CONFERENCE ON FAST/SPS
From Research to Industry

BOOK OF ABSTRACTS

WARSAW, 16–18.10.2023

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Łukasiewicz Research Network – Poznań Institute of Technology
6 Ewarysta Estkowskiego St., 61-755 Poznan, Poland
Tel. +48 61 850 49 10
E-mail: office@pit.lukasiewicz.gov.pl
https://pit.lukasiewicz.gov.pl/

Content editing
Dariusz Garbiec, Marcin Chmielewski

Technical editing
Alicja Januszkiewicz, Małgorzata Słodzinka, Marcin Tomkowiak

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Tel. +48 61 639 50 60
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https://wieland.com.pl/

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From Research to Industry

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Łukasiewicz Research Network – Institute of Microelectronics and Photonics
32/46 Lotników Ave., 02-668 Warsaw, Poland
Tel. +48 22 548 78 16
E-mail: sekretariat@imif.lukasiewicz.gov.pl
https://imif.lukasiewicz.gov.pl/

CO-ORGANIZERS

Łukasiewicz Research Network – Poznań Institute of Technology
6 Ewarysta Estkowskiego St., 61-755 Poznan, Poland
Tel. +48 61 850 49 10
E-mail: office@pit.lukasiewicz.gov.pl
https://pit.lukasiewicz.gov.pl/

Łukasiewicz Research Network – Krakow Institute of Technology
73 Zakopiańska St., 30-418 Krakow, Poland
Tel. +48 12 261 83 24
E-mail: sekretariat@kit.lukasiewicz.gov.pl
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KEY SPONSORS

Dr. Fritsch Sondermaschinen GmbH
8 Dieselstraße St., 70736 Fellbach, Germany
Tel. +49 711 51832 0
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GeniCore Sp. z o.o.
133 Wólczyńska St., 01-919 Warsaw, Poland
Tel. +48 22 857 04 77
E-mail: genicore@genicore.pl
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SPONSORS

FCT Systeme GmbH
Rauenstein, Gewerbepark 16,
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AMAZEMET Sp. z o.o.
27 Jana Pawła II Ave, 00-867 Warsaw, Poland
Tel. +48 573 481 303
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27/27 Walerego Sławka St., 30-633 Krakow, Poland
E-mail: info@mdsd.pl
https://www.mdsd.pl/

Norimat
51 Rue de l'Innovation, 31670 Labège, France
Tel. +33 6 49 77 76 36
E-mail: contact@norimat.com
https://www.norimat.com/

NETZSCH Instrumenty Sp. z.o.o.
9 Halicka St., 31-036 Krakow, Poland
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70 Years
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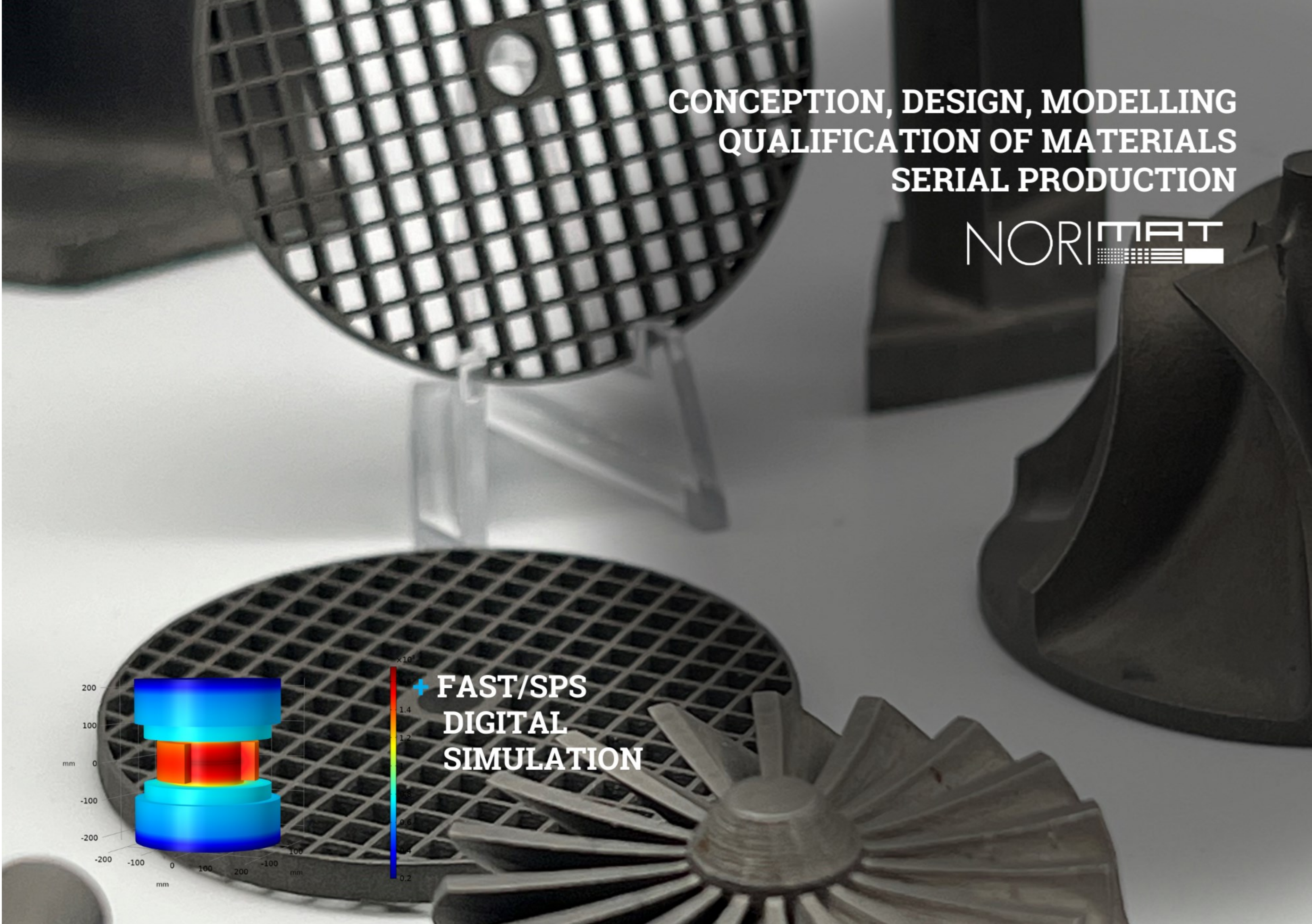
70 Years FAST/SPS

DR. FRITSCH

SPS is GREEN


CONCEPTION, DESIGN, MODELLING
QUALIFICATION OF MATERIALS
SERIAL PRODUCTION
NORIMAT
+ FAST/SPS
DIGITAL
SIMULATION

October 16–18, 2023
Monday, 09.00–23.00
Tuesday, 09.00–21.00
Wednesday, 09.00–15.30

Airport Hotel Okęcie
24 Komitetu Obrony Robotników St.,
02-148 Warsaw, Poland

CONFERENCE PROGRAMME

DAY 1
OCTOBER 16, 2023 (MONDAY)

09.00–09.30 **Participants Registration**

09.30–09.45 **Opening Ceremony**

09.45–10.30 **Keynote Lecture I**
Session Chairman: Dariusz Garbiec
Field Assisted Sintering Technologies for Greener and Better Materials
Olivier Guillon, Institute of Energy and Climate Research: Materials Synthesis and Processing Forschungszentrum (IEK-1), Jülich, Germany

10.30–11.15 **Keynote Lecture II**
Session Chairman: Marcin Chmielewski
Strategies to Overcome the Main Challenges of Spark Plasma Sintering Process and to Design Materials with Tailored Properties
Claude Estournes, Université de Toulouse, Toulouse, France

11.15–11.30 **Coffee Break**

11.30–12.30 **Session I**
Session Chairman: Olivier Guillon

11.30–11.50 **Metallic Tool in FAST/SPS**
Alexander Laptev, Łukasiewicz Research Network – Poznań Institute of Technology, Poznań, Poland

11.50–12.10 **A CoNi-based High Entropy Superalloy Processed by Spark Plasma Sintering**
Ahad Mohammadzadeh, IMDEA Materials Institute, Getafe, Madrid, Spain

12.10–12.30 **Remarkable Irradiation Resistance of ODS-HEA Alloys Sintered Using the Spark Plasma Sintering Technique**
Łukasz Kurpaska, National Centre for Nuclear Research, Otwock, Poland

12.30–12.45 **Coffee Break**

12.45–15.00 **Session II**
Session Chairman: Alexander Laptev

12.45–13.15 **70 years of FAST/SPS Sintering, 70 Years Dr. Fritsch**
Jens Huber, Dr. Fritsch, Fellbach, Germany

13.15–13.45 **U-FAST^{COMPACT} – the First Step Into Sintering Adventure**
Katarzyna Jach, GeniCore Sp. z o.o., Warsaw, Poland

13.45–14.00 **Latest Developments and Trends for the Industrial Application of FAST/SPS Technique**
Benjamin Luthardt, FCT Systeme GmbH, Frankenblick, Germany

14.00–14.15 **Ultrasonic Atomization, a Novel Technology for Powder Production with Tailored Chemical Compositions**
Bartosz Morończyk, AMAZEMET Sp. z o.o., Warsaw, Poland

14.15–14.30 **Resonant Alternating Current Sintering (RACS) Technology**
Andrzej Koleżyński, Materials Design, Systems & Devices, Krakow, Poland

14.30–14.45 **The Imminent Surge of FAST/SPS Technology: Paving the Way to Mainstream Production in the Next Decade**
Romain Epherre, Norimat, Labège, France

14.45–15.00 **Advanced Materials Characterisation Techniques – Netzsch Portfolio**
Krzysztof Hodor, NETZSCH Instrumenty Sp. z.o.o., Krakow, Poland

15.00–16.30 **Lunch Time**

16.30–18.00 **Poster Session**

19.00–23.00 **Conference Dinner**

DAY 2
OCTOBER 17, 2023 (TUESDAY)

09.00–16.00 **Industrial Session**

09.00–09.05 **Opening of the 2nd day of the Conference**

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Session Chairman: Piotr Klimczyk
The Use of SPS for Sustainable Development of High Entropy Alloys Using Pre-alloyed Commodity Powders
José Manuel Torralba, IMDEA Materials Institute, Getafe, Madrid, Spain

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10.20–12.00 **Session III**
Session Chairman: José Manuel Torralba

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Mickaël Coeffe Desvaux, IRCER – University of Limoges, Limoges, France

10.40–11.00 **High Performance Duplex Ceramics For Efficient Machining Of Nickel Superalloys**
Piotr Klimczyk, Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland

11.00–11.20 **MAX-based Composites Made by SPS: Structure Formation and Unique Properties**
Volf Leshchynsky, Łukasiewicz Research Network – Poznań Institute of Technology, Poznań, Poland

11.20–11.40 **Micromechanical Testing of Magnetron Sputtered W-Ti-B Coatings from SPS Targets**
Tomasz Mościcki, Institute of Fundamental Technological Research PAS, Warsaw, Poland

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Igor Veremchuk, Helmholtz-Zentrum Dresden-Rossendorf, Dresden, Germany

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Beata Leszczyńska-Madej, AGH University of Krakow, Krakow, Poland

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Mirosław J. Kruszewski, Warsaw University of Technology, Warsaw, Poland

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Paweł Nieroda, AGH University of Krakow, Krakow, Poland

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Juliusz Leszczyński, AGH University of Krakow, Krakow, Poland

13.50–14.10 **Synthesis and Consolidation of Bi_2Te_3-based Thermoelectric Materials with the Use of SPS/FAST Technique**
Bartosz Bucholc, Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

14.10–15.30 **Lunch Time**

16.00–21.00 **Guided City Tour**

DAY 3
OCTOBER 18, 2023 (WEDNESDAY)

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Session Chairman: Rafał Zybała
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Martin Jackson, The University of Sheffield, Sheffield, UK

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10.20–12.00 **Session V**
Session Chairman: Martin Jackson

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Abdullah Riaz, University of Rostock, Rostock, Germany

10.40–11.00 **Quantitative Analysis of Influence of SPS Process Parameters on the Porous Materials Structure Using Combined EBSD and Computer Assisted Software**
Szymon Nosewicz, Institute of Fundamental Technological Research PAS, Warsaw, Poland

11.00–11.20 **The Investigation of $Al/Mg_2B_2O_5w$ Composites Sintered by SPS and HPHT Methods: Chemical Interaction, Microstructure and Mechanical Properties**
Yuliia Rumiantseva, Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland

11.20–11.40 **In-Depth Analysis of the Influence of Bio-Silica Filler on the Properties of Mg Matrix Composites**
Anna Dobkowska, Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

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Fatima Nisar, Institute of Fundamental Technological Research PAS, Warsaw, Poland

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12.30–14.10 **Session VI**
Session Chairman: Abdullah Riaz

12.30–12.50 **Sputtering Targets Obtained by Induction Hot Pressing (IHP) and Spark Plasma Sintering (SPS) Methods**
Krzysztof Mars, AGH University of Krakow, Krakow, Poland

12.50–13.10 **The Effect of Process Optimization on Microstructure Evolution and Mechanical Properties of Low BPR Mechanically Alloyed CoCrFeNi High Entropy Alloy**
Artur Olejarz, National Centre for Nuclear Research, Otwock, Poland

13.10–13.30 **β-Type Titanium and Zirconium-Based Alloys Produced via Sustainable Spark Plasma Sintering of Nanocrystalline Powders**
Mateusz Marczewski, Łukasiewicz Research Network – Poznań Institute of Technology, Poznań, Poland

13.30–13.50 **Detailed Analysis of Microstrostructure and Properties of Copper Sintered by SPS Method**
Kamil Kaszyca, Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

13.50–14.10 **Characterization Of FeCrAl-Y_2O_3 ODS Alloys With the Additions of Ti and V Consolidated By SPS**
Tomasz Stasiak, National Centre for Nuclear Research, Otwock, Poland

14.10–14.30 **HEBM-FAST/SPS as a Way of Obtaining Composite Tool Material Made from WC-Ti & WC-Ti6Al4V Powder Mixtures**
Wiktoria Krzyżaniak, Łukasiewicz Research Network – Poznań Institute of Technology, Poznań, Poland

14.30–15.30 **LUNCH TIME**

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I. ORAL PRESENTATIONS

Olivier Guillon[1], Chen Cao[1] Apurv Dash[1,2], Robert Mücke[1]
[1]Institute of Energy and Climate Research (IEK-1): Materials Synthesis and Processing,
Forschungszentrum Jülich, Jülich, Germany
[2]DTU Energy, Copenhagen, Denmark

Field Assisted Sintering Technologies for Greener and Better Materials

Abstract

Processing of ceramics – especially sintering – requires large amount of energy and time. The use of renewable electricity may profoundly change the ceramic industry towards more efficiency and sustainability. The fundamental understanding of field-assisted sintering of oxide ceramics is thus more than ever key to develop new sintering techniques and devices.

A systematic study of the electric field effects requires well-controlled experiments. In particular, by using moderate fields and monitoring the sample temperature, it is possible to separate thermal from athermal effects. The combined influence of mechanical stress also needs to be considered. We therefore rely on a unique, instrumented field-assisted sinter-forge equipped with laser scanners. With this approach, it is possible to measure in-situ the sintering behavior and viscous parameters (sintering stress, different viscosities, viscous Poisson's ratio) as function of material, field conditions (strength, frequency) and density. In addition, the microstructure and especially the evolution of grain size is quantified. Finally, parallel with electroplastic deformation of ceramics, also controlled by diffusional processes, is made.

Acknowledgement

Funding form the DFG Priority Program SPP 1959 "Fields Matter" is greatly acknowledged.

Claude Estournes
University of Toulouse, Toulouse, France

Strategies to Overcome the Main Challenges of Spark Plasma Sintering Process and to Design Materials with Tailored Properties

Abstract

The spark plasma sintering process (SPS) permits the densification of a wide variety of materials (metals, alloys, ceramic, polymer and composites) at lower temperature compare to conventional processes. This breakthrough sintering technique is able to create highly dense materials with micro-structural control and a very short processing time. Although it has experienced a very strong growth in the last three decades in terms of publications and patents, its industrialization is still in its infancy. This is mainly due to the fact that pieces of simple geometries have been densified so far, and that this technology is considered to suffer from a lack of reproducibility and productivity.

We will try to show in this presentation, how to overcome the main challenges of this technology (reproducibility, multiple-sampling and 3D complex shape parts) and how it and its variants (High Pressure: HP-SPS, Cold-SPS and Flash-SPS) can be used to obtained nanostructured materials with tailored properties.

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

Metallic Tool in FAST/SPS

Abstract

Tool is the central part of any FAST/SPS setup. The FAST/SPS tool must be strong enough to withstand the applied pressure at sintering temperature. The typical material of the FAST/SPS tool is graphite. However, the conventional grades of graphite have a low compressive strength, prone to brittle fracture and are low wear resistive. These grades of graphite enable FAST/SPS with 30–50 MPa at a temperature of up to 2500°C. The special graphite grades allow a pressure of up to 200–230 MPa. However, these grades are extremely expensive. An alternative tool material is a hot work tool steel (e.g., AISI H13). Steel tools are applicable up to 550–600°C with a pressure of 400–1000 MPa depending on temperature. Another alternative to graphite is nickel-based alloys like Inconel 718 or MAR-M-247 which are applicable up to 700–750°C with a pressure of up to 600–700 MPa. The next possibility to replace graphite in the FAST/SPS tooling is the application of Mo-based TZM (Ti-Mo-Zr) alloy. The manufacturer of this alloy Plansee SE recommends its application between 700°C and 1400°C. However, this recommendation is not proven in the FAST/SPS technique so far. We believe that realistic is the application of TZM at a temperature of up to 1000–1050°C with a pressure of 300 MPa. The further increase in the operation temperature by 100–150°C can be potentially achieved by application of MHC (Mo-Hf-C) alloy with the increased strength and higher recrystallization temperature. However, the literature does not provide the information on application of MHC alloy in FAST/SPS practice. The important drawback of metallic tool is the low electrical resistivity leading to overheating of the power supply chain. The shortcoming of hot work tool steels and especially of superalloys is the low thermal diffusivity resulting in a prolonged cooling time. Also, the metallic tools need more energy for heating than a graphite tool with the same geometry. Finally, a special investigation requires the primary creep of metallic tool working at FAST/SPS conditions. The paper provides some examples of application of metallic tool in FAST/SPS.

Ahad Mohammadzadeh[1, 2], Alessandro De Nardi[1, 3], Dariusz Garbiec[4], Jose Manuel Torralba[1, 5]
[1]Imdea Materials Institute, Madrid, Spain
[2]University of Maragheh, Maragheh, Iran
[3]Politecnico di Torino, Torino, Italy
[4]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland
[5]Universidad Carlos III de Madrid, Leganes, Spain

A CoNi-Based High Entropy Superalloy Processed by Spark Plasma Sintering

Abstract

In this study, a novel single face-centered cubic (FCC) phase CoNi-based high entropy superalloy (HESA) was successfully produced using fully prealloyed gas atomized powders and spark plasma sintering (SPS). The alloy design process incorporated high entropy alloy (HEA) principles and thermodynamic calculations (CALPHAD) to predict phase transformations at various production stages, including powder production, SPS processing, and heat treatment of the HESAs. The resulting alloys exhibited a relative density of 99.9%, ultimate tensile strength (UTS) of 800 MPa, and a hardness of approximately 400 HV_1. Comprehensive microstructural and fractographic analyses demonstrated the absence of detrimental secondary and/or topologically closed-packed (TCP) phases within the developed alloy. Thermal analysis results revealed that an increase in the entropy of mixing resulted in a correspondingly high volume fraction of the gamma prime (γ') phase (70%) and an elevated solvus temperature of 1165°C. These findings highlight the significant impact of HEA principles in the design of CoNi-based superalloys, accelerating the development of this alloy family for advanced manufacturing technologies.

Acknowledgement

This investigation was supported by the European Union Horizon 2020 research and innovation program (Marie Sklodowska-Curie Individual Fellowships, Grant Agreement 101028155). A. De Nardi would like to acknowledge the grant "Research Initiation Fellowship 2023" (Excelencia María de Maeztu).

Łukasz Kurpaska[1)], Sri T. Nori[1)], Per Erik Vullum[2)], Damian Kalita[1)], Witold Chrominski[1)], Yanwen Zhang[3, 4)]

[1)]National Centre for Nuclear Research, Otwock, Poland
[2)]Norwegian University of Science and Technology, Trondheim, Norway
[3)]Idaho National Laboratory, Idaho Falls, USA
[4)]University of Tennessee, Knoxville, USA

Remarkable Irradiation Resistance of ODS-HEA Alloys Sintered Using the Spark Plasma Sintering Technique

Abstract

In the past few years, oxide dispersion strengthened (ODS)-concentrated solid solution alloys (CSAs) emerged as potential structural materials for Gen. IV nuclear reactors. They can possess the best radiation resistance even at high temperatures (>400℃), proving that these materials can be considered one of the strongest candidates for Generation IV nuclear reactor technologies. So far, these remarkable properties have been demonstrated in limited studies [1, 2]. It is known that one of the possible adverse effects of high-temperature irradiation is chemical redistribution on grain boundaries and possibly inside of the strengthening particles, which can alter the stability of phases and local properties of the material (predominantly mechanical properties). Hence, this work examines the high-temperature radiation-induced chemical redistribution phenomenon in some NiCo-based ODS-CSAs, an utterly new family of materials successfully sintered using the Sparc Plasma Sintering technique.

The ODS-NiCoFe, ODS-NiCoFeCr, and ODS-NiCoCr were studied following Ni^{2+} irradiation at 580℃ via scanning transmission electron microscopy (STEM)-electron energy loss spectroscopy and high-resolution TEM to reveal the possible chemical redistribution in the irradiated regions and then compare with the pristine region. The interfaces between the matrix and nanooxide particles and the grain boundaries showed significant chemical redistribution during irradiation. These regions are considered to act as defect sinks. We showed that chemical segregation indicating Cr and Fe depletion and Ni and Co enrichment occurs near the grain boundaries, specifically in Cr-containing ODS-CSAs. We also conducted a nanoindentation campaign to collate structural properties, local chemical redistribution effect, and mechanical properties of ion irradiated samples. All such key findings culminated in evaluating the high-temperature radiation resistance of the alloy family ODS-CSAs.

Literature:

[1] Y. Guo, M. Li, C. Chen, P. Li, W. Li, Q. Ji, Y. Zhang, and Y. Chang, Intermetallics, 117, 106674 (2020).

[2] C. Lu, M. Li, P. Xiu, X. Wang, G. Velisa, L. Jiang, K. L. More, J. D. Poplawsky, Y. Chang, Y. Zhang, and L. Wang, J. Nucl. Mater., 557, 153316 (2021).

Acknowledgement

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Jens Huber
Dr. Fritsch Sondermaschinen GmbH, Fellbach, Germany

70 Years of FAST/SPS Sintering, 70 Years Dr. Fritsch

Abstract

Even among scientists not many people know that industrial FAST/SPS sintering dates back far longer than they assume. Many people still think that FAST/SPS has been invented somewhen in the 1980ies in Japan.

Therefore, it is time to have a look back: Although the first patents and tests date back to at least 1906, it took long time until the first industrial mass production came up. It took until the early 1950ies, when an entrepreneur from Germany has been approached by a manufacturer of diamond tools. He explained to him that the diamond tools used at that time could perform much better if not so many diamonds would carbonize due to the long cycle times of the sinter processes used at that time. The entrepreneur was not hesitating and came up with the first industrial FAST/SPS sinter press in 1953 developed specifically for industrial needs. His name was Dr. Fritsch. As it seems, the machine concept was very convincing and the demand grow every year. Today, FAST/SPS is the main production route for diamond tools and Dr. Fritsch is still the largest manufacturer in that industry. Since then, many other industries have discovered the advantages of FAST/SPS sintering. For example for the generation of hydrogen, thermoelectric materials, friction materials such as motorcylce brake pads or brakes for high speed trains such as the ICE, TGV and Shinkansen. For all these applications, the improved performance of the products is what convinces the customers of Dr. Fritsch to use FAST/SPS.

Katarzyna Jach, Marcin Rosiński
GeniCore Sp. z o.o., Warsaw, Poland

U-FAST^{COMPACT} – the First Step into Sintering Adventure

Abstract

The key aspect of FAST technology is the ability to perform the sintering process at a much more favourable conditions than other available methods. GeniCore U-FAST technology, thanks to its advanced heating system based on a unique pulse shape allows achieving better results compared to other available SPS devices. The advantages include lower sintering temperature with positive effect on limiting grain growth, homogeneity of the resulting material and also high energy efficiency.

U-FAST^{COMPACT} have all the same functionality as the GC model, but in a compact size. This device is suitable for R&D purposes and is intended for scientific institutes and universities. It is designed to be used on a smaller scale for research and development activities.

The aim of the presentation is to show the possibilities of material development using U-FAST^{COMPACT}, from creative and unique ideas to design of the properties and as a result obtaining a product that is able to make consumers satisfied.

Acknowledgement

The project is co-financed by the National Center for Research and Development as part of the Path for Mazovia program “Development of a production line of world-class innovative SPS/FAST devices using hybrid heating of sintered powders”.

Benjamin Luthardt, Jürgen Hennicke
FCT Systeme GmbH, Frankenblick, Germany

Latest Developments and Trends for the Industrial Application of FAST/SPS Technique

Abstract

In the past two decades, the field assisted sintering technique (FAST), also known as spark plasma sintering (SPS), has been successfully established for rapid sintering applications. With the focus on the implementation of this technique into industrial applications, the achieved scientific results could have been transferred in practical. Together with a multiplicity of industrial partners and based on its own R&D activity in its in-house technology center, FCT Systeme GmbH could implement these FAST/SPS techniques successfully in customized plant concepts.

With more than 40 years of experience and its wide portfolio of different sintering technologies, FCT is covering the complete range from laboratory plants up to highly specialized industrial furnaces.

The company has certainly committed to the development of customized and efficient plants which achieve a maximum of performance and service life. A team of highly skilled engineers and scientists is working together in close cooperation with global customers to develop solutions with the purpose of sustainable process optimization.

Innovative hybrid concepts are the latest trend in sintering technology: in the FAST/Hybrid trend-setter, the classic hot-pressing method has been complemented with an additional direct heating of the compacted powder part using impulse-direct current (FAST/SPS). Compared with conventional methods, this hybrid combination goes beyond and enables much more new opportunities.

FCT's new hybrid plants allow previously unrealisable opportunities for the development of innovative materials used in energy, electric mobility, semiconductor and/or aerospace industry as well as other areas relevant for the future (e.g. energy-efficient production).

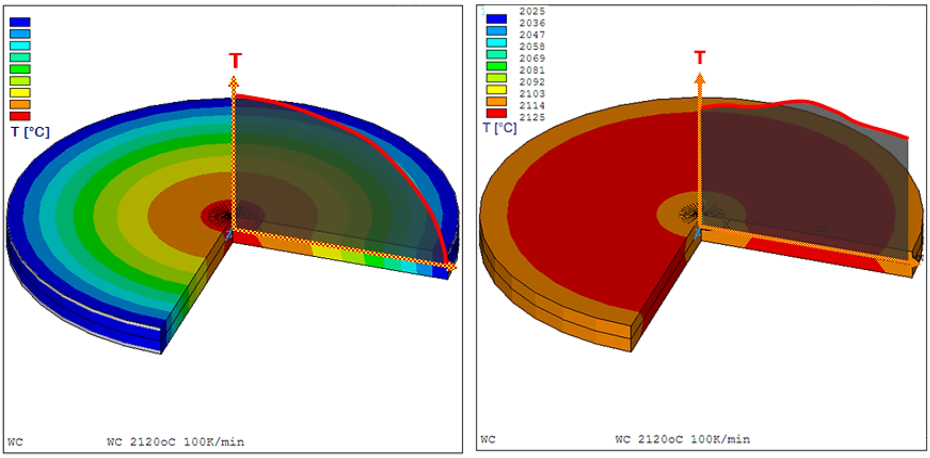

Fig 1. Comparison between conventional FAST/SPS and FAST/Hybrid of a WC sample-disc (D=200 mm)

Literature:

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Bartosz Morończyk[1, 2], Tomasz Choma[1, 2], Bartosz Kalicki[1, 2], Łukasz Żrodowski[1, 2]
[1]AMAZEMET sp. z o.o., Warsaw, Poland
[2]Warsaw University of Technology, Warsaw, Poland

Ultrasonic Atomization, a Novel Technology for Powder Production with Tailored Chemical Compositions

Abstract

The limited availability of custom, high-quality powders with tailored chemical compositions is a constraint in developing new materials for FAST and SPS technologies. Standard powder production routes, like mechanical alloying, that attempt to provide such powders often suffer from unintentional contamination from the container or milling ball materials, affecting feedstock powder quality. Also, the irregular shape and small size of powders produced by mechanical alloying promote their oxidization. On the other hand, the industrial gas atomization process can deliver quality without contamination from the milling process. Still, industrial devices require large facilities, and it is economically not viable to produce small batches of powders. In this study, we present an innovative method of ultrasonic atomization that circumvents these limitations and offers a reliable pathway for producing small custom batches of spherical powders.

The ultrasonic atomization process employs ultrasonic waves to disperse liquid metals into fine particles, creating custom powders with specific chemical compositions. Master alloys can be prepared directly in the device from the raw elements and from any shape or form of feedstock. The method produces highly spherical powders with aspect ratios above 0.9 and no satellites. Particle size distribution (PSD) can be controlled with a chosen frequency of vibrations. Atomization at higher frequencies of vibrations results in smaller particles size with narrow PSD. Future development is needed to increase the production capacity of the device.

Manufactured powders have minimal surface oxidation promoting good bonding between the particles during the sintering. This reduction is attributed to the smaller size of the ultrasonic atomization device and the minimized exposure to oxidation-prone conditions. Furthermore, due to the highly spherical morphology of the produced particles, these powders present a minimized surface-to-volume ratio. Moreover, the spherical shape of the powders improves their flow characteristics and packing density, thereby increasing their sinterability.

Andrzej Koleżyński
Materials Design, Systems & Devices LLC, Krakow, Poland

Resonant Alternating Current Sintering (RACS) Technology

Abstract

Materials Design, Systems & Devices LLC (MDSD™) was founded in 2018 as a spin-off of AGH University of Krakow, Poland, by the scientists employed there and InnoAGH LLC.

MDSD is now an independent company, focusing on the design and construction of scientific and research equipment as well as the synthesis, characterization and theoretical modeling and description of the properties of functional materials for energy conversion. The presentation will be devoted to our company, Materials Design, Systems & Devices LLC, the company's business profile, our offer and our flagship product, a device for various powder materials densification using our innovative resonant alternating current sintering (RACS™) method.

In the RACS method, the energy for sintering is provided in the form of high-frequency alternating current pulses, generated by a resonant power supply coupled to a matching transformer. This method enables efficient consolidation of materials by sintering, particularly ionic materials, due to the AC current not causing ion migration and component segregation. The advantage of the method is the high efficiency in the energy transfer between power supply and sintered powder, which is achieved by matching the resonant circuit to the dies with the consolidated powder and operating the system in resonance. In addition, the placement of the matching transformer in close proximity to the die, allows the use of short current leads, which effectively reduces energy losses.

This allows highly efficient consolidation of metallic, ceramic, semiconductor, nanocrystalline, submicron, and micron powder materials, in the electrothermal sintering process.

Arnaud Fregeac, Jennifer Mackie, Yannick Beynet, Romain Epherre
Norimat, Toulouse, France

The Imminent Surge of FAST/SPS Technology: Paving the Way to Mainstream Production in the Next Decade

Abstract

FAST/SPS has gained recognition as an R&D process capable of manufacturing high-performance parts across a broad spectrum of materials. Recent years have witnessed significant breakthroughs in overcoming the technology's two primary constraints: production scalability and geometry limitations, owing to significant research and technological advancements.

This conference will center on the advancement of producing fully dense 3D complex shapes using FAST/SPS. An innovative and versatile approach, combining additive manufacturing and FAST/SPS for densification, will be showcased, along with original use cases involving diverse ceramic and metal materials designed for applications in the aeronautic, spatial, or defense sectors.

Addressing production challenges, the key concerns revolve around standardization and a comprehensive understanding of the technology. To tackle these issues, the development of digital tools becomes imperative. These tools facilitate the optimization of the manufacturing process to ensure excellent repeatability and control. Furthermore, through leveraging deep statistical analysis of the wealth of data generated by the FAST/SPS process, it has been demonstrated that FAST/SPS technology can achieve capabilities on par with the requirements of the automotive manufacturing sector. In addition, various digital tools that offer support to users of FAST/SPS will be presented, covering the steps from conception and feasibility through to production and quality control monitoring. These modules represent a transformative step towards FAST/SPS 4.0.

Krzysztof Hodor
Netzsch Instrumenty Sp. z o.o., Krakow, Poland

Advanced Materials Characterization Techniques – Netzsch Portfolio

Abstract

Wielding over 60 years of experience, Netzsch Analyzing & Testing provides top-of-the-line customized solutions in thermal analysis, rheology, the determination of thermophysical properties, and fire testing. Our representative offices around the globe, along with our large subsidiaries in China, Japan, the USA, and Korea, allow us to deliver our unparalleled service and support to customers in academia, industry, and materials testing worldwide.

Used in the fields of polymers, life sciences, energy solutions and electronics – as well as in high-temperature applications such as glass, ceramics, metals, alloys, building materials and inorganics – the instrumentation in our comprehensive portfolio is developed and manufactured in-house, in deliberate collaboration with our customers to ensure their utmost satisfaction. Technical excellence, innovative technology, uncompromising quality standards and first-rate software make us to one of the world's leading manufacturers in our trade, with exceptional service offerings rounding out our unsurpassed reputation.

Headquartered in Germany with over 600 employees worldwide, Netzsch Analyzing & Testing joins Grinding & Dispersing and Pumps & Systems as one of three business units in the Netzsch Group – an internationally recognized, owner-managed technology company with an annual turnover of over €720M. With more than 4,000 employees in 50 countries, the Netzsch Group boasts a sweeping global sales and service network, ensuring proximity to our customers no matter where on the planet they happen to be.

S Venkatesh Kumaran[1),2)], Dariusz Garbiec[3)], José Manuel Torralba[1),2)]
[1)]IMDEA Materials Institute, Madrid, Spain
[2)]Universidad Carlos III de Madrid, Leganes, Spain
[3)]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland

The Use of SPS for Sustainable Development of High Entropy Alloys Using Pre-Alloyed Commodity Powders

Abstract

One of the main problems in developing high entropy alloys (HEAs) at an industrial level is the need to use a large number of alloying elements considered critical and/or strategic. This work proposes a way to avoid the direct use of these raw materials by using pre-alloyed powders of superalloys that already contain these alloying elements. In this way, the critical aspect of the raw materials is eliminated, as these powders can be manufactured by recycling scrap from the corresponding superalloys. For this purpose, SPS is a very suitable technology, as it allows, with short sintering cycles and relatively low temperatures, to achieve fine-grained, homogeneous and fully dense materials. In this work, three non-equiatomic HEAs are developed, based on an almost equiatomic composition of Co, Fe, Ni and Cr, with different amounts of Mo in a much lower percentage. For this purpose, gas atomized powders of Ni625, INVAR36, CoCr75, 316L and Fe49Ni manufactured for other applications, are used as starting raw materials. Once the materials have been manufactured by SPS, they are characterized structurally and microstructurally and their mechanical properties at room and high temperature are evaluated. It is shown that this novel route allows to obtain high-performance HEAs avoiding a direct source of critical raw materials in an efficient and cheaper way than using elemental powders as starting material.

Mickaël Coëffe Desvaux[1, 2], Hatim Saidi[1, 2], Tatiana Tabares-Medina[1, 2], Pascal Marchet[1], Marion Vandenhende[1], Mickaël Joinet[2], Nicolas Pradeilles[1], Alexandre Maître[1]

[1]University of Limoges, Limoges, France

[2]THALES Avionics, Moirans, France

Tunable Microstructures of AlN-Based Ceramics by Pressure Assisted Sintering Methods (SPS, HP): Influence on Electrical and Thermal Properties

Abstract

Aluminium nitride (AlN) is a well-known ceramic thanks to its high electric as well as their thermal performances. AlN monoliths are commonly used in electronic and opto-electronic, as a substrate or heat dissipator (*i.e.* LED packaging). Nevertheless, full densification of AlN requires the combination of high temperature and pressure. AlN sintering is mostly performed by hot-pressing (HP) or isostatic pressing (HIP or CIP associated with conventional sintering). In this study, the influence of sintering processes (HP and SPS) on the microstructural features of AlN-based specimens has been investigated. In order to compare properly the working properties of both sintered samples, sintering temperature and processing time have been optimized to reach similar relative density and median grain size from SPS and HP. The comparative study pointed out the importance of sintering process on secondary phases formation and spatial distribution of these latter within ceramic microstructure. HP induces the formation of secondary phases' grains located at AlN triple points. However, SPS allows the formation of rope-like secondary phases located at AlN grain boundaries. Hence, it has been possible to tune the AlN microstructure according to sintering process.

Moreover, the reaction sequence which leads to the secondary phases appears to be strongly dependent on the sintering method. In this way, this latter modifies the chemical composition of secondary phases.

Finally, properties of AlN ceramics obtained from both HP and SPS processes have been compared. For instance, electrical resistivity appeared strongly dependent of secondary phases nature and spatial distribution.

Piotr Klimczyk[1], Kinga Momot[1], Marcin Podsiadło[1], Jolanta Laszkiewicz-Łukasik[1], Yuliia Rumiantseva[1], Agnieszka Gubernat[2]
[1]Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland
[2]AGH University of Krakow, Krakow, Poland

High Performance Duplex Ceramics for Efficient Machining of Nickel Superalloys

Abstract

Ceramic matrix composites (CMCs) are of high interest, mainly due to their high hardness, low abrasion and high chemical resistance, also at elevated temperatures. Their main disadvantage is low fracture toughness and low strength, which often lead to catastrophic failure during exploitation. CMCs in which oxide and carbide or nitride phases coexist are particularly promising materials. Oxide-carbide based group of CMCs includes composites based on aluminum oxide with the addition SiC whiskers, titanium carbide, titanium nitride, as well as widia (WC-Co) and tungsten carbide. It was found that the best performance is demonstrated by composites with mutually interpenetrating carbide and oxide phases (oxides/carbides volume ratio ≈50/50), with a duplex microstructure.

In this work, dense Al_2O_3–ZrO_2–MC ceramics, where MC means one of the following metal carbides: TiC, WC, WTiC, ZrC, were obtained by spark plasma sintering (SPS). The volume composition of all composites was fixed and equal 46:12:42 for Al_2O_3:ZrO_2:MC respectively. The microstructure and physical-mechanical properties of the materials were investigated and compared. In addition, the round cutting tools (RNGN geometry) were manufactured from each material and their cutting properties were assessed during turning tests of Inconel 718 alloy.

The obtained composites were characterized by a relative density of 97–100% and a Young's modulus of 320–470 GPa, depending of metal carbide admixed. Scanning electron microscopy observations revealed quite homogeneous microstructures of the composites, while perfect duplex structure, consisting of mutually penetrating oxide and carbide phases, was obtained only for the composite containing tungsten carbide, because this composite had the most similar grain sizes of both phases (both oxides and tungsten carbide were in submicron range). Al_2O_3–ZrO_2–WC composite was also characterized by the highest Vickers hardness (HV_1=21.5 GPa), fracture toughness (K_{Ic}=5.8 MPa·$m^{1/2}$) and cutting performance of all investigated composites.

Acknowledgement

This work was carried out within the framework of the DuplexCER project, entitled "High performance duplex ceramics for efficient machining of nickel superalloys", co-founded by The Polish National Centre for Research and Development within the framework of the M-ERA.NET research programme (Agreement no. M-ERA.NET3/2021/82/DuplexCER/2022).

Volf Leshchynsky, Dariusz Garbiec
Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland

MAX-Based Composites Made by SPS: Structure Formation and Unique Properties

Abstract

Combining high-energy ball milling and spark plasma sintering is one of the most promising technologies in materials science. The mechanical alloying process enables the production of nanostructured composite powders that can be successfully spark plasma sintered in a very short time, while preserving the nanostructure and enhancing the mechanical properties of the composite. Composites with MAX phases are among the most promising materials. In this study, Ti/SiC composite powder was produced by high energy ball milling and then consolidated by spark plasma sintering. During both processes, Ti_3SiC_2, TiC and Ti_5Si_3 phases were formed. Scanning electron microscopy, energy-dispersive X-ray spectroscopy and X-ray diffraction study showed that the phase composition of the spark plasma sintered composites consists mainly of Ti_3SiC_2 and a mixture of TiC and Ti_5Si_3 phases which have a different indentation size effect. The indentation size effect at the nanoscale for Ti_3SiC2, TiC+Ti_5Si_3 and SiC-Ti phases is analyzed on the basis of the strain gradient plasticity theory and the equation constants were defined. The friction and wear behavior were investigated at temperatures ranging from 20 to 700°C and test loads from 10 to 40 N, revealing self-lubricating effects during sliding. A near-zero wear effect at high temperatures was found for the composites sintered from non-stoichiometric Ti/SiC powder. The resulting composites exhibited hardness of 810–860 HV_{10} and fracture toughness of 5.5–6.5 MPa $m^{1/2}$ for the samples sintered from Ti_3SiC_2-based powder and hardness of 1100–1200 HV_{10} and fracture toughness of 5.0–5.3 MPa $m^{1/2}$ for the samples sintered from Ti/SiC powder.

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Tomasz Mościcki, Rafał Psiuk, Dariusz Jarząbek
Institute of Fundamental Technlogical Research PAS, Warsaw, Poland

Micromechanical Tests of Magnetron Sputtered W-Ti-B Coatings from SPS Targets

Abstract

The main problem of using super-hard and thermally resistant ceramic as protective layers is their brittleness and difficulties with adhesion to a much softer substrate. The last research has reported on flexible hard ceramic coatings prepared by magnetron sputtering. Among them tungsten diborides represent a new class of coatings which are simultaneously super-hard, tough and resistant to cracking. Alloying of WB_x with transition metal like titanium, zirconium or tantalum leads to considerable improvement of mechanical and tribological properties compared to undopped borides. In this stdies these special mechanical properties of W-Ti-B coatings were obtained thanks to the use of high power impulse magnetron sputtering (HIPIMS) which provide the suitable balance of energy at deposited surface. In this study the micromechanical tests were used for measurement of superhardness H >42 GPa, high fracture toughness 3.5±0.1 MPa·$m^{1/2}$ and those properties are better than for commercially used TiN coatings. Also nanopilar compressing and Stoney equation for compressive strength and film stress designation were used respectively. Alloyed with titanium coatings are well adherent to nitrided steal substrates, incompressible and wear resistant. HiPIMS sputtering from a single SPS target reduces the cost of equipment (only one magnetron) and enables to decrease the deposition temperature to 300°C, which makes it possible to use it for larger dimension tools.

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Igor Veremchuk[1)], Yuri Grin[2)], Denys Makarov[1)]
[1)]Helmholtz-Zentrum Dresden-Rossendorf e.V., Institute of Ion Beam Physics
and Materials Research, Dresden, Germany
[2)]Max Planck Institute for Chemical Physics of Solids, Dresden, Germany

Spark Plasma Sintering for Synthesis of Transition Metal Oxides

Abstract

The solid-state synthesis of transition metal oxides (TMO's) is a challenging task. Slow diffusion and mass transfer of reagents are characteristic of such solid-state reactions (SSR's). In this context, spark plasma sintering (SPS) seems to emerge a promising and technologically applicable synthetic route to obtain TMO's. We successfully conducted SSR of Ti_2O_3 synthesis using SPS with dc-current acting as an accelerator of the diffusion-controlled processes between TiO_2 and Ti [1]. Further, this approach was applied to directly synthesize different TMO's (titanium oxides [2], molybdenum oxides [3], tungsten oxides [4], and chromium oxides [5]). Among the advantages of such synthetic routes, we would like to stress: i) simple pre-experiment preparation (i.e., mixing of the initial powders); ii) simultaneous compaction and shaping of products; iii) short synthesis time (i.e., from minutes to about the few hours), iv) enormous accuracy (i.e., ≅0.1 at % of oxygen) as well as v) high degree of reproducibility.

New types of electrochemical using SPS SSR was recently performed in our laboratories using SPS. By sintering TiO_2 (insulator) with WO_2 (metal) mixed in different proportions, we obtained solid solution based on rutile (i.e., TiO_2) structure. However, an appearance of elemental tungsten cannot be avoided while performing the synthesis with graphite foils as separators between the reacting mixture and the punches. To shed light on the mechanism of such an electrochemical process we performed two reactions, applying the opposite polarity of dc-current pulses, to the placed in graphite die layers of unmixed TiO_2 and WO_2. The further combined metallographic-EDX investigation of the polished cuts of the reacted specimens revealed that in the case when WO_2 was under positive pole (i.e., being an anode) free W is forming at "+"-electrode, whereas the switch of the polarity results in the formation of tungsten inclusion on the phases border between reactants. Thus, elemental tungsten seems to be the product of electrochemical reduction of WO_2. Avoiding this reaction, a single phase $Ti_{1-x}W_xO_2$ is obtained replacing the graphite foils by tungsten ones.

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Beata Leszczyńska-Madej[1)], Marcin Madej[1)], Anna Wąsik[1)], Dariusz Garbiec[2)]
[1)]AGH University of Krakow, Krakow, Poland
[2)]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland

Investigation of Microstructure and Tribological Behavior of Al-SiC Composites with High SiC Content Processed through Spark Plasma Sintering

Abstract

The microstructure and tribological properties of Al-70wt% SiC and Al-90wt% SiC composites produced by spark plasma sintering have been investigated. Due to their attractive thermal, physical and mechanical properties, aluminum matrix composites with high volume fractions of silicon carbide (>50%) have become of great interest as a potential material for multifunctional electronic packaging and cryogenic applications. The SPS process has been carried out in a vacuum atmosphere under various conditions. Composites were obtained with densities close to theoretical (96–98%) and with a uniform distribution of the carbide phase in the matrix. X-ray diffraction, optical microscopy and scanning electron microscopy with EDS analysis were used to characterise the microstructure. Mechanical properties were determined by hardness measurements and a three-point bending test. The tribological properties of the composites were determined using a T-05 block-on-ring tribometer. The results showed that the composite with the highest SiC content (90 wt%) had a higher hardness (2537 HV_1) and flexural strength (242 ± 15MPa) with a lower wear resistance. The weight loss of this composite was 0.43 and 0.76% for friction at loads of 100 and 200 N, respectively, and was 360 and 270% higher than that of composites with the lower SiC phase content. The wear rate was three times higher for the Al-90wt%SiC composites.

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**Mirosław Kruszewski[1)], Konrad Cymerman[1)], Jakub Flaga[1)],
Marcin Chmielewski[2,3)], Dorota Moszczyńska[1)], Łukasz Ciupiński[1)]**
[1)]Warsaw University of Technology, Warsaw, Poland
[2)]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland
[3)]National Center for Nuclear Research, Swierk, Otwock, Poland

Fabrication and Evaluation of Co-based Diffusion Barriers for Skutterudite Thermoelectric Materials Obtained via Pulse Plasma Sintering

Abstract

One of the key aspects determining the broad commercialization of thermoelectric modules is their durability. To guarantee long-term durability, stable and effective diffusion barriers must be used to separate the thermoelectric material from the solder or connectors. Among the numerous parameters that should be paid attention to is thermodynamic stability, which is related to the diffusion phenomenon, and matching the coefficients of linear thermal expansion (CTEs) of the module's component materials, thanks to which stresses arising as a result of temperature changes are minimized.

This work aims to design and fabricate stable and effective diffusion barriers to be used in thermoelectric modules based on skutterudites. Co-Ni-Cr, Co-Ni-W, Co-Cr, and Co-W systems were selected for the tests. The chemical composition of the barriers has been chosen to ensure maximum compatibility in terms of thermal expansion with the $CoSb_3$ material. At the work's initial stage, several consolidation tests via pulse plasma sintering (PPS) technique were performed to determine the optimal conditions for the barrier fabrication. The density, microstructure, and CTE of the obtained materials were investigated. In the next stage of the work, joints between the tested barriers and $CoSb_3$ were manufactured. Two ways of producing joints were used: (1) a single-stage process of PPS consolidation of the diffusion barrier together with $CoSb_3$, and (2) a two-stage process in which the diffusion barriers were initially PPSed and then joined with $CoSb_3$ during its consolidation. The microstructure and phase composition of the joint area as well as the electrical contact resistance, were investigated in the as-fabricated state and after prolonged annealing. The results of this work were also discussed in the context of other works presented in the literature.

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Paweł Nieroda
AGH University of Krakow, Krakow, Poland

Superionic Thermoelectric Materials Based on Cu_2X (X = S, Se) Sintered Using the SPS and the "SPS Melting" Method

Abstract

Cu_2Se and Cu_2S belong in recent years to the most intensively studied thermoelectric materials, due to the very high values of the thermoelectric figure of merit *ZT* parameter. In this work, Cu_2S and Cu_2Se powders were sintered using the self-constructed SPS apparatus equipped with two independent power sources, i.e., with direct current (DC) and alternating current (AC) to reduce the migration of copper ions, which is the main reason limiting the use of these materials in thermoelectric generators for the conversion of thermal energy into electricity. Additionally, a one-stage method (synthesis and densification) for obtaining Cu_2Se sinters, using a modified SPS technique called here the "SPS melting method" was developed. The new approach consists of placing a special graphite container in a graphite die, which which enables the initial reaction of the substrates at a temperature of T=750 K and then re-melting the material at a temperature above the melting point in one process. The chemical composition after sintering was investigated employing scanning electron microscopy (SEM) combined with the energy-dispersive spectroscopy (EDS) method, and the phase composition was determined with X-ray diffraction (XRD). The uniformity of the thermoelectric properties of the materials was examined by scanning thermoelectric microprobe (STM). Measurements of thermoelectric transport properties, i.e., electrical conductivity, the Seebeck coefficient and thermal conductivity in the temperature range from 300 to 965 K were carried out. Based on these results, the temperature dependence of the thermoelectric figure of merit *ZT* was determined as a function of temperature for Cu_2Se samples densified by the SPS and the 'SPS melting' method. The negative influence of the DC current used in the SPS process in the case of sintering superionic thermoelectric materials was shown. On the other hand, the use of AC current allows the preservation of all the advantages of the SPS technique, such as, e.g. the high speed of the process, but additionally shows that the migration of ions does not occur. The new proposed approach of one-stage preparation of Cu_2Se sinters (SPS melting) seems to be suitable also for many other congruently melting materials.

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Juliusz Leszczyński, Paweł Nieroda, Andrzej Koleżyński
AGH University of Krakow, Krakow, Poland

FAST/SPS Sintering of Tetrahedrites $Cu_{12-X}(Tm^1Tm^2)Sb_4S_{13}$ (Tm^x – Mn, Fe, Co) for Thermoelectric Applications

Abstract

Low cost, low toxicity and a complex, flexible crystal structure allowing numerous modifications make tetrahedrites an interesting group of thermoelectric materials. The strategy of introducing multiple dopants into the structure of semiconducting thermoelectrics has worked well for most known materials, yielding significant improvements in thermoelectric properties. Tetrahedrites belong to the copper sulphide group, in which high copper ion mobility and ionic conductivity have been found, which brings some challenges during current-assisted sintering. In addition, the investigated materials in the temperature range used during synthesis, densification and thermoelectric measurements may undergo phase transitions which presents additional challenges during densification. Therefore, in the study undertaken, it was decided to introduce several dopants into one atomic position of the tetrahedrite, which should, by significantly modifying the structure and introducing additional disorder, improve the thermoelectric properties of the materials obtained. The SPS sintering method using AC current was chosen as the densification method for the synthesised powders.

Tetrahedrites co-doped with two dopants in the copper sublattice $Cu_{11.5}(Tm^1,Tm^2)_{0.5}Sb_4S_{13}$ (Tm^x – transition metal) were obtained by means of a high-temperature reaction of high-purity elements and subsequent sintering using the SPS method. The obtained pellets were characterised by structural (XRD) and microstructural (SEM-EDX) methods to confirm their chemical and phase composition and homogeneity. Electrical conductivity, Seebeck coefficient and thermal conductivity were studied from room temperature to 720 K and used to calculate thermoelectric figure-of-merit ZT. The different SPS sintering strategies used in the densification of the materials obtained and their advantages and disadvantages in the context of sulphide tetrahedrites are presented and discussed.

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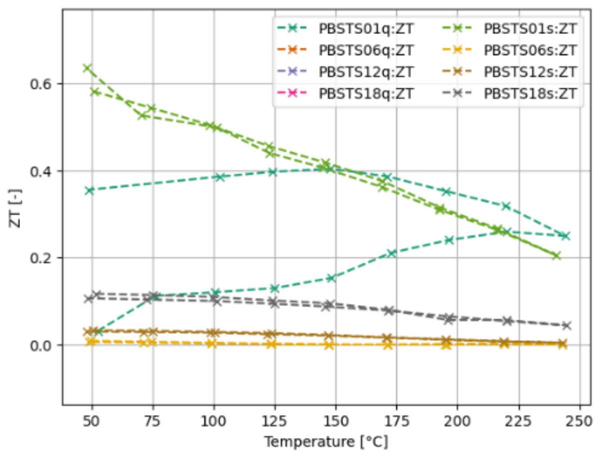
Bartosz Bucholc[1,2], Kamil Kaszyca[1,3], Krystian Kowiorski[1], Konrad Krzyżak[1], Agata Strojny-Nędza[1], Grzegorz Kuderski[1], Krzysztof Mars[3], Anna Dobkowska[1,4], Marcin Chmielewski[1,5], Rafał Zybała[1,4]

[1]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland
[2]Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland
[3]AGH University of Krakow, Krakow, Poland
[4]Warsaw University of Technology, Warsaw, Poland
[5]National Centre for Nuclear Research, Otwock, Poland

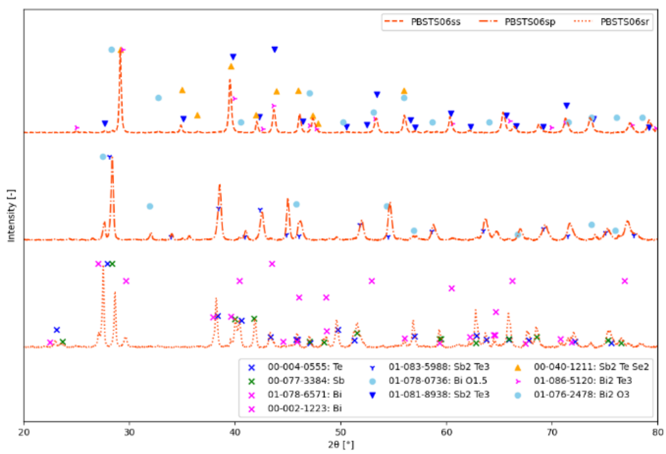
Synthesis and Consolidation of Bi_2Te_3-based Thermoelectric Materials with the Use of SPS/FAST Technique

Abstract

The pursuit of sustainable and efficient energy conversion has driven significant interest in thermoelectric materials (TM), particularly those based on bismuth telluride (Bi_2Te_3). Bi_2Te_3 is a compound first observed over sixty years ago and since then its thermoelectric performance has been steadily improved. Amongst methods used for enhancing their properties, doping should be mentioned. In this study, a novel approach for fabricating TM based on Bi_2Te_3 is presented. Selenium (Se) and antimony (Sb) doped materials were synthesized through the energy-efficient self-propagating high-temperature synthesis (SHS) process and subsequently consolidated with the use of the self-developed spark plasma sintering (SPS) apparatus. Due to employing the SHS, it was possible to obtain powders of *n*-type $Bi_2Te_{3-y}Se_y$ and *p*-type $Bi_{0.5}Sb_{1.5}Te_{3-y}Se_y$ in a remarkably reduced time compared with the application of conventional synthesis technique in quartz tubes. The resulting powders were then sintered and thoroughly investigated. Comprehensive characterization using X-ray diffraction (XRD) and scanning electron microscopy (SEM) was performed in order to assess and analyze the microstructural evolution in the subsequent processing stages. In addition to structure and microstructure studies, developed materials were thoroughly characterized in terms of thermal conductivity and electrical properties (Seebeck coefficient and conductivity as functions of temperature) in order to evaluate their potential for application in thermoelectric devices, e.g. thermogenerators. The analyses were conducted with the use of techniques such as the four-probe method and laser flash analysis (LFA). By evaluating these fundamental parameters, the thermoelectric figure of merit (ZT), a critical metric indicating the materials' overall efficiency in converting waste heat into useful electrical energy, was ascertained. The most promising results were obtained for material with an electrical conductivity of type *p* - $Bi_{0.5}Sb_{1.5}Te_{2.9}Se_{0.1}$ with ZT=0.6 at 320 K.



a)



b)

Fig. 1. a) ZT values of p-type material series, b) XRD patterns of exemplary material of sr – raw powder mixture, sp – material after SHS reaction, ss – material after SPS sintering.

Our findings showcase the promising potential of combining SHS synthesis with SPS consolidation, allowing for the rapid production of high-performance thermoelectric materials.

Acknowledgement

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Martin Jackson
The University of Sheffield, Sheffield, United Kingdom

Exploitation of FAST/SPS to Recycle Surplus Metal Powder for Sustainable Solutions and Near-Net Shape Components

Abstract

Field-assisted sintering technology also known as spark plasma sintering is starting to be recognised as a potential route for metals processing and near net shaping for a range of sectors. FAST/SPS is effective way of rapidly consolidating powder and particulate feedstocks, including waste streams such as machining swarf into shaped billets with as-forged properties. FAST/SPS can also be used as an intermediate step prior to conventional closed die forging or hot rolling (FAST-forge and FAST-roll, respectively) or subsequent step after cold-isotatic pressing (CIP-FAST) and additive manufacturing (AddFAST). The solid-state technique has also been demonstrated to be an effective way to functionally grade and diffusion bond different alloys in the same FAST billet (FAST-DB). In this paper I will summarise some of the developments at The University of Sheffield around FAST/SPS over the last couple of years with examples from different material types for a range of different sectors, such as defence, biomedical, aerospace and automotive.

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Abdullah Riaz, Christian Polley, Eberhard Burkel, Hermann Seitz
University of Rostock, Rostock, Germany

Field-Assisted Sintering of Load-Bearing Ti6Al4V-Barium Titanate Piezoelectric Scaffolds for Bone Tissue Engineering

Abstract

A critical-size bone defect in load-bearing areas is a challenging clinical problem in orthopaedic surgery. Currently used titanium alloy (Ti6Al4V) scaffolds feature a high biomechanical stability but lack electrical activity, which hinders their further use. In preliminary studies we have fabricated piezoelectric and biocompatible calcium titanate and barium titanate ceramics by means of field-assisted sintering. The piezoelectric properties of fabricated scaffolds were analysed and compared with the piezoelectric properties of natural bone. Our recent study is focused on the fabrication of Ti6Al4V-barium titanate bulk composite scaffolds to combine the biomechanical stability of Ti6Al4V with electrical activity through barium titanate. A hollow cylindrical Ti6Al4V is additively manufactured by electron beam melting and combined with barium titanate powder for joint processing in field-assisted sintering. Scanning electron microscope images on the interface of the Ti6Al4V-barium titanate composite scaffold showed that after sintering, the Ti6Al4V lattice structure bounded with the barium titanate matrix without its major deformation. The Ti6Al4V-barium titanate scaffold had average piezoelectric constants of (0.63 ± 0.12) pC/N directly after sintering due to partial dipole alignment of the barium titanate tetragonal phase, which increased to (4.92 ± 0.75) pC/N after a successful corona poling. Moreover, the nanoindentation values reveal that the Ti6Al4V is the harder and stiffer part in the Ti6Al4V-barium titanate composite scaffold. Such a scaffold has the potential to treat critical-size bone defects in load-bearing areas and guide tissue regeneration by piezoelectric stimulation.

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Szymon Nosewicz[1], Grzegorz Jurczak[1], Witold Chromiński[2], Jerzy Rojek[1], Kamil Kaszyca[3], Marcin Chmielewski[3]
[1]Institute of Fundamental Technological Research PAS, Warsaw, Poland
[2]Warsaw University of Technology, Warsaw, Poland
[3]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

Quantitative Analysis of Influence of SPS Process Parameters on the Porous Materials Structure Using Combined EBSD and Computer Assisted Software

Abstract

The proposed study demonstrates the experimental, numerical, and theoretical attempt to describe the microstructure of nickel aluminide (NiAl) samples manufactured by spark plasma sintering using electron backscatter diffraction and computer-assisted software. The purpose of the paper was to present the evolution of the structural microscopic and macroscopic parameters – grain size, shape and boundary contact features – and their relation to the main SPS process parameters – temperature and pressure. The use of the electric current and the additional external pressure results in significant changes in the microstructure of the samples, such as the occurrence of lattice reorientation resulting in grain growth, an increase in the grain neighbors, or the evolution of grain ellipticity, circularity, grain boundary length, and fraction [1]. Moreover, the numerical simulation of heat conduction via a finite element framework was performed to analyze the connectivity of the structures evaluating local heat fluxes, deviation angles, and effective thermal conductivity and studying them in the context of the microstructural porosity. Finally, the effective thermal conductivity of 2D EBSD maps was compared with those obtained from FEM simulations of 3D micro-CT structures [2]. The relationship between the 2D and 3D results was derived by using the analytical Landauer model.

Literature:

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Yuliia Rumiantseva[1)], Piotr Klimczyk[1)], Jolanta Laszkiewicz-Łukasik[1)], Marcin Podsiadło[1)], Karolina Chat-Wilk[1)], Serhiy Lysovenko[2)], Olha Kushch[2)]
[1)]Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland
[2)]V. Bakul Institute for Superhard Materials of NASU, Kyiv, Ukraine

The Investigation of Al/$Mg_2B_2O_5$w Composites Sintered by SPS and HPHT Methods: Chemical Interaction, Microstructure and Mechanical Properties

Abstract

The chemical interaction of magnesium borate whiskers under high temperature conditions at moderate pressure (SPS method, 50 MPa, 1300°C) and high pressure (HPHT, 7.7 GPa, 2000°C) has been studied. Thermodynamic modelling was carried out with the assistance of the ThermoCalc program and the findings were compared with results from XRD phase analysis.

For samples sintered through the SPS method, the presence of MgO, B_2O_3, and $AlBO_3$ phases was established. HPHT-sintered samples exhibited the presence of MgO, Al_2O_3, and $Al_{0.72}B_2Mg_{0.2}$ phases. Notably, both sintering methods showed indications of the $MgAl_2O_4$ phase. X-ray diffraction analysis revealed that the $MgAl_2O_4$ phase was more prominent in the case of SPS sintering, evident from the peak heights' increasing. This suggests that pressure application suppresses the chemical interaction between aluminium powder and magnesium borate microfibers. Furthermore, pressure hindered the formation of the glass-like B_2O_3 phase, as confirmed by SEM investigations' results. In turn, it led to a lower amount of unreacted aluminium in SPS-sintered composites. Consequently, SPS-sintered samples exhibited better mechanical properties (HV_1=13.0 ± 2 GPa; HV_5=11.15 ± 1.2 GPa; E=195 GPa) compared to composites sintered through the HPHT synthesis method (HV_1=4.71 ± 0.44; HV_5=3.88 ± 0.15; E = 75 GPa).

The outcomes shows the substantial influence of the sintering method on the structure formation and chemical interaction of aluminum powders with magnesium borate whiskers and points on the potential of the "Al-$Mg_2B_2O_5$w" system for obtaining promising material with a perovskite structure – $Mg_2Al_2O_4$, possessing unique properties, such as no phase transition up to its melting point (2105°C), high strength at elevated temperature, high chemical inertness against both acidic and basic slag, low thermal expansion coefficient, high thermal shock resistance and unusual inertness to high-fluence neutron irradiation.

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Anna Dobkowska[1),2)], Mirosław Jakub Kruszewski[2)], Rafał Zybała[1)], Izabela Zgłobicka[3)]

[1)]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland
[2)]Warsaw University of Technology, Warsaw, Poland
[3)]University of Bialystok, Bialystok, Poland

In-Depth Analysis of the Influence of Bio-Silica Filler on the Properties of Mg Matrix Composites

Abstract

A novel metal matrix composites (MMC) with Mg matrix reinforced with natural filler in the form of Didymosphenia geminata frustules (algae with distinctive siliceous shells) are presented in this work. Pulse plasma sintering (PPS) was used to manufacture Mg-based composites with 1, 5 and 10 vol.% ceramic filler. As a reference, pure Mg was sintered. The results show that the addition of 1 vol.% Didymosphenia geminata frustules to the Mg matrix increases its corrosion resistance by supporting passivation reactions. Addition of 5 vol.% the filler supports microgalvanic reactions leading to the greater corrosion rate. Higher content than 5 vol.% the filler causes significant microgalvanic corrosion due to the greater micro-galvanic effect of the composites containing 10 and 15 vol.% diatoms. The results of contact angle measurements show the hydrophilic character of the investigated materials, with slightly increase in numerical values with addition of amount of ceramic reinforcement. With the increasing addition of Didymosphenia geminata frustules, an increase in strength with a decrease in compressive strain is observed. In all composites an increase in microhardness was attained. The results clearly indicate that filler in the form of Didymosphenia geminata frustules may significantly change the most important properties of pure Mg, indicating its wide potential in the application of Mg-based composites with a special focus on biomedical use.

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Fatima Nisar[1)], Jerzy Rojek[1)], Szymon Nosewicz[1)], Marcin Chmielewski[2)], Kamil Kaszyca[2)]

[1)]Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland

[2)]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

Thermo-Electric Model for FAST/SPS Sintering in Discrete Element Framework

Abstract

A discrete element model is developed to simulate two-way coupling of thermal and electrical problem in electric current assisted sintering (ECAS) process. ECAS is a powder consolidation process which combines uniaxial pressure with temperature, generated via current (Joule heating), to enhance cohesive inter-particle bonds and hence densification. A wide range of materials, both conductive and nonconductive ones, can be sintered using ECAS process. Here we focus on conductive materials, therefore we investigate coupled current and temperature phenomena in sintered materials.

The pipe network model is used to model both heat transfer and electric current between particles. The electrical and thermal conductance of the pipes connecting centres of spherical particles model is based on the authors' own model proposed in [1]. The conductance is dependent on the material conductivity and the radii of the necks formed and growing between particles during sintering. Moreover, particle-particle contact resistance is also considered and added to the model.

The model is verified by comparison with finite element simulations of benchmark examples. The thermal, electrical and thermoelectrical problems have been defined for simple three particle configurations and analysed both with the DEM and FEM. Heat fluxes, temperature and current distribution obtained in the DEM analyses were found to be consistent with the more established but complex and time consuming FEM model.

Thereafter, the DEM model was applied for more complex problems. It was used to determine thermal and electrical conductivity of porous material. For calibration and validation of the model, copper and NiAl samples are manufactured using ECAS/SPS with different processing parameters. Experimental results for electrical and thermal conductivity of these partially sintered porous materials are then compared to the DEM results [2].

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Krzysztof Mars[1,2], Rafał Zybała[3]
[1]AGH University of Krakow, Krakow, Poland
[2]Materials Design Systems & Devices LLC, Krakow, Poland
[3]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

Sputtering Targets Obtained by Induction Hot Pressing (IHP) and Spark Plasma Sintering (SPS) Methods

Abstract

The sputtering targets work as a stable and long-lived evaporation source in many physical vacuum deposition processes. The target, in the form of a circular plate, is made up of the material to be sputtered and deposited on the substrates. After pumping the vacuum chamber down and introducing working gas (usually Ar), a negative voltage is applied to the target to create the plasma. The positive ions available in the plasma are accelerated towards the target and hit atoms that are ejected from a surface by momentum transfer. These atoms are condensed on the substrates to build up a layer.

The paper presents selected examples of the targets obtained in the Sn-Se, Cu-Se, Sb-Te, Ge-Sb-Te, Mg-Si, Al-Cr, and Al-Ti-Cr-Fe-Ni systems. The target materials were produced by a liquid phase synthesis in a quartz ampoule (SnSe, Cu_2Se, Sb_2Te_3, $Ge_2Sb_2Te_5$). Magnesium silicide (Mg_2Si), AlCr alloy, and high entropy alloy (AlTiCrFeNi) were produced from elemental powders by self-propagating high-temperature synthesis (SHS). The laboratory set-up of unique design enabled maintaining adiabatic conditions during the exothermic reaction, which proceeded very rapidly in an oxygen-free environment, yielding a powder product. The received materials in the form of ingots or pellets were powdered by mechanical milling using a Fritsch 6 planetary mill in the presence of isopropyl alcohol. induction hot pressing (IHP) and spark plasma sintering (SPS) methods were subsequently used for the consolidation/densification of the powder. Dense sintered compacts with a diameter of 50 mm were used to produce the sputtering targets. Characterisation of the targets comprised phase analysis by X-ray diffraction (XRD), studies of morphology and chemical composition by scanning electron microscopy (SEM), and energy dispersive X-ray spectrometry (EDS). The resulting targets were installed on the magnetron gun and tested in the vacuum apparatus. The enhanced performance of the targets was discussed with reference to sintering conditions and compared with the data on the properties of similar materials produced by other synthesis/processing routes.

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Artur Olejarz[1)], Wenyi Huo[1,2)], Maciej Zieliński[1)], Damian Kalita[1)], Edyta Wyszkowska[1)], Łukasz Kurpaska[1)]
[1)]National Centre for Nuclear Research, Otwock, Poland
[2)]Nanjing Forestry University, Nanjing, China

The Effect of Process Optimization on Microstructure Evolution and Mechanical Properties of Low BPR Mechanically Alloyed CoCrFeNi High Entropy Alloy

Abstract

Powder metallurgy is a promising alternative for modern alloys, including high-entropy ones. The primary assumption of this group of techniques is to mix all the desired elements in a proper condition and then synthesize them to obtain a bulk material. Besides the many advantages of this group of methods, the most significant challenges are the scale of acquired samples and the undesired phase formation. To overcome the first issue, we decided to reduce the ball-to-powder ratio (BPR) to find the possibilities to make manufacturing high-entropy alloys via powder metallurgy processes more industrially scalable.

In our work, the mechanical alloying process was implemented to mix Co, Cr, Fe, and Ni powder's equiatomic composition. The 40 hours of milling time as a commonly used parameter was used. As an innovative approach, we introduce the short, highly efficient pre-milling process of Cr and Ni powders, which have the most considerable particle size and present the lowest diffusion coefficients. As a next step, we improve the efficiency of the mechanical alloying process by increasing the milling speed up to 350 rpm and reducing the milling breaks after each milling interval from 15 to 5 minutes. The very low BPR 5:1 was employed to improve the productivity of the process.

Samples were sintered using the spark plasma sintering technique. The sintering temperature was 950°C, and the sintering pressure was 50 MPa. The heating rate was 100°C/min, and samples were held for 10 minutes at the target temperature. Moreover, heat treatment was performed under a protective atmosphere at 850°C and 1050°C for 12 hours, followed by water quenching.

Manufactured samples presented a high densification level reaching even 97% of theoretical density. The sample milled in one milling cycle showed coarse (<50 μm) Cr-rich particles evenly distributed in a sample's volume. Two different structures inside the particles have been found: the grain BCC structure is surrounded by the dark grey ring. The annealing process promoted the homogenization of coarse particles and a matrix phase, where some Co-, Fe- and Ni-rich areas can be detected after the sintering process. The pre-milling process implementation resulted in the reduction of size particles. The fine particles

(<1 μm) formed agglomerates. A bimodal grain size structure has been found with a smaller grain size nearby the Cr-rich particle agglomerates. X-ray diffraction patterns revealed the formation of the different carbides depending on the chosen process. The sample milled in one milling cycle presents the $M_{23}C_6$ in both sintered and annealed forms. The pre-alloyed samples present both $M_{23}C_6$ and M_7C_3 after sintering. However, the annealing at a lower temperature promoted the $M_{23}C_6$ formation, whereas the higher temperature caused M_7C_3. The coarse particles in one milling cycle showed significantly higher hardness than the fine particles, especially after high-temperature annealing. The hardness of a FCC matrix phase after the improved two-milling cycle process is 408 HV_1, the highest value of all manufactured samples. Moreover, the annealing at lower temperatures results in hardness stability, and the sharp hardness drop after higher temperature annealing is observed. In conclusion, we demonstrated that using a low BPR ratio could produce HEA with stable microstructure and improved mechanical properties.

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**Mateusz Marczewski[1)], Piotr Siwak[2)], Flávia Pires Rodrigues[3)], Lucas Dorna de Paula[3)],
Kamil Kowalski[2)], Mieczysław Jurczyk[4)], Dariusz Garbiec[1)]**

[1)]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland
[2)]Poznan University of Technology, Poznan, Poland
[3)]Paulista University – UNIP, São Paulo, Brazil
[4)]University of Zielona Gora, Zielona Gora, Poland

β-Type Titanium and Zirconium-Based Alloys Produced Via Sustainable Spark Plasma Sintering of Nanocrystalline Powders

Abstract

The increasing life expectancy is causing several issues in the locomotor system as well as dental care. Living standards improvement is further possible through the development of commercial osteoarticular and dental implants. It is possible by the manufacturing of the new generation body-centred cubic structured Ti-based and Zr-based implants with a lower risk of stress-shielding failure and lower toxicity. Moreover, using Zr instead of Ti allows for further decreasing stress-shielding effect and the number of MRI artefacts, which increases the implant availability. In the present work, the combination of two nanotechnologies: mechanical alloying (mechanochemical synthesis driven by the competitive cold welding and fracturing of powder particles in the high-energy ball mill) and spark plasma sintering (sintering with Joule heating caused by the current flowing through the sample) were used to allow energy efficient and sustainable manufacturing of the proposed materials. Selected Zr and Ti alloys were produced in the ternary Ti-Zr-Nb system and their crystal structure after every processing stage was analyzed with X-ray diffraction and Rietveld refinement. The correlation between alloys manufacturing parameters (especially sintering temperature), composition, crystal structure, and properties such as nanoindentation modulus were evaluated with the aim to develop an alloy with a modulus about 40% lower than titanium Grade 2. The formation of nanostructure in the powder after a short milling time was finally confirmed, and the significant influence of zirconium on the lattice constant of the body-centred cubic phase was highlighted. Spark plasma sintering seems to allow the development of new materials in the medicine and other advanced materials-related areas.

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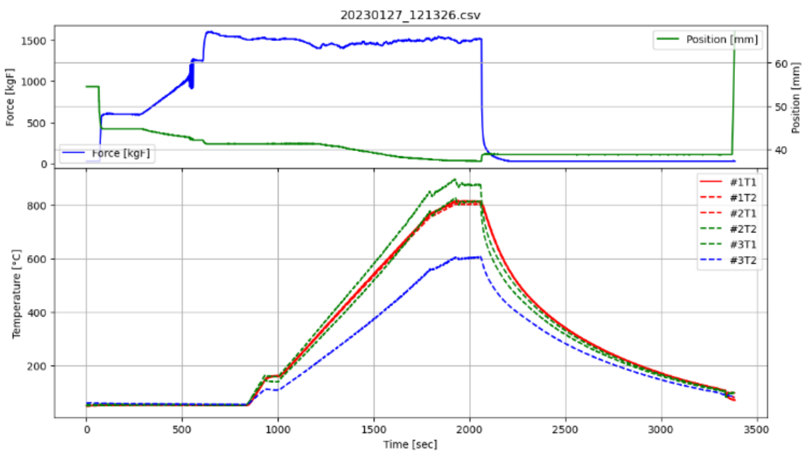
Kamil Kaszyca[1,3], Fatima Nissar[2], Krystian Kowiorski[1], Konrad Krzyżak[1], Agata Strojny-Nędza[1], Grzegorz Kuderski[1], Bartosz Bucholc[1,2], Rafał Zybała[1,4], Jerzy Rojek[2], Marcin Chmielewski[1,5]

[1]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland
[2]Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland
[3]AGH University of Krakow, Krakow, Poland
[4]Warsaw University of Technology, Warsaw, Poland
[5]National Centre For Nuclear Research, Otwock, Poland

Detailed Analysis of Microstrostructure and Properties of Copper Sintered by SPS Method

Abstract

A detailed description of SPS process parameters' influence on sintered copper microstructure and selected properties will be presented. The new functionalities of SPS aparatus after its modernization will be presented. The information will be provided on temperature measurement system, force control system and process parameters control system. We used the custom, modular SPS apparatus acquisition system allowing to determine 6 temperatures using contact measurement, an infra-red sensor for non-contact temperature measurement, position, force and electrical power supply characteristics (Fig. 1).



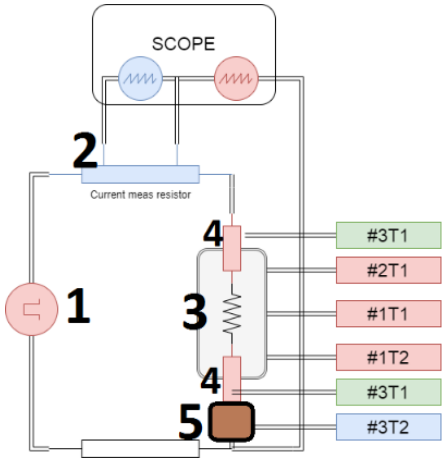


Fig. 1. Process parameters and SPS measurement system diagram. 1 – Power supply, 2 – current sensing resistor, 3 – graphite die, 4 – graphite punches, 5 – graphite distancer. #XTn – temperature sensor

A detailed study of copper samples produced using different process parameters (sintering temperature, time and pressure) and their influence on the progress of the sintering process was carried out. Changes in microstructure were analyzed using scanning electron microscopy, and electrical and thermal properties were characterized as a function of material density. Obtained results are useful for deeper understanding of phenomena's that take place during SPS process, as well it allows to determine dynamic temperature gradient characteristics inside die during heating stage. Acquired date will be later used for large-scale sintering model validation.

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Tomasz Stasiak[1)], Jarosław J. Jasiński[1)], Wojciech Chmurzyński[1)], Łukasz Kurpaska[1)], Marcin Chmielewski[1, 2)], Magdalena Wilczopolska[1)], Maciej Zieliński[1)], Marcin Kowal[1)], Jacek Jagielski[1)]

[1)]National Centre for Nuclear Research, Otwock-Swierk, Poland
[2)]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

Characterization of FeCrAl-Y_2O_3 ODS Alloys with the Additions of Ti and V Consolidated by SPS

Abstract

FeCrAl-Y_2O_3 ODS alloys are promising materials for new-type nuclear reactors, in which operating conditions will be harsh – temperatures of up to 700°C and high irradiation dose of up to 200 dpa. ODS alloys are expected to withstand those conditions due to nanometric oxides homogenously distributed in the bcc matrix. These oxides improve high-temperature mechanical resistance and act as sinks for radiation-induced defects.

In this study, the FeCrAl-Y_2O_3 alloys with the addition of Ti (to improve corrosion resistance) and V (to increase strength) were investigated. The samples were prepared by mechanical alloying and consolidated by SPS. Most ODS alloys are prepared by powder metallurgy to facilitate the formation of nanometric oxides and fine grains of the matrix phase. However, the most common consolidation methods are hot isostatic pressing or hot extrusion. Nevertheless, SPS presents many advantages, such as a very short time, minimal grain growth, and no texture. The bulk samples reveal matrix bcc with a grain size of about 1 μm and nanometric (15–50 nm) oxides. Moreover, it was found that the addition of V increases the hardness from 3.8 GPa to 4.8 GPa. The conducted annealing at 1020°C led to the homogenization of the microstructure and increased relative density above 96%.

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Wiktoria Krzyżaniak[1)], Jakub Wiśniewski[1,2)], Grzegorz Adamek[2)], Dariusz Garbiec[1)]
[1)]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland
[2)]Poznan University of Technology, Poznan, Poland

HEBM-FAST/SPS as a Way of Obtaining Composite Tool Materials from WC-Ti & WC-Ti6Al4V Powder Mixtures

Abstract

Due to the geopolitical factors and depletion of natural resources, alternatives for cobalt-based cemented carbides have been searched for many years. Nowadays, there is still no possibility to replace this tool material completely, even though there are plenty of potential candidates. The aim of the research was to produce CRM-limited composite tool materials, based on powder mixtures consisting of tungsten carbide with titanium or titanium alloy Ti6Al4V. The powders with 75% and 95% mass percentage of tungsten carbide were high-energy ball milled with rotational speed of 500 rpm and milling time of 5 minutes and 3 hours. Obtained powder mixtures were sintered by FAST/SPS with various process parameters, including compaction pressure of 50 MPa, different heating rates, sintering temperatures in the range from 1600 to 2000°C, holding times from 1 to 10 minutes and different cooling conditions. Sintered materials were examined by Vickers hardness and fracture toughness tests, due to being the key factors of using tool materials in their primary technological purpose. Compared results were used to choose materials, which are characterized by the best properties combination, which means Vickers hardness value of more than 2000 HV_{10} and fracture toughness value of more than 7 $MPa \cdot m^{1/2}$. Additionally, microstructure of chosen materials was also examined, using X-ray diffraction spectra and scanning electron microscopy, including elements distribution maps. Research showed that obtained materials have a potential of being competitive to cobalt-based cemented carbides which are used commonly as cutting inserts. Moreover, there is a possibility of manufacturing near net shape cutting tiles made from developed materials based on WC-Ti and WC-Ti6Al4V powders by FAST/SPS.

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II. POSTERS

Krzysztof Nowik[1)], Rafał Zybała[2)], Zbigniew Oksiuta[1)]
[1)]Bialystok University of Technology, Bialystok, Poland
[2)]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

Manufacturing and Properties of 12Cr Ferritic ODS Steel with Zr Addition

Abstract

Ferritic oxide-dispersion strengthened (ODS) steels belong to the group of structural nuclear materials. Chemical composition of modern ferritic ODS alloys is exclusively based on low activation elements (reduced activation ferritic steel – RAF), usually of type Fe-(12–14)Cr-W-Ti-Y_2O_3. The classic route of producing ODS steel is the powder metallurgy, which involves mechanical alloying of powders with subsequent sintering, most often by the hot isostatic pressing (HIP). However, the dissemination of sintering methods other than HIP, such as spark plasma sintering (SPS), taking into account its advantages (primarily the relatively short sintering time), may be a promising alternative. Nowadays, ODS RAF steels are characterized by good tensile and creep strength, especially at elevated temperatures, accompanied with great oxidation and neutron irradiation resistance. However, they usually exhibit poor ductility and impact fracture properties.

Initially, ODS alloys contained pure Y_2O_3 oxide in the form of unfavorable, large-sized precipitates. Later, it was established that the addition of some alloying elements in minor content (usually <1 wt.%) favors the formation of a complex Y-O based nano-oxides bonded with added element (e.g. Y-Ti-O). Although the size of oxides can be optimized by adding Ti, the more recent studies have focused on modifying their chemical composition by adding different elements, such as Al, Hf or Zr. Therefore, efforts were undertaken in this study to substitute Ti by Zr and to determine its impact on the properties of the alloy.

This work presents major outcomes of the manufacturing process of Fe-12Cr-2W-0.5Zr-0.3Y_2O_3 RAF ODS steel. The alloy was obtained by mechanical alloying of elemental powders in Ar atmosphere, which were subsequently consolidated by SPS. Principal results, especially in terms of microstructure and mechanical properties, concerning both the bulk and consolidated material, are highlighted in this study.

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Oksana Haponova[1,2], Viacheslav Tarelnyk[3], Tomasz Mościcki[1], NataliiaTarelnyk[3], Jacek Hoffman[1]
[1]Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland
[2]Sumy State University, Sumy, Ukraine
[3]Sumy National Agrarian University, Sumy, Ukraine

A New Method for the Formation of Tribotechnical Coatings by the Method of Electrospark Alloying

Abstract

The wear of the contact joints parts, in particular of the anti-friction purpose, is the reason for the disbalancing of the unit due to the change in the size of the worn parts, which leads to the instability of the equipment, loss of productivity and reduction of product quality. Molybdenum disulfide MoS_2 is a well-known solid lubricant that is chemically and thermally stable up to 600°C, which guarantees its unchanged presence in the composite both during the manufacturing process and during the operation of the material. There are known methods of surface treatment, which make it possible to obtain a sulfomolybdenum coating during sequential or simultaneous saturation of Mo and S steel surfaces. The method of electrospark alloying (ESA) expands the possibilities of obtaining tribological coatings. The purpose of the work is to improve the quality of the surface layer of steel parts involved in friction pairs by developing a method of obtaining a wear-resistant tribological coating containing molybdenum disulfide on their surface by the method of electrospark alloying. Coatings on austenitic steel were studied, because a large number of important parts of pumping equipment assemblies are made of complex alloyed steels of the austenitic class. The coating was obtained under different processing modes. Metallographic, X-ray microspectral, X-ray structural analyzes were used, microhardness and tribotechnical properties were determined for analyze the quality of the obtained coatings. As a result of studies of the process of sulfomolybdenation of metal surfaces by the ESA method, it is shown that the coatings consist of 3 zones: the upper "white" hardened layer, the diffusion zone and the base metal. The hardness of the "white" layer is up to 1438 HV, depending on the processing mode. Electron microscopic studies of the obtained coatings showed that the formed layer has a homogeneous composition. X-ray structural analysis of the coatings showed that there is a MoS_2 26–44% in the surface, depending on the processing mode. Tribological studies confirm the effectiveness of the proposed surface treatment technology based on the ESA method.

Acknowledgement

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Agata Strojny-Nędza[1)], Marcin Chmielewski[1)], Kamil Kaszyca[1)], Grzegorz Kuderski[1)]
Krystian Kowiorski[1)], Rafał Zybała[1)], Bartosz Bucholc[1)], Szymon Nosewicz[2)]
[1)]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland
[2)]Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland

Effect of Diamond Particles Size on Microstructure and Materials Properties of Cu-C Composites Manufactured by SPS Method

Abstract

Combination of extreme service conditions and complex thermo-mechanical loadings, e.g. in electronics or power industry, requires using advanced materials with unique properties. Dissipation of heat generated during the operation of high-power electronic elements is crucial from the point of view of their efficiency. Good cooling conditions can be guaranteed for instance by using materials with very high thermal conductivity, low thermal expansion coefficient and designing the heat dissipation system in an accurate manner.

These requirements can be fulfilled by the metal-diamond composites with the copper as a matrix which has high thermal conductivity. Copper-5vol.% diamond composite powders mixtures were prepared by mechanical alloying and next sintered using spark plasma sintering method (SPS). The effect of diamond particles size on the microstructure, mechanical and thermal properties were investigated in details.

The selected conditions of SPS process allowed to obtain composites with uniform distribution of carbon phase and high relative density. The developed procedure of preparation of Cu-diamond mixtures, SPS conditions, the results of investigation of microstructure (SEM) and the measurements of thermal (thermal conductivity, thermal expansion coefficient and specific heat) and mechanical properties will be presented. The obtained results confirmed that the developed composites can be useful as heat dissipation materials.

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Krystian Kowiorski[1)], Kamil Kaszyca [1,3)], Konrad Krzyżak[1)], Agata Strojny-Nędza[1)], Grzegorz Kuderski[1)], Bartosz Bucholc[1,2)], Rafał Zybała[1,4)], Marcin Chmielewski[1,5)]

[1)]Łukasiewicz Research Network - Institute of Microelectronics and Photonics, Warsaw, Poland
[2)]Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland
[3)]AGH University of Krakow, Krakow, Poland
[4)]Warsaw University of Technology, Warsaw, Poland
[5)]National Centre For Nuclear Research, Otwock, Poland

Thermal and Electrical Stability of Cu-Graphene and Ni-Graphene Materials Obtained by SPS/FAST Technique

Abstract

The continuous miniaturisation of electronic products, accompanied by increasing power density in semiconductor systems, requires the use of materials that provide rapid heat dissipation. When an electronic device is operating, the flow of electric current through its components generates heat and consequently an increase in temperature. This can lead to adverse changes in the thermophysical, mechanical and functional properties of the materials in the overheating area. Therefore, the problem of rapid of heat dissipation in electronic systems is one of the most important issues in modern electronics. issues in modern electronics. Copper and nickel are attractive as a matrix material because of good thermal and electrical conductivity, but also very good mechanical properties, like plasticity. The effect of graphene addition on the microstructure, mechanical, thermal and electrical properties were investigated in this paper. The graphene used was obtained by Hummers' method and then reduced by chemical reduction. Cu-1vol.% graphene and Ni-1vol.% graphene powder mixtures were prepared by mechanical alloying and next sintered using spark plasma sintering method (SPS). Measurements of the thermal stability of the tested materials were carried out on the basis of thermogravimetric tests Tg/DSC in a protective atmosphere in the temperature range of RT–750°C. Statistical studies of the distribution of the Seebeck coefficient and the possible correlation between the microstructure and local electrical properties of the tested material were performed. The scanning electron microscopy (SEM) was used to observe the changes occurring in the material structure. Thermal properties such as specific heat, thermal diffusivity, thermal conductivity and linear expansion coefficient as a function of temperature were analyzed.

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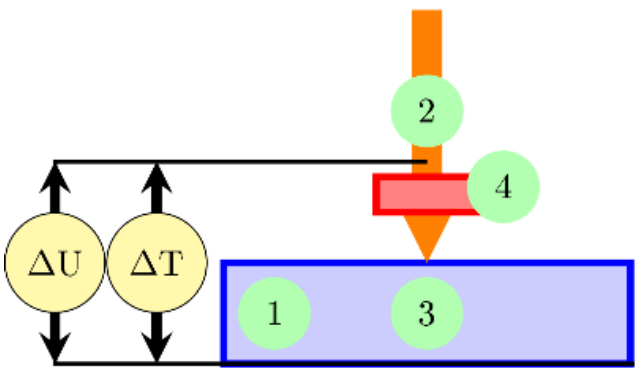
Grzegorz Kuderski[1)], Agata Strojny-Nędza[1)], Bartosz Bucholc[1)], Rafał Zybała[1)], Kamil Kaszyca [1)], Krystian Kowiorski[1)], Marcin Chmielewski[1)], Anna Gajewska-Midziałek[2)]
[1)]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland
[2)]Łukasiewicz Research Network – Warsaw Technological Institute, Warsaw, Poland

A Novel Method for Quick Characterization of Heat-Transfer Materials for Electronic Devices

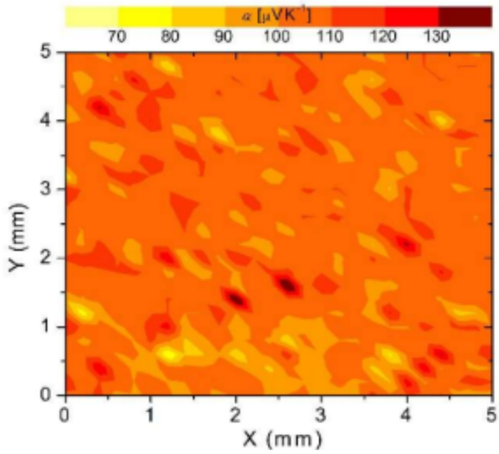
Abstract

The combination of severe operating conditions and complex thermomechanical stresses, e.g. in the electronics or energy industries, requires the use of advanced materials with unique properties. The dissipation of heat generated during the operation of high power electronic elements is very important in terms of their efficiency. Good cooling conditions can be ensured by using materials with very high thermal conductivity, low coefficient of thermal expansion and by designing the heat dissipation system correctly.

Fabrication of high quality bulk materials and layers must be supported with development of methods for their properties determination. This work presents a new method that enables a possibility of quick, non-destructive characterization of composite materials' obtained by SPS technique surface uniformity. Presented method is based on Seebeck coefficient (α) mapping – this parameter is very sensitive and allows to determine the material heterogeneity. During Measurement a heated probe contacts with a points on the surface and measures the generated Seebeck voltage – Fig. 1a shows the idea of mapping device. Seebeck coefficient is determined during a simultaneous measurement of generated voltage and temperature difference. The values of α in the function of location is stored in matrix (Fig. 1b) and represented by colormaps (Fig. 1c).



a)



b)

$$\alpha_{(x,y)} = \begin{bmatrix} \alpha_{0,0} & \alpha_{1,0} & \alpha_{2,0} & \alpha_{3,0} \\ \alpha_{0,1} & \alpha_{1,1} & \alpha_{2,1} & \alpha_{3,1} \\ \alpha_{0,2} & \alpha_{1,2} & \alpha_{2,2} & \alpha_{3,2} \\ \alpha_{0,3} & \alpha_{1,3} & \alpha_{2,3} & \alpha_{3,3} \end{bmatrix}$$

c)

Fig. 1. a) Idea of Seebeck coefficient mapping method 1 – ambient temperature, 2 – heated probe, 3 – measured sample, 4 – heater block; b) a Seebeck coefficient container matrix; c) an example colormap of Seebeck coefficient

A validation procedure used for interpretation of example results is presented with explanation of crucial parameters.

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**Konrad Krzyżak, Rafał Zybała, Marcin Chmielewski,
Kamil Kaszyca, Krystian Kowiorski, Agata Strojny-Nędza,
Bartosz Bucholc, Michał Gajewski, Grzegorz Kuderski**
Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

Precise Surface Machining of Materials Sintered by SPS Method

Abstract

The possibilities for precision machining of materials produced by spark plasma sintering are presented. Precise cutting and surface treatment (grinding, polishing) of the different material types (metals and alloys, ceramics, composites, semiconductors, etc.) into various shapes and sizes of samples is of key importance for carrying out measurements and tests for a wide range of materials application. Materials produced by sintering are often characterized by very difficult mechanical processing, which requires the use of unusual devices and tools. The available machine tool for precise cutting is characterized by a spindle with a high torque and high rigidity, and the use of diamond blades allows you to process difficult-to-process materials in the range of several micrometers. Surface treatment together with scanning electron microscopy imaging and roughness measurement allow to prepare materials for further more precise and advanced research. Presented technological equipment and professional operators experience guaranties precise, high quality and repeatability of the machined materials.

Kinga Momot[1)], Piotr Klimczyk[1)], Beata Leszczyńska-Madej[2)], Marcin Podsiadło[1)], Agnieszka Gubernat[2)]
[1)]Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland
[2)]AGH University of Krakow, Krakow, Poland

Advanced Ceramic Cutting Tools Sintered by HPHT and SPS Methods

Abstract

This paper presents a concept of ceramic composites produced at Łukasiewicz – Krakow Institute of Technology by two different sintering methods: Spark plasma sintering and high pressure – high temperature sintering. Both produced composites are intended for machining Inconel 718 superalloy. The microstructure of composites, as well as mechanical, tribological and cutting properties were investigated. Nickel-based superalloys have become an increasingly important material, particularly in the aerospace and automotive industries. However, the main problem in machining these materials is their low thermal conductivity and hardening during cutting.

The first material is a composite belonging 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 presence of thermodynamically unstable cBN, this composite was produced using the HP-HT sintering method with a pressure of 7.7 GPa, and a temperature of 2100°C, which provided optimal conditions for cBN phase preservation, thus achieving a high hardness of the entire composite (26.4 GPa). The second composite was sintered in ternary Al_2O_3–ZrO_2–WC system in the volume ratios of 42:12:46 respectively, using spark plasma sintering at 1550°C and 63 MPa. The composites were used to produce cutting tools for machining Inconel 718 alloy and were compared with commercial materials from both the superhard and ceramic groups. The materials designed at Łukasiewicz – Krakow Institute of Technology were characterised by similar cutting tool life to commercial inserts (criterion V_B=0.3) when working under the same conditions. The cBN insert performed for an average of 12 minutes, which is comparable to a commercial super-hard tool from a leading manufacturer. In contrast, the insert produced by the SPS method displayed slightly better machining ability than the commercial insert and ran for about 11 minutes.

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This work was carried out within the framework of the DuplexCER project, entitled „High performance duplex ceramics for efficient machining of nickel superalloys", co-founded by The Polish National Centre for Research and Development within the framework of the M-ERA.NET research programme (Agreement no. M-ERA.NET3/2021/82/DuplexCER/2022). Presented results are also partly derived from work carried out within the „Implementation doctorate V" programme.

Grzegorz Kubicki[1,2], Jakub Wiśniewski[1], Joanna Półrolniczak[1], Jarosław Jakubowicz[2]
[1]Łukasiewicz Research Network—Poznan Institute of Technology, Poznan, Poland
[2]Poznan University of Technology, Poznan, Poland

Application of Spark Plasma Sintered MAX Phase Powders for Cold Spraying Technology

Abstract

MAX phases are promising materials for coatings used for high-temperature applications, especially to increase wear and corrosion resistance of the coated material. In this work, HEBM-SPS technique has been used for fabrication of different MAX phase powders suitable for kinetic spraying. The microstructure of Ti_3AlC_2, Ti_3SiC_2, Ti_2AlC and Cr_2AlC powders has been studied using scanning electron microscope, showing lamellar grain structure typical to MAX phases. A hybrid aerosol cold spraying (aerosol deposition + low pressure cold spray) system has been employed in order to deposit thick MAX phase coatings, with the exception of Ti_3SiC_2 coating, where the deposition resulted in thin layer. The resulting coatings the exhibited low amount of cracks and voids. The porosity of the structure has gradually decreased as the coating was closer to the sprayed surface, signifying that continuous shot-peening/hammering effect took place, that densified previously deposited layers. The spraying process has not affected the phase structure as no additional peaks were found in the X-Ray Diffraction spectra. The coatings have shown increased resistance to wear during high temperature friction. The formation of titanium dioxide (in forms of anatase and rutile) outer layer at high temperature was proposed as the reason for increased wear resistance of coating at high temperatures. The research has proven that synthesized powders are suitable for ACS deposition that results in unique coating suitable for high temperature applications. Further measurements will be made in order to fully assess the mechanical and functional properties of the MAX phase coatings, such as nanoindentation, tensile adhesion testing and potentiodynamic testing.

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Rafał Rubach[1], Paweł Figiel[2], Anna Biedunkiewicz[2], Dariusz Garbiec[1]
[1]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland
[2]West Pomeranian University of Technology, Szczecin, Poland

Microstructure and Mechanical Properties of (Ti,Mo)C Reinforced Ti Matrix Composites Produced by FAST/SPS

Abstract

Spark plasma sintering is a modern method sintering of powder materials and is used to consolidate the powder at lower temperatures and shorter times compared to conventional powder metallurgy techniques. The allows materials characterized by high density and low grain growth to be obtained. The composites based on a titanium matrix, whose ceramic reinforcements are embedded in a metal matrix combine the properties of metals, among others plasticity and ductility as well as the properties of ceramics, such as. high hardness. By appropriately adjusting the matrix and the reinforcement in composite materials, combinations of various properties can be consciously of materials.

In this work, nanocrystalline $Ti_{1-x}Mo_xC$ (x≈0.15) powder manufactured by sol-gel method, and mixed with high purity, microcrystalline titanium (APS <45µm) in the high-energy ball milling (15 min/400 RPM) was used to obtain homogenous mixtures powder to the production of titanium matrix composites (TMCs). The content of ceramic reinforced was varied by 10, 20, and 80 wt%. Prepared powder mixtures were spark plasma sintered using two types of regimes: typical one with a heating rate of 100°C/min and holding time 10 mins, with sintering at temperature ≥1300°C under compacted pressure ≥50 MPa, and a rapid one with a heating rate 500°C/min and holding time 10 mins, sintered at temperature ≥1300°C under compacted pressure ≥50 MPa. In this study, the density, hardness, and fracture toughness of sintered compacts were analyzed. The sintered compacts were characterized by high density and increased Vickers hardness HV_{30} by more than 100% than sintered compact without reinforcement. Besides, the microstructural observation by SEM-EDS was conducted. The phase composition and crystalline size were determined by XRD.

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Jakub Wiśniewski, Wiktoria Krzyżaniak, Dariusz Garbiec
Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland

Tribological Behavior in a Wide Temperature Range of Ti_3AlC_2 MAX Phase Obtained by FAST/SPS

Abstract

MAX phases, a family of layered ternary carbides or nitrides with the general formula $M_{n+1}AX_n$ (n=1, 2, or 3), have garnered significant attention in recent years due to their exceptional combination of metallic and ceramic properties. Among these MAX phases, Ti_3AlC_2 has emerged as a promising material for various high-temperature applications, owing to its remarkable thermal stability, mechanical strength, and oxidation resistance. This poster presents a comprehensive investigation into the tribological behavior of the Ti_3AlC_2 MAX phase over an extensive temperature range. Our study focuses on the material's frictional properties and specific wear rates, with a particular emphasis on its performance at elevated temperatures. The Ti_3AlC_2 MAX phase specimens were synthesized via the FAST/SPS method to ensure high-density, phase-pure samples. Tribological tests were conducted using a pin-on-disk configuration, covering temperatures ranging from room temperature to 700°C. At elevated temperatures, the Ti_3AlC_2 MAX phase demonstrates an intriguing tribological behavior. The material exhibits a decreasing coefficient of friction in increasing test temperature, reaching 0.16 at a maximum test temperature of 700°C, which is more than five times lower than in the case of testing at room temperature (0.84). This behavior can be attributed to the unique combination of its intrinsic properties, including its layered structure and excellent thermal stability, as well as its self-lubricating effect. Our findings highlight the exceptional tribological performance of the Ti_3AlC_2 MAX phase across a wide temperature range, making it a promising candidate for applications requiring both high-temperature stability and minimal wear. This research contributes to our understanding of MAX phases and their potential in advanced engineering and industrial applications.

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**Maria Wiśniewska[1,3], Alexander M. Laptev[1], Alex Sullivan[2],
Luca Celotti[2], Mirosław Szybowicz[3], Dariusz Garbiec[1]**
[1]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland
[2]Azimut Space GmbH, Berlin, Germany
[3]Poznan University of Technology, Poznan, Poland

3YSZ-10CNT Composites Manufactured by Spark Plasma Sintering Technology

Abstract

The space industry is one of the areas in which dependable materials of the highest value should be used. spark plasma sintering (SPS), also known as field assisted sintering techniques (FAST) was used to manufacture demonstrators of heat shields suitable for use in that industry. However, when manufacturing relatively large parts from non-electrical conductive powders (with a diameter of 75 mm) using FAST/SPS, there is a risk of thermal gradients and thus a non-homogenized microstructure of sintered bulks. To counteract this, 10 vol.% carbon nanotubes were added to partially stabilized zirconia powder with a uniform dispersion of 3 mol% yttria (3YSZ), which increased its electrical conductivity. This, combined with an optimized graphite tool setup, helped reduce thermal gradient up to 40°C. The development of tool geometry allowed the manufacturing demonstrators by near net shape FAST/SPS process. Additionally, FAST/SPS parameters like temperature, compaction pressure, heating rate, and holding time were also optimized. The samples were then analyzed using scanning electron microscopy, and their relative density, Vickers hardness, and thermal properties were measured. The resulting thermal conductivity was below 2.5 W/mK in the temperature range of RT–700°C. Furthermore, SEM micrographs and element distribution maps confirmed the uniform distribution of CNTs in the zirconia-based composites. X-ray diffraction was conducted for phase analysis and indicated the presence of both tetragonal and monoclinic phases in powder mixtures, whereas in the sintered sample only the tetragonal phase was present. The surface of the heat shield demonstrator was coated at the end with a protective coating using vacuum cold spraying (VCS) technology.

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Maria Wiśniewska[1], Mateusz Marczewski[1], Alexander M. Laptev[1], Agnieszka Krawczyńska[2], Małgorzata Lewandowska[2], Tomasz Mościcki[3], Dariusz Garbiec[1]

[1]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland
[2]Warsaw University of Technology, Warsaw, Poland
[3]Institute of Fundamental Technological Research Polish Academy of Sciences, Warsaw, Poland

Process of Up-Scaling the FAST/SPS Method Based on Production Sputtering Targets

Abstract

Field assisted sintering techniques/spark plasma sintering (FAST/SPS) technique is widely used in powder metallurgy technology as one of the non-conventional sintering techniques, which is based on the electrical, thermal, and mechanical fields that interact. FAST/SPS technique has a lot of significant advantages, like a limitation in grain growth, a lower sintering temperature, and a much shorter sintering time. One of the limitations of using this method is the maximum size of the obtained samples, which results from the technical parameters of the device. This work presents the process of upscaling the diameter of produced samples. All tests were performed on an HP D 25 SPS furnace from FCT Systeme. Performed FEM modeling allowed the design of tool geometry that was used for the manufacturing samples of 100 mm in diameter, which is 1.2 times bigger than the maximum diameter declared by the producer. Additional plates and spacers, both made of carbon-reinforced fiber carbon (CFRC), and full thermal insulation of the tool setup were applied. It allowed for a significant reduction in energy consumption during the sintering. Optimization of the tool design as well as process parameters allows to manufacturing of multi-component sputtering targets with a diameter of 100 mm, which will be used for the deposition of thin coatings by high-power impulse magnetron sputtering (HiPIMS). The sintered targets were characterized using scanning electron microscopy and energy-dispersive X-ray spectroscopy for microstructure analysis, X-ray diffraction for phase identification, and measurements of density and electrical conductivity. A difference in density in the diameter was observed, nevertheless, a homogeneous phase composition was present, which proved the effectiveness of the upscaling for the target manufacturing.

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Joanna Półrolniczak[1),2)], Grzegorz Kubicki[1),2)],
Mateusz Marczewski[1)], Jarosław Jakubowicz[2)]
[1)]Łukasiewicz Research Network – Poznań Institute of Technology, Poznan, Poland
[2)]Poznan University of Technology, Poznan, Poland

Synthesis of Ti_2AlC Using Spark Plasma Sintering

Abstract

Ti_2AlC is a member of the MAX phases family, which can be also named binary metal carbide or nitride, where M is a transition metal, A is an A-group element, and X is carbon or nitrogen. Multilayer compounds possess a combination of properties of ceramics and metals such as low density, low hardness, low friction coefficient, high thermal shock resistance, high electrical conductivity, good machinability, excellent corrosion and oxidation resistance. There are many possible applications for MAX phases, thanks to these properties, for example in engine components exposed to high temperatures and friction wear.

This work presents selected results from the two-step synthesis of phase Ti_2AlC. In the first step, the powder has been mechanically alloyed in a planetary ball mill. The pre-prepared powder has been analysed for phase composition using X-ray diffractometry. After a few hours of mechanical alloying phase changes have been analysed. The powder prepared in this way has been sintered using FAST/SPS at temperatures in the range from 1100 to 1300°C. To confirm the synthesis and phase composition, the obtained sinters have been also analysed by X-ray diffractometry.

The combination of mechanical alloying and spark plasma sintering gave excellent results. Work on the synthesis of phase X resulted in obtaining a sample with almost 100% phase purity. In the next step, the developed methodology for obtaining the pure MAX phase will be used to produce samples for tribological tests and for testing mechanical properties.

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Marcin Brykała, Wojciech Chmurzyński, Marcin Chmielewski, Magdalena Gawęda, Kinga Suchorab, Katarzyna Mulewska, Jarosław Jasiński
National Centre for Nuclear Research, Otwock-Swierk, Poland

Effects of Various Synthesis Methods and Additives on the Properties of Sintered Doped Zirconia for the Nuclear Applications

Abstract

One of the alternative for utilization the minor actinides separated from spent nuclear fuel is the incorporation into the inert ceramic matrices and transmutation to short-life nuclides. The most effective candidate for this purpose is cubic zirconia. The identifying synthetic methods that yield reproducible control over shape, size uniformity, volume and homogeneity of incorporated Minor Actinides is important. At this study, materials-substrates obtained by two different techniques were compiled and compared. The first technique was "wet route" synthesis using the complex sol-gel process (CSGP), and the second "dry route" based on a mechanical alloying process in a planetary ball mill under the argon atmosphere. Both techniques were applied to synthesis stabilized zirconia with neodymium or/and cerium oxide with content of 10 mol% as surrogates of potential minor actinides. The spark plasma sintering (SPS) technique was applied to consolidate the prepared powder mixtures. Afterwards, material microstructure, phase composition and properties were assessed using ICP-MS&LIBS, TG-DSC, dilatometry, laser flash method, Raman spectroscopy, SEM observation and SEM-EDS mapping, XRD phase analysis, Vickers hardness and surface nanoindentation testing. The presented results enable significant in-depth characterization of this critical material that could operate in extreme temperature conditions and high radioactivity. In addition, it is possible to assess the impact of the selected method of powders preparation and type of additives on the properties of the sintered material. Such a comparison is also important in the context of obtaining other advanced materials used in nuclear reactors.

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Julia Popis[1], Sabina Lesz[1], Małgorzata Karolus[2]
[1]Silesian University of Technology, Gliwice, Poland
[2]University of Silesia, Chorzow, Poland

Characteristics of Fe-B-La Alloys Used for Hydrogen Storage

Abstract

Currently, in the energy market, natural resources such as crude oil, natural gas, and brown coal are predominantly used. Projections regarding energy needs create a necessity to search for new alternative energy sources. Hydrogen can be an innovative material that replaces the currently used natural resources. The reason for this is the significant energy (approximately 120 MJ/kg) obtained through the combustion process of hydrogen, as well as the absence of pollutants generated in this process, as water is the resulting product. The main challenge lies in the storage and transportation of hydrogen in its gaseous form. High-pressure tanks used for its storage are large, require low temperatures, and involve additional costs (e.g., liquefaction).

A future-oriented solution is the storage of hydrogen through absorption in solid materials (alloys, composites, intermetallic compounds). Materials used for hydrogen storage in applications such as automotive industry should have high gravimetric density, easy absorption/desorption at moderate temperatures and pressures, low material cost, and ecological safety. Therefore, the aim of the project is to produce Fe-B-La alloys selected based on literature research and investigate their properties. However, unlike the mentioned manufacturing methods, they will be produced using mechanical alloying (MA) method.

The $Fe_{82}B_{16}La_2$ and $Fe_{82}B_{14}La_4$ alloys were produced using the method of mechanical alloying and then subjected to sintering, for example, using the spark plasma sintering (SPS) technique.

The powders obtained from the mechanical alloying process were subjected to X-ray diffraction (XRD) and scanning electron microscopy (SEM) analysis to examine their structure and morphology. Subsequently, the sintered samples will be examined using X-ray diffraction for phase composition analysis and scanning electron microscopy for detailed microstructural analysis. Electrochemical impedance spectroscopy (EIS) will be conducted to investigate the electrochemical properties of the fabricated material.

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**Alexander Ahrend[1], Olaf Keßler[1], Hermann Seitz[1],
Marcel Wetegrove[2], Abdullah Riaz[1]**
[1]University of Rostock, Rostock, Germany
[2]Leibniz Institute for Plasma Science and Technology, Greifswald, Germany

Development and Characterization of a High-Entropy Alloy (HEA) for Catalysts in Anion-Exchange Membrane Water Electrolysis

Abstract

High-entropy alloys (HEAs) are alloys consisting of multiple (at least five) homogeneously distributed elements in one solid solution phase. Due to their material variety, new properties for different applications can be achieved. In this case, a HEA with high activity in the hydrogen evolution reaction (HER) under alkaline conditions was developed to be used as a catalyst in anion-exchange membrane water electrolysis. Therefore, a HEA with common and affordable elements in an equiatomic ratio ($Al_{20}Co_{20}Cr_{20}Fe_{20}Ni_{20}$) was chosen to compare the electrocatalytic activity to HEAs with rare and expensive elements (e.g. Pt, Au, Ti).

HEAs can be prepared from many metallurgical operations. The chosen HEA was mechanical alloyed followed by consolidation using field assisted sintering technique (FAST), also called spark plasma sintering (SPS). Firstly, the particle size, distribution and morphology were investigated depending on different milling times, rotation speeds, ball sizes and ball-to-powder-weight-ratios (BPWR) in a planetary and high-energy ball mill. Afterwards, the composition and microstructure of the HEA phases were studied by X-ray diffraction (XRD), energy dispersive X-ray analysis (EDX) and scanning electron microscopy (SEM). The HEA powder milled for 48 h with 300 rpm, a ball size of 10 mm and a BPWR of 10:1 in the planetary ball mill were most homogeneously distributed in particle size (50 µm) and consisted of a single phase (fcc, a=3.650 Å).

This powder was successfully consolidated by SPS at 1.000, 1.100 and 1.200°C with a pressure of 50 MPa and a heating rate of 100 K/min. The sintered targets (Ø: 20 mm, h: 5 mm) will be characterized in density, microstructure and composition followed by a magnetron sputtering process. With these experimental investigations in combination with numeric simulations, the final aim is to sinter targets of 4 inches diameter for the catalytic application in anion-exchange membrane water electrolysis.

Acknowledgement

The CatHEA project 'Metal-supported membrane electrode units with high-entropy alloy cathodes for alkaline polymer membrane water electrolysis' is being carried out in

collaboration with the Leibniz Institute for Plasma Science and Technology (INP Greifswald) and Center for Fuel Cell Technology (ZBT Duisburg).

The overarching goal of this project is the production of a functional model of a low-cost, precious metal-free, high-performance membrane electrode unit for alkaline electrolysis.

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Natasha Sweeney Fort[1], Richard Thackray[1], Hugh Hamilton[2], Gill Thornton[3], Martha Briceno de Gutierrez[2], Xinjiang Hao[3], Andy Fones[2]
[1]University of Sheffield, Sheffield, England
[2]Johnson Matthey Technology Centre, Sonning Common, England
[3]Liberty Powder Metals, Middlesborough, England

Effect of Heat Treatment on Microstructure and Corrosion Behaviour of FAST Processed Cathodically Modified 17-4PH Stainless Steel

Abstract

Corrosion resistance is a principal feature of stainless steels due to the formation of an adherent, passive Cr-rich oxide. However, when operating in reducing acidic environments, such as those found in fuel cells, this protective layer is subject to dissolution, which exposes the substrate to the corrosive media. Stainless steels' ability to resist corrosion can be improved by alloying with platinum group metals – a process known as cathodic modification.

17-4PH is a martensitic, precipitation hardenable stainless steel. Heat treating 17-4PH causes the precipitation of Cu-rich particles. By impeding the motion of dislocations, the Cu precipitates strain the lattice and produce a strengthening effect. Typically, heat treating precipitation hardening stainless steel also enhances corrosion resistance due to the homogenisation of alloying elements. This study presents the impact of heat treatment on cathodically modified stainless steel – a topic which has not been previously researched.

Field assisted sintering (FAST) was used to consolidate water atomised 17-4PH stainless steel doped with 0.5 wt.% Ru and 0.5 wt.% Pd under varying sintering temperatures (1000–1100°C) and times (300–900 s). The sintering pressure and heating rate were fixed at 50 MPa and 100°C/min, respectively. The as-sintered samples were subjected to the H1075 heat treatment which involved solution treating at 1050°C for 0.5 hours followed by a precipitation hardening step at 579°C for 4 hours. The microstructure and elemental composition of the as-sintered and heat treated materials were analysed using optical microscopy, SEM, EDS and TEM. Open circuit potential and potentiodynamic polarisation techniques evaluated the corrosion behaviour of the as-sintered and heat treated samples in 0.05 M H_2SO_4 at room temperature. The results showed that Cu-rich particles approximately 20 nm in size precipitated following heat treatment. Ru and Pd doped samples sintered for 300 s showed the best precipitation hardening response following heat treatment (429 HV_1 and 427 HV_1, respectively). All other samples showed either little change or deterioration in hardness following heat treatment.

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**Jarosław Woźniak, Mateusz Petrus, Dorota Moszczyńska,
Tomasz Cygan, Kamil Broniszewski, Andrzej Olszyna**
Warsaw University of Technology, Warsaw, Poland

The Consolidation of WC Ceramics Using MAX Phases As a New Family of Sintering Activators

Abstract

Non-oxide ceramic materials, like silicon carbide, tungsten carbide, or silicon nitride, show several unique properties compared to other materials. They exhibit fascinating properties, including high melting temperature, extraordinary hardness, exceptional stiffness, high resistance to chemical agents, and stability of their properties over a wide temperature range. Unfortunately, achieving full dense ceramic sintered compacts is difficult due to their strong covalent bond, low grain-boundary diffusion coefficient, and the inevitable presence of oxide contaminations on the raw materials. Consequently, it is necessary to apply high processing temperatures, high pressure, novel sintering technique, or the use of sintering aids. Another of their disadvantages, significantly limiting the application potential, is the low fracture toughness. One of the methods of increasing fracture toughness is the production of ceramic matrix composites. The ideal material to act as a reinforcement should improve fracture toughness without reducing other properties, such as hardness or thermal stability. One of the latest trends in research on the production of dense ceramic sinters with high mechanical properties is the simultaneous use of novel sintering techniques combined with the use of a new group of sintering aids. The new group of sintering additives uses the phenomenon of decomposition of the phases used to produce compounds responsible for the intensification of sintering processes and also playing the role of reinforcing phase. One such material group is the MAX phases, which have been gaining popularity recently. During their decomposition, products are formed that react with oxides on the surface of ceramic particles, modifying the sintering process through transient plastic phase processing and introducing the liquid phase sintering (LPS). So far, the influence of the Ti_3AlC_2 phase on the sintering process of such TiB_2, B_4C, and ZrB_2 materials has been described. It has been shown that the sintered temperature is reduced with a simultaneous improvement in mechanical properties.

Moreover, the world literature lacks research on the influence of MAX phases on other ceramic materials, such as SiC, WC, or Si_3N_4. Their use has potentially many benefits. It will enable sinter production with high mechanical properties while significantly reducing the sintering temperature. It will also allow for greater control and the possibility of designing the microstructure. The scientific goal is to provide new knowledge on MAX phases, which simultaneously act as a sintering additive facilitating the densification process and as a precursor for the formation of a reinforcing phase. The WC+xTi_3AlC_2 composites were

produced (x=0, 3, 6, 9 vol.%). The influence of the MAX phases on the degree of sinter consolidation, as well as the mechanical properties of the sinter (i.e., hardness, fracture toughness), was tested. In addition, the sinters were subjected to microstructure analysis using SEM microscopy.

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**Kamil Broniszewski, Jarosław Woźniak, Tomasz Cygan,
Marek Kostecki, Andrzej Olszyna**
Warsaw University of Technology, Warsaw, Poland

Mechanical and Electrical Properties of SiC – Graphene Composites Obtained Using Spark Plasma Sintering

Abstract

Silicon carbide (SiC) matrix composites have garnered significant attention in materials science and engineering due to their exceptional mechanical and thermal properties. In recent years, the incorporation of graphene into SiC composites has emerged as a promising approach to enhance their overall performance. This study investigates the effects of graphene addition on the mechanical properties and electrical conductivity of SiC-based composites, produced using the spark plasma sintering (SPS) method. The utilization of SPS proved to be a pivotal factor in fabricating high-density SiC-graphene sintered compacts at the temperature of 2000°C. The fabrication process involved the milling of powder substrates, followed by compaction and densification using the SPS technique. The composites were prepared with varying graphene content (0.25–3 %wt.) to analyze its influence on the final properties. Scanning electron microscopy (SEM) and X-ray diffraction (XRD) were utilized to examine the microstructure and phase composition of the composites. The investigation of physical properties was an essential aspect of this study to comprehend the overall behavior of the SiC-graphene composites. The density of the fabricated composites was measured using the Archimedes method to evaluate the degree of densification achieved through the SPS process. It was observed that the incorporation of graphene did not significantly alter the density of the composites. Additionally, Young's modulus was measured using the ultrasonic technique. The mechanical properties of the SiC-graphene composites were evaluated through comprehensive testing, including hardness and fracture toughness measurements. Moreover, the electrical conductivity of the SiC-graphene composites was investigated. It exhibited a considerable enhancement due to the introduction of graphene.

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Tomasz Cygan, Marek Kostecki, Jarosław Woźniak, Andrzej Olszyna
Warsaw University of Technology, Warsaw, Poland

Sintering Behaviour, Microstructure and Mechanical Properties of Ultra-High Temperature Ceramics from TiB_2/ZrB_2-SiC/Graphene Systems

Abstract

Ultra-high temperature ceramics (UHTCs), are defined as ceramic materials with melting point higher than 3000°C. Most of them are binary compounds where boron, carbon, or nitrogen combine with one of the early transition metals (Zr, Hf, Ti, Nb, Ta). This result in strong covalent bonds which are responsible for their high melting point, high hardness, good wear resistance and chemical stability. In addition, due to varying degrees of metallic bond character, they exhibit higher electrical and thermal conductivities than oxide ceramics. Such combination of properties allow ultra-high temperature ceramics to work in extreme conditions of high temperature, radiation levels, chemical reactivities or heat fluxes.

In particular, UHTCs are investigated for use in aerospace industry as hypersonic vehicles, scramjet propulsion and rocket components. Although, from such specific applications emerge also specific requirements. In hypersonic flight, one of the challenges is the bow shock at sharp leading edges, producing temperatures in excess of 2000°C, high heat fluxes, as well as highly reactive dissociated gas species. Due to high thermal conductivity, superior oxidation resistance and high strength at elevated temperatures, diboride ceramics (like ZrB_2, HfB_2, TiB_2) are most often investigated for such applications among UHTCs. What limits it wider use though, is poor sinterability and moderate fracture toughness.

The aim of this work was to obtain dense diboride ultra-high temperature ceramics with improved mechanical properties, by preparing composites from TiB_2/ZrB_2-SiC/graphene systems. Materials were produced with the use of powder metallurgy and consolidated using the Spark Plasma Sintering technique. The essential properties (density, hardness, Young's modulus, fracture toughness) of the composites were thoroughly investigated, and in depth analysis of microstructure was conducted. As summary, the influence of the SiC/graphene addition on various properties of TiB_2/ZrB_2 ultra-high temperature ceramics was presented.

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**Anna Dobkowska, Aleksandra Zielińska, Mirosław Jakub Kruszewski,
Bogusława Adamczyk-Cieślak, Jarosław Mizera**
Warsaw University of Technology, Warsaw, Poland

Corrosion Resistance of 316L Oxide Dispersion Strengthened (ODS) Steel

Abstract

High-temperature and harsh conditions require the use of advanced and highly resistant materials. Nuclear reactors are a very good example of an environment with such conditions, and oxide dispersion strengthened (ODS) 316L steel is suitable to build them. This is a material with desirable properties such as good workability, ductility, and mechanical integrity at very high temperature exceeding 1000°C.

In this study, to improve the corrosion properties of 316L steel, yttrium was added to the matrix, to form oxides *in situ* during fabrication. Materials for this research were manufactured through powder metallurgy techniques and subsequently compared with the reference materials (316L commonly available rods). The effectiveness of the yttrium addition in corrosion resistance improvement was investigated in acidic solutions (water-based solution of HNO_3). The electrochemical tests and corrosion rate measurements were carried out to determine the corrosive properties. Next, the corrosion damage on the surfaces after immersion in acidic solution was characterized using scanning electron microscopy (SEM) and optical profilometry.

The results revealed that the addition of 5% wt. Y caused the formation of Y_2O_3 which is confirmed by XRD analyses. Nevertheless, $Cr_{23}C_6$ also form, and its presence may decrease mechanical and corrosion properties of the steels. The additional coating of BN used during SPS protect the bulk material and limits carbon contamination during sintering. The addition of 5% wt. Y increase the corrosion resistance of 316L, however, localized corrosion is still observed on the surface of the alloy.

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NOTES

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