CONFERENCE ON FAST/SPS
From Research to Industry

BOOK OF ABSTRACTS

KRAKOW, 20–22.10.2025

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October 20–22, 2025
Monday, 09.00–23.00
Tuesday, 09.00–19.00
Wednesday, 09.00–13.30

Łukasiewicz Research Network –
Krakow Institute of Technology
73 Zakopiańska St.
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CONFERENCE PROGRAMME

DAY 1
OCTOBER 20, 2025 (MONDAY)

09.00–09.30 **Registration of participants**

09.30–09.45 **Opening of the conference**

09.45–10.30 **Keynote presentation**
Chair: Dariusz Garbiec
A New Paradigm in Alloy Design, Based on High Entropy, for Developing Advanced PM Materials while Avoiding the Direct Use of Fundamental Metals
Jose M. Torralba, Charles III University of Madrid/IMDEA Materials Institute, Madrid, Spain

10.30–10.50 **Coffee break**

10.50–12.30 **Session I**
Alloys and Metallic Materials
Chair: Jose M. Torralba

10.50–11.10 **Advanced HEA-Ti_3SiC_2 Composites Processed by FAST/SPS: Tailoring Microstructure and Strength through Reinforcement Control**
Beata Leszczyńska-Madej, AGH University of Krakow, Krakow, Poland

11.10–11.30 **FAST/SPS-Based Manufacturing of Co-Cr-Fe-Mn-Ni High-Entropy Alloy**
Anna Kopeć-Surzyn, AGH University of Krakow, Krakow, Poland

11.30–11.50 **The Influence of Mo and TiC Content in Titanium Matrix Composites on the Microstructure, Mechanical, Tribological, and Corrosion Properties Obtained by the FAST/SPS Process**
Paweł Figiel, West Pomeranian University of Technology, Szczecin, Poland

11.50–12.10 **Spark Plasma Sintering of Hybrid Reinforced Ferritic Alloys: Microstructural Evolution and Corrosion Behavior**
Bartosz Bucholc, Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

12.10–12.30 **Effect of Yttrium Oxide Content on the Properties of Austenitic ODS Steels Manufactured by SPS Method**
Grzegorz Kuderski, Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

12.30–13.00 **Coffee break**

13.00–14.40 **Session II**
Advances in FAST/SPS Processing and Technology
Chair: Diletta Giuntini

13.00–13.20 **A Digital Twin for Design and Optimization of SPS Tooling**
Alexander Laptev, Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland

13.20–13.40 **The Future of FAST/SPS in Fusion: Scale-Up of Advanced SMART Materials**
Monica Keszler, Forschungszentrum Jülich GmbH, Jülich, Germany

13.40–14.00 **Evaluation of High Heating Rates in FAST/SPS Sintering: Benefits, Challenges, and Limitations**
Jakub Wiśniewski, Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland

14.00–14.20	**Is What We See Is Really What We Get? Process Data Correction Approach** *Kamil Kaszyca, Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland*
14.20–14.40	**From Superalloys to Intermetallics: The Role of U-FAST Technology in the Transformation of High-Temperature Materials** *Katarzyna Jach, GeniCore Sp. z o.o., Warsaw, Poland*
14.40–15.30	Lunch
15.30–16.30	Session III **Magnetic Materials** *Chair: Jan Raethel*
15.30–15.50	**Advanced Powder Metallurgy as Key for High-Performance Soft Magnetic Components** *Johannes Trapp, Fraunhofer Institute for Manufacturing Technology and Advanced Materials IFAM, Dresden, Germany*
15.50–16.10	**Directly Recycling Hot-Deformed Nd-Fe-B Magnet Scrap via Spark Plasma Texturing** *Monica Keszler, Forschungszentrum Jülich GmbH, Jülich, Germany*
16.10–16.30	**Tailoring the Properties of Cr_{2}-xV$x$$O_{3}$ Compounds through Spark Plasma Synthesis** *Igor Veremchuk, Helmholtz-Zentrum Dresden-Rossendorf, Dresden, Germany*
16.30–17.30	Poster session & exhibition
20.00–23.00	Conference dinner

DAY 2
OCTOBER 21, 2025 (TUESDAY)

09.00–09.05	**Opening of the second day of conference**
09.05–09.50	Keynote presentation *Chair: Alexander Laptev* **Spark Plasma Sintering of Light Ceramics Based on Boron Carbide or Boron Phosphide. Comparison and Analysis of Their Sintering Ability and Mechanical Performances** *Alexandre Maitre, University of Limoges, Limoges, France*
09.50–10.10	Coffee break
10.10–11.30	Session IV **Borides** *Chair: Piotr Klimczyk*
10.10–10.30	**Manufacturing of Boride-Based Composites Utilizing SPS Technique** *Zbigniew Pędzich, AGH University of Krakow, Krakow, Poland*
10.30–10.50	**Impact of Precursor Morphology on the Synthesis and Properties of TaB_{2} Fabricated via Reactive Spark Plasma Sintering** *Jan Słomiński, Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland*
10.50–11.10	**The Influence of Different Coatings on the Tribological Behavior of Plasma-Sintered Ti-TiB_{2} Materials at the Nanoscale** *Wojciech Łoński, Łukasiewicz Research Network – Institute for Sustainable Technologies, Radom, Poland*
11.10–11.30	**Influence of Substrate Temperature on Properties of W-Ti-B and W-Ta-B Coatings on Cemented Carbide Tools Deposited by HiPIMS** *Rafał Psiuk, Institute of Fundamental Technological Research PAS, Warsaw, Poland*
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12.00–13.20	Industrial session *Chair: Rafał Zybała*
12.00–12.20	**Innovative Developments in Spark Plasma Sintering (SPS) Equipment Driven by Application Demands** *Damian Karpowicz, GeniCore Sp. z o.o., Warsaw, Poland*

12.20–12.40	**Materials Design, Systems & Devices LLC: Innovating Advanced Sintering Technologies** *Juliusz Leszczyński, Materials Design, Systems & Devices LLC, Krakow, Poland*
12.40–13.00	**Graphite as a Tool for SPS Molds** *Kristian Beno, TOP Grafit s. r. o., Topolcany, Slovakia*
13.00–13.20	**Sintering Ceramic Materials for Industrial Application** *Jens Huber, Dr. Fritsch Sondermaschinen GmbH, Fellbach, Germany*
13.20–14.00	**Lunch**
15.00–19.00	**City tour**

DAY 3
OCTOBER 22, 2025 (WEDNESDAY)

09.00–09.05	**Opening of the third day of conference**
09.05–09.50	**Keynote presentation** *Chair: Abdullah Riaz* **Double-Tough Ceramics: Leveraging Multiscale Toughening via Material Design, FAST/SPS and Bayesian Optimization** *Diletta Giuntini, Eindhoven University of Technology, Eindhoven, The Netherlands*
09.50–10.10	**Coffee break**
10.10–11.10	**Session V** **Functional Materials** *Chair: Johannes Trapp*
10.10–10.30	**Field-Assisted Sintering of Interconnected Porous Lithium Sodium Potassium Niobate Piezoceramics for Bone Regeneration** *Abdullah Riaz, University of Rostock, Rostock, Germany*
10.30–10.50	**Development of FAST/SPS-Processed Targets for Magnetron Sputtering of LiPON Solid-State Ionic Conductors** *Mateusz Marczewski, Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland*
10.50–11.10	**FAST-Delivered Ceramics for K-ion Cell Components** *Wiktoria Krzyżaniak, Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland*
11.10–11.40	**Coffee break**
11.40–12.40	**Session VI** **Advanced Ceramic Composites** *Chair: Marcin Chmielewski*
11.40–12.00	**A Comparative Analysis of SPS and HPHT Sintering Methods for Advanced Ceramics Production** *Yuliia Rumiantseva, Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland*
12.00–12.20	**Demonstrator of the Heat Shields Manufactured by FAST/SPS and ACS Technology** *Maria Wiśniewska, Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland*
12.20–12.40	**The Second Life of WC in Oxide-Carbide Duplex Ceramics** *Piotr Klimczyk, Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland*
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CONTENTS

I. ORAL PRESENTATIONS

Jose M. Torralba[1, 2], Alberto Meza[2], S. Venkatesh Kumaran[3], Amir Mostafaei[4], Ahad Mohammadzadeh[5], Sama Safarloo[2], Lucía García de la Cruz[1], Paula Alvaredo[1], Mónica Campos[1]

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[5]University of Maragheh, Maragheh, Iran

A New Paradigm in Alloy Design, Based on High Entropy, for Developing Advanced PM Materials while Avoiding the Direct Use of Fundamental Metals

Abstract

The problems we face when designing new alloys are increasing, particularly with regard to critical and strategic elements. These issues are becoming more prevalent as the use of these critical elements increases exponentially with the introduction of non-fossil fuel-based energy. We must change the design paradigm by developing methodologies that maximize the potential for recycling, moving inexorably towards sustainable metallurgy.

This paper will present examples of this new approach, based on the concept of the 'commodity method' and powder technology. This methodology enables us to recycle complex scrap and e-waste concentrates more effectively, as all elements can be used to contribute positively to the design, with the high entropy concept acting as the main driver.

Beata Leszczyńska-Madej, Ewa Małkowska,
Anna Wąsik, Marcin Madej, Paweł Nieroda
AGH University of Krakow, Krakow, Poland

Advanced HEA-Ti_3SiC_2 Composites Processed by FAST/SPS: Tailoring Microstructure and Strength through Reinforcement Control

Abstract

This study presents the development of advanced high-entropy alloy (HEA) composites reinforced with Ti_3SiC_2, fabricated via the FAST/SPS technique. The work focuses on tailoring the microstructure and mechanical properties by controlled variation of processing parameters and reinforcement content.

The FAST/SPS method effectively consolidated dense HEA-Ti_3SiC_2 composites with refined microstructures and minimal porosity. The use of rapid heating rates and short sintering times suppressed grain growth and promoted strong particle bonding. Mechanical performance strongly depended on Ti_3SiC_2 content; the composite containing 10 wt% reinforcement exhibited the best balance of strength, ductility, and microstructural uniformity.

Sintering temperature showed a non-linear influence on material properties: at higher temperatures, improved matrix hardness and stronger interfacial bonding were observed, especially at elevated Ti_3SiC_2 contents. Meanwhile, samples with lower reinforcement levels exhibited grain growth and slight deterioration of matrix hardness with increasing temperature. Microstructural analysis confirmed uniform dispersion of the reinforcement phase and stable interfacial bonding.

Mechanical testing demonstrated progressive improvements in compressive strength and hardness with increasing Ti_3SiC_2 content, reaching a peak at 15 wt% reinforcement. This behavior was attributed to synergistic effects of enhanced particle distribution and interfacial bonding.

Overall, the study shows that moderate addition of Ti_3SiC_2 to a Cantor-type HEA matrix significantly enhances mechanical performance without compromising microstructural integrity. These findings offer valuable insights for optimizing processing conditions of next-generation HEA-based composites with potential applications in aerospace, automotive, and energy sectors.

Acknowledgement

This research was funded by the Ministry of Science and Higher Education of Poland, grant number 16.16.180.006.

Anna Kopeć-Surzyn, Marcin Madej, Beata Leszczyńska-Madej
AGH University of Krakow, Krakow, Poland

FAST/SPS-Based Manufacturing of Co-Cr-Fe-Mn-Ni High-Entropy Alloy

Abstract

The Co-Cr-Fe-Mn-Ni high-entropy alloy (HEA) was manufactured using field-assisted sintering technology/spark plasma sintering (FAST/SPS), as this technique offers precise control over sintering parameters, enabling tailored microstructures and enhanced properties in complex alloy systems such as HEAs.

The study was conducted in two stages. In the first, samples were sintered at temperatures ranging from 875°C to 975°C under a compaction pressure of 50 MPa for 15 mins in vacuum. In the second stage, sintering was performed at 1050°C under the same compaction pressure, with varied sintering times ranging from 7.5 to 60 mins. The resulting densities, hardness values, microstructures and near-surface layers of the sintered samples were thoroughly analyzed.

The results showed a clear increase in both hardness and density with rising temperature and extended sintering time. Samples sintered at 975°C reached up to 346 HV_1, while those processed at 1050°C exceeded 600 HV_1. Microstructural analysis confirmed the formation of a homogeneous, single-phase FCC solid solution with fine, evenly distributed pores indicating effective densification.

Acknowledgement

Research project partly supported by program „Excellence Initiative – Research University" for the AGH University of Krakow, application number 10651.

Paweł Figiel[1], Rafał Rubach[2], Anna Biedunkiewicz[1], Mirosława Pawlyta[3, 4], Dariusz Garbiec[2]
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The Influence of Mo and TiC Content in Titanium Matrix Composites on the Microstructure, Mechanical, Tribological, and Corrosion Properties Obtained by the FAST/SPS Process

Abstract

This study examines the properties of titanium matrix composites reinforced with 10 wt% and 20 wt% titanium-molybdenum carbides ($Ti_{1-x}Mo_xC$), with Mo content variations of x=0.05, 0.15, and 0.20. These composites were fabricated using spark plasma sintering (SPS), with optimal consolidation parameters determined experimentally. The composite powders consisted of microcrystalline titanium and sol-gel-synthesized nanocrystalline (Ti,Mo)C.

A key innovation in this study was the introduction of an isothermal hold at 800°C for 5 mins during SPS, which enabled near-full densification of the composites (~100% relative density). Structural characterization was performed using X-ray diffraction (XRD), while microstructural analysis employed scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Corrosion resistance was tested in a 3 wt% NaCl solution via potentiodynamic polarization. Mechanical properties were evaluated through Vickers hardness (HV_{30}), fracture toughness (K_{1C}), Young's modulus (ultrasonic testing), and wear resistance (ball-on-disk method). The optimized process parameters yielded composites with fine grain structures and a uniform dispersion of the reinforcing phase.

Phase analysis revealed the presence of α-Ti, α′-Ti martensite, β-Ti (Mo), and TiC, with the composition highly dependent on Mo content. This allowed a detailed investigation into how Mo levels and TiC proportions influence composite performance. Moreover, composites with the highest α′-Ti martensite content showed the lowest hardness (529–535 HV_{30}), Young's modulus (140–152 GPa), and TiC content, but exhibited superior fracture toughness (9.29–10.34 $MPa{\cdot}m^{1/2}$).

Bartosz Bucholc[1,2], Rafał Zybała[1,3], Kamil Kaszyca[1], Grzegorz Kuderski[1], Anna Dobkowska[4], Katarzyna Pietrzak[2]

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Spark Plasma Sintering of Hybrid Reinforced Ferritic Alloys: Microstructural Evolution and Corrosion Behavior

Abstract

Oxide dispersion-strengthened (ODS) ferritic steels are promising materials for demanding high-temperature applications due to their excellent creep resistance, corrosion resistance, and mechanical stability. In standard ODS alloys, reinforcement is provided by Y_2O_3 nanoparticles, which serve as stable dispersoids within the ferritic matrix. Their nanoscale distribution contributes to grain refinement, effective dislocation pinning, and improved microstructural stability.

In this study, the reinforcement concept was extended toward a hybrid strategy by combining conventional Y_2O_3 with additional Al_2O_3 nanoparticles to form hybrid reinforced ferritic alloys (HRFAs). Powders with a fixed Y_2O_3 content and varying Al_2O_3 additions were prepared by high-energy mechanical alloying and subsequently consolidated via spark plasma sintering (SPS), offering refined control over densification and grain evolution compared to conventional hot isostatic pressing or extrusion.

Microstructural characterization was the primary focus and employed scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS) to assess grain morphology, dispersoid size, and spatial distribution. Near-surface chemistry and oxide states were examined by Auger electron spectroscopy (AES) and X-ray photoelectron spectroscopy (XPS). For contextualization of environmental stability, electrochemical measurements in chloride-containing solution: open circuit potential (OCP), linear sweep voltammetry (LSV), and electrochemical impedance spectroscopy (EIS) were performed.

The results demonstrate that Y_2O_3 and Al_2O_3 behave differently under MA-SPS processing, leading to distinct microstructural features and heterogeneous oxide distributions. These differences strongly influenced grain evolution and near-surface chemistry, with measurable consequences for hardness and corrosion resistance in chloride environments. Together, the findings highlight the complexity of hybrid reinforcement strategies.

Acknowledgement

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Grzegorz Kuderski[1)], Anna Dobkowska[2)], Jarosław Mizera[2)], Dorota Moszczyńska[2)], Kamil Kaszyca[1)], Krystian Kowiorski[1)], Marcin Chmielewski[1)], Rafał Zybała[1)]

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Effect of Yttrium Oxide Content on the Properties of Austenitic ODS Steels Manufactured by SPS Method

Abstract

Nowadays, ODS steels are known as best candidates for high temperature and tough working conditions operations. Their extraordinary properties, such as ductility, machinability, temperature stability and corrosion resistance, make them perfect candidates for nuclear reactor material.

The aim of the presented study was to determine the effect of yttrium oxide addition on the properties of oxide-strengthened austenitic steel (ODS). The investigated 304L steel was strengthen by Y_2O_3 by weight percent: 1%, 2%, 3%. The process of samples manufacturing consisted of mechanical grinding of the powders in a ball mill and sintering using SPS method.

Detailed analysis of fabricated materials will be presented. Surface morphology, crystal structure, chemical and phase composition, corrosion rate, Brinell hardness, conventional yield strength, tensile strength, uniform elongation, and total elongation were characterized and investigated.

The work included the characterization of powders (PSD, SEM, EDS), and characterization of the microstructure of sintered (light microscopy, SEM, XRD, EDS, EBSD, XRF), corrosion resistance tests (electrochemistry, corrosion rate in a salt chamber, FTIR, SEM, and EDS of corrosion products), mechanical properties (Brinell hardness, static tensile strength test).

The research showed that yttrium oxide has a huge impact on the tested properties of 304L steel. Yttrium oxide significantly increased conventional yield strength, tensile strength, Brinell hardness, and corrosion resistance. The use of the SPS method enabled very rapid powder sintering, preventing over-growth of grains.

Acknowledgement

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Alexander M. Laptev
Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland

A Digital Twin for Design and Optimization of SPS Tooling

Abstract

Transitioning SPS technology from research to industry demands attention to cost, product quality, reproducibility, and effective marketing. Herein, the proper design and exploitation of the SPS tooling are crucial. The optimized tooling design involves selecting materials and shaping the tooling parts to facilitate uniform sintering with minimal energy consumption. The effective use of tooling depends on optimizing the sintering parameters (such as the temperature, number of sintering segments, applied pressure, heating rate, etc.). All these tasks can be fulfilled using either the trial-and-error approach or a digital twin of the SPS tooling. The last strategy is both time-efficient and cost-effective, matching the trends of Industry 4.0. The developed digital twin (DT), called Leonardo 2.0, utilizes ANSYS software for finite element modeling of the temperature distribution within the SPS tooling and the sintered sample. The digital twin operates under the control of an operating file that specifies the tooling geometry, materials of tooling parts, meshing settings, the sintering cycle, and the results presentation. Additionally, an external database supplies the digital twin with relevant material data. The calculation procedure is supported by a virtual PID temperature controller embedded in the digital twin. All initial parameters can be easily adjusted with minor modifications to the operating file, requiring no programming skills. The predefined output options include temperature contours throughout the entire SPS setup and/or in the sintered sample at the end of sintering, the electric power required, and the temperature development at four key points within the sintered sample. The ANSYS post-processing features provide additional output options. The developed DT Leonardo 2.0 simulates the sintering of discs but can be customized to model the sintering of rectangular samples and other simple and complex shapes. Fig. 1 schematically shows the interaction of the digital twin with ANSYS and the material database.

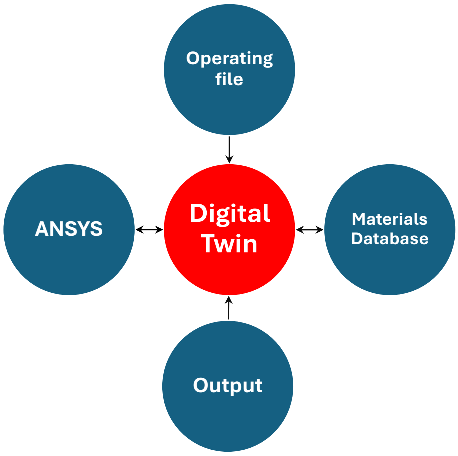

Fig. 1. Control and interaction in the DT Leonardo 2.0

Acknowledgement

Research funded by Łukasiewicz Research Network – Poznań Institute of Technology, project no. S-6665-1-2025.

Monica Keszler, Jie Chen, Aparna Binu Varghese, Martin Bram, Andrey Litnovsky
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The Future of FAST/SPS in Fusion: Scale-Up of Advanced SMART Materials

Abstract

The plasma-facing walls of fusion power plants must be made of materials that can withstand many challenges, including long-term stability under fusion plasma exposure and suppression of volatile oxide formation in accident conditions. A promising class of alloys, known as self-passivating metal alloys with reduced thermo-oxidation (SMART), has arisen with potential to fulfil these requirements. These alloys are tungsten-based matrices with chromium as a passivating element coupled with yttrium as an active element. So far, the most promising oxidation resistance has been observed in alloys with submicron grain sizes. Field-assisted sintering technology/spark plasma sintering (FAST/SPS) of mechanically alloyed tungsten, chromium, and yttrium powders has been utilized to produce SMART materials of optimal grain size, homogeneous microstructure, and well-balanced properties. Optimization of FAST/SPS parameters for scale-up, with the goal of producing 100×100 mm^2 demonstrator wall components, is ongoing. Future activities involve the demonstration of durability of new SMART components under reactor-relevant plasma conditions as well as potential automation of production for large-scale manufacturing.

**Jakub Wiśniewski[1, 2], Wiktoria Krzyżaniak[1],
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[2]Poznan University of Technology, Poznan, Poland

Evaluation of High Heating Rates in FAST/SPS Sintering: Benefits, Challenges, and Limitations

Abstract

Field assisted sintering technology (FAST/SPS), is widely recognized as one of the most efficient consolidation techniques for powders, enabling the production of dense materials within exceptionally short processing times. Among the various process parameters that govern the efficiency and quality of sintering, the heating rate is one of the most important one. It directly determines not only the kinetics of densification but also the microstructural development of the material. While in principle extremely high heating rates are achievable in modern FAST/SPS systems, their implementation is far from straightforward and brings about a number of opportunities as well as challenges that must be carefully addressed.

The presented work focuses specifically on the feasibility of attaining very high heating rates and on the technological and process-related factors that inherently limit their applicability. Special attention is given to the influence of the dimensions and geometry of the sintering tools, which strongly affect the efficiency of energy transfer and thereby the maximum attainable heating rate. By analyzing these constraints, the work highlights where the practical boundaries of the process are likely to occur.

Another important aspect under investigation is the effect of elevated heating rates on the homogeneity of sintered materials. As heating rates increase, the risk significant thermal gradients within large or geometrically complex compacts becomes a critical concern. These gradients can lead to inhomogeneous densification, local variations in microstructure, and ultimately reduced performance of the final component. Furthermore, the phenomenon of local overheating of graphite tools, particularly under extreme heating conditions, is emphasized as a decisive factor that influences both the stability of the sintering process and the lifetime of the tooling.

By combining experimental findings with process analysis, this work aims to deliver an overview of the role of heating rate in FAST/SPS. Both the benefits – such as reduced processing time and potential for refined microstructures – and the limitations associated with pushing the heating rate to its extremes are discussed. The insights obtained are intended to support more reliable process design, improved reproducibility, and practical optimization of FAST/SPS technologies.

Acknowledgement

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Is What We See Is Really What We Get? Process Data Correction Approach

Abstract

The aim of this presentation is to examine the differences between sintering curves recorded by the acquisition system and the actual physical parameters affecting the sample. Accurate analysis of sintering process data is crucial for process design and optimization. Precise determination of sample shrinkage parameters also represents an important step in modeling the spark plasma sintering (SPS) process. This work introduces a multi-step approach for correcting process data obtained with an FCT H-HP D 25 device. Corrections for temperature, applied pressure, and sample deformations are discussed and presented. Data analysis was performed using Python with the SciPy package.

Acknowledgement

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From Superalloys to Intermetallics: The Role of U-FAST Technology in the Transformation of High-Temperature Materials

Abstract

Superalloys have long been the benchmark for structural applications in extreme environments, including gas turbines, jet engines, and nuclear reactors. Their exceptional resistance to creep, oxidation, and corrosion at elevated temperatures makes them indispensable in high-performance systems. However, the production of superalloys is associated with significant drawbacks: high material and processing costs, energy-intensive manufacturing, and complex post-processing requirements. These limitations have prompted the search for alternative materials that can deliver comparable performance with improved sustainability and cost-efficiency.

Intermetallic compounds have emerged as promising candidates due to their high melting points, excellent oxidation resistance, and relatively low density. Moreover, they are often composed of more abundant and less critical raw materials. Despite these advantages, their widespread adoption has been hindered by intrinsic brittleness and poor sinterability, which make conventional processing methods ineffective or economically unviable.

The U-FAST (upgraded field assisted sintering technology) developed by GeniCore offers a breakthrough solution. This advanced sintering method enables:
- rapid densification of intermetallics without compromising structural integrity,
- significantly lower sintering temperatures, reducing grain growth and enhancing mechanical properties,
- precise control over microstructure, which is essential for brittle materials,
- energy-efficient and scalable processing suitable for industrial deployment.

Recent experimental studies conducted by GeniCore demonstrate that U-FAST can successfully consolidate intermetallic compounds that were previously considered unprocessable using standard SPS techniques. This opens new avenues for replacing superalloys in high-temperature applications, particularly in aerospace, energy, and defense sectors. U-FAST thus represents a transformative step in the development of next-generation materials for extreme environments.

Acknowledgement

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Advanced Powder Metallurgy as Key for High-Performance Soft Magnetic Components

Abstract

By 2030, an estimated 300 billion kWh of electrical energy could be conserved through the deployment of advanced, energy-efficient materials in electrical applications like electric motors and transformers. However, conventional approaches relying on electrical steel sheets and standard manufacturing methods such as rolling and punching have reached their efficiency limits. Emerging materials, particularly nanocrystalline soft magnetic alloys with elevated iron content, offer a promising alternative, though their practical application has been constrained by stringent processing requirements, notably a rapid required heating rate of 100 K/min during heat treatment to realize nanocrystallization without grain growth.

This work demonstrates the successful processing of such alloys using the powder metallurgical technique FAST/SPS, which can easily achieve the necessary heating rates. Nanomet ribbons, after being coated and wound into toroidal cores, were thermally treated via FAST/SPS, resulting in nanocrystalline components that exhibited significantly reduced magnetic losses of 7.5 W/kg at 1 T and 1 kHz. This is significantly surpassing current commercial materials such as non-oriented electrical steel (NO20: 55 W/kg) and soft magnetic composites (Somaloy 130i 5P: 93 W/kg).

These findings underline the potential of nanocrystalline alloys processed by FAST/SPS for the next generation of high-efficiency magnetic applications.

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Directly Recycling Hot-Deformed Nd-Fe-B Magnet Scrap via Spark Plasma Texturing

Abstract

Due to its specialized microstructure, hot-deformed Nd-Fe-B magnet scrap currently has no direct recycling route. Standard powder metallurgical procedures would destroy the microstructure due to high temperatures. However, a combination of field assisted sintering technology/spark plasma sintering (FAST/SPS) and spark plasma texturing (SPT) has shown to be an effective method of preserving this microstructure while densifying magnet scrap into new magnets. This work describes the optimizations made in using FAST/SPS and SPT as alternatives to hot-pressing and hot-deformation, respectively, in order to maintain the unique anisotropic microstructure of hot-deformed Nd-Fe-B. These processes combined generated magnets with an energy product above 200 kJ/m^3. With further grinding and shaping, these magnets were formed into the desired dimensions for use in a water pump motor and tested as demonstrator parts. The 100 wt% recycled magnets reached roughly 95% of the electromotive force of a rotor containing commercially produced hot-deformed magnets.

Acknowledgement

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Tailoring the Properties of $Cr_{2-x}V_xO_3$ Compounds through Spark Plasma Synthesis

Abstract

The solid-state synthesis of transition metal oxides (TMO's) is often hindered by sluggish diffusion and mass transfer inherent to solid-state reactions (SSR's). Spark plasma sintering (SPS) has emerged as a powerful and technologically viable synthetic route to overcome these limitations. The DC current pulses inherent to SPS accelerates diffusion-controlled processes, as demonstrated by the successful synthesis of Ti_2O_3 directly from TiO_2 and Ti [1]. This approach has since been extended to other oxide systems, including titanium oxides [2], molybdenum oxides [3], tungsten oxides [4], chromium oxides [5], as well as mixed transition metal oxides. Among the advantages of SPS-assisted SSR are: i) simple preparation of starting mixtures; ii) simultaneous synthesis, compaction, and shaping of final products; iii) dramatically reduced synthesis times (minutes to a few hours); iv) high compositional precision (≈0.1 at % of oxygen); and v) excellent reproducibility.

In this work, we focus on the $(1\text{-}x)Cr_2O_3\text{-}(x)V_2O_3$ quasi-binary system, which was synthesized across the entire composition range using SPS. This approach enabled the stabilization of the oxide solid solutions that are otherwise challenging to obtain by conventional synthesis routes. The rapid heating, short dwell times, and pulsed DC current inherent to SPS not only ensured full densification but also facilitated the formation of new phases within hours.

Comprehensive investigations were carried out on the crystal structure, electrical transport, and magnetic properties of the synthesized compositions. The interplay between chromium and vanadium cations, including their mixed valence states, gives rise to rich structural and electronic versatility. These features are particularly attractive for advanced functional applications, including thermoelectrics, spintronics, and other electronic technologies.

Thus, the $(1\text{-}x)Cr_2O_3\text{-}(x)V_2O_3$ quasi-binary system exemplifies how SPS can be employed not only to accelerate classical solid-state synthesis but also to access a broad family of materials with tunable multifunctional properties.

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Spark Plasma Sintering of Light Ceramics Based on Boron Carbide or Boron Phosphide. Comparison and Analysis of Their Sintering Ability and Mechanical Performances

Abstract

The ultra-light ceramics like boron carbide and boron phosphides show numerous fields of potential applications, in particular military (ballistic protection) or nuclear (neutron moderators). Indeed, these ceramics exhibit exceptional working properties such as high thermal conductivity and mechanical properties (*e.g.* hardness), high capacity to absorb neutrons. These promising features can be interpreted considering the unusual characteristics of B-B, B-C, B-P chemical bonds.

Fully-dense boron carbide and boron phosphide ceramics were shaped and sintered by spark plasma sintering. Their densification behavior and microstructural evolution were examined as a function of the physical-chemistry features of raw powders (*e.g.* stoichiometry, grain size distribution), the thermal stability and reactivity of main boron-based phases and the sintering additives. As an example, the mechanism of thermal destabilization for low temperature boron phosphide phase (BP) has been deeply investigated by coupling in-situ/ex-situ TEM and XRD characterizations. This information has been relevant to establish the optimized SPS treatment for manufacturing full dense and micro-sized boron phosphide specimens.

Therefore, the spark plasma sintering mechanisms of carbide and boron phosphide-based ceramics were investigated using both an analytical model and densification kinetic data under isothermal and isobaric conditions. Combining this study with fine microstructure observations by TEM makes the determination of sintering mechanism possible. So, it appears that the mechanisms responsible for SPS are similar for two types of ceramics and are particularly governed by the dislocation movement.

In addition, mechanical characterizations were carried out on the sintered parts and gave promising results, particularly in terms of Vickers hardness, with values close to 35–40 GPa for both ceramic phases.

Acknowledgement

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Manufacturing of Boride-Based Composites Utilizing SPS Technique

Abstract

Manufacturing of composites with titanium diboride or zirconium diboride matrices containing dispersed carbides phase could be performed as a reactive sintering process. Boron carbide and suitable intermetallic compounds could be used for this purpose. Presented work concerns technological aspect of SPS supported synthesis of TiB_2/SiC and ZrB_2/SiC composites derived from B_4C and intermetallic from Ti-Si or Zr-Si systems. Phase compositions and microstructures are described in details. It was confirmed that dense polycrystalline composites could be achieved in the one-step process. Chemical reactions leading to boron carbide decomposition and boride phase formation were conducted with efficiency reaching 99%. Proposed procedures are suitable to produce in one technological step well densified parts composed of boride (TiB_2 or ZrB_2) and silicon carbide phases arranged as a continuous, interpenetrating structures.

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Impact of Precursor Morphology on The Synthesis and Properties of TaB_2 Fabricated via Reactive Spark Plasma Sintering

Abstract

Tantalum diboride belongs to the ultra-high-temperature ceramics family and is distinguished by exceptional characteristics such as high Young's modulus, superior thermal conductivity, remarkable thermal stability, and excellent corrosion resistance. However, its inherently low diffusion coefficient complicates densification through conventional sintering techniques. Spark plasma sintering combined with reactive sintering emerges as a promising approach, facilitating rapid consolidation of challenging-to-sinter phases while reducing processing costs by using elemental precursors. A crucial factor in optimizing the SPS process is the particle size of the starting powders, which significantly influences the microstructure, mechanical properties, onset temperature, and kinetics of the synthesis reaction. Utilizing fine precursor powders enhances reactivity but may also provoke uncontrolled reaction pathways. This study explores how the morphology of the precursors affects the SPS reactive sintering of TaB_2 and assesses the structural and mechanical properties of the resulting ceramics. Reactive SPS enables the simultaneous synthesis and densification of TaB_2 from elemental tantalum and boron powders through a solid-state combustion reaction.

The advantage of combining SPS with reactive sintering lies in achieving near-theoretical density ceramics with fine microstructure and favourable mechanical characteristics in a reduced processing time and temperature compared to traditional sintering routes.

Acknowledgement

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The Influence of Different Coatings on the Tribological Behavior of Plasma-Sintered Ti-TiB_2 Materials at the Nanoscale

Abstract

This study aims to investigate the tribological behavior of plasma-sintered titanium and titanium diboride composite materials, with a particular focus on the effect of various protective coatings applied to their surfaces. Ti-TiB_2 composites are known for their high hardness, thermal stability, and resistance to wear, making them attractive candidates for applications involving high mechanical loads and surface interactions. However, their tribological performance can be further enhanced through the application of suitable PVD coatings, which may reduce friction and wear while improving durability under nanoscale contact conditions.

The experimental approach involves the selection and application of four distinct coating types, chosen for their potential to modify surface interactions and improve tribological properties. The tribological tests were conducted using a nanotribometer, which enables precise measurement of friction coefficients, wear depth, and wear track morphology under controlled loading and environmental conditions.

The study will explore how different coatings influence wear mechanisms such as adhesion, abrasion, and delamination, and how these effects correlate with the microstructural features of the coated surfaces. By analyzing the performance of each coating, the research will contribute to a deeper understanding of coating-substrate interactions and provide insights into the design of advanced surface treatments for Ti-TiB_2-based materials.

Nanoscale tribological studies are important because they allow for understanding of the wear and friction mechanisms that occur at the level of actual surface contacts in modern devices, such as mechanical microelements, implants, and components of precision technological systems.

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Influence of Substrate Temperature on Properties of W-Ti-B and W-Ta-B Coatings on Cemented Carbide Tools Deposited by HiPIMS

Abstract

Tungsten diborides are very promising candidates as protective coatings for tools in machining and forming. Alloying such material may not only affect its properties but also interface characteristics between coating and substrate. Here, we present deposition of $W_{0.76}Ta_{0.24}B_{2.5}$ and $W_{0.76}Ti_{0.24}B_{2.5}$ layers on cemented carbide cutting tools by high power impulse magnetron sputtering (HiPIMS). Targets used for deposition were manufactured via SPS. Coatings were deposited at substrate temperatures ranging from 200 to 500°C with step of 100°. During deposition the optical emission spectroscopy (OES) was performed to obtain characteristic signal of highly energetic plasma in HiPIMS process. Coatings properties were evaluated via nanoindentation and micropillar compression. Hardness of obtained films were heavily influence by substrate temperatures. Ta-alloyed coatings reached its maximum hardness of 41.3 GPa at 500°C while Ti-alloyed reached its maximum hardness of 42.7 GPa at 400°C. It was also revealed, by X-ray diffraction, that 300°C is the minimum temperature for obtaining crystalline layers. At 200°C hardness decreases below 30 GPa due to amorphous structure. Micropillar compression tests revealed that yield strength can reach 10.3 GPa. Additionally, coatings with addition of titanium have shown small range of plastic deformation during the tests. Such behavior is probably demonstrating relatively high fracture toughness of such material. It was further revealed via cube corner indentation that fracture toughness can reach 3.7 $MPa \cdot m^{1/2}$ for coatings alloyed with titanium. Adhesion of coatings was evaluated via scratch-test. While adhesion of coatings with addition of tantalum was better already at 300°C, titanium addition has caused the coatings to adhere significantly better at higher substrate temperatures.

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Innovative Developments in Spark Plasma Sintering (SPS) Equipment Driven by Application Demands

Abstract

GeniCore tracks application requirements, which directly drive innovation in the design of its spark plasma sintering (SPS) devices. In response to evolving market needs, the company developed the advanced U-FAST Glovebox system – an integrated solution combining the MBRAUN inert gas glovebox with U-FAST SPS technology. This system enables uninterrupted processing in an inert atmosphere, eliminating exposure of oxygen- and moisture-sensitive materials, thereby achieving ultra-fast densification with custom-shaped current pulses under 1 millisecond, reducing grain growth and optimizing material properties.

Another innovation is the power supply of the U-FAST Compact PRO. The latest technological upgrade allows for higher heating rates at the same power level compared to other solutions, with a pulse rise time below 1 ms and currents up to 5 kA. The system supports three heating modes: DC (SPS) alone, AC (induction) alone, and an independent combination controlled within a single PLC unit, enabling specialized sintering processes that combine the advantages of both methods. Induction heating offers energy-efficient, rapid-response temperature control. The device is also equipped with vacuum connectors prepared for high-voltage sintering.

Both devices are designed for R&D and low-volume production of advanced materials such as bulk metallic glasses (BMG), high-entropy alloys (HEA), transparent ceramics, and thermoelectrics. Collaborations with leading scientific centers confirm their ability to meet stringent requirements and accelerate development in the electronics, energy, aerospace, and other sectors. Importantly, materials developed on a small scale to minimize resource use can be easily scaled up to industrial volumes using the same process parameters with devices built for high-volume production.

This future-proof platform provides strict environmental control and versatile process scalability, representing a significant advancement in plasma sintering technology.

Acknowledgement

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Materials Design, Systems & Devices LLC, Krakow, Poland

Materials Design, Systems & Devices LLC: Innovating Advanced Sintering Technologies

Abstract

Founded in 2018 as a spin-off from AGH University of Krakow, Poland, Materials Design, Systems & Devices LLC (MDSD™) has evolved into a company focused on the design and construction of scientific and research equipment as well as synthesis, characterization and theoretical modeling of various functional materials.

This presentation highlights our company's profile, core offerings, and our flagship products: a state-of-the-art devices (RACS25™ and RACS15™) for the densification of various powder materials, built around our unique and highly efficient resonant alternating current sintering (RACS™) technology. This technology delivers energy for sintering via high-frequency alternating current pulses generated by a resonant power supply and a matching transformer. This process is particularly effective for consolidating ionic materials, as the AC current prevents ion migration and component segregation. The system's high efficiency is achieved by matching the resonant circuit to the powder-filled die, and the close proximity of the matching transformer to the die, minimizing energy losses through the use of short current leads.

This proprietary electrothermal sintering process allows for the highly efficient consolidation of a wide range of materials, including metallic, ceramic, semiconductor, nanocrystalline, submicron, and micron powders, enabling the creation of advanced functional materials for diverse applications.

The presentation also provides an overview of a new ultra high-temperature sintering (UHS) device, a product under development with a commercial launch planned for the coming year.

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Top Grafit s.r.o., Topoľčany, Slovakia

Graphite as a Tool for SPS Molds

Abstract

Graphite is one of the 2 crystalline forms of carbon. It has unique properties, such as an excellent thermal and electrical conductivity, and a very unusual behavior at high temperatures: unlike for most materials, the mechanical properties will increase as the temperature raises, up to a range of around 2500°C. It can furthermore be quite easily machined.

In this presentation, we'll explain the production of graphite to you and describe its main properties, especially in regard to SPS.

Top Grafit is a well-known graphite machine shop located in Topoľčany, Slovakia. Our company was established in 1997 and remained independent from any graphite producer. This enables us to be in the position to offering the best suitable grade for each application. In our longer than 25 years history, we constantly grew up and enlarged our structure up to the current level of 50 skilled collaborators. Even if our main market remains Europe, we are now active on 4 continents and, thanks to our well recognized expertise and service, we have been serving many of our customers since the establishment of our company. Our structure allows us a high flexibility and we are delivering our products to OEMs, leading research institutions as well as large scale industrial customers. We offer conventional and CNC turning and milling but also most modern technologies such as waterjet cutting.

Supplementary to genuine graphite and carbon products, we also deliver CFC (carbon fiber carbon composites) parts as well as insulation (soft and hard felt) and flexible graphite foils.

Jens Huber
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Sintering Ceramic Materials for Industrial Application

Abstract

FAST/SPS sintering of metallic materials has long been state of the art. Major applications are the diamond tool and friction materials industries. However, sintering ceramics by means of FAST/SPS is much more challenging. The lack of electrical conductivity, the brittleness of the material and its reaction with the mould material make it more difficult to get fast results. This talk covers several materials such as B_4C, Al_2O_3, ZrO_2, TiB_2 as well as others. Applications for these materials range from defence to sputtering targets, jewellery and semiconductors. Additionally, we will also have a look on graded materials and heterogeneous materials, e.g. bonding of ceramic powders onto a metallic surface.

While FAST/SPS is already used by various industries for sintering ceramic materials, many issues still require investigation: the risk of cracks in the workpiece, contamination of the target material by the mould material and the difficulty of temperature measurement in the mould are just a few of the main challenges to overcome.

Process parameters such as heating rate, specific pressure at a given time, process atmosphere, mould design, cooling parameters, insulation, material composition of the part and heating current application are only some of the parameters that need to be taken into account. There are many factors influencing material behaviour, and therefore process control, that a strictly scientific approach does not yet seem possible. Therefore, this talk describes the possibilities for processing different materials with FAST/SPS technology, rather than an in-depth case study of a single material.

While the material development is the first step and pre-condition for any industrial production, we will also need to have a closer look on the FAST/SPS machines and automation systems which are able to handle the required production volumes and weights.

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Double-Tough Ceramics: Leveraging Multiscale Toughening via Material Design, FAST/SPS and Bayesian Optimization

Abstract

The traditional labeling of ceramics as brittle materials has been challenged by the last decades of efforts in toughening them with a variety of strategies. Multiscale designs, tailored additions of material constituents and controlled phase transformations have shown promising results. Both the processing and mechanical characterization of toughened ceramics, however, require innovative approaches.

We show here how multiple toughening strategies can be combined into bulk all-ceramic materials consisting of alumina and zirconia. Specifically, we present an optimization-driven approach to creating a double-tough ceramic material with a brick-and-mortar microstructure, where the zirconia mortar surrounding the alumina bricks is itself transformation-toughened, engineered with the goal of simultaneously achieving high strength and fracture toughness levels. As the design of such a material, driven by multiscale toughening mechanisms, requires a laborious trial-and-error approach, and in turn the processing parameters space is extremely broad, we propose a Bayesian optimization framework as an integral part of our methodology to streamline and accelerate the design process.

FAST/SPS is instrumental to achieving our goal. Together with promoting both densification and the development of a textured (brick) microstructure, it also enables the retention of nanograined zirconia with a prevalence of tetragonal phase in the mortar. Bayesian optimization guides the experimental campaign to expedite the optimization of the material's mechanical properties. We use a Gaussian process to emulate the material's mechanical response and implement a cost-aware batch optimization to efficiently identify optimal design process parameters, accounting for the cost of experimentally varying them. Given its process efficiency, varying the FAST/SPS parameters is less experimentally "expensive" than varying the powder preparation steps.

As a result, we develop a bio-inspired all-ceramic composite that exhibits an exceptional balance between bending (704 MPa), and fracture toughness (13.6 MPa·m$^{1/2}$).

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Field-Assisted Sintering of Interconnected Porous Lithium Sodium Potassium Niobate Piezoceramics for Bone Regeneration

Abstract

The formation of interconnected, hierarchical pores in piezoceramic scaffolds is crucial for effective bone regeneration. The porous structure supports nutrient transport and cell migration, and the piezoelectric effect provides electromechanical stimulation that promotes osteogenic activity. However, sintering interconnected porous ceramics with enhanced mechanical and electrical properties remains challenging as it requires maintaining a well-defined porous structure while achieving high densification. In this study, we fabricated porous lithium sodium potassium niobate (LNKN) using a field assisted sintering technique and zinc oxide tetrapods (t-ZnO) as a thermally stable sacrificial template. A high relative density of ~97% was achieved through sintering the mixed powders. Subsequently, the chemically unstable t-ZnO was selectively removed by citric acid-based etching, resulting in a network of interconnected pores. X-ray diffraction and energy-dispersive X-ray spectroscopy confirmed the complete removal of t-ZnO. Scanning electron microscopy revealed well-distributed interconnected pores on the surface and throughout the bulk. The resulting pores had lengths of ~60 µm with diameters of ~3–4 µm (corresponding to the length and the diameter of the t-ZnO arms). The piezoelectric characterization revealed direct and inverse piezoelectric constants of 10.0±1.7 pC/N and 112.3±17.6 pm/V, respectively, and the capacitance was 20.9±0.2 pF. These results show the potential of this approach to fabricate multifunctional piezoelectric scaffolds for advanced bone tissue engineering applications.

Acknowledgement

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Development of FAST/SPS-Processed Targets for Magnetron Sputtering of LiPON Solid-State Ionic Conductors

Abstract

Thin-film batteries are considered to be a key enabling technology for next-generation mobile applications, including wearable and portable electronics, medical devices, and sensors. The further development of these technologies is strongly linked to advances in flexible and stretchable power sources, which are of strategic importance for the European market. A critical component of thin-film batteries is the solid-state electrolyte, such as LiPON (lithium phosphorus oxynitride), which offers enhanced safety and enables operation at elevated temperatures.

The primary challenge in LiPON fabrication is the enhancement of the deposition of high-quality films from LiPO (Li_3PO_4, lithium phosphate) targets. In the DEPOION project, conducted in collaboration with Empa, BFH (Switzerland), and The Batteries Sp. z o.o. (Poland), the challenge is addressed by developing next-generation LiPO targets using FAST/SPS (field assisted sintering technique/spark plasma sintering) technology. This approach facilitates the fabrication of mechanically stable LiPO targets that are resistant to cracking. Furthermore, it facilitates their integration with conductive copper substrates as foil, thereby constituting an alternative to conventional bonded targets. Moreover, it facilitates the design of hybrid architectures that combine non-conductive LiPO with conductive Cu by adapting the shape of developed targets based on the race track after the conducted sputtering. Conductive aluminium particles are also investigated for their potential in doping, with the aim of enhancing power transfer and deposition performance.

These developments not only enhance compatibility with RF-MS (radio frequency magnetron sputtering) but also pave the way for the utilisation of HiPIMS (high power impulse magnetron sputtering) and MAR-HiPIMS (microwave assisted reactive high power impulse magnetron sputtering), offering higher plasma density, improved ionisation, and potentially superior deposition rates and film quality.

However, it should be noted that the research is still in its early stages, and there are many challenges that still need to be addressed. These include enabling sufficient power transfer for HiPIMS, optimising target architecture, and ensuring reproducibility. The presentation will therefore share the current state of progress and highlight the key findings.

Acknowledgement

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FAST-Delivered Ceramics for K-ion Cell Components

Abstract

The increasing demand for sustainable and scalable energy storage systems has intensified the search for alternatives that reduce dependence on critical raw materials (CRMs) such as lithium. Among the emerging candidates, all-solid-state potassium-ion batteries (KIBs) could be a promising and economically viable alternative due to potassium's natural abundance and low cost. However, there are still some challenges to be addressed in the development of chemically stable solid electrolytes and compatible electrode materials suitable for integration into all-solid-state KIB architectures.

Presented study explores the potential of FAST/SPS (field assisted sintering technique/spark plasma sintering) in the rapid synthesis and densification of ceramic-based materials for use in all-solid-state KIBs. A series of ceramic electrolytes and electrode materials were processed using FAST/SPS, which resulted in a significant reduction in processing time when compared to conventional manufacturing methods. In the interest of monitoring phase evolution and compositional stability, with particular attention given to potential secondary phases, a structural characterization approach via X-ray diffraction (XRD) was carried out. In addition to the successful fabrication, presented work demonstrates the potential of FAST/SPS as a tool for rapid chemical and phase composition design. It has been suggested that FAST/SPS could enable rapid synthesis and consolidation of different material formulations in a fraction of the time typically required. This has the potential to accelerate the identification of CRM-reduced, high-performance materials suited for all-solid-state KIBs.

The results suggest that FAST/SPS could potentially play a dual role as both a processing technique and materials design accelerator. Its use in developing ceramic components for all-solid-state potassium-ion batteries could be a meaningful step toward sustainable and resource-resilient energy storage solutions.

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A Comparative Analysis of SPS and HPHT Sintering Methods for Advanced Ceramics Production

Abstract

A critical comparative analysis of two pressure-assisted ceramic consolidation methods – spark plasma sintering (SPS) and high-pressure high-temperature (HPHT) sintering – was conducted for diamond-based, cBN-based, and oxide-based ceramics, with results summarized in Table 1. For diamond and cBN composites [1, 2], only HPHT enables direct grain-to-grain bonding, superior thermal conductivity, and maximum mechanical strength; while SPS and UHP-SPS allow consolidation without graphitization, minor grain boundary phases may persist, influencing overall performance. For Al_2O_3 and $MgAl_2O_4$ ceramics, both HPHT and SPS can produce dense, transparent ceramics, but HPHT more reliably eliminates pores and defects – leading to higher transparency and thermal conductivity [3]. SPS is capable of nearly equivalent properties if processing is optimized, though there is a slightly higher risk of residual color centers, minor carbon contamination, or electrical leakage due to rapid sintering and die materials. For perovskite YAP ($YAlO_3$) ceramics [4], HPHT sintering was found to enable full densification, optimized optical properties, and superior thermal stability, while SPS offers rapid prototyping and the potential for controlled do-pant retention. Future advances should focus on overcoming scaling limitations, enhancing process control, and broadening application potential. For HPHT, this requires scaling up chamber size and improving temperature/pressure uniformity. SPS will benefit from modular, multi-chamber systems, real-time digital process monitoring, and innovative die materials, enabling high-throughput production with maintained microstructural uniformity. Integrating AI-driven optimization and multi-field effects (pressure, electric, and controlled atmospheres) will be key to advancing efficiency and consistency for both processing approaches.

Table 1. Some properties of SPS and HPHT-sintered materials

	cBN Composites	**Diamond (binderless)**	**$MgAl_2O_4$ Spinel**	**Al_2O_3**	**YAP ($YAlO_3$) Perovskite**
Electrical Properties	Wide-gap, $\sigma < 10^{-10}$ S/cm	Wide-gap insulator, $\sigma < 10^{-14}$S/cm	Insulator, $\sigma < 10^{-14}$ S/cm	Insulator, $\sigma < 10^{-13}$ S/cm	Wide-gap dielectric, $\sigma \sim 10^{-10}$ S/cm
Optical Properties	Band gap: ~6.2 eV	Band gap: ~5.4 eV, Optical transmittance (UV–IR)	Band gap: ~7.8 eV, Transmit-tance>85% (UV–Vis)	Band gap: ~8.8 eV, Transmittance >80% (trans-parent grade)	Band gap: ~5.3 eV, Opti-cal transparency (UV–Vis: 300–700 nm)
Thermal conductivity (λ) and Stability	λ: ~12–15 W/mK, Tmax: ~1400°C (air)	λ: 2100 W/mK, Tmax: ~1100°C (air)	λ: 15–20 W/mK, Tmax: ~2135°C	λ: 30–35 W/mK, Tmax: >2000°C	λ: ~12–14 W/mK, Tmax: ~2050°C
SPS Conditions	1300–1400°C, 60–150 MPa	1300–1400°C, 4–5 GPa (UHP-SPS)	1200–1500°C, 50-100 MPa	1300°C, 75 MPa, 5-30 min	1400°C, 50–100 MPa (est.)
Key Properties & Advantages of SPS-sintered materials	Hardness> 21 GPa, Young's modu-lus> 470 GPa, Metastable processing	Grain boundary formation, Metastable, $\sigma < 10^{-14}$ S/cm	Transparency, Fine grains, Optical use	Density: 99.5%, Grain size: <1 μm, Lower cost, Rapid processing	>99% phase, Dense polycrys-talline, Phase purity control
HPHT Conditions	1900–2200°C, 5.5–8 GPa	1500–2000°C, 5–25 GPa	1000–1600°C, 2–5 GPa	1000–1800°C, 2–5 GPa	1200–1600°C, 2–8 GPa
Key Properties & Advantages of HPHT-sintered materials	Hardness > 26 GPa, Young's modu-lus> 580 GPa, Stability	Near-theoretical density, Direct bonding, Superior bonding	Hardness: 15–20 GPa, Controlled stoichio-metry	Density: >99%, Grain size: <1 μm, Hardness > 18 GPa, Superior purity	Enhanced mechanical properties, Controlled microstructure

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Demonstrator of the Heat Shields Manufactured by FAST/SPS and ACS Technology

Abstract

This presentation is showing the developed technology allowing the production of ZrO_2-CNTs composites for heat shields using the FAST/SPS technique. Heat shields are one of the key components of vehicles traveling outside the Earth's atmosphere, hence, it is so crucial that they meet a number of requirements, especially those related to thermal properties. FAST/SPS is one of the unconventional powder material sintering techniques and allows for the production of materials with unique properties. This paper presents the methodology by which it was possible to produce a monolithic heat shield using FAST/SPS. Zirconium dioxide (ZrO_2) is characterized by properties that make it applicable in a wide range of areas, and carbon nanotubes CNTs are an allotropic variety of carbon. The electrical conductivity of MWCNTs is significantly higher than that of ZrO_2, and that property makes the combination of these materials, with a final composition of 3YSZ-10MWCNT, a favorable choice for producing a thermal shield demonstrator by FAST/SPS. The ACS (aerosol cold spraying) technology was then applied to deposit a protective coating that protects the demonstrator surface from the oxidation of nanotubes. The developed technology makes it possible to produce a demonstrator of the monolithic heat shield. In order to scale up that methodology, it is necessary to overcome technological limitations, which for now include the limited diameter of samples that can be produced using FAST/SPS.

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The Second Life of WC in Oxide-Carbide Duplex Ceramics

Abstract

Sintered carbides (WC-Co) are widely utilized across numerous industries, especially in the production of cutting tools and wear resistant components. However, the classification of both tungsten and cobalt as critical raw materials (CRMs) raises concerns about the future sustainability of their supply and environmental impact.

Our research focused on the development of ceramic matrix composites (CMCs) in which oxide (Al_2O_3, ZrO_2) and carbide (WC) phases form an interpenetrating "duplex microstructure", hereinafter referred to as DuplexCER. Such CMCs can be successfully produced using tungsten carbide recovered from WC-Co scrap. The elimination of Co, the partial replacement of WC with oxide phases and the use of recycled WC can reduce the need for CRM and have a positive impact on the environment.

The two grades of DuplexCER composites, containing oxides and tungsten carbide, were obtained by spark plasma sintering. The first grade contained 42 vol.% WC, while the second contained 26 vol.% WC. Both grades were produced in two variants. The “fresh” commercial WC powder was used for the first variant and the recycled one for the second variant. Composites had relative density of 98–100% and a Young's modulus of 400–470 GPa, depending of composition and sintering parameters. SEM observations showed good homogeneity of both composites. Best material had the hardness of 21.5 GPa and fracture toughness of 5.8 $MPa \cdot m^{1/2}$. The cutting performance of tools made from DuplexCER composites was significantly better than that of reference commercial advanced ceramics when turning Sleipner hardened steel. The lifetime of DuplexCER tools made from composites containing recycled tungsten carbide was comparable to that of tools containing commercial tungsten carbide. This work contributes to the implementation of the assumptions of a Circular Economy and European Green Deal. However, to achieve the desired outcome, it will be necessary to optimize and scale up the cemented carbide recycling technology.

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Structural and Property Evaluation Mg-Based Alloys Prepared by MA and SPS

Abstract

The growing interest in lightweight, biocompatible materials has intensified research on multicomponent magnesium-based alloys, particularly those modified with biologically and functionally active elements. In this study, a novel Mg-Zn-Ca-Au-Er alloy system was developed in powder form using mechanical alloying (MA), followed by consolidation through spark plasma sintering (SPS).

Powder mixtures were subjected to high-energy ball milling for 13, 20, and 30 h to investigate the effect of milling time on the homogeneity, morphology, and phase formation. The process led to the refinement of particles and gradual solid-state alloying. The powders were then sintered via SPS at optimized parameters to achieve near-full densification while preserving the fine-grained microstructure.

Material characterization was carried out. Scanning electron microscopy (SEM) revealed significant changes in particle morphology and sintered structure, with increasing milling time enhancing homogeneity. X-ray Diffraction (XRD) analysis confirmed the formation of new intermetallic phases and provided insight into the evolution of crystallite size and phase stability after sintering. Vickers hardness testing demonstrated an increasing trend with extended milling duration, indicating the strengthening effect of refined microstructure and solid-solution hardening. Porosity measurements showed that SPS enabled high densification efficiency.

The addition of elements such as Zn, Ca, Au, and Er aimed to synergistically enhance the alloy's mechanical properties and corrosion behavior, particularly in potential biomedical applications. Ag and Er are known for their antibacterial and osteogenic properties, while Ca and Zn support bone regeneration and bioresorption. The results highlight the potential of mechanical alloying combined with SPS for designing new multifunctional Mg-based materials with tailored properties for advanced structural or biomedical use.

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Effect of Mn, Nb, and Cu Additions on the Mechanical Properties of Zirconium Sintered via Spark Plasma Sintering (SPS)

Abstract

Zirconium is a material characterized by its high melting point and good mechanical strength, while also being easily machinable and weldable. Currently, zirconium is primarily manufactured using casting methods; however, spark plasma sintering (SPS) presents a promising alternative or complementary approach for producing zirconium in bulk form. SPS offers several advantages, including relatively low sintering temperatures and short processing times, which help preserve fine microstructures and improve mechanical performance. The technique enables precise control over grain size and densification, resulting in enhanced purity and homogeneity compared to conventional casting.

The aim of this research was to investigate the influence of 2.5 wt% additions of manganese (Mn), niobium (Nb), and copper (Cu) on selected mechanical properties of zirconium sinters. These alloying elements are expected to positively affect corrosion resistance, creep resistance, and mechanical performance. Due to the flammability of zirconium powders, mechanical alloying was not applied. Instead, the powders were mixed in water and isopropyl alcohol. The materials were sintered using the spark plasma sintering (SPS) method at temperatures ranging from 900°C to 1200°C, under a compaction pressure of 35 MPa, with a holding time of 1 min. The following properties were examined: density, Young's modulus, hardness, coefficient of friction, and specific wear rate.

The addition of niobium (Nb) increases the sintering temperature, while copper (Cu) reduces it. Samples doped with Cu and manganese (Mn) exhibit lower densities compared to pure zirconium. Moreover, Mn contributes to an increase in Young's modulus. Higher wear rates were observed in samples containing Mn and Cu across all applied loads.

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Sintering and Tribology in Nanoscale of ($Ti-TiB_2$)-Based Materials

Abstract

$Ti-TiB_2$ (50/50 by wt%) composites sintered by means of spark plasma sintering have brilliant tribology characteristic in sliding conditions, however, in oscillating conditions they seem to be susceptible to fatigue wear, cracking and pitting. This work shows results of wear resistance tests of $Ti-TiB_2$ composites with different contents of hexagonal boron nitride. Samples was obtained at heating rate 100°C/min, compaction pressure 50 MPa, dwell time 15 min, temperature 1350°C. Tribological tests was performed by means of nanotribometr NTR2, CSM Instruments, using 80 mN load at oscillating conditions. The aim was to determine the friction coefficient and the nature of wear while minimizing the effect of load on fatigue wear under oscillatory motion conditions. Composite materials content 0, 1, 5, and 15% of hexagonal boron nitride by weight. Materials with 5 and 15% of hexagonal boron nitride was sintered at different temperatures (1350–1550°C depending on the sample), because at lower temperatures high roughness was obtained, indicated by porosity. Increase in sintering temperature results in lower porosity and lower roughness possible to obtain. Microscopic observations revealed destruction mechanism of composite material worn against 100Cr6 steel ball at low load. The results obtained indicate low fatigue wear in the wear trace. The main mechanism is abrasion wear. The effect of the presence and concentration of hexagonal boron nitride is not significant at low load conditions. This is due to the structure of hexagonal boron nitride, which consists of easily shareable layers to which sufficient force must be applied to break the bonds. Force applied was probably too low to achieve the effect of the presence of those layers. Maximum Hertzian shear stress estimated for disc during tests was 230–280 MPa, Li Y. et al. [1] calculate shear stress between hBN layers of 300 MPa, which is slightly higher than stresses during tribotests.

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The Effect of SiC Alloying on the Selected Properties of Fe-Based Composites Produced by Spark Plasma Sintering

Abstract

The paper presents the influence of the chemical composition of the feedstock material and grain size on selected mechanical properties of the final product. The study was conducted using two types of composites: iron and Distaloy SA with characteristic SiC reinforcement. Composite mixtures were prepared from iron or Distaloy SA powders with SiC contents of 2.5, 5, 7.5, 10, and 12.5%. Samples were produced using a spark plasma sintering (SPS) 10-4 device after determining optimal sintering parameters. The study resulted in a material with a high density of up to 93–98% of pure iron and favorable mechanical properties.

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Multiscale Investigation of Nickel – Silicon Carbide Composites Sintered by SPS Method

Abstract

Silicon carbide (SiC) is characterised by good thermal and electrical conductivity, and high hardness and mechanical strength. Due to its unique properties SiC is applied as the reinforcement in metal matrix composites to improve their thermal, mechanical and wear properties. Ni-SiC composites are widely used in the automotive industry (pistons, brake discs), aviation and aerospace industries (engine components), as structural materials for the molten salt reactors, as well as in electronics (as the layered materials).

Comprehensive investigation of the influence of the main process parameters of spark plasma sintering on the mechanical and microstructural properties of nickel-silicon carbide composites at various scales. Microstructure analysis performed by scanning and transmission electron microscopy revealed a significant interfacial reaction between nickel and silicon carbide due to the decomposition of silicon carbide. The chemical interaction of the matrix and reinforcement results in the formation of a multicomponent interphase zone formed by silicides ($Ni_{31}Si_{12}$ or/and Ni_3Si) and graphite precipitates [1]. The influence of SPS on Ni-SiC composites was revealed by implementing a multiscale experimental tests at three different scales (macroscopic, microscopic, and nanoscale) [2]. The nanomechanical properties of composite components: metal matrix, ceramic reinforcement, and the interface have been evaluated by nanoindentation testing. Next, the deformation, strength, and fracture behaviour of the bonding zone of composite components was determined by a microcantilever bending test on the microscale. Finally, a uniaxial tensile test has been carried out to designate the effective mechanical performance of nickel-silicon carbide composites at a macroscopic level.

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Microstructural Characterization and Thermal Analysis of SPS Sintered Ce/Nd Doped Zirconia Ceramics for Nuclear Applications

Abstract

For many years, zirconium oxide (zirconia, ZrO_2) has been regarded as a promising material for inert matrix fuel (IMF) in nuclear applications due to its low neutron absorption cross section, high chemical and thermal stability, and elevated melting point. This study characterizes the thermal properties – specifically the thermal expansion coefficient, specific heat and thermal conductivity – of zirconia-based ceramics doped with neodymium and/or cerium oxide as minor actinide surrogates. Samples were synthesized using two techniques: a wet synthesis via the complex sol-gel process (CSGP) and a dry mechanical alloying (MA) method in a planetary ball mill under an argon atmosphere. Consolidation of the doped zirconia powders was performed by spark plasma sintering (SPS). Thermal analysis (TG-DSC, Dilatometry, Laser Flash), SEM imaging, and XRD were used to assess the microstructure, phase composition, and thermal behavior of the materials. Results reveal detailed insights into the influence of synthesis method and dopants on thermal characteristics, indicating that Ce and Nd incorporation as actinide surrogates decreases thermal conductivity and increases expansion. Such issues are essential for designing zirconia-based ceramics with controlled thermal properties for nuclear applications.

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Optimization of the HEBM Process to Provide the Optimal Particle Size in the MAX Phase as Feedstock for MXene Etching

Abstract

MXenes are a group of materials obtained by selectively etching A element from MAX phase structure. Preparation of a good-quality MAX phase is the first critical stage in MXene production, which determines chemical and phase impurities, flake size and functional properties. One of the most critical parameters of feedstock material is the particle size distribution (PSD). This poster presents the optimization process of production the optimal MAX phase material in terms of particle size distribution. The research was conducted on Ti_3AlC_2 MAX phase obtained by pre-alloying the precursor powders in high energy ball milling (HEBM) process and synthesis using FAST/SPS. To reduce the average particle size of synthesized MAX phase HEBM process was used again. The milling was conducted using Pulverisette 5 Classic Line (Fritsch GmbH, Idar-Oberstein, Germany) planetary ball mill. The optimization focused on following process parameters: milling tools material, milling media size, rotational speed and process control agent content. To reduce D_{50} parameter under 10 μm, addition of isopropyl alcohol as process control agent was necessary. Comparison between milling tools made of stainless steel and WC-Co showed no significant impact on particle size distribution. Reduction of milling ball size from Ø10 mm to Ø3 mm provides lower average particle size, however, it increases required time of milling and rotational speed. Processes conducted at 200 RPM and 400 RPM resulted in significant differences in impurities content. X-ray diffraction revealed large quantity of the milling tools material in the MAX phase after 400 RPM process. To prevent the material transfer lower rotational speed and WC-Co milling tools were chosen. In conclusion, the optimal particle size distribution can be obtained using WC-Co milling tools with 10:1:0.5 ball-powder-isopropanol weight ratio at 200 RPM.

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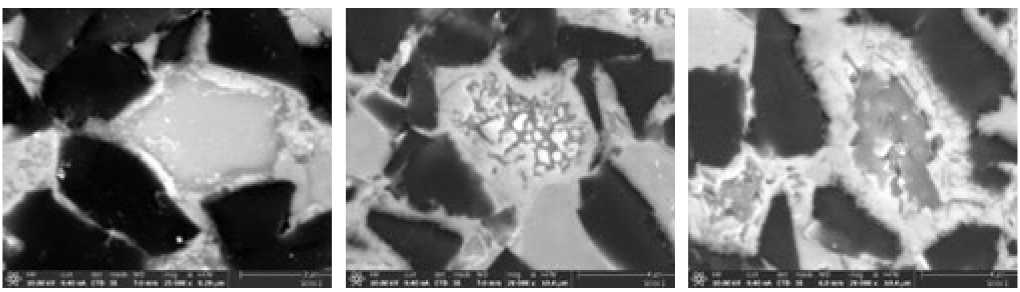
HPHT Sintering of cBN-TiN-Ti_3SiC_2 Composite: Phase Evolution and Mechanical Properties

Abstract

Composites with cBN can be generally classified as those with high (>70 vol.%) or low cubic boron nitride content. cBN composites with a relatively high content of ceramic bonding phase can be effectively used in advanced machining processes, including the turning of hardened steels, cast irons, and especially nickel-based superalloys. Titanium compounds, such as TiC or TiN, which have been widely used as ceramic binders in superhard composites for years, exhibit excellent high-temperature properties and good oxidation resistance, although their ductility remains low. Therefore, it is desirable to look for innovative bonding phases that combine both optimal chemical and mechanical properties.

This study aimed to manufacture the cBN-TiN-Ti_3SiC_2 composites (marked as BNT) via high pressure – high temperature sintering technique at a pressure of 7.7 GPa and temperature range of 1700–2100°C. These composites belong to the group of superhard materials, containing 65% vol. of cubic boron nitride (cBN) and a multi-component bonding phase in the form of titanium compounds. Due to the highly reactive Ti compounds forming the bonding phase, many reactions occurred during sintering, resulting in a final phase composition of the BNT composites that differed significantly from the initial composition.

The sinters exhibit a bi-modal structure, with large crystallites separated by fine crystalline material. As the sintering temperature increased, enhanced diffusion within the bonding phase was observed, promoting the development of a heterogeneous structure reminiscent of eutectic formations.



1700°C 1900°C 2100°C

Fig. 1. Microstructure of BNT composites sintered at 1700, 1900 and 2100°C

The EDS analysis confirmed the multiphase nature of the sinters. The intensive diffusion of the composite components, specifically silicon, which occurred as the sintering temperature increased, enables to conclude that under the applied sintering conditions an intensive phase transformation took place within the bonding phase. Given the known instability of the Ti_3SiC_2 phase under elevated pressure and temperature, its thermal decomposition was anticipated. This decomposition likely initiated in situ the formation and evolution of a complex, multi-component bonding phase.

Despite the relatively short sintering time of 1 min, the cBN-TiN-Ti_3SiC_2 system underwent a complex phase evolution within the applied temperature range. From a thermodynamic standpoint, the observed reactions are strongly non-equilibrium.

Acknowledgement

Presented results are derived from work carried out within the „Implementation doctorate V" programme.

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Spark Plasma Sintering of ZrB_2-1Cu-Steel Matrix Composites

Abstract

Modern sintering methods such as spark plasma sintering (FAST/SPS) are widely used for the production of metal matrix composites. Spark plasma sintering can be used to obtain fully dense material at lower sintering temperature and short times (a few minutes) to the conventional methods.

The main objective of the study was to determine the effect of the copper addition (1 wt%) and powder mixture preparation method on selected properties and microstructure of the steel matrix composites reinforced with different content of the ZrB_2 phase (5 and 10 wt%). Two methods were used to prepare composite mixtures: mixing in a Turbula and milling in a planetary mill. For comparison purposes, AISI 316L steel powder mixtures with 1% copper were produced. The sintering process was carried out using the spark plasma sintering at a temperature of 1100°C, a compaction pressure of 35 MPa and a time of 5 mins in an argon atmosphere.

The density, Young's modulus, hardness, corrosion and tribological properties of the sintered compacts were investigated. The microstructure and phase composition were characterized using scanning electron microscopy (SEM) and X-ray diffraction. A very high degree of densification was achieved for the sintered materials. The test results showed that adding 1 wt% copper has a positive effect on the density, Young's modulus, hardness, abrasion resistance and corrosion resistance of the sintered composites. Aditionally, higher content of ZrB_2 in the steel matrix is responsible for the improvement in Young's modulus, microhardness and abrasive wear resistance with the reduction of the corrosion resistance of sintered composites. At the same time, the method of powder preparation significantly affected the microstructure and properties of the composites. An inhomogeneous distribution of ZrB_2 was observed in the steel matrix when mixing in Turbula was used. Milling of powders in a planetary mill contributed to the fragmentation and homogeneous dispersion of ZrB_2 particles in the matrix.

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Comparison of Pressure Sintering Methods for ZrB_2-HfB_2 Boride-Based Composites

Abstract

Sintering of ZrB_2-HfB_2-based ceramics containing either 20 vol.% SiC, B_4C, WC, $MoSi_2$ or $CrSi_2$ was conducted using different routes: hot pressing (HP), spark plasma sintering (SPS) and high pressure high temperature (HPHT). Among the three employed techniques, the HP sintering process resulted in the best for ZrB_2-HfB_2-based composites in terms of density and microstructure homogeneity.

The HP sintering was a relatively long-lasting process, 30 mins dwell time was much longer than that applied in SPS (5 mins) or HPHT (1 min). SPS and HPHT promoted the retention of the original batched.

The SPS technique intensified the boride phases homogenization and the $(Zr,Hf)B_2$ solid solution formation. Parallel, such an intensive process trapped water remains in the microstructure and thus oxygen reacted with borides, producing zirconium and hafnium oxides in quite significant amounts.

The HPHT technique led to the less homogenous materials. A short time of sintering did not allow for the solid solution formation, so the final materials were composed of the initial ZrB_2 and HfB_2 phases as well as unreduced oxides and introduced additives.

The performed investigations undermine the importance of the intensive SPS and HPHT techniques for sintering relatively not very fine (micrometric) powders. Furthermore, the tests showed that to remove oxides passivating the boride particles carbides were the most effective. The undoubted advantage of using silicides was the significant reduction of the sintering temperature by 500–600°C. However, when silicides were added, the obtained composites had a complex phase composition, which can beneficially affect the mechanical, thermal and chemical properties of ultra-high-temperature ceramics.

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Reactive Spark Plasma Sintering of HfB_2: Effects of Precursor Morphology and Sintering Parameters on Phase Formation and Microstructure

Abstract

Hafnium diboride (HfB_2) is recognized as one of the most promising ultra-high temperature ceramics (UHTCs) because of its unique combination of outstanding physicochemical properties. These include an exceptionally high melting point (3250°C), high values of Young's modulus (550 GPa) and Vickers hardness (20 GPa), along with remarkable thermal and electrical conductivity. Furthermore, the material's excellent chemical and high-temperature oxidation resistance positions HfB_2 as a promising solution for critical components in extreme environments across aerospace, defense, nuclear, and energy industries. The fabrication of dense HfB_2 still faces considerable challenges. Conventional multi-step processing routes often involve complex synthesis, lengthy milling and energy-intensive sintering at very high temperatures, which hinder cost-effectiveness and scalability.

In this study, reactive spark plasma sintering (RSPS) was applied as an alternative route to synthesize HfB_2 directly from elemental precursors, thereby simplifying processing and enhancing densification. Figure 1(a) presents an SEM micrograph of a specimen after reactive sintering, revealing spherical hafnium grains partially transformed into a diffusion-formed HfB_2 phase through the reaction of Hf with B. The work particularly examines the role of sintering temperature and precursor morphology to boride phase formation, densification, and microstructural development. Figure 1(b) shows the fine-grained microstructure of HfB_2 obtained under the optimal RSPS conditions. The findings underline the potential of RSPS as an innovative, cost-effective method for producing dense HfB_2 ceramics, relevant as targets for protective coating and components exposed to severe operating environments.

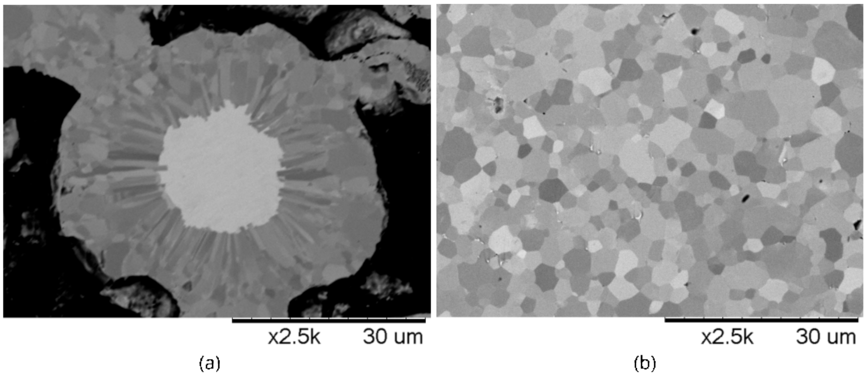

Fig. 1. SEM micrographs of (a) specimen after RSPS with spherical hafnium grains partially transformed into a diffusion-formed HfB_2 phase resulting from the reaction between Hf and B; (b) microstructure of HfB_2 obtained under optimal RSPS processing conditions

Acknowledgement

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Deposition of Aluminium-Doped LiPON Films by Microwave-Assisted Reactive Radiofrequency Magnetron Sputtering Using Spark Plasma Sintered Customized Targets

Abstract

Thin-film batteries are a key enabling technology for next-generation mobile applications, including wearable and portable electronics, medical devices, and sensors. The further development of these technologies is strongly linked to advances in flexible and stretchable power sources, which are of strategic importance for the European market. A critical component of thin-film batteries is the solid-state electrolyte, such as LiPON (lithium phosphorus oxynitride), which offers enhanced safety and enables operation at elevated temperatures.

On challenge in LiPON fabrication is the enhancement of the deposition of high-quality films from LiPO (Li_3PO_4, lithium phosphate) targets. In particular the deposition speed must be increased to reach industrial viable levels. In the DEPOION project, conducted in collaboration with Łukasiewicz Research Network – Poznań Institute of Technology, BFH (Switzerland), and The Batteries Sp. z o.o. (Poland), the challenge is addressed.

Within this part of the DEPOION project, we address the deposition of aluminium doped LiPON films using microwave assisted radiofrequency magnetron co-sputtering. The deposition reactor is a modified hexagonal HEX L chamber, (Korvus Technologies, UK), which can be equipped with various plasma diagnosis tool. The reactor is equipped with 3 electron cyclotron resonance elementary micro wave sources (AuraWave, Sairem, FR). The additional large volume microwave plasma, serves an efficient means to activate molecular dinitrogen at the vicinity of the substrate, allowing to reduce the nitrogen content in the reactor chamber. The objective is to increase the sputtering efficiency.

The thin films thus produced, including Al-LiPON content gradient films, are analysed using various analytical techniques including, XRD, SEM and ellipsometry.

First promising results recently obtained with be presented and discussed.

Acknowledgement

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Self-Propagating High-Temperature Synthesis (SHS) Initiated by Local Ignition for the Synthesis of Mg_2Si-Based Thermoelectric Materials

Abstract

Magnesium silicide was obtained from elemental powders using high temperature self-propagating synthesis. The synthesis were carried out in the reactor supported by induction heater. The pre-compacted green body pellets, produced from a homogeneous mixture of magnesium and silicon in stoichiometric quantities corresponding to Mg_2Si compound were placed into a graphite die. The sample was degassed under vacuum conditions at 200°C. The reaction was initiated by means of a resistive heating element located in the vicinity of the base of the cylindrical sample. The specially designed laboratory set-up allowed the preservation of adiabatic conditions throughout the exothermic reaction, which proceeded rapidly in a oxygen-free environment within the temperature range of 500–900°C, resulting in the formation of a powdered product. Characterization of the samples comprised phase analysis by X-ray diffraction (XRD), analysis of morphology and chemical composition by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS). Multiphysics model of SHS has been formulated and compared with experimental data. Simulation results show a good agreement with experimental thermocouple readings. Some deviation of experiment and model might be a consequence of the microstructure change during SHS process illustrated by X-ray computed tomography results. The properties of magnesium silicide obtained by SHS were discussed with the refence to the synthesis parameters and similar materials produced by the other synthesis/processing routes.

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Fabrication of Thermoelectric Modules Using Materials Produced by the SPS Technique

Abstract

Thermoelectric phenomena is widely used for both cooling and energy harvesting purposes. The key component of module is semiconductor material not only efficient in energy conversion, but also mechanically durable and chemically stable. Spark plasma sintering (SPS) has emerged as an effective technique for the fabrication of advanced semiconductor materials with tailored thermoelectric and mechanical properties. In this study, we present the small-scale production of thermoelectric modules for energy generation created under COTEG project. Using SPS technique allowed to achieve polycrystalline, fine grain material with excellent mechanical and thermoelectric properties, which allowed 50×50 mm^2 thermoelectric module fabrication. Fifty thermoelectric modules were prepared as part of the project. This work demonstrated the effectiveness of using SPS-produced materials in the small-scale production of thermoelectric modules.

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Electrospark Deposition by Using Nanoparticle-Containing Paste

Abstract

In the modern era of rapid scientific and technological development, there is an increasing demand for the creation of new materials with enhanced performance characteristics. Metal components of machines and equipment are subject to intensive wear, which is one of the main causes of their loss of functionality. Friction and wear lead to significant energy and material losses, making the improvement of wear resistance of working surfaces a highly relevant problem. One promising approach to address this challenge is the use of nanomaterials, which, due to their unique properties, allow for significant enhancement of the physicomechanical characteristics of coatings. Nanostructured layers provide high hardness, corrosion resistance, and extended service life of components. Traditional coating methods, such as CVD, PVD, or laser cladding, require complex and expensive equipment and have certain limitations. Therefore, methods that combine ease of implementation, energy efficiency, and environmental friendliness are attracting increasing attention. One such method is electrospark deposition (ESD), which enables the formation of coatings with high adhesion and the possibility of localized treatment of critical components. The use of nanoparticle-containing pastes in the ESD process opens new opportunities for obtaining nanostructured layers with improved properties. Investigating the structure formation processes of such coatings is a relevant task in contemporary materials science.

For the experiments, Armco iron was used as the substrate, while the alloying electrode was molybdenum obtained by the SPS technique from molybdenum powder. Coatings were formed on the "Elitron-22A" installation under various process conditions. The ESD process was conducted in the sequence: application of the carbon nanoparticle-containing paste to the treated surface followed by ESD using the molybdenum electrode. Multi-walled and single-walled carbon nanotubes dispersed in a polymer matrix via ultrasonic treatment were used as nanoparticles.

The results demonstrated that the use of nanoparticle-containing pastes during ESD promotes the formation of uniform and continuous coatings compared to those without nanoparticles. Optical and electron microscopy confirmed a uniform distribution of single-walled carbon nanotubes at a 0.01% concentration, while increasing the nanotube content (0.2–0.6%) led to agglomeration and reduced coating continuity. Microstructural analysis revealed three distinct zones in the coatings: an upper “white” layer, a diffusion zone, and the substrate. The addition

of nanotubes increased the thickness and continuity of the layer as well as microhardness. Specifically, the incorporation of 0.01% single-walled nanotubes increased hardness threefold (from 446 to 1438 HV). Further increasing the nanotube concentration did not yield additional improvements. Microstructural studies showed grain refinement and the formation of a homogeneous structure in the presence of nanotubes. Elemental analysis indicated that the molybdenum diffusion zone was 12–14 μm, while the carbon content in the coating remained essentially unchanged.

Thus, the use of nanoparticle-containing paste during ESD enables the formation of homogeneous and continuous coatings. The optimal effect is achieved with 0.01% single-walled carbon nanotubes, resulting in uniform filler distribution, increased thickness, and enhanced hardness (up to 1438 HV). Increasing the nanotube concentration beyond the optimal value leads to agglomeration and decreased coating quality. The addition of nanotubes contributes to grain refinement and the formation of a nanostructured layer, which can improve the operational properties of the surface.

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