO₂ by transmission electron microscope (TEM) and their high temperature in-situ micropillar compression studies inside a scanning electron microscope (SEM). Our studies on flash-sintered YSZ show that YSZ exhibits high inelastic strain (~ 8%) primarily due to phase transformation toughening below 400°C. At higher temperatures, crack nucleation and propagation are significantly retarded and prominent plasticity arises mainly from dislocation activities. The holding time and current density limit after the onset of flash for flash-sintered TiO₂ significantly affect the microstructure and mechanical behavior. High dislocation density and stacking faults have been observed in the flash-sintered TiO₂ under TEM. The presence of high-density defects generated during flash sintering plays a major role in the overall microstructure and mechanical behavior of ceramics.

FLASH SINTERING OF CERAMIC FILMS: THE INFLUENCE OF SURFACE TO VOLUME RATIO

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Key Words: Flash, thickness, surface, volume.

To establish the effect of sample thickness on the flash onset, we measure the sintering behavior of thin and thick films of 3 mol% yttria, the following is noted. (i) The onset of flash moves to a higher value for the thinner specimens. (ii) The power transition falls within a narrow band when the power is normalized with respect to surface rather than volume. (iii) Sintering of thinner specimens require a higher current density limit. These results point to a deeper significance of black body radiation than its use in estimating the specimen temperature in a steady state of flash.

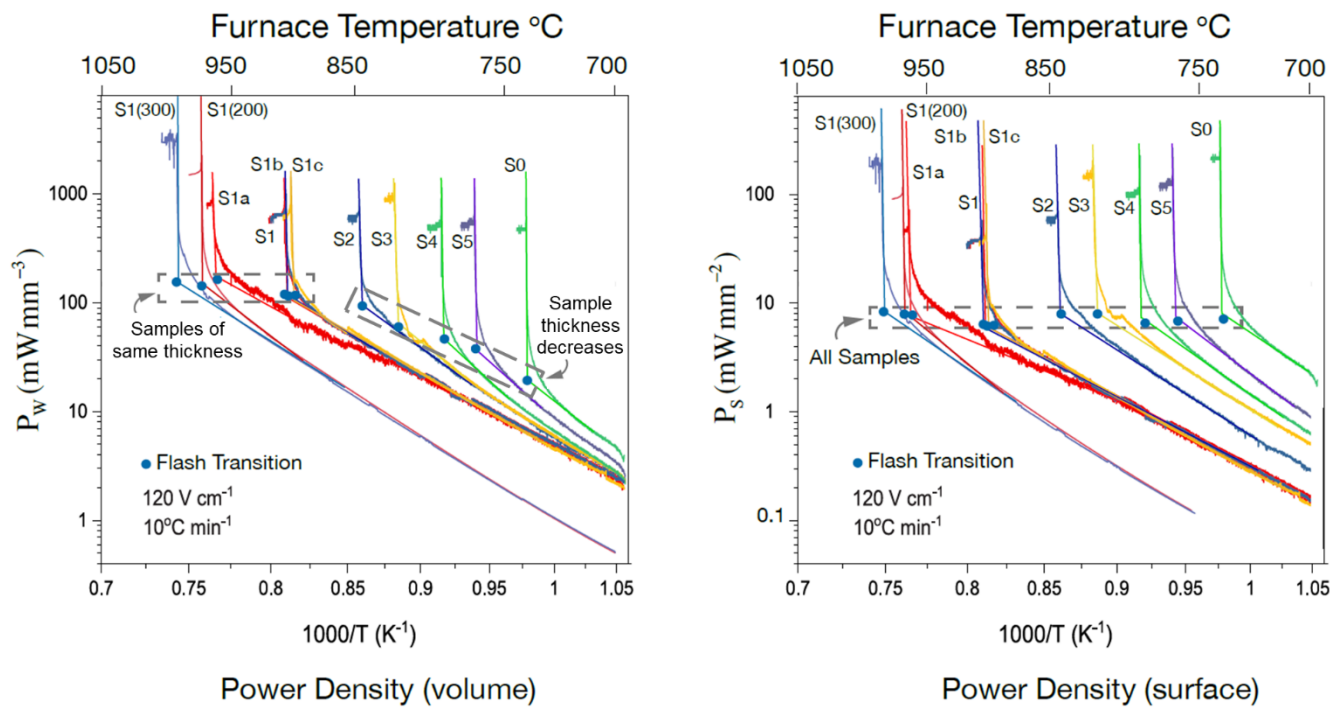


Figure 7 – Arrhenius plots of power for specimens with variable thickness, normalized respect to volume (left) and surface (right).

TRANSITION TO PARTIAL ELECTRONIC CONDUCTIVITY AT THE ONSET OF FLASH MEASURED BY IN-SITU IMPEDANCE SPECTROSCOPY

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Key Words: flash sintering, electronic conduction, In-situ impedance spectroscopy

We show that the early onset of Flash in single crystals of cubic zirconia is accompanied by a transition to electronic conductivity. The transition is measured just before, during and just after the cusp of the onset. The finding has the following significant implications:

- (i) The metal-ceramic electrodes, which are blocking to electrical transport of ions, which necessitates redox reactions of the type $O + 2e \rightarrow O^{2-}$, yield to becoming non-blocking since the electrons can be transported via the external circuit to maintain high overall conductivity.
- (ii) The blackening and abnormal grain growth seen near the electrodes most likely occur before the onset of the flash.
- (iii) The ideal transport number to avoid (ii) should be approximately 0.5. We are seeking to measure it in our laboratory.
- (iv) The onset of electronic conductivity implies the generation of electrons and holes, which can recombine to produce electroluminescence.

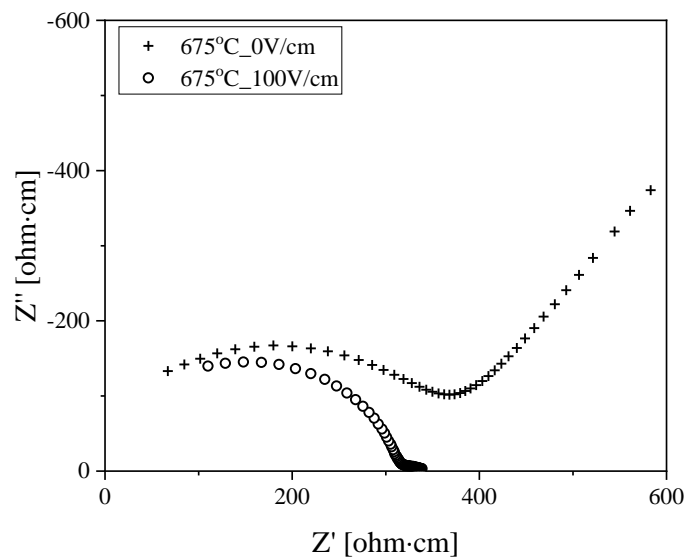


Figure 8 – Nyquist plot of 8YSZ single crystal at near the flash temperature when applied electric field.

IN-SITU MEASUREMENTS OF THE ELASTIC MODULUS OF ZIRCONIA POLYCRYSTALS HELD IN A STATE OF FLASH INDUCED BY AN ELECTRIC FIELD

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Key Words: Flash; Elastic Modulus; 3Y-TZP; damping.

Partially Stabilized zirconia is known to show the phenomenon of flash under applied electric fields. A proposed mechanism for flash is the introduction of nonlinear lattice vibrations at the onset of flash which may further soften the shear modulus of the lattice. This softening would in turn lower the energy barrier for the formation of defects. Here, in-situ measurements of elastic modulus of 3Y-TZP during flash for different current densities is estimated by comparing the modal response from the experiments against the simulations. Results in Figure 1 compare the modulus with and without (baseline) flash showing a lower modulus during flash for that corresponding sample temperature. Hence, supporting a scientific explanation behind flash.

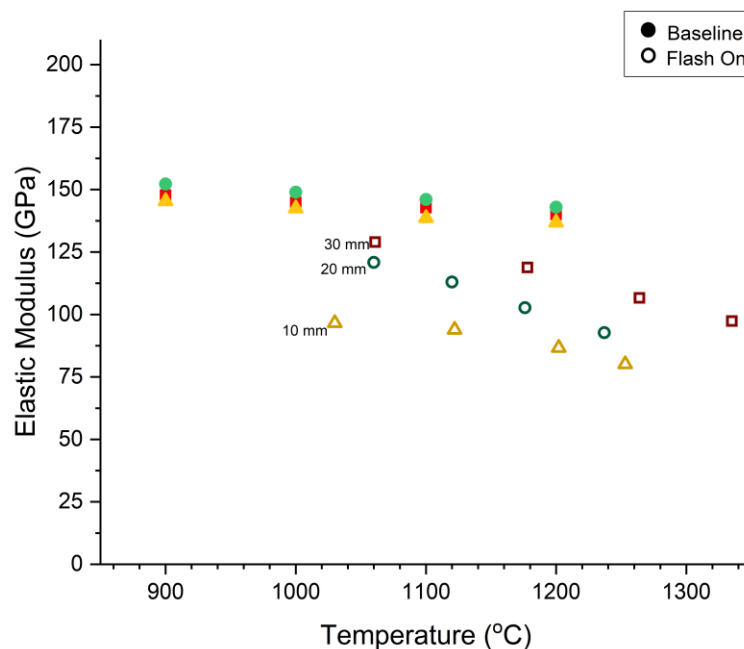


Figure 9 – Elastic Modulus of 3Y-TZP. Here, the solid points represent the baseline values in no flash state and hollow points are the values during flash for different flash lengths.

CURRENT RATE FLASH OF CARBON FIBERS

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UNUSUAL ATOM DISPLACEMENTS IN TiO_2 DURING FLASH SINTERING

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Key Words: Flash sintering, Pair Distribution Function, Atomic displacements, Synchrotron, In-situ

We report on in-situ measurements of displacements for Ti and O atoms within a TiO_2 unit cell during flash sintering, where one species (O) moves much more than the other. The heart of these investigations is focused on the hypothesis that colossal concentrations of defects, in the form of Frenkel pairs are generated which lead to abnormal kinetics of diffusion and phase transformations. These experiments will continue to provide new information which can be built into atomistic simulations to provide a scientific basis for the flash phenomena.

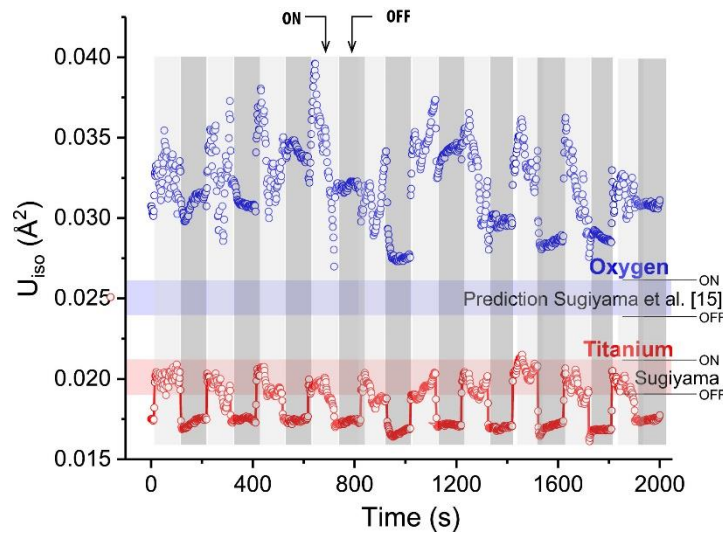


Figure 1. Measurements of U_{iso} for O (blue) and Ti (red) atoms. The bands show the estimates from baseline data with single crystals at specimen temperatures for the ON and OFF periods of the flash cycles.

POWDERS OF FOUR ELEMENTAL OXIDES TRANSFORMED AND SINTERED BY REACTIVE FLASH

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Key Words: Reactive, flash, synthesis, sintering, oxides.

The cubic phase of a complex oxide of Li, alumina doped lithium lanthanum zirconate, is fraught with issues related to the loss of lithium and unwelcome phase transformations [1]–[3]. Here we show that the cubic phase of this complex oxide can be made by reactive flash sintering with elemental oxides of Li, La, Zr and Al. Transformation and sintering occur at a furnace temperature lower than 700°C in ambient atmosphere. The possibility of discovering new, high-conductivity solid-state electrolytes of lithium through chemistry is greatly enhanced by the viability of the reactive flash sintering process.

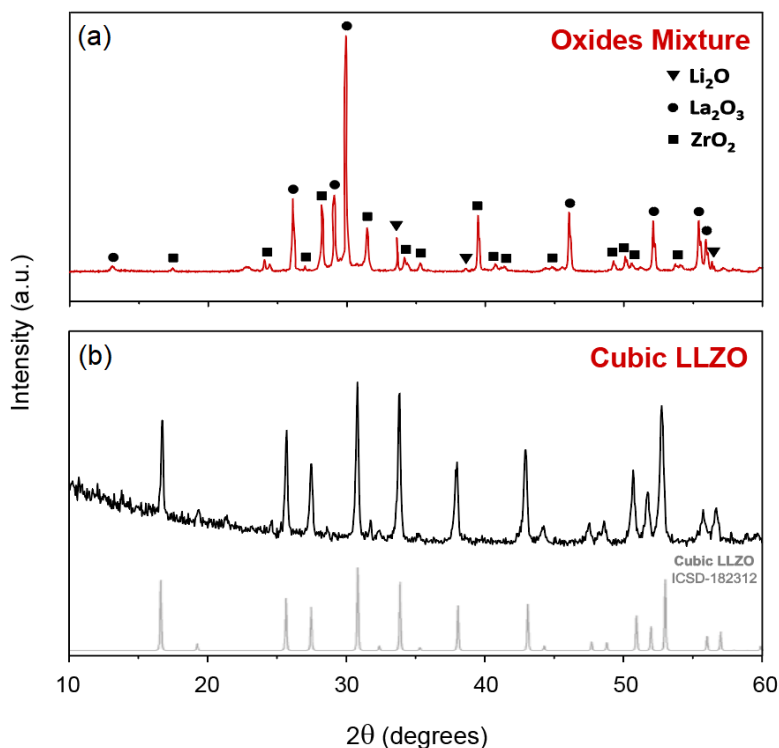


Figure 10 – X ray diffraction pattern for (a) the initial oxide powders and (b) their transformation to cubic LLZO after reactive flash.

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LATTICE SOFTENING

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Key Words: phonons, Frenkel Defects, Flash Sintering, Elastic Modulus, In-situ.

In-situ measurements of the elastic modulus with 8YSZ in the state of flash reveal a large softening in the presence of the electric field. This discovery may explain the lowering of the energy barrier for the formation of Frenkel defects. The results also support the consequences from recent molecular dynamics calculations (Jongmann and Wolf) that reveal that Frenkels can form by the injection of phonons at a high rate, while holding the specimen above the Debye Temperature. These points will be raised for discussion.

A SHORT REVIEW OF FS MECHANISMS

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Key Words: Defect nucleation; Electrochemical triggered; Joule heating runaway

The current views on flash sintering (FS) will be reviewed with focus on mechanism research [1]. The mechanism of FS has been under debate since its discovery and three main mechanisms were suggested: defect nucleation, Joule heating runaway and electrochemical reactions. We believe that thermal runaway is essential for FS initiation; by calculating the thermal runaway of the green body, the onset temperature can be predicted. Since sintering under AC stimulus was achieved, electrochemical reactions can account for FS only if breaking the symmetry is dealt with. Symmetry breaking is indeed possible due to imperfect contacts and uneven kinetics of, e.g., redox reactions at different facets in oxides. The effect of electrode material on threshold conditions and of electrochemical reactions on the final microstructure were also discovered. The work done in our group showing that the FS onset conditions of highly Fe doped strontium titanate change under different pO₂, unties the link between flash onset and ionic defect concentrations [2]. However, the influence of charged defects and of electrode materials on the flash process and threshold conditions cannot be ignored. Clear evidence for the nucleation of a new phase using in-situ XRD analysis was also shown. Further research to unveil all the effects affecting all stages of FS and their relations is needed. The evidence for a formation of a new phase during the flash found by Raj group might be connected to formation of a softened / liquid phase. Moreover, since phase transition limits temperature rise, it might account for the low temperatures measured using in-situ XRD. Lastly, since both liquid formation and electrochemical reactions are a trigger for thermal runaway, we believe that all the suggested mechanisms are linked. Further research for discovering proof of liquid formation and electrode effects will shed light on the triggers of thermal runaway thus solving the problem of FS mechanism. Stages II and III of the flash are yet to be understood, with currently clear hints toward the importance of point defects and of huge temperature gradients.

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B-SIALON-BASED CERAMIC COMPOSITES BY COMBUSTION SYNTHESIS AND SPARK PLASMA SINTERING

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Key Words: combustion synthesis, spark plasma sintering, ceramic composites, sialons, boron nitride, titanium nitride, silicon carbide.

Spark-plasma sintering (SPS) of composite ceramics in the β - Si_5AlON_7 - system (SiC, TiN, BN) was studied. An effective mechanism of intensification of sintering processes was revealed due to the introduction of h-BN, having a scaly structure and playing the role of a solid lubricant, which improves the compressibility of the sintered powder mixtures under load. For composites containing 0–30 wt. % h-BN, 0–40 wt. % β -SiC, and 0–40 wt. % TiN, with different character distribution of components by volume (homogeneous, layered and gradient), the optimal parameters of the PCA were established, ensuring the achievement of a high level of relative density (not less than 95%) and functional characteristics.

According to XRD results, the raw powders of β - Si_5AlON_7 , h-BN, and TiN did not contain impurity phases while β -SiC had trace amounts of Si_3N_4 . According to SEM results, all as-synthesized powders appeared largely as agglomerates. Their specific surface was about $1.3 \text{ m}^2/\text{g}$ for β - Si_5AlON_7 powders, and from 9.8 to $22.8 \text{ m}^2/\text{g}$ for h-BN, β -SiC, and TiN fine powders. After ball milling, the specific surface increased by a factor of 4–6.

The addition of h-BN improves the compactibility of sintered powder mixtures. Under a compressive stress of 50 MPa at 600°C , the initial value of ρ_{rel} exceeds 80% for the compact containing 30 wt. % BN and 60% for that of pure β - Si_5AlON_7 (Figure 1). In parallel, an increase in h-BN content suppresses the consolidation processes due to formation of liquid eutectics. At 30 wt. % BN, the temperature dependence of ρ_{rel} becomes much more aligned.

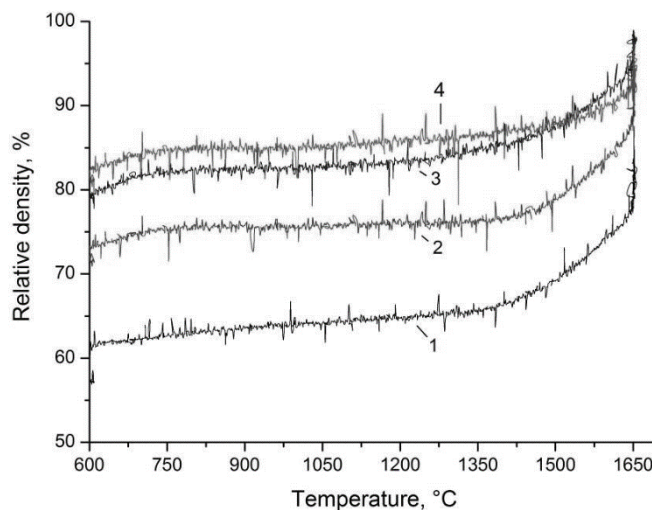


Figure 1. Relative density ρ_{rel} as a function of temperature T for: β - Si_5AlON_7 (1), β - Si_5AlON_7 -BN (10 wt. %) (2), β - Si_5AlON_7 -BN (20 wt. %) (3), and β - Si_5AlON_7 -BN (30 wt. %) (4); $T_{\text{max}} = 1650^\circ\text{C}$.

The main structural characteristics (relative density, phase composition, microstructure) and physics-mechanical properties and functional characteristics of the obtained samples of composite ceramics (flexural strength, electrical conductivity, linear thermal expansion coefficient, thermal shock and wear resistance, etc.) and dependencies between them were researched.

The achieved level of physics-mechanical properties and functional characteristics confirms that productive and energy efficient methods of SHS and SPS can be successfully applied in the development of promising ceramic materials and products based on sialons for use in extreme conditions.

EVIDENCE FOR MICROSTRUCTURE-DEPENDENT HYSTERESIS IN SCO MOLECULAR CERAMICS PREPARED BY COOL-SPS

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Key Words: Cool-SPS, low temperature sintering, spin crossover materials, functional molecular ceramics.

In recent decades, the development of new molecular materials with spin transition has aroused a growing interest from scientists in the field of information and communication technologies [1]. These compounds have the capacity to change their electronic state under the effect of an external disturbance such as temperature, pressure, or light irradiation, with important consequences on their structural, magnetic, or optical properties making them attractive for potential applications in the field of sensors, memories, molecular motors, or smart pigments. If the relations between the properties of these compounds and the crystalline structures are well established [2], the effects related to their microstructure were recently highlighted [3] and are still being discussed. Recently, the efficiency of Cool-SPS for the sintering of fragile materials at low temperature was established [4] Cool-SPS allowed the first molecular ceramics to be obtained at ICMCB [5]. Current work aims to develop new molecular ceramics from functional materials such as spin-transition complexes, to extend their diversity and to establish relationships between the microstructures obtained, their physical properties, and their switching behaviors. The compound $[\text{Fe}(\text{Htrz})_2(\text{trz})](\text{BF}_4)$ was chosen as starting material because the switching of the Fe^{2+} ion between a diamagnetic low spin state (LS, $S=0$), and a paramagnetic high spin state (HS, $S=2$) is carried out with a large thermal hysteresis ($\sim 40\text{K}$) above the room temperature [6]. Moreover, the importance of the microstructure on this compound is known [7], and recent work has shown a clear evolution on this scale following several cycles or heat treatments [8]. In this work, first molecular ceramics from SCO materials have been developed by Cool-SPS, the optimal sintering conditions will be discussed, the influence of the sintering parameters (temperature, pressure, etc.) on the structural and morphological properties will be studied, and the correlation between microstructure and hysteresis loop after sintering will be highlighted (Figure 1). The future work, within the framework of this thesis, aims to pay attention to a further characterization of the ceramics elaborated in order to investigate the influence of the sintering conditions on the physical properties of the powders and to study in detail the relationship between the microstructural properties and the physical properties. SPS treatment conditions will be optimized to obtain denser ceramics while controlling their microstructure.

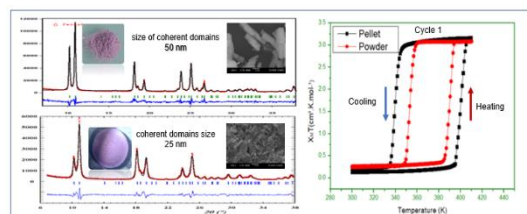


Figure 11 – Profile matching of the PXRD pattern, TEM images and VSM first cycle of the $[\text{Fe}(\text{Htrz})_2(\text{trz})](\text{BF}_4)$ respectively before and after sintering.

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W-Cr SOLID SOLUTION: COMPARISON OF ALLOYING IN SPS AND BY BALL MILLING

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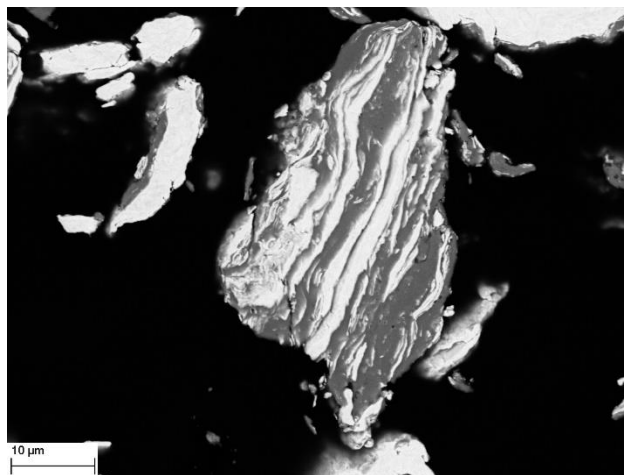
Key Words: tungsten-chromium alloys, Spark Plasma Sintering, mechanical alloying

Tungsten alloys currently represent prospective candidates to replace tungsten in the first wall applications in future fusion facilities. They are anticipated to suppress unfavorable mechanical properties of commercially pure tungsten and/or to gain advantages such as ability of self-passivation under accidental conditions. The self-passivating alloys are designed to minimize possible consequences related mainly to a LOCA (Loss of Coolant Accident) event with simultaneous air ingress into the reactor vessel.

Self-passivating tungsten alloys contain additions such as Cr with the ability to form compact and thermally stable oxide scale. As soon as the oxide scale is formed, activated tungsten oxides cannot be released to the surroundings. W-Cr with 10 wt.% of Cr have shown the best passivation properties.

W and Cr are elements with a very different melting point. At the onset of tungsten melting (3422 °C) the temperature is far beyond the boiling point of chromium (2 672 °C). Thus, standard casting methods are very inconvenient for this system. Therefore, tungsten-chromium alloys are usually prepared by mechanical alloying route. However, this method possesses several drawbacks such as impurity introduction stemming out of the principles of mechanical alloying and long processing times (usually tens of hours to days).

In this study, W-Cr alloys were prepared by direct alloying in SPS. The W-Cr system possesses a miscibility gap; thus, to enhance alloying, sintering was performed above the gap. In some cases, further alloying enhancement was reached by short ball milling of W-Cr powder and subsequent sintering. The results are summarized and compared to mechanically alloyed samples. Feasibility of W-Cr alloys production by SPS alloying is discussed.



Powder particle after short ball milling process. The powder consists of pure tungsten and pure chromium layers.

FLASH JOINING OF GRAPHITE WITH POLYMER DERIVED CERAMIC INTERLAYER

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Key Words: Flash joining; polymer derived ceramics; graphite; joining; SiOC

High-temperature ceramics for structural applications are often characterized by poor machinability and very high melting temperature. Therefore, it is difficult to manufacture them into net-shape large components with complex geometry, with manufacturing technologies such as casting, plastic forming and machining not being viable. Therefore the development of novel joining technologies for high temperature ceramics is of considerable interest. However, ceramic joining is a challenging task because of their high melting temperature; their resistance to plastic deformation; and their brittle behavior (which can cause failure when thermal stresses are developed).

The recent discovery of flash processes and, namely, of flash SPS opens new possible processing routes for the production of ceramic joints. In this work, we investigate the feasibility of a flash joining process for graphite, one of the most refractory materials known. It is shown that effective graphite-graphite joints can be produced by flash SPS in very a few seconds (6 to 10 s) using a pre-ceramic resin as an interlayer.

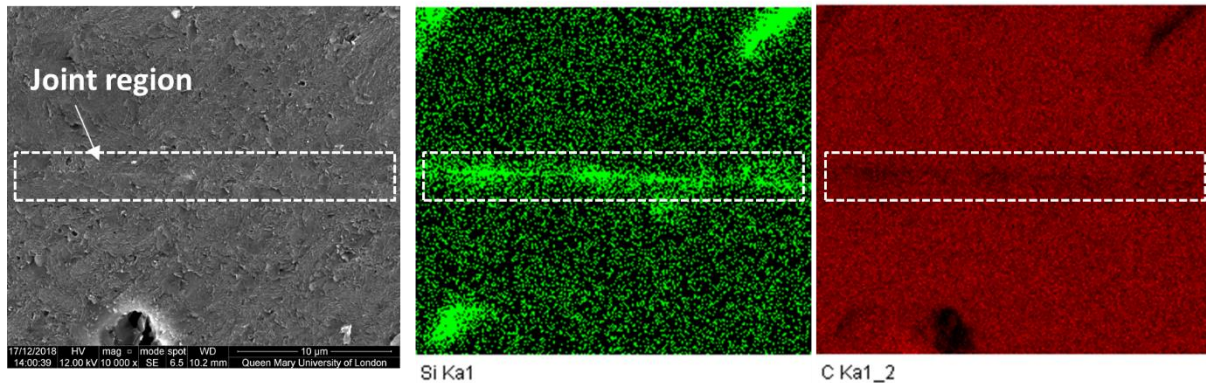


Figure 12 – Flash joined graphite samples.

THE EFFECT OF HIGH CURRENT DENSITIES ON IRON-CARBON ALLOY THIN FILMS

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Key Words: Abnormal grain growth, electromigration, high current densities, electron microscopy, iron thin films

The recently discovered flash sintering method for preparing high quality oxide materials can be applied to the preparation of high performance nanocrystalline metals as well. Just as for the oxide materials, it is possible to use electric fields and currents to enhance densification of metal powders while limiting grain growth, however, the exact mechanism is still under discussion. The goal of our study is to understand how electric currents effect impurity redistribution and grain growth in fine grained metals.

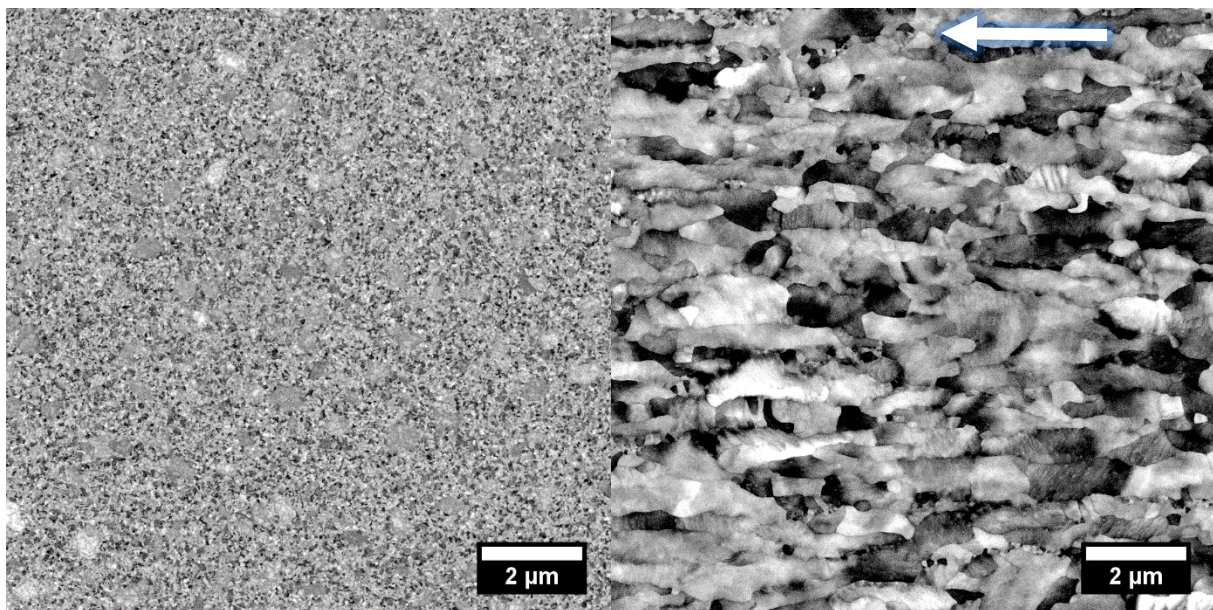


Figure 1 - Microstructure of a 110 nm thick iron-carbon film before (left) and after (right) current treatment of 4 MA/cm² at 550°C. The arrow shows the direction of current flow.

Thin nanocrystalline iron films with carbon concentrations of up to 1 wt.% are prepared as a model system and to compare with the behavior of nanocrystalline bulk samples. The films are then annealed at 460°C to allow for grain growth before they are heated to a temperature between 470 and 650°C and exposed to high current densities comparable to those experienced during the sintering process of bulk materials. The evolution of the microstructure, morphology and carbon concentration are investigated using electron microscopy and x-ray diffraction as a function of temperature, current density and time. Extensive effects of the electrical current on C redistribution and grain growth are observed, including strong coupling between grain growth and C content. The formation of microstructures comparable to rolled materials with grains that are elongated along the direction of the current are found. The various microstructural observations will be summarized and possible explanations will be discussed.

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EFFECT OF ELECTRIC CURRENT ANNEALING IN PHASE TRANSITION OF MN-AL ALLOY

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Key Words: Electric current annealing, phase transition, Mn-Al alloy, magnetic material

The electronic structure of any material can be modified when it is exposed to high density electric currents or high strength electric fields, caused by the increased electronic/ionic mobility. Electromigration effects can have desirable uses (1), but also be a problem, for example in IC circuit design (2). However, the increased electronic/ionic mobility can be used to tailor the material properties by modifying e.g. phase formation, phase stability, density of defects etc. Our goal is to understand, by theoretical (DFT calculation) and experimental approaches, and utilize these effects in the processing of hard magnetic materials and to quantify the influence of the electric current on microstructure and magnetic properties.

In our work, we investigated the influence of the electric current density on the phase formation in Mn-Al alloys during the heat treatment process. For this, $Mn_{54}Al_{46}$ melt spun ribbons, with typical cross-section of 0.03x5 mm were heat treated under applied electric current in a home build device. The device allows the sample temperature and resistance to be measured simultaneously. This approach allows to decouple the macroscopic Joule heating and the effect of electric current during annealing by detecting changes in the phase transition temperature through resistance variations.

The results have shown that increasing the current density from 2.5 (solid line) to 30A/mm² (dashed line) the phase transition temperature was shifted to 55°C towards lower temperature, as shown in Figure 1a. This can be seen by the resistance drop over the annealing temperature. This drop is related to the transition of hcp structure to the tetragonal L1₀ phase, which presents lower resistance due to the ordered structure. It worth mentioning that the phase transition happened in a temperature window of 75°C for both cases. The phase transition was also confirmed by magnetic measurements (Figure 1b), where the initial state (as-spun) is non-magnetic and the L1₀ is ferromagnetic.

The systematic shift in the phase transformation temperature was observed with increasing current density. Based on these results, new possibilities of changing the phase stability conditions and, consequently, microstructure can be used to enhance magnetic properties of Mn-Al alloys.

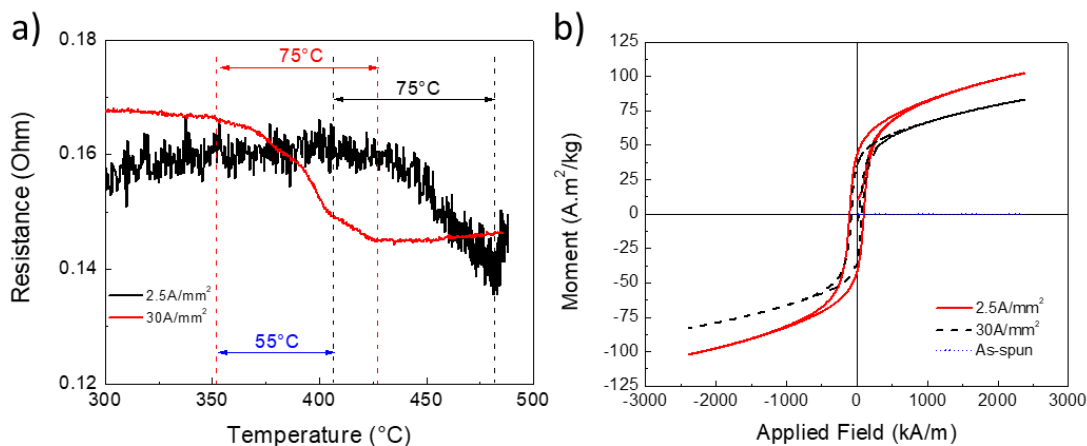


Figure 13 – Resistance change vs. temperature (a) and magnetic properties of the electrically annealed samples (b), as a comparison, the initial state (as-spun) is also shown.

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INSIGHTS INTO REACTIVE FLASH SINTERING OF $\text{MgO-Al}_2\text{O}_3\text{-(8YSZ)}$ BY IN-SITU SYNCHROTRON X-RAY DIFFRACTION

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Key Words: Flash sintering, Spinel, Phase transformation, Synchrotron, X-ray diffraction

Recently, flash sintering has been expanded to synthesize complex oxides in a few seconds at low furnace temperatures. It is called 'Reactive Flash Sintering (RFS)'. Here, we study reactive flash sintering of $\text{MgO-Al}_2\text{O}_3$ to MgAl_2O_4 . 8YSZ was added as a flash catalyst, since MgO and Al_2O_3 could not be flashed by itself within our experimental condition. We show that one-step flash experiment produces polycrystals of high density, also the transformation of magnesia and alumina into single-phase spinel.

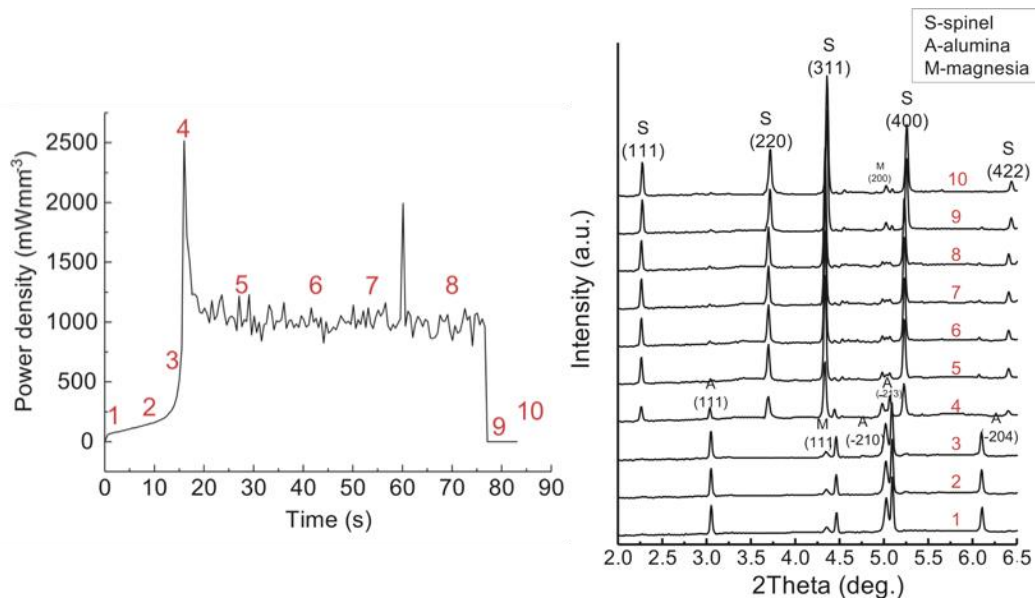


Figure 1. Power density curve for flash sintering and in-situ XRD analysis of the composite.

FLASH SINTERING OF ZIRCONIA/ALUMINA POWDERS

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Key Words: Flash sintering, particle atomic layer deposition, yttria-stabilized zirconia, alumina

Yttria-stabilized cubic zirconia (YSZ) is the most-common electrolyte material for solid oxide fuel cells due to its reasonable oxygen-ion conductivity and chemical stability. To achieve suitable ionic conductivities, YSZ ceramics must be near theoretical density, requiring sintering temperatures around 1450°C. In 2011, it was demonstrated that flash sintering densifies YSZ in a few seconds at 750°C.¹ During flash sintering, an electric field is applied across the sample and at a certain threshold temperature, the sample conductivity and power dissipation rapidly increase causing densification. Since densification occurs in just a few seconds, grain growth can be difficult to control. During conventional sintering of YSZ ceramics, researchers have demonstrated that the addition of a small quantity of aluminum oxide (Al_2O_3) pins grain boundaries, reducing YSZ grain size.² However, the effect of small quantities of Al_2O_3 addition on the grain growth of flash sintered YSZ has not been studied.

In this work, atomic layer deposition (ALD) was used to deposit a conformal film of amorphous Al_2O_3 on the surface of YSZ particles. Particle ALD is a gas-phase deposition technique that homogeneously disperses a small quantity of Al_2O_3 throughout the green body to control grain growth during flash sintering. Flash sintering experiments showed that the addition of 2.2 wt% Al_2O_3 by Particle ALD increased the flash temperature of YSZ from 760°C to 880°C at an electric field of 300 Vcm^{-1} (Figure 1). Scanning electron microscopy was used to determine the effects of the Al_2O_3 films on grain growth and microstructural evolution during flash and conventional sintering. Transmission electron microscopy and energy dispersive X-ray spectroscopy were used to characterize the as-deposited Al_2O_3 films.

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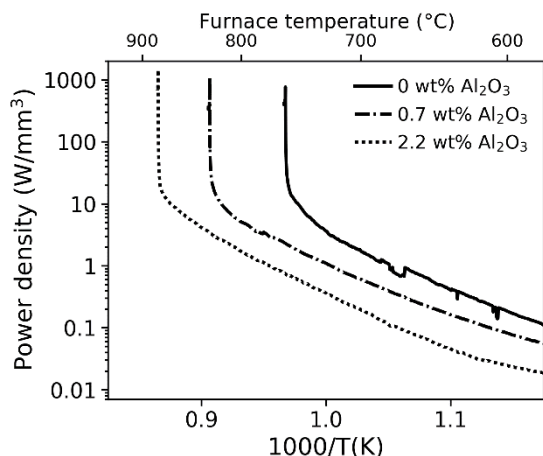


Figure 14 – Power dissipation for samples with 0, 0.7, and 2.2 wt% Al_2O_3 addition.

THE INFLUENCE OF CARBON ON THE MICROSTRUCTURE OF SINTERED ALUMINA

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Key Words: Sintering, Alumina, Carbon, Microstructure, Sintering Atmosphere.

Alumina is one of the most used ceramic materials, and as such understanding its sintering and densification processes is important. It is known that the sintering behavior is strongly affected by dopants, such as MgO, which promotes sintering and limits grain growth. The present study focuses on the influence of carbon on the sintering of alumina. Commercial ready-to-press (RTP) alumina specimens were sintered to full density (98%) at 1600°C for 2 h in air and in a graphite furnace using flowing He, a reducing atmosphere. The specimens sintered in graphite furnace resulted in a black color, an indication of the high carbon content originating from the large amount of organic compounds in RTP powders. In comparison, alumina specimens sintered in air, where the carbon decomposes during sintering at elevated temperatures, were white. Sintering with carbon in under He resulted in specimens with a finer microstructure. The presence of carbon retards grain growth, most probably by solute drag. A uniform segregation of carbon to the grain boundaries of alumina was shown by atom probe tomography [1]. In order to evaluate the wear resistance of the sintered alumina, the time (normalized by area) to section specimens with a diamond wafer blade was determined. The time to section specimens containing carbon was more than 40 times longer compared to the specimens sintered in air. The combination of reducing atmosphere and high carbon content has a positive effect on the microstructure and mechanical properties of alumina.

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DENSIFICATION OF CLASSIC AND FRAGILE FERROELECTRICS BY COOL-SPS

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Key Words: Cool-SPS, low temperature sintering, fragile materials, dense ceramics, classic ferroics

This presentation will illustrate the investigation by Cool-SPS (Spark Plasma Sintering) of the sintering of fragile ferroelectric materials. The efficiency of this advanced technique to sinter, stabilize or even synthesize fragile materials has been established by previous studies conducted at ICMCB [1,2].

Fragile materials have a limited thermodynamic stability that prevents their conventional sintering, i.e. pressureless densification at high temperature ($T > 1000^{\circ}\text{C}$). However, the characterization and use of ferroelectric materials require dense samples. An access to the ceramic form for fragile ferroelectrics could allow avoiding the difficulties inherent to the elaboration and characterization of thin films or single-crystals. In this presentation, it will be shown that Cool-SPS can be used to obtain highly densified ceramics of classic ferroic materials such as Rochelle salts, KDP, etc. Dense ceramics of such fragile ferroelectrics were obtained in less than 2 hours at low temperature ($< 200^{\circ}\text{C}$). As these classic ferroics have been studied for decades in single-crystal form, their successful sintering offers an opportunity to investigate their ferroic properties in polycrystalline form. The structure and micro-structure of the obtained ceramics will also be presented. Moreover, with previous reports concerning the sintering of KDP (and also proton conductor CsH_2PO_4 [3]) by CSP (Cold Sintering Process), advantage will be taken of the opportunity to compare both approaches and highlight their complementarity.

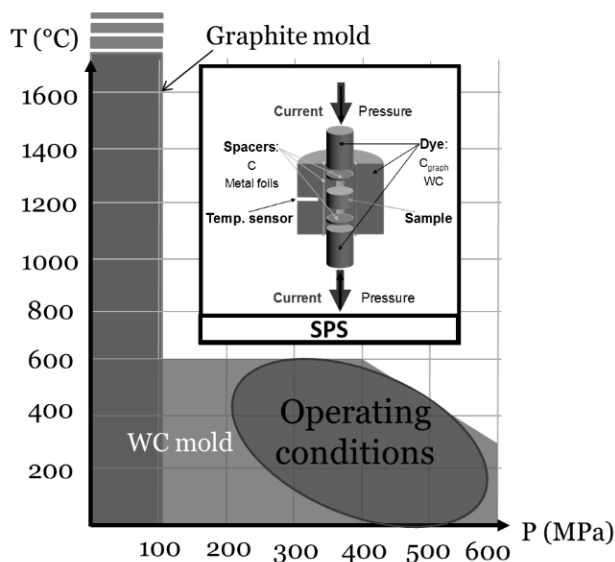


Figure 15 – Cool-SPS operating conditions used to sinter fragile materials.

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EUTECTIC MICROSTRUCTURES BY FLASH SINTERING

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Ceramic composites made of alumina and monazite can be densified by flash sintering, whereas the individual components are difficult to flash sinter, raising an intriguing question as to what role interfaces and point defects play. Examples from alumina and monazite composites, contrasted with monazite and YSZ composites are shown. With a high current density, novel eutectic microstructures formed in the hot zone of alumina/monazite composites may lead to interesting mechanical properties, with the shell region composed of polycrystalline equiaxed grains. In contrast, YSZ/monazite composites produce a conventional microstructure after flash sintering.