1st CONFERENCE ON FAST/SPS
From Research to Industry

BOOK OF ABSTRACTS

POZNAN, 25-26.10.2021

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CONFERENCE PROGRAMME

DAY 1
OCTOBER 25, 2021 (MONDAY)

09.00–09.30	**Registration of participants**
09.30–09.45	**Opening of the 1st day of the Conference**
09.45–10.30	**KEYNOTE PRESENTATION** Session chairman: Dariusz Garbiec
09.45–10.30	**Application of FAST/SPS for the processing of advanced materials** (stationary) Olivier Guillon, Institute of Energy and Climate Research: Materials Synthesis and Processing (IEK-1), Forschungszentrum Jülich GmbH, Jülich, Germany
10.30–10.50	**COFFEE BREAK**
10.50–12.10	**SESSION I** Session chairman: Alexander Laptev
10.50–11.10	**Structural changes and pseudo-piezoelectricity in FAST/SPS prepared calcium titanate – towards novel implant materials** (stationary) Abdullah Riaz, University of Rostock, Rostock, Germany
11.10–11.30	**Ceramic particulate reinforced aluminium alloys consolidated by spark plasma sintering for brake disc applications** (stationary) Johannes Trapp, Fraunhofer Institute for Manufacturing and Advanced Materials (IFAM), Dresden, Germany
11.30–11.50	**Numerical determination of the effective thermal conductivity of porous material manufactured by FAST/SPS** (stationary) Jerzy Rojek, Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland
11.50–12.10	**ENGEMINI: A digital asset paving the way towards SPS industrialization** (stationary) Antoine Van der Laan, Norimat, Labège, France
12.10–12.30	**COFFEE BREAK**
12.30–13.50	**SESSION II** Session chairman: Piotr Klimczyk
12.30–12.50	**Development of MMC, CMC and MAX-based composites by FAST/SPS** (stationary) Dariusz Garbiec, Łukasiewicz Research Network – Metal Forming Institute, Poznan, Poland
12.50–13.10	**Bimodal grain size structure formation by SPS and severe deformation** (stationary) Volf Leshchynsky, Łukasiewicz Research Network – Metal Forming Institute, Poznan, Poland

13.10–13.30	**Impact of CFRC plates on energy and temperature during spark plasma sintering** (stationary) Alexander Laptev, Łukasiewicz Research Network – Metal Forming Institute, Poznan, Poland
13.30–13.50	**Development of high entropy alloys by field assisted sintering using commodity powders** (stationary) Venkatesh Kumaran Sivagnana, IMDEA Materials Institute, Getafe, Madrid, Spain
13.50–15.00	**LUNCH**
15.00–16.40	**SESSION III** Session chairman: Abdullah Riaz
15.00–15.20	**Recycling of out of specification AM metal powders using FAST** (remotely) Simon Graham, The University of Sheffield, Sheffield, UK
15.20–15.40	**FAST-AM: A novel technique for creating Ti site-specific properties via additive manufacturing** (remotely) Cameron Barrie, The University of Sheffield, Sheffield, UK
15.40–16.00	**Deformation behaviour of a FAST diffusion bond processed from Ti-6Al--4V/Ti-6Al-2Sn-4Zr-2Mo powders** (remotely) Oliver Levano Blanch, The University of Sheffield, Sheffield, UK
16.00–16.20	**Channeling current to create unique temperature profiles in titanium alloys during FAST processing** (remotely) James Pepper, The University of Sheffield, Sheffield, UK
16.20–16.40	**Hall-Petch effect in binary and ternary alumina / zirconia / spinel composites** (remotely) Olivia Graeve, University of California San Diego, La Jolla, USA
16.40–17.00	**COFFEE BREAK**
17.00–18.20	**SESSION IV** Session chairman: Olivier Guillon
17.00–17.20	**Deformation behaviour of a novel Ni-based superalloy processed via spark plasma sintering** (remotely) Antonio Potenciano, Universidad Carlos III de Madrid, Leganés, Madrid, Spain
17.20–17.40	**The influence of composition in the creep strength of a ferritic ODS steel consolidated by SPS** (remotely) Alberto Meza, IMDEA Materials Institute, Getafe, Madrid, Spain
17.40–18.00	**Spark plasma sintering consolidation of ceramics based on Portland clinker** (remotely) Jose M. Torralba, IMDEA Materials Institute, Getafe, Madrid, Spain; Universidad Carlos III de Madrid, Leganés, Madrid, Spain
18.00–18.20	**Heating pathways in spark plasma sintering** (remotely) Eugene A. Olevsky, San Diego State University, San Diego, USA
18.20–18.40	**COFFEE BREAK**
18.40–19.00	**SESSION V – POSTER SESSION** (STATIONARY)
20.00–22.00	**CONFERENCE DINNER**

DAY 2
OCTOBER 26, 2021 (TUESDAY)

09.00–09.10	**Opening of the 2nd day of the Conference**
09.10–10.30	**SESSION VI** Session chairman: Johannes Trapp
09.10–09.30	**Ultrashort sintering of amorphous Zr-based AMZ4 bulk metallic glass and preparation of Cu-based AMC4 and Diamond composite in GeniCore U-FAST machine** Maweja Kasonde, GeniCore Sp. z o.o., Warsaw, Poland
09.30–09.50	**FAST/SPS in industrial production** (stationary) Jens Huber, Dr. Fritsch Sondermaschinen GmbH, Fellbach, Germany
09.50–10.10	**GeniCore – Examples of PPC and SPS made materials in industrial applications** (stationary) Damian Karpowicz, GeniCore Sp. z o.o., Warsaw, Poland
10.10–10.30	**Scaling up the production of short fiber reinforced – ultra high temperature composites by SPS** (remotely) Miguel A. Lagos, TECNALIA, Donostia-San Sebastián, Spain
10.30–10.50	**COFFEE BREAK**
10.50–12.10	**SESSION VII** Session chairman: Jerzy Rojek
10.50–11.10	**Effect of spark plasma sintering on microstructure and properties of Alumix-based composites reinforced with B4C particles** (remotely) Anna Wąsik, AGH University of Science and Technology, Krakow, Poland
11.10–11.30	**Characteristics of the surface layer of Ti-Mo/TiC nanocomposites obtained by Field Assisted Sintering Technique** (stationary) Paweł Figiel, West Pomeranian University of Technology, Szczecin, Poland
11.30–11.50	**SHS synthesis of tellurides using SPS apparatus** (stationary) Kamil Kaszyca, Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland
11.50–12.10	**Properties of spark plasma sintered bulks and coatings made of tungsten diboride alloyed with Cr, Mo, Re and Zr** (stationary) Rafał Psiuk, Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland
12.10–13.10	**CLOSING OF THE CONFERENCE / LUNCH**
13.10–14.00	**Visiting Łukasiewicz Research Network – Metal Forming Institute**

CONTENTS

I. ORAL PRESENTATIONS

Olivier Guillon, Martin Bram
Institute of Energy and Climate Research: Materials Synthesis and Processing (IEK-1),
Forschungszentrum Jülich GmbH, Jülich, Germany

Application of FAST/SPS for the processing of advanced materials

Abstract

Field Assisted Sintering Technique / Spark Plasma Sintering and related Electric Current Assisted Sintering techniques enable to process materials and components relevant to many applications. This talk will give an overview of the possibilities offered to develop and produce advanced ceramic, metals and composite materials for energy conversion and storage devices. Oxides like gadolinium-doped ceria, yttria, lithium cobalt oxide and lithium lanthanum zirconate, non-oxide ceramics like MAX phases, metals like tungsten alloys will be considered as examples.

The following aspects will be addressed:
- developing strategies to sinter materials which are prone to evaporation, chemical expansion or decomposition;
- taking advantage of alternative tool materials to increase the maximum pressure;
- developing strategies to sinter high-temperature and refractory materials;
- reducing temperature gradients to avoid microstructure gradients and residual stresses;
- scaling up of the technology while maintaining homogeneous parts;
- combining FAST/SPS and flash sintering/cold sintering technologies.

Reference:
M. Bram et al., Adv. Eng. Mater. 2020, 22, 20000051.

Acknowledgement

Parts of the work were funded by the Deutsche Forschungsgemeinschaft (DFG) in the framework of the priority program SPP 1959 (Fields Matter). The work on battery materials was funded by the Bundesministerium für Bildung und Forschung (BMBF) in the framework of the EvaBATT project.

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

Structural changes and pseudo-piezoelectricity in FAST/SPS prepared calcium titanate – towards novel implant materials

Abstract

The treatment of critical size bone defects is still a challenge. If bone defect exceeds a certain limit, then external material is needed to support bone and to guide tissue regeneration by physical stimulation. Promising effects of electrical stimulation on bone cell growth has let to interest in using piezoelectric ceramics for tissue repair. Nevertheless, the usage of piezoelectric ceramics is still concerning due to the toxicity of commonly known piezoelectric ceramics, which exhibit ion dissolution in biological fluids. Thanks to calcium titanate, this limitation can be overcome because it is a non-cytotoxic ceramic material. Although, it has an orthorhombic crystal structure and do not exhibit piezoelectric behaviour. However, in the present study such behaviour is observed in nanostructured pure and doped calcium titanate prepared by sol-gel synthesis and FAST/SPS. This behaviour is referred to as pseudo-piezoelectricity since it is generated by distorted structure which is formed during densification by FAST/SPS. *In-situ* high-energy X-ray diffraction studies have been performed to investigate this behaviour. Strain and defects in the nanostructured bulk material led to its piezoelectric response. It is also observed that doping of nanostructured calcium titanate increased the strain and defects in the structure of calcium titanate compared to the pure one. This led to a stronger pseudo-piezoelectric effect in the doped samples. The piezoelectric constants are comparable with the piezoelectric constants of natural bone. Moreover, the pseudo-piezoelectric behavior can be tuned by doping. This particular response of pure and doped calcium titanate is of great interest in biomedicine because it can facilitate cell-material interaction and trigger bone cells for their growth.

Acknowledgement

This work was financially supported by the German Research Foundation Graduate School *welisa* (project no. 1506).

Johannes Trapp[1], Thomas Schubert[1], Andreas Storz[2], Thomas Weißgärber[1]
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[2]Sigma Materials, Erkrath, Germany

Ceramic particulate reinforced aluminium alloys consolidated by spark plasma sintering for brake disc applications

Abstract

Despite their attractive mechanical and physical properties, aluminium-based metal matrix composites (AMC's) have not yet been able to meet expectations of a widespread use. This relates mainly to the difficulties in processing and machining of these materials, which make them very costly compared to conventional solutions. Mainly friction applications, e.g. brake discs, have long been a market of such aluminium matrix composites.

Using Spark Plasma Sintering (SPS), it is possible to produce fully dense net-shape bulk compacts in very short processing times. The densification behaviour and sintering response of SiC particle reinforced aluminium alloys consolidated by using SPS was studied depending on the processing parameters. The electrical conductivity of the powder mixtures plays a crucial role within the SPS process. In case of no electrical contact to the used steel die, a minimum electrical conductivity of the powder is a need for a fast heating up. Highly loaded Al/SiC powder mixtures especially show a significant drop of the conductivity depending on the SiC content. With a sufficient pre-treatment of the powder mixtures or compound powders, a beneficial increase of their electrical conductivities and consolidation behaviour can be achieved.

Jerzy Rojek[1], Szymon Nosewicz[1], Tharmaraj Ramakrishnan[1],
Kamil Kaszyca[2], Marcin Chmielewski[2]
[1]Institute of Fundamental Technological Research, Polish Academy of Sciences, Warsaw, Poland
[2]Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland

Numerical determination of effective thermal conductivity of porous materials manufactured by FAST/SPS

Abstract

Numerical model aimed to determine the effective thermal conductivity of porous materials manufactured by FAST/SPS will be presented. The model will be developed within the discrete element method (DEM). The DEM is a relatively new modelling method in which material is represented by an assembly of spherical particles interacting with one another. Sintered porous media can be considered as sphere particles connected by necks, which are created during sintering process. Therefore the discrete element method employing bonded spherical particles is a suitable tool to model thermal problems of such systems.

The authors' own formulation of the discrete element method for heat conduction analysis has been developed. The model is based on the thermal pipe-network approach. It employs lumped capacitances concentrated at the particles centres which are connected by heat conducting bars (thermal pipes). The governing equations are based on the balance of the rate of heat storing in the lumped capacitances and rate of heat flow through the pipes and any other contributions of heat transfer.

The heat flux in a thermal pipe is expressed in terms of the temperatures in the connected nodes and the effective thermal conductance. The effective thermal conductance of the pipe is determined using the analytical approximation of finite element results obtained in simulations of heat flow in systems consisting of two spheres connected by a cylinder. The radius of cylinder equivalent to the neck radius is determined from the criterion of volume conservation during sintering.

The DEM is applied to simulation of transient heat flow in cylindrical samples built from spherical particles representing NiAl powder particles at different stage of sintering. The steady state temperature field is used to determine the effective thermal conductivity from the Fourier law of heat conduction. Different particle configurations corresponding to different porosities are considered. Numerical results are validated using own experimental results.

Acknowledgement

Authors acknowledge funding by National Science Centre, Poland, within project no. 2019/35/B/ST8/03158.

Antoine Van der Laan, Yannick Beynet, Romain Epherre
Norimat, Labège, France

ENGEMINI: a digital asset paving the way towards SPS industrialization

Abstract

Spark plasma sintering is an efficient powder densification technique. It is used to sinter a wide variety of materials (ceramics, metals, alloys, composites, etc.). Compared to conventional processes, it allows to reduce production time while enhancing material performances. However, to obtain the desired product with the required density and material properties, this process mainly relies on a trial-and-error approach. Thus, despite being a promising solution for several industries, FAST/SPS is still mostly used at the research scale. Convinced by its industrial potential, Norimat has invested in the development of digital simulation in support of SPS technology to help it face these challenges.

The digital model developed at Norimat is capable of accurately predicting the electro-thermal and mechanical behavior of materials sintered by FAST/SPS. The main benefit of this simulation is that it can be efficiently used for any type of material, sample dimensions and FAST/SPS apparatus. Thanks to this digital model, it is possible to predict features such as the thermal and/or density gradients inside the sample and during the full SPS cycle. This allows to anticipate these behaviors and then, to optimize the SPS conditions to obtain the desired products within a very limited number of experimental trials. Another main benefit is the prediction of the stress concentration inside the mold, which limits and avoids mold breaking during the FAST/SPS experiments. Thus, using this digital model enables the SPS user to break the empirical testing approach, which is a significant asset from an industrial point of view.

Based on the efficiency of the digital model tested on multiple use cases, Norimat has decided to launch a groundbreaking application, called ENGEMINI. It offers any end user a complete toolkit to design, simulate and manage its own FAST/SPS production. ENGEMINI constitutes a new step forward towards the industrialization of FAST/SPS.

Dariusz Garbiec, Volf Leshchynsky, Alexander Laptev,
Maria Wiśniewska, Rafał Rubach, Jakub Wiśniewski
Łukasiewicz Research Network – Metal Forming Institute, Poznan, Poland

Development of MMCs, CMCs and MAX-based composites by FAST/SPS

Abstract

Since 2011 Field Assisted Sintering Technology / Spark Plasma Sintering (FAST/SPS) is an important sintering technique at Łukasiewicz – Metal Forming Institute in Poland. During these 10 years a number of MMCs, CMCs and MAX-based composites have been synthesized using HP D 25/3 FAST/SPS furnace. The series of investigated advanced materials includes Mg-SiC, Al-Al_2O_3, Al-SiC, AA7075-SiC, AA7075-B_4C, Ti-SiC composites. Currently, the 3YSZ-CNTs, ZrB_2-HfB_2-SiC, WC, WC-Ti, WC-Co, W-B and Ti_3SiC_2 MAX-phase-based composites are under investigation. In some cases the FAST/SPS technique was combined with KOBO extrusion at manufacturing of profiles for automotive industry. Recently, the FAST/SPS technique was applied for sintering of a near-net-shaped nose cone for thermal protection system. The new activities at the Institute include the application of high-energy ball milling at manufacturing of composite powders for FAST/SPS as well as FEM modeling for optimized design of FAST/SPS tool. In particular, the modeling was used at design of FAST/SPS setup for manufacturing of PVD targets with a diameter of 100 mm sintered at 1800°C. The application area of materials manufactured at Łukasiewicz – Metal Forming Institute is mainly the space industry, automotive, gas/oil industry, metal forming and machining industry. In particular, a new cobalt-free (WC-Ti) cemented carbide tool is now under investigation.

Acknowledgement

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Volf Leshchynsky[1)], Dariusz Garbiec[1)], Piotr Siwak[2)]
[1)]Łukasiewicz Research Network – Metal Forming Institute, Poznan, Poland
[2)]Poznan University of Technology, Poznan, Poland

Bimodal grain size structure formation by SPS and severe deformation

Abstract

Combining Severe Deformation and Spark Plasma Sintering is one of the most promising technologies in materials science. The mechanical alloying (MA) and cyclic extrusion (KOBO) are promising severe deformation methods. MA allows the production of nanostructured composite powders which can be successfully spark plasma sintered (SPS) in a very short time while preserving the nanostructure and enhancing the composite mechanical properties. KOBO allows to refine the grain structure due to development of dynamic structure formation mechanisms such as recrystallization, strain aging and oth. The microstructure and mechanical properties of AA7075 with a coarse-fine-grained laminated microstructure produced by SPS and the KOBO technique were investigated. The EBSD analysis results reveal the formation of a deformed and partially recrystallized ultrafine grain microstructure owing to the generation and development of shear bands during KOBO extrusion. The ultimate tensile strength of the AA7075 alloy rose after SPS-KOBO severe deformation up to 422 MPa, with high tensile elongation of about 33%. The indentation size effect at the nano-scale is analyzed on the basis of the Strain Gradient Plasticity theory and the deformation mechanisms are defined. The obtained results show that the SPS-KOBO extrusion technique allows a bimodal laminated fine gradient grain microstructure to be obtained thanks to deformation and dynamic recrystallization, which result in both high strength together with good ductility. The new heterogeneous AA7075 alloys obtained by the SPS-KOBO combined techniques demonstrate that microstructural heterogeneities can assist in overcoming the strength-ductility trade-off.

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Impact of CFRC plates on energy and temperature during Spark Plasma Sintering

Abstract

Spark Plasma Sintering (SPS) is a technology used in both labs and industry. A number of SPS facilities with different size and capacity were developed. For example, FCT Systeme GmbH offers the standard SPS furnaces enabling sintering of parts with a diameter from 30 to 400 mm. The use of large-sized tool especially at high temperature requires a proper thermal insulation to diminish both energy consumption and temperature inhomogeneity. The thermal insulation is usually realized by graphite felt and Carbon Fiber-Reinforced Carbon (CFRC) plates. Whereas the thermal insulation by graphite felt reduces radiation from tool surface, the CFRC plates hold up the heat flow from SPS tool toward water-cooled electrodes. At the present time the effectiveness of CFRC plates is not sufficiently studied especially in respect to reduction in energy demand. It is worthy of note, that CFRC has a relative large ohmic resistance. Thus, CFRC is a heat source in electric circuit directly contacting the water-cooled electrodes. In the present paper, the impact of CFRC plates on required power, total sintering energy, and temperature distribution was investigated by experiments and by Finite Element Modeling (FEM). The study was performed at a temperature of 1000°C with a graphite dummy mimicking an SPS setup. A rather moderate influence of CFRC plates on power and energy demand was found. Furthermore, the cooling stage becomes considerably longer. However, the application of CFRC plates leads to a significant reduction in the axial temperature gradient. The comparative analysis of experimental and modeling results showed the good accuracy of the FEM method at prediction of temperature distribution and electric current. However, a discrepancy between measured and calculated voltage and consumed power was observed. This issue must be further investigated, considering the influence of AC harmonics in the DC field.

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Development of High Entropy Alloys by field assisted sintering using commodity powders

Abstract

High-Entropy Alloys (HEAs) are one of the most rapidly emerging materials, and the powder metallurgy (PM) route offers an interesting possibility to manufacture HEAs. But one of the main drawbacks of the powder metallurgy route is the unavailability of fully prealloyed powders in the market, which makes powder route development an expensive alternative to the ingot metallurgy route. The possibility of using commodity powders, which are commercially available in large quantities and at competitive prices, to produce HEAs presents a completely new and competitive scenario for obtaining viable alloys for high-performance applications. In this work, we have used five different commodity powders, which are Ni 625, Invar36, CoCrF75, SS 316L and Fe49Ni, to develop three HEAs using different combinations of the above five powders. The powders were simply mixed and sintered using Field Assisted Hot Pressing and Spark Plasma Sintering at a temperature of 1000°C, 15 minutes dwell time and 50 MPa pressure, where full density was achieved. After sintering, a heat treatment was performed at 1200°C for 24 hours to homogenize the alloy. The resultant microstructure was a single-phase FCC with no precipitates or intermetallics. This work demonstrates that this alternative and innovative route to develop HEAs is possible and opens a completely new field of work with multiple possibilities to manufacture HEAs by PM route at a competitive price.

Acknowledgement

We gratefully acknowledge the research funding support provided by IMDEA Materials Institute, Getafe, Madrid, Spain.

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Recycling of out of specification AM metal powders using FAST

Abstract

Additive manufacturing (AM) with metal powders requires specific particle sizes and morphologies, with laser powder bed fusion (LPBF) generally requiring a particle size range of 20–60 µm. The gas atomisation processes used to produce these metal powders are not efficient, and so the resulting particle size range is much greater than desired. Therefore, there are increasing quantities of 'waste' metal powders with particle sizes undesirable for additive manufacturing. Powder manufacturers are seeking secondary processing routes for these powders to create value and improve sustainability in the supply chain.

Field assisted sintering technology (FAST) is extremely versatile regarding input powder size and morphology, with even machining swarf material shown to be processable. Therefore, FAST can be used to consolidate these metal powders into bulk material, which can then be formed into various shapes. Alternatively, exploring the possibility to directly create complex shapes via FAST is appealing.

Here, some preliminary work is presented on the FAST processing of a high strength aluminium alloy powder, originally developed for use in LPBF. The effect of varying FAST temperature and dwell time, along with subsequent heat treatments, has been studied to achieve full density and optimal mechanical properties, comparable to AM produced material. Tensile and fatigue testing has shown that the FAST produced material exceeds cast material after the same heat treatments, although with slightly lower ductility than AM material, with further analysis required to understand the cause of this.

Further optimisation of the FAST processing could further improve mechanical properties, with a view to producing near-net shape components directly with FAST using shaped graphite tooling. This would allow for the effective recycling of these otherwise waste powders, whilst producing useful components for various potential industries.

Acknowledgement

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Cameron Barrie, Martin Jackson, Iain Todd
The University of Sheffield, Sheffield, United Kingdom

FAST-AM: a novel technique for creating Ti site-specific properties via additive manufacturing

Abstract

The appealing properties of titanium have led to much research into processing methods that can increase application and reduce its prohibitive manufacturing cost. From this work came an opportunity to create novel titanium properties through the design of complex interpenetrating grain microstructures. These were produced by combining the loose FAST powder bed with a pre-built structure placed into the mould, which then bonds with the powder during sintering to become one part. Producing these inclusion structures with Additive Manufacturing gives them a distinctive columnar grain morphology and texture, contrasting to the surrounding powder grains when bonded and creating an architectured composite microstructure with a high degree of control over the design. In this work, we demonstrate that this FAST-AM technique successfully creates parts with this complex architecture. The samples show a precise arrangement of varying grain sizes and texture with high-quality bonding and densification. These results demonstrate the technique's viability and provide confidence in the novel properties that result from its composite microstructure. The combination of large, texturally-aligned AM grains and smaller, equiaxed matrix grains leads to potential in complex loading properties and the ability to design site-specific mechanical and functional behaviour into the part. Use of AM construction creates open-ended design possibilities for the composite architecture and the ability to create precise structures with very fine degrees of control, giving excellent tailorability and adaptability to meet the needs of a specific part. Verifying this FAST-AM process method leads to a unique opportunity to create novel performance properties in small titanium parts with a high degree of control, while increasing application opportunity for these two major technologies.

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Deformation behaviour of a FAST diffusion bond processed from Ti-6Al-4V/Ti-6Al-2Sn-4Zr-2Mo powders

Abstract

Many of the aerospace components are exposed to extreme service stress states and temperatures, which in some applications may require overengineering to avoid compromising the components performance when a single alloy is used. A potential solution to this issue could be the combination of dissimilar titanium alloys in subcomponent regions, achieved through diffusion bonding of powders via field assisted sintering technology (FAST-DB) and subsequent hot forging (FAST-forge).

In this project, a study was undertaken of the tensile deformation behaviour and strain localisation across a dissimilar titanium alloy solid-state bond. The powder was processed above and below the beta transus temperatures to study the deformation behaviour of different microstructures. Two different approaches were used to test the diffusion bond. The first approach tested ASTM standard FAST-DB specimens with digital image correlation (DIC) to evaluate the macroscale behaviour of the bond. The second approach used a micro tester with optical DIC to evaluate the mesoscale behaviour of the bond. The work demonstrated that the diffusion bond remains intact and that tensile failure occurs in the lower strength alloy and is independent of the grain crystal orientation.

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Channeling current to create unique temperature profiles in titanium alloys during FAST processing

Abstract

With the constant drive for low cost Titanium production, tecniques such as Field Assisted Sintering Technology and our understanding of their potential (FAST) have become increasingly vital. In order to further improve FAST as a technique and overcome one of the primary scale limitations for its use in industry, a solution has been proposed for maintaining homogeneous temperatures across the sample region. By restricting and channelling current using insulating Boron Nitride sprays, tailored current densities and temperature profiles have been generated for Ti-6Al-4V samples. With a clear Beta transus, Ti-6Al-4V has a distinct temperature transition which may be determined from final microstructure. This distinction was used to determine the impacts of these insulation profiles through comparison with predictive Finite Element Models. A series of discs were created with the same processing recipe for four differing temperatures with varying insulation profiles to determine the potential, and any limitations of the technique through their densities and spatially resolved microstructures. It was found that the Boron Nitride insulation profiles applied to graphite foils resulted in improved, and spatially resolved, rates of densification and unique predictable microstructures in a single sintering step. In addition, the inclusion of BN in the FAST process has demonstrated a significant reduction of required power and time to achieve these results. With predictive backing from the FEM model using FKM-GTN powder modelling and Optical/SEM imaging we conclude that this is the direct result of the shaped current profiles and resulting variation in resistive heating during sintering. Additionally, the accuracy of the FEM model was further validated here for predictive use in future works.

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Hall-Petch effect in binary and ternary alumina / zirconia / spinel composites

Abstract

We present the Hall-Petch correlation between microhardness values and grain sizes for single, binary (50-50 vol.%), and ternary (33-33-33 vol.%) phase composites of yttria-stabilized zirconia (8Y-ZrO_2), spinel ($MgAl_2O_4$), and alumina (Al_2O_3) samples produced by spark plasma sintering. Both the intrinsic grain microhardness values and the Hall-Petch constants were found to be significantly higher for alumina among the single-phase samples, and for the alumina-containing composites for the multiphase specimens. The microhardness values of all binary and ternary composites were observed to follow a rule of mixtures for both sub-micron (250-400 nm) and micrometer (1-1.5 mm) grain sizes. The fracture toughness values for all the specimens were also determined using the Vickers indentation method. Among all studied ceramic composites, single phase Al_2O_3 and a binary composite of 8Y-ZrO_2 / Al_2O_3 attained the highest hardness and toughness values of 20 GPa and 5.8 MPa•m$^{1/2}$, respectively. The number of slip systems could be the main reason for the improved mechanical performance of the alumina and alumina-containing composites. Particularly, alumina consists of only 17 slip systems, while both zirconia and spinel are more ductile and contain 12 principal and 24 secondary, and 24 slip systems, respectively. In general, multiphase composites were found to have superior hardness and fracture toughness compared to the individual single-phase constituents. The results from the current study, which connect the grain sizes of the materials produced from an equal amount of constituent phases and their corresponding mechanical response, are of interest for advanced engineering applications requiring both higher hardness and improved fracture toughness.

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Deformation behaviour of a novel Ni-based superalloy processed via spark plasma sintering

Abstract

Astroloy is a Ni based Superalloy with good mechanical properties at higher temperatures than the well-known Inconel 718. Correctly processed Astroloy allows higher operating temperatures and, thus, more efficient jet-engines with lower carbon and NOx emissions. A candidate component to be produced with such material is a turbine casing. In the turbine casing, the ability to contain is critical during operation of the jet-engine, and consequently it needs a combination of high strength and ductility at elevated temperatures. Processing Astroloy via spark plasma sintering will not only minimize the amount of scrap material and energy consumption compared to traditional manufacturing techniques, but it will also enhance microstructure control, including porosity and alloying elements control.

The main objective of this work is to explore the high temperature deformation behaviour of Astroloy obtained via spark plasma sintering. The material was subjected to compression testing in a thermo-mechanical simulator GLEEBLE 3800 over wide temperature and strain rate ranges. These tests represent physical simulation of industrial forging process. Physical simulation of forging allows to save almost 1 ton of expensive Ni-based superalloy simultaneously reducing carbon footprint, time required for development of effective forging process and related labour cost. The effect of temperature on stress–strain curves was analyzed, and strain rate sensitivity was estimated. Activation energy was calculated, and deformation mechanisms were investigated. Processing maps are generated in order to show the optimum conditions for Astroloy processing. The effect of deformation temperature and strain rate on microstructure evolution and forgeability of the material was studied as well.

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The influence of composition in the creep strength of a ferritic ODS steel consolidated by SPS

Abstract

The Gen IV nuclear reactors have been called to be the next power generation technologies in terms of security, efficiency and, specially, carbon-free. Thus, the materials that conform them have to exhibit outstanding properties such as a proper microstructural stability or an excellent mechanical behaviour. Such features are met by the ODS ferritic steels, which are reinforced by a fine, homogeneous dispersion of nanometric oxides that confer them a superior performance at both room and high temperatures (~650°C). However, the current ODS steels display some issues such as a minor strength to toughness ratio or an improvable creep performance. For this, the incorporation of an oxide former (Y-Ti-Zr-O) that increases the precipitation of nano-oxides would be suitable to affront these issues. Hence, three new ODS steel compositions have been processed and compared with a reference ODS ferritic steel previously studied in the literature. Following a powder metallurgy route involving a mechanical alloying and a consolidation by spark plasma sintering (SPS), the ODS ferritic steels have been manufactured. This field assisted sintering technique (FAST) has allowed an almost full densification of the powders while simultaneously avoiding an excessive grain growth of the sintered samples. The developed steels have been characterised at all the processing stages in terms of microstructure, studying the milled powders and the SPS-consolidated samples. Analogously, the mechanical performance of the steels has been studied by means of microhardness measurements, microtensile tests and small punch creep tests (SPCT). These last tests have proved the excellent creep behaviour of the consolidated ODS ferritic steels against the creep phenomenon.

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Spark plasma sintering consolidation of ceramics based on Portland clinker

Abstract

Portland clinker is the main component in cement and concrete manufacturing. These materials are obtained through a setting process and their main use is the construction industry. However, Portland clinker could be used for alternative applications, as a structural material, refractory material or as a wear-resistant ceramic, if it is manufactured by alternative procedures.

Some studies have been conducted on alternative methods of processing Portland clinker. These studies reveal that some alternative consolidation routes, such as spark plasma sintering (SPS), can promote a higher densification, a good level of mechanical properties and a good wear behavior. However, literature about some physical properties, such as the electrical properties of the sintered clinker, is limited.

In this work we have analyzed the electrical properties of different sintered clinkers. The samples where consolidated using SPS in order to obtain densities close to the theoretical one. The effect of this alternative processing route on the electrical properties was evaluated. We have characterized the densification degree of the resulting materials, the variation of the electrical conductivity at room temperature and at high temperature, and the complex impedance at different frequencies. The results show the influence of the different phases on the electrical conductivity. Regarding the complex impedance, non-linear regimes were observed at low frequencies, and a stabilization at high frequencies.

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Heating pathways in spark plasma sintering

Abstract

High-rate field-assisted processing involves transient thermal and non-thermal phenomena, which, if properly managed, provide unique environment for densification and microstructure retention. Thereby, an efficient field-assisted sintering process utilizes conditions of controlled non-equilibrium. For practical realization of this concept the in-depth analysis of the role of electrical current pathways in mass transport during field-assisted sintering is necessary. The impact of the electric current on sintering is revealed by introducing the three heating modes during spark plasma sintering enabling the different electric current density under the same temperature. The experimental evidence of the electric current effect on sintering is demonstrated by the determination of the electric current related constitutive parameters obtained from the three heating modes. By considering the explicit influence of the electric current effect on the spark plasma sintering densification mechanism, the constitutive equations describing the spark plasma sintering of powders are developed. The densification mechanism is determined by the inverse regression of the new spark plasma sintering constitutive equations and by utilizing the experimental results on powder consolidation with and without the participation of the electric current effect. The derived equations show the possibility of the facilitation of the dislocation movement by an intrinsic current effect other than a change of the densification mechanism. The developed constitutive equations for spark plasma sintering can be applied to obtain densification mechanisms for other electrically conductive materials subjected to field assisted sintering. The enhancement of the dislocation motion and the reduction of the flow stress under the electric current passage are experimentally proven by the mechanical strength tests and using the X-ray diffraction results.

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Ultrashort sintering of amorphous Zr-based AMZ4 bulk metallic glass and preparation of Cu-based AMC4 and diamond composite in GeniCore U-FAST machine

Abstract

The GeniCore Upgraded Field Assisted Sintering Technology U-FAST was applied to sintering of a commercial Zr-based AMZ4 bulk metallic glass, and to prepare a composite of diamond grits in an amorphous Cu-based AMC4 alloy. It is the utmost preference that the metal alloy matrices be in amorphous state upon sintering in these materials. The cooling rates achieved in the U-FAST machine reach 250°C/min in the temperature range 1400°C–800°C, and 100°C/min in the temperature range 800°C to room temperature. Copper and Titanium appear to be the main elements contributing to boundary diffusion processes during solid state sintering. The XRD, SEM and DSC analysis of the sintered Zr-based bulk AMZ4 compacts showed the benefit of U-FAST method as an enabler for the production of fully amorphous samples with 100% relative density when sintering at 420°C/480s and 440°C/60s. The hardness values for fully amorphous samples over HV1 519 surpass cast materials and 1625 MPa compressive strengths are comparable to commercial cast products. The advantage of the U-FAST technology in this work is attributed to the high heating and cooling rates inherent to ultra-short pulses, which preserved the metastable structures and achieved a better temperature control during the process. The geometry and the material of the sintering capsule dies and punch determine the thermal inertia and pressure distribution inside the compacts, thus affect the properties of near net shape compacts.

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FAST/SPS in industrial production

The unique FAST/SPS-sintering process is employed in an increasing number of industries and fields of R&D. Up to now, more than 1000 FAST/SPS sinter presses have been installed worldwide by Dr. Fritsch alone – about 98% of them at production companies. Having its origin in the diamond tool industry, FAST/SPS has spread into many other industries. Back in 1953, not even the inventors would have imagined the diverse fields of application for this sintering technique. Today, this technology is entering such diverse fields as the diamond tools and friction materials industry, sputter target manufacturing, heat sink production, the hard metal and ceramics industry as well as universities and R&D-institutes. The list is getting longer every year and with new developments coming up it keeps growing and growing.

I. INTRODUCTION

Most users are already familiar with traditional hot-pressing techniques before switching to FAST/SPS sintering. Hot-pressing is one of the widely established methods used to consolidate cold-pressed or loose powders. However, most people associate hot-pressing with long sinter cycles and low flexibility. FAST/SPS sintering takes another approach. FAST stands for “Field Assisted Sintering Technique”, SPS for “Spark Plasma Sintering”. The mold is directly connected to electric power. The resistivity of the mold material and the powder part or green compact generate the heat directly inside the mold. Not the furnace, nor the atmosphere inside the furnace are heated up. The heat is only generated where it is needed. This results in very high heating rates and additionally a significant increase in the sintering activity of fine metal powder aggregates. Depending on the part size, sinter cycles of only a few minutes can be achieved. Further, this process lowers the sintering temperature and pressure compared to that required in traditional sintering processes. The diamond tool industry was one of the first industrial adaptors of the FAST/SPS process because it solved an issue the industry was struggling with. If diamonds which are embedded in a metal matrix are sintered for too long time, they will carbonize. As a result the diamond tool won´t cut into hard materials anymore. So the idea was to establish a sintering process which is so fast (or FAST) that the diamonds won´t carbonize. Additionally, the new sintering method allowed to reduce the sintering temperatures which further decreased the risk of carbonization and reduced the sinter cycle time compared to traditional sintering techniques.

II. FIELD ASSISTED SINTERING TECHNIQUE

The basic idea of sintering with electric current is quite old. Resistance heating of hard metal powders was patented by American inventor George F. Taylor as early as 1933 [1]. Since then, the

[1] G.F. Taylor, U.S. patent 1896854, “Apparatus for Making Hard Metal Compositions”, February 7th, 1933.

technique evolved and is known under many different names. SPS, Spark Plasma Sintering, is the most commonly used one, however, from a technical point of view it is not correct. There is no evidence that a Spark or a Plasma exists. The term "SPS" has been created by marketing motivations. Most manufacturers nowadays prefer the term FAST. Other terms found in literature are "Rapid Hot-Pressing", "Direct Current Sintering", amongst many others. Due to the high cost related to generating a pulsed current and the unproven effect of it, the optional pulsing is much less common for industrial applications.

Latest research suggests that there is no evident difference between sintering with pulsed or un-pulsed current. The same improved sinter results (compared to traditional sintering) can be achieved by all FAST/SPS sintering techniques [2]. Typical materials and applications of FAST/SPS are:
- hard metals,
- technical ceramics,
- multi-layer composite materials,
- Functionally Graded Materials (FGMs).

For applications such as:
- sputter targets for surface coatings,
- abrasive-resistant parts for agricultural machines,
- friction materials for brakes and clutches,
- diamond tools (metal-diamond-alloys),
- thermoelectric materials for waste-heat applications,
- ballistic protection plates,

to name just a few of the most common ones.

All kind of materials can be sintered, may it be conductive, non-conductive, semi-conductive or even if it changes its conductivity during the heating process. In all these cases, the advantages of a FAST/SPS process are coming into effect, mainly:
- reduction of grain growth thanks to steep heating rates;
- effective use of energy because heat is generated where it is needed.

In some industries, a good result is defined as the full densification of the material, e.g. in the sputter target industry. For other applications, the material must have an exactly defined porosity, for example for stainless steel filters. The exact control of all process parameters is a very important reason why researchers as well as manufacturers switch from un-precise traditional furnaces to modern-style FAST/SPS-sinter presses.

Besides (hard) metals and steel, typical applications include the sintering of technical ceramics, such as B_4C, AlN, TiB_2, Al_2O_3 and others. For sputter target applications, AlN has already been sintered with 300mm diameter using a Dr. Fritsch FAST/SPS sinter press which proves the technology not only suitable for sintering large dimensions in metals, but also in ceramic materials.

However, this is not the end of the development process. The new Dr. Fritsch MSP-5 allows sintering of specimen with diameters of 400mm, providing an electrode size of diameter 450mm. The daylight opening is 500mm and the machine features an hydraulic cylinder with 500t pressing

[2] „International Powder Metallurgy Directory" (January 4th, 2012): 2011 Hagen Symposium. A Review of Spark Plasma Sintering by Prof. Bernd Kieback, Director of Fraunhofer IFAM branch lab Dresden and the Institute for Materials Science at the Technical University of Dresden (Germany). Summary has been published by Dr. Georg Schlieper.

force. As of now, it is the world´s most compact 500t FAST/SPS sinter press, a fact which is vital for the economic success in the industry, where floor space is expensive and installation costs are to be reduced to a minimum. In order to keep productivity high, the FAST/SPS sinter press MSP-5 can be connected to a MSC-5 cooling stating. The sinter goods are transferred into the cooling station under vacuum and inert gas. This allows to increase the productivity by up to 100% compared to single-chamber machines. The first machine has already been sold and will be installed in South Korea within 2022.

Furthermore, Dr. Fritsch offers a wide range of turnkey automation solutions which allow a fully automated operation of FAST/SPS production lines without any human interaction. Such automation solutions are usually implemented by customers who operate at least 2 and often more than 10 or 20 machines in one production line.

III. CONCLUSION

FAST/SPS sintering is the answer to many issues industries and researchers around the world are dealing with. It allows to sinter materials with exactly pre-defined characteristics and combines this precision with a very fast and energy-efficient manufacturing process. The development of new materials can be accelerated significantly compared to traditional hot-pressing techniques. In fact, the sinter quality and achievable density comes close or is comparable to Hot-Isostatic-Pressing (HIP) but at a fraction of the cost and with much less grain growth. While FAST/SPS is already commonly used in R&D at universities, up to now only the diamond tool industry has established this technique as a standard production process. Other industries are catching up, and within the next years a strong increase of industrial FAST/SPS applications is expected to be seen.

Dr. Fritsch supports this approach with the development and production of customized highly effective mass production equipment and automation solutions which are all reality proven and based on almost 70 year of experience in building FAST/SPS sinter presses and automation equipment. Being the world´s largest FAST/SPS manufacturer, both in turnover and machines produced annually, Dr. Fritsch can support its customers in an unrivaled holistic approach backed by decades of industrial production experience.

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GeniCore – examples of PPC and SPS made materials in industrial applications

Abstract

SPS technology (Spark Plasma Sintering) also called FAST technology (Field Assisted Sintering Technology) enables effective consolidation of pure metal powders, metal alloys, and ceramic and metal composites using pulsed direct current. The key aspect of FAST technology is the possibility to carry out the sintering process at a significantly lower temperature than other available methods. This has a beneficial effect on limiting the growth of grain and ensures much better parameters of the created materials. The advantages of FAST also include a very short sintering time and uniformity of the resulting material, as well as high energy efficiency related mainly to the direct generation of heat in the material.

The further SPS technology development allowed to upgrade the FAST technology and create the U-FAST device which represents the state-of-the-art sintering technology. The U-FAST technology allow to sinter materials from metallic powder and obtain groundbreaking material properties which allows the final product to be implemented into high-end industrial applications.

The U-FAST technology represents low-voltage sintering which is beneficial for most of materials. There are however materials like diamond which require different approach and in that case the high-voltage Pulse Plasma Compaction technology may be used. The generation of high-voltage pulses is possible thanks to high-current electronic key in the capacitors battery discharge circuit. One of the factors behind the usage of the high-current electronic keys is the formation of rectangular impulses with the duration of several hundred microseconds and adjustable amperage of several kA. The examples of materials made by both mentioned technologies will be presented.

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Scaling up the production of short fiber reinforced – ultra high temperature composites by SPS

Abstract

The novel class of ultra-high temperature (UHT) ceramic-matrix composites (CMCs), recognized as UHTCMCs, was extensively studied within the Horizon 2020 European research project entitled Next Generation Ceramic Composites for Harsh Combustion Environment and Space (C^3HARME). The applications of UHTCMCs developed in C^3HARME were thermal protection tiles and hot components for propulsion such as rocket nozzles and chamber inserts.

This work presents the scale up of the production of this kind of composites by Spark Plasma Sintering. The processing route was based in ball milling of ceramic powder with the reinforcement and subsequent SPS sintering. Large components were manufactured and characterized. Large pieces of 400 mm diameter and 6 mm thickness were successfully obtained. In addition, huge blocks of 170 mm diameter by 40 mm thickness were also successfully manufactured. From these pieces, different prototypes were machined for testing.

Microstructure and thermomechanical properties of different pieces were studied including thermal diffusivity up to 1950°C, thermal expansion coefficient, bending and tensile strength up to 1500°C. In addition, some components were tested in real application conditions for validation.

In general, homogeneous composites were obtained but some tendencies in properties depending on the pressing direction were observed. These materials were able to sustain very harsh testing conditions. This scale up opens the possibility of new applications for these kind of materials besides the space market. Some indications about the machining and fabrication of different components will be also included.

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Effect of spark plasma sintering on microstructure and properties of alumix-based composites reinforced with B_4C particles

Abstract

Spark plasma sintering (SPS) has become a promising technique in the production of composite materials as it combines fast heating with a high degree of densification due to the applied compaction pressure during the sintering. In this research, SPS was applied to fabricate aluminum alloy-based composites reinforced with B_4C particles. The effect of the percentage of weight of reinforcement and the time of sintering on the microstructure and mechanical properties was investigated. AA7075 powder (Alumix 431) was used as a matrix material. The B_4C particles were introduced into the matrix as reinforcement in various weight percents of 3; 5 and 10. The particle size of the matrix material and the strengthening phase was 86 and 0.8 μm, respectively. The sintering temperature was 500°C, and the pressure of 80 MPa was constant throughout the process. The holding time was changed from 3 to 10 minutes. The microstructure of the composite materials was investigated using light microscopy (LM) and scanning electron microscopy (SEM). Sintered composites were characterized by a fine-grained microstructure with a strengthening phase located at the grain boundaries of the aluminum grains. As a result of the SPS process and cooling, fine precipitation of intermetallic phases from a matrix material was observed in the microstructure. The chemical composition analysis in the microareas indicated the presence of phases rich in the Mg, Zn, and Cu in microstructure. The relative density of the sintered composites reached 97% and remained at the same level for all variants of the weight fraction of the strengthening phase and the sintering conditions. Extending the sintering time allowed higher values of flexural strength and hardness to be achieved. The highest flexural strength values were obtained for composites with the lowest B_4C particle content and oscillated between 618 and 629 MPa. An inverse relation was observed for the hardness measurements, where the highest value was achieved by the composite with the addition of 10 wt. % of B_4C (122 HB). Fractographic studies showed a plastic character within the matrix and in the areas where particles occur, the phenomenon of brittle fracture.

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Characteristics of the surface layer of Ti-Mo/TiC nanocomposites obtained by Field Assisted Sintering Technique

Abstract

Field Assisted Sintering Technique (FAST/SPS) is a rapidly developing processing technique in which different powders can be consolidated rapidly with simultaneous application of pressure and current. During the sintering of titanium and nanocrystalline $Ti_{0.9}Mo_{0.1}C$ powders mixture physicochemical processes took place, as a result of which Ti-Mo/TiC type composites were obtained. In FAST processes two types of nanocrystalline carbides were used: $Ti_{0.9}Mo_{0.1}C$ and $Ti_{0.9}Mo_{0.1}C/C$ in carbon shell. The content of the reinforcement phase was 10 and 20 wt. %. The influence of sintering temperature of the composite with the highest content of the reinforcing phase was analyzed. The nearly full dense composites were obtained. Phase composition of the composites was analyzed based on X-Ray diffraction (XRD). The distribution of the particles on the Ti matrix was imaged using optical microscopy (OM) and scanning electron microscopy (SEM) with the help of electron backscatter diffraction analysis (EBSD). We also evaluated the microstructure, morphology, mechanical and tribological properties of composites. It was observed that the hardness, Young modulus and surface roughness increased with an increase in the content of the reinforcing phase in titanium matrix. Moreover, the phase composition of the native surface layer of TMMCs was studied using Grazing Incidence X-Ray diffraction (GIXRD), X-ray photoelectron spectroscopy (XPS), Raman spectroscopy (RS) and nanoindentation technique. This analysis revealed the presence of thin TiO_2 in the top layer of the composites. The spectra identified both crystalline and amorphous titanium oxides. Besides, the non-stoichiometric titanium carbide corresponding to $TiC_{0.67}$ (Ti_3C_2) was also detected on the composites surface.

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SHS synthesis of tellurides using SPS apparatus

Abstract

The development of innovative applications must be supported not only by scientific analysis and basic research of novel materials, but also finding new ways to synthesise well-known semiconductor materials in simplier and faster way. Nowadays a few main directions within thermoelectric society can be identified. One group of research subjects focuses on adopting known methods of synthesis to fabricate thermoelectric material witchoud many stages of synthesis in vacuum quatrz ampules, milling, another vacuum quartz anealing, milling and sintering. Simplification of tellurides' fabication process has a huge potential for thermoelectric applications, where lack of good-quality material aviability limits the implementation potential. This work shows the way of fabrication of antimony telluride using the SPS apparatus as a vacuum furnace with very low thermal inertia, presents material properties and compares them to material fabricated in the traditional way.

Acknowledgement

The presented results are part of the doctoral dissertation carried out as part of the "implementation doctorate" programme conducted within consortium of the Łukasiewicz Research Network – Institute of Microelectronics and Photonics and AGH University of Science and Technology.

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Properties of spark plasma sintered bulks and coatings made of tungsten diboride alloyed with Cr, Mo, Re and Zr

Abstract

Tungsten borides due to theirs properties can be an alternative to superhard materials. While they possess high hardness, chemical and thermal resistance, they are easier to manufacture than diamond and cubic boron nitride. Tungsten borides in bulk form can be produced without employing high pressures (>5GPa), and in case of coatings there is no need to use processes with high plasma density. To enhance the properties of tungsten diboride (WB_2), we have synthesized and characterized solid solutions of this material with chromium, molybdenum, rhenium and zirconium. The obtained materials were subsequently deposited as a coatings. Various concentrations of these transition-metal (TM) elements, ranging from 0.0 to 24.0 at. %, on a metals basis, were made. Spark plasma sintering (SPS) was used to synthesize these refractory compounds from the pure elements. Elemental and phase purity of the both samples (sinters and coatings) were examined using energy-dispersive X-ray spectroscopy (EDS) and X-ray diffraction (XRD), and microindentation was utilized to measure the Vickers hardness under applied loads of 200 gf. XRD results indicate that the solubility limit is below 8 at. % for Mo, Re, Zr, and below 16 at. % for Cr. Above this limit both diborides $(W,TM)B_2$ are created. Addition of TM caused decrease of density and increase of hardness and electrical conductivity of sinters. In the case of coatings with Zr the hardness grows even more and reaches 40 GPa – superhardness limit. Deposited coatings $W_{1-x}TM_xB_{2-z}$ (x=0.08—0.24, z=0.2—0.07) are homogenous, smooth, hard and also refractory in case of addition of zirconium. One hour annealing with 700°C did not change structure and improve hardness of zirconium alloyed films.

Acknowledgement

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II. POSTERS

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Spark plasma sintering of bimodal titanium as a matrix for further composites development

Abstract

The constant and dynamic development of technology requires the use of materials with better and better properties. Titanium belongs to light material group and is characterized by a low density of 4.51 g/cm^3, biocompatibility, good corrosion resistance and very good ductility, which is relate to the good fracture toughness. The used of Ti under friction conditions is limited due to the poor tribological properties. The composites based of titanium matrix which ceramic reinforcements are embedded in a metal matrix combine the properties of metals with the properties of ceramics, incl. high hardness, which leaded to an increased in tensile and compressive strength, higher wear resistance and allow these materials to work at higher temperatures. By appropriately adjusting the matrix and the reinforcement in composite materials, combinations of various properties can be consciously of materials. One of the most promising methods of producing composites is spark plasma sintering (SPS). The SPS method 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 to obtain materials characterized by high density and low grain growth. The study attempts to development of titanium matrix for used in future Ti-TiMoC composites. In the work, sintered compacts were produced by spark plasma sintering method using two powders, microcrystalline Ti and nanocrystalline Ti powders and their mixtures, which were produced in two ways using a Turbula T2F shake-mixer and a planetary mill PULVERISETTE 5 classic line at argon atmosphere. The sintering process was conducted using an HP D 25/3 was carried out at a temperature of 900°C for 5 min, with a heating rate of 400°C/min at a uniaxial compaction pressure of 50 MPa in an argon atmosphere. The diameter of the obtained specimens was 20 mm and the high was about of 3.5 mm were, for which the density, hardness and fracture toughness and the microstructural observation by light microscope was conducted. The sintered compacts of characterized of high density. The planetary mixing of powders allowed better homogenization that shaker mixing, which results got material characterized of hardness 727 HV_{10} and fracture toughness 6,60 MPa·m$^{1/2}$ at close to 100% compaction (50µTi-50nTi).

Acknowledgement

The research work was carried out as part Łukasiewicz Research Network – Metal Forming Institute internal project No. BS.901.0106 entitled "Development of a technology for consolidation of composites based on titanium with the addition of nc-$Mo_xTi_{1-x}C$ type carbide by spark-plasma sintering (SPS)" and as part of the 4th edition of the Implementation Doctorate financed by the Ministry of Science and Higher Education entitled "Development of sintering conditions by spark plasma sintering (SPS) method of titanium powders and nc-$Mo_xTi_{1-x}C$ carbide in order to obtain nanocomposites characterized by high wear resistance, fracture toughness and corrosion".

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Mechanically alloyed and SPS consolidated Ti-20Ta-7Mn-5Mg alloys

Abstract

The industry still finds new applications for titanium alloys, such as in aerospace, construction or medical. Titanium comes with excellent corrosion resistance, a good strength-to-weight ratio, and has relatively low density. Introducing additives to pure elements affects almost every aspect of the main component, i.e., mechanical, physical and thermal properties. Thanks to powder metallurgy, it is possible to obtain bulk material from the powder form without a porosity in the structure. Still, it requires setting correct sintering temperatures (favourably the lowest possible to maintain starting grain size), proper compression pressure (the highest possible, but lower than compression strength under elevated temperature for die and punch material) and time for each sintering step (the shortest for warming up and heating stage). In this study, mechanically alloyed titanium alloy (Ti-20Ta-7Mn-5Mg) powder has been sintered in the form of discs using an SPS machine. Every disc has been made out of 7 grams of powder. Subsequently, we investigated the influence of the sintering temperature (750°C, 850°C, 950°C) on the porosity, hardness and piston displacement charts under the same sintering conditions for all samples. With increasing temperature, porosity was decreasing 20,88±1,04%, 3,46±0,57% and 1,93±0,17%, respectively. Microhardness was increasing accordingly 482±159HV0,1 551±32,3HV0,1 and 548±8,22HV0,1. Due to the presence of porosity, there is an enormous variation in results for 750°C sample. Comparing the temperature chart with the piston displacement chart, it is possible to determine whether the consolidation process has been finished too soon with the given parameters or the heating stage should be further elongated. It must be pointed out that keeping samples at an elevated temperature long enough affects the grain size and influences the metastable structures if any have been achieved after mechanical alloying.

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Mechanically alloyed Ti-20Si_3N_4 based composites

Abstract

Silicon-based non-oxide ceramics (Si_3N_4+TiN) have attracted much attention as engineering materials, mainly due to their good mechanical and chemical properties such as high hardness, chemical stability and corrosion resistance, high melting point and reliablity at elevated temperatures. Si_3N_4 based composites have great potential as cutting-tool and other high temperature applications. Other authors have reported that TiN/Si3N4 based nanocomposites with excellent mechanical properties and conductivity can be processed through a chemical route, as well as, mechanical milling and sintered by SPS. They applied Si_3N_4 and TiN nano powders as a precursors.

In this work authors investigate mechanical alloying process of Ti-20Si_3N_4 powders. The specimens were than consolidated by high frequency induction heating sintering and cold pressing followed by free-sintering. The structure, microstructure and porosity, hardness were investigated by XRD, SEM and Vickers tester, respectively.

After mechanical alloying nearly amorphous structure was obtained which could be interesting precursor for consolidation, due to higher reactivity than standard microstructure. The MA process takes 30h, using Spex shaker-type mill, under argon atmosphere.

Authors decided to investigate the consolidation process using two techniques: traditional cold pressing (1000 MPa) followed by free-sintering (1300°C/120min) and high frequency induction heating sintering (50MPa, 1000°C/3min).

Free-sintering results in highly porous samples (porosity level was over 30%). HFIH samples despite lower sintering parameters (pressure, temperature and time) showed nearly full densification (0,4% porosity). Moreover, after HFIH the grain size was almost two times lower than after traditional consolidation. The phase compositions were the same for both consolidation techniques and consist of Si_3N_4, Ti_5Si_3, TiN. The obtained composites show relatively high hardness of about 1650 HV.

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The influence of WC content on microstructure and mechanical properties of titanium metal matrix composites

Abstract

Titanium and its alloys are applied in aircraft, automobile, arms, biomedical and chemical industries through a property such as high specific strength, excellent corrosion resistance, good creep resistance, and good high-temperature strength. Nevertheless, Ti-based alloys possess low wear resistance, which limits their applications. In order to improve the mechanical properties of Ti-based alloys, composites materials can be fabricated. In this work, the Ti-based composites with a variable quantity of WC were produced. The influence of chemical composition on "in-situ" composites formation was examined. Obtained composites were characterized in terms of their phase composition, microstructure, and mechanical properties. The phase composition after sintering consists of matrix phases - Ti(α) and Ti(β), and reinforcement phases $TiWC_2$ and WC. The grain size of sintered composites remains in ultra-fine grained range. Ti-WC systems are characterized by high strength properties, which enable them to use in the aircraft, automotive and chemical industries.

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Tribological properties of Ti_3SiC_2-based composites manufactured by FAST/SPS

Abstract

Ti_3SiC_2 is one of the most common and examined compound from MAX phases group of materials. Ternary carbides or nitrides of transitions metals and elements of the 13th or 14th group are called MAX phases. Their unique properties characteristic for both, metals and ceramic, are the reason for the widespread interest in these materials. Ti_3SiC_2 MAX phase has application potential in order to usage in high-temperature wear conditions thanks to relatively high hardness with good fracture toughness. Despite many studies on this phase, few scientists have decided to analyze tribological properties over a wide temperature range.

The aim of this study was to obtain Ti_3SiC_2-based composites by FAST/SPS method in order to analyze tribological behavior in wide temperature range from RT to 700°C. To produce the samples, Ti_3SiC_2-based composite powder was used. Sintering was carried out in the HP D 25/3 SPS furnace with the following parameters: sintering temperature of 1400°C, holding time of 15 min, compaction pressure of 60 MPa, heating rate of 200°C/min. X-ray diffraction and SEM-EDS observations confirmed presence of Ti_3SiC_2, $TiSi_2$, TiC and SiC phases in the microstructure of sintered compacts. Density, Vickers hardness and fracture toughness measurements were performed to characterize physical and mechanical properties of the obtained samples. Specific wear rate (SWR) and coefficient of friction of the sintered composites were determined in *ball-on-disc* wear tests in temperature range from room temperature (RT) to 700°C. Average coefficient of friction values were approximately decreasing with increasing test temperature, ranging from 0.82 to 0.54 for RT and 700°C, respectively. SWR values increased from 218.88×10^{-6} mm^3/Nm for RT, up to 1093.25×10^{-6} mm^3/Nm for 200°C, then decreased significantly to -17.89×10^{-6} mm^3/Nm for testing in temperature of 700°C.

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Microstructure and crystal structure analysis of Cr_2AlC MAX based powders produced with the combination of mechanical alloying and spark plasma sintering

Abstract

The unique group of materials called MAX phases are gaining more interest every year. Thanks to their unique properties (high temperature resistance, wear resistance, self-healing capabilities etc.) they are studied for various applications, for example surface coating material. While vapor deposition technologies require precursor materials in the form of a solid, other coating technologies, like thermal spraying require the initial material to be in form of a powder. One of the most promising routes for obtaining a highly pure MAX phase is the mechanical alloying of powders and their sintering. FAST/SPS has been widely used to obtain pure, highly dense samples. We present an alternative MA-SPS approach, where the material remains in the powder state to be used in powder-based technologies. The elementary powders of Cr, Al, C were used as a precursor for Cr_2AlC MAX phase synthesis. The milling process was conducted using a planetary ball mill (Pulverisette 5 Premium Line, Fritsch) using WC-Co milling media under Ar atmosphere. The milled powder was then sintered using FAST/SPS equipment (HP D 25/3, FCT Systeme) at 1100°C for 30 min. under vacuum. In order to keep the material in powder form, no pressure was applied during sintering. The process resulted in a high-purity Cr_2AlC powder (and solid for sintering with pressure). The pressure during sintering had little to no effect on the phase content of the sintered material. Analysis of the sintering curve suggests that the exothermic crystallization of Cr_2AlC started at a temperature between 590–630°C. The created powder had a polycrystalline, agglomerated morphology with grain sizes ranging between 1–3 μm, while the particle size ranged between 5–50 μm.

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Tribological properties of Cu-Al_2O_3 coatings obtained by vacuum cold spraying and spark plasma sintering

Abstract

Metal matrix composite (MMC) coatings are candidates for wear-critical applications in the automotive and aircraft industries because of their good heat conductivity, wear resistance and stable chemical properties. Such coatings can be obtained using spraying methods including vacuum cold spray (VCS), which is a new, low cost and low energy-consuming method for coating deposition. This technology can give prospective results in powder mixture spraying. It is shown in this work how the combination of VCS and SPS technologies resulted in better adhesion strength and tribological properties of Cu-Al_2O_3 coatings. Firstly, Cu-based powder mixtures with different contents of Al_2O_3 were prepared using a planetary mixer. Both the components had a mean particle size of ~1 μm. The powder mixtures were deposited on a low carbon steel substrate using vacuum cold spraying technology, obtaining coatings with a thickness of ~100 μm. One part of the coated samples was sintered using FAST/SPS technology (HP D 25/3 FCT Systeme) at 850°C/1 min/10 MPa pressure under vacuum. Adhesion tests of the coatings were conducted according to the ASTM C633 standard. In the next step, the tribological properties of the obtained coatings were tested using a ball-on-disc tribometer (T-21, Łukasiewicz Research Network – Institute for Sustainable Technologies, Poland). The results of the friction tests were presented as coefficient of friction and calculated wear rate graphs.

The results of the research are very promising. Spark plasma sintering increased the adhesion strength of the coatings. The sintering process also influenced the tribological properties of the sprayed coatings, substantially reducing the coefficient of friction as well as the wear rate.

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The influence of grain size on the properties of Al_2O_3 sintered by the SPS method

Abstract

Alumina is a ceramic material commonly used in industry, for example as abrasive, polishing or refractory material. Other uses for alumina include machining tools, electronics components, implants, and more. Many aspects of sample manufacturing and processing can affect the properties of the obtaining material. The grain size of the powder is one of the very important factors affecting the properties of the material. Our experiment was meant to check the influence of grain size on the properties of Al_2O_3 sintered by the SPS method. Three Al_2O_3 powders of different grain size were used: TAIMEI with purity 99,9% and grain size 0,1 μm, Almatis CL370 with purity 99,9% and grain size 2,5 μm, Pyzalski with purity 99,9% and grain size 40 μm. The Spark Plasma Sintering method of compacting the powder was used for sintering the samples. The processes were carried out on an FCT HP D5 device. Parameters of the processes were in the range: 1250–1600°C, 3–15 kN, 10 min. Selected physical properties of sintered material were analyzed, such as density, open porosity and Young's modulus. Density and open porosity were calculated using Archimedes method. Young modulus was determined with use ultrasonic measurement method. Ultrasonic flaw detector Epoch III by Panametrics was used. Surface topography as well as surface and profile roughness parameters were analysed. The TOPO 02 contact profilometer was used to measure the surface texture. Device was equipped with an inductive measuring sensor with a 5 μm radius and 90 degrees angle of diamond tip.

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Preparation and properties of Al_2O_3–ZrO_2–WC composites

Abstract

The results of research on the development of oxide-carbide ceramic composites intended for machining of nickel superalloys are presented. Nickel superalloys can be admittedly machined with superhard cBN-based composite tools, but this approach is usually too expensive for common industrial processes. Therefore, ceramics (which are hard, thermally stable and cheap, but brittle) are being improved all the time to increase their performance. Tungsten carbide seems to be an ideal candidate as a strengthening phase in oxide ceramics because of its exceptional hardness, strength and wear resistance. Investigations of the oxide-carbide ceramics sintered in ternary Al_2O_3–ZrO_2–WC system are presented in this work. The powders of the selected compounds were mixed using a ball mill in volume ratios of 42:12:46, respectively. The prepared mixture was then sintered via Spark Plasma Sintering (SPS) and Hot Pressing (HP) methods at the temperature in the range of 1450-1550°C for SPS and 1550-1650°C for HP sintering. The obtained composites were characterized by a density of 98-100% of the theoretical density value and a Young's modulus of 430-470 GPa over the entire range of temperatures for both HP and SPS method. Scanning Electron Microscopy observations revealed a highly homogeneous microstructure, regardless of sintering method and temperature. An increase in the sintering temperature caused a slight increase in the grains size, but did not affect the formation of heterogeneity of the structure. Vickers hardness tests indicated that the obtained materials have a high hardness of approximately 19-20 GPa. The fracture toughness of the composites was found to be about 7-8 $MPa{\cdot}m^{1/2}$. Tribological tests have determined a coefficient of friction which is less than 0.4. This value is relatively low and stable over the whole range of test durations compared to other Al_2O_3 based ceramics. One representative sample was selected to perform a three-point bending test which showed that the bending strength of the composite reached values in the range of 650-700 MPa. At the final stage of the research, machining tests were carried out on Inconel 718, which showed that the average tool life of the Al_2O_3–ZrO_2–WC composite was comparable to a highly advanced ceramic from a leading tool manufacturers.

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Densification and processing technology of new type of thermoelectric materials

Abstract

Thermoelectric harvesting of low-temperature waste heat offers great opportunities for sustainable energy production, but the low conversion ratio of currently used thermoelectric materials creates a need to search for new, more efficient ones. On the other hand, new materials, despite having a large figure of merit (ZT), are mechanically very weak, which makes their technological application limited and generate challenges for their processing. The results of research on the development of manufacturing technology of a new type of thermoelectric materials for conversion of low temperature waste heat into electricity are presented. In the framework of the TERMOD project, the two types of thermoelectric materials, based on PbBiTe n-type and PbSnTe p-type systems, were synthesized. The powders were sintered by the SPS (Spark Plasma Sintering, HPD5 FCT system, Germany) method at the pressure of 50 MPa. Various heating rates (25–100°C/min.), sintering temperatures (300–650°C) and sintering durations (1–30 min.) were tested to find an optimal SPS process conditions for each material. The best achieved values of Young's modulus of sintered materials were 34 GPa for PbBiTe n-type and 55 GPa for PbSnTe p-type, respectively. SPS technique ensures desirable properties of sintered product, but HPD5 device gives possibility to obtain rather small batch of material at one time in relation to the costs incurred, i.e. the costs of matrices, electricity, operators' work, etc. In order to increase the efficiency of the production process of thermoelectric elements, dedicated extrusion technology was developed. It was assumed that the selection of the appropriate extruder geometry, strain rate and process temperature will enable the production of high quality material, not only on a laboratory scale.

A special device, enabling almost continuous production of materials with a length of max. 400 mm at max. temperature of 700°C and the pressure of 550 MPa (for die entry diameter of 30 mm) was constructed. The results of the work and research conducted will find potential application in the industrial production process of thermoelectric modules.

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Surface topography inspection after turning of Inconel 718 by Wiper insert and special insert made by SPS method

Abstract

This paper focuses on the parametric and nonparametric description of surface topography after turning in dry and wet conditions. The carried out research involved the longitudinal turning tests of Inconel 718, conducted in a range of variable feeds *f*. Tests were conducted using two types of cutting inserts: WNMG 080408-FW KC5010 with Wiper geometry (as a reference) and special insert with ISO type of WNMG size 080408 SPS-sintered binderless tungsten carbide. The shape of the special insert was obtained by means of EDM. Surface topography measurements were conducted with the application of Nanoscan 855. The performed analysis includes parametric and non-parametric evaluation of the obtained surfaces. The conducted research shows that the application of wet machining can lead to the reduction of 3D surface roughness parameters compared to values reached after the dry machining for both of used cutting inserts. In dry machining stickings have appeared on the machined surface. The stickings on the machined surface probably originate from the generated chips or, more strictly, from the fuzzed edge which arises at the location of the smallest thickness of the cut material. The sticking not appear during wet machining. Application of coolant to lead heat out of the cutting zone, also creates a lubricating film between the cutting edge and machined material. The coolant also washes chip fragments preventing their sticking to the machined surface. In none of the considered cases, formation of sticking on the machined surface has been observed. The chips generated during cutting were also analyzed. Discoloration of the obtained chips was observed, especially at the lowest tested feed. Although the SPS-insert did not have a protective layer on its surface, the wear and surface roughness were similar to the reference one.

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Improving SPS sintered Si_3N_4 based ceramics through addition of SiC whiskers

Abstract

The work present the results of silicon nitride ceramic modification via using addition of SiC whiskers in order to improve mechanical properties of sintered materials. To activate the hard-to-sinter silicon nitride powder, oxides additions: Al_2O_3 and Y_2O_3 in the amount of 6 mas % to the Si_3N_4 matrix were used. The content of SiC whiskers in the produced composites was 20% and 50% by weight. The sintering parameters of the initial mixtures were optimized to obtain the highest possible sinter properties, such as: apparent density, Young's modulus, Vickers hardness and indentation fracture toughness. All of the materials were sintered using SPS/FAST technique in the temperature range of 1550°C÷1700°C (every 50°C), sintering duration was 5 min and applied pressure was 63 MPa. SPS sintering of Si_3N_4 matrix materials with oxides resulted in maximum values of physical and mechanical properties already at 1600°C and did not change significantly with increasing the temperature to 1700°C (average: ρ=3.21 g/cm^3, E=297 GPa, HV1=16.2 GPa, $K_{IC(HV)}$=5.4 MPa·m$^{1/2}$). The application of the 20% SiC whiskers addition to the Si_3N_4 matrix with oxides resulted in the improvement of Young's modulus, Vickers hardness and fracture toughness, but the apparent density did not change significantly. Similarly, the properties of sintered materials in the temperature range 1600°C÷1700°C reached optimal values, which were on average ρ=3.19 g/cm^3, E= 310 GPa, HV1= 18.5 GPa, $K_{IC(HV)}$= 6.1 MPa·m$^{1/2}$. In this way, an improvement in mechanical properties, including Young's modulus and hardness, was achieved by about 12%, which was confirmed during the SEM observations, where the phenomenon of crack bridging was observed. The addition of 50% of SiC whiskers into the Si_3N_4 matrix and SPS sintering in optimum temperature for Si_3N_4 resulted in a decrease in the properties of the sintered materials, which may be caused by to low temperature of SPS process for such a high content of SiC whiskers addition.

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Mechanical alloying of magnesium-based alloys

Abstract

Modern materials engineering uses advanced laboratory and research equipment as well as computer techniques that enable the selection of appropriate chemical composition, for a specific material with beneficial properties [1]. An important aspect is the selection of suitable materials for biomedical applications, as very high requirements are placed upon them. The ones used for implants must be biocompatible, have adequate porosity [2].

A apt material that meets the requirements for biomedical implants described in this paper is an alloy based on magnesium with the addition of zinc, calcium, and, the rare-earth element, praseodymium, produced by mechanical alloying. It is a method of manufacturing non-equilibrium materials, making possible to create a microstructure or even a nanostructure. The basis of the process are the deformations, that occur cyclically (welding, crushing, and re-welding). The processes that take place result in the reduction of grain sizes and the creation of new grain boundaries. Using also the powder metallurgy method, it is possible to obtain materials with a strictly defined chemical composition and a high degree of purity. Materials produced by this method are characterized by controlled porous structures, fine-grained structure, and isotropic properties [3].

In the present manuscript, Mg-Zn-Ca-Pr alloy was prepared and analysed. The alloy powder with a nominal composition of $Mg_{65}Zn_{30}Ca_4Pr_1$ was prepared via the mechanical alloying method. The samples varied in milling time and were as follows: 8, 13, 20, and 30 hours. On the $Mg_{65}Zn_{30}Ca_4Pr_1$ powder samples prepared by mechanical alloying, the phase analysis, Vickers microhardness testing, and scanning electron microscopy were conducted.

This research is a part of a preliminary study, the $Mg_{65}Zn_{30}Ca_4Pr_1$ alloy powder will be taken into future consideration as a sintering material by Spark Plasma Sintering (SPS) technique.

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