

**1<sup>st</sup> Interdisciplinary Annual  
PhD Conference  
on Material Science  
and Innovative Technologies  
*InterMST 2022***

**Book of Abstracts**

Krakow, 19–20 May 2022



**Łukasiewicz**

Krakow Institute of Technology

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## TABLE OF CONTENTS

<b>Welcome to the 1<sup>st</sup> <i>Inter</i>MST 2022 .....</b>	<b>4</b>
<b>General information .....</b>	<b>5</b>
<b>Conference Programme .....</b>	<b>7</b>
<b>Abstracts .....</b>	<b>12</b>
<b>Index of Authors .....</b>	<b>46</b>

## **WELCOME TO THE 1<sup>st</sup> *InterMST 2022***

*Dear PhD Conference Participants,*

*On behalf of the Scientific Committee and the Organizing Committee we are pleased to welcome you on the InterMST 2022 conference – Interdisciplinary Annual PhD Conference on Material Science and Innovative Technologies hosted on-line by the Łukasiewicz Research Network – Krakow Institute of Technology, 19–20 May.*

*The conference is devoted to exploring the richness of approaches, methodologies, and themes of the discipline in order to showcase a wide range of studies and provide a picture of the current state of research in the field of material science and innovative technologies. The interdisciplinary approach of the conference highlights the trajectories of the various scientific disciplines which allow for a progress in material science and innovative technologies – from the engineering and exact sciences to the natural sciences and medical disciplines.*

*“Book of Abstracts” comprises 29 extended abstracts that have been carefully selected on the basis of a peer review process. It includes state of the art in scientific considerations related to innovative materials and material characterization, advances in casting technology, high temperature and high entropy materials, advances in coatings technologies and finally additive technologies and advances in biomedical and optical technologies.*

*On behalf of the conference hosts, we would like to express our gratitude to the members of the Scientific Committee, the members of the Organizing Committee, and all the Authors for their effort and willingness to take part in the InterMST 2022 conference – Interdisciplinary Annual PhD Conference on Material Science and Innovative Technologies.*

*Yours faithfully,*

*Katarzyna M. Marzec*

*Tomasz Dudziak*

*Małgorzata Grudzień-Rakoczy*

*Krakow, May 2022*

## GENERAL INFORMATION

This conference is devoted to exploring the richness of approaches, methodologies, and themes of the discipline in order to showcase a wide range of studies and provide a picture of the current state of research in the field of material science, engineering and innovative technologies. The interdisciplinary approach of the conference highlight the trajectories of the various scientific disciplines which allow for a progress in material science and innovative technologies – from the engineering and exact sciences to the natural sciences and medical disciplines. The conference is addressed to PhD student. It is possible to publish an article in the “Journal of Applied Materials Engineering” (JAME) up to 6 months after the conference.

The conference topics include:

- Modern materials for harsh conditions (corrosion degradation)
- Coatings (thermal spray coatings, CVD, PVD, slurry coatings, and others)
- Mechanical properties (creep, tensile, bending, micro bending, micro scale tensile, and others properties)
- Heat treatment (various processes inducing higher performance of materials)
- Computing simulations (Cellular automata (CA), neural network modelling (NNM), and others)
- Additive manufacturing (SLM, SLS, and others)
- Non-destructive techniques (tomography, XRD scanning, and others)
- Microstructure characterisation (SEM, SEM-FEG, TEM, STEM, and others)
- Tribology and wear properties
- High temperature ceramics (borides, oxides, carbides)
- Others related to Material Science and Engineering.

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*Marcin Małysza*

*Marcin Podsiadło*

*Adam Tchórz*

## PROGRAMME OF THE CONFERENCE

### DAY 1

**9.00**      **Opening session:** *Katarzyna M. Marzec, Tomasz Dudziak, Małgorzata Grudzień-Rakoczy*

**Session I: Innovative materials and material characterization – chair Rafal Nowak**

- |       |   |                         |   |
|-------|---|-------------------------|---|
| 9.15  | SYNTHESIS AND CHARACTERIZATION OF $MgAl_2O_4$ SPINEL VIA MODIFIED SOL-GEL METHOD  | <i>Tiago Oliveira</i>   | Federal University of Rio Grande do Sul, Department of Materials Engineering, Porto Alegre, Brazil  |
| 9.35  | PASSIVE MAGNETIC METHODS AS NOVEL NON-DESTRUCTIVE TECHNIQUE OF DIAGNOSTIC FOR STEEL WIRE ROPES  | <i>Paweł Mazurek</i>    | AGH University of Science and Technology, Krakow, Poland  |
| 9.55  | APPLICATION OF X-RAY COMPUTER TOMOGRAPHY IN RESEARCH AND QUALITY ASSESSMENT OF CERAMIC MOLDS FOR PRECISE CASTING OF RESPONSIBLE MACHINE PARTS | <i>Adam Tchórz</i>      | Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland   |
| 10.15 | INSIGHT INTO THE DIFFUSION OF ELECTRICALLY ACTIVE AND INACTIVE IMPURITIES   | <i>Adrianna Wójcik</i>  | Łukasiewicz Research Network – Institute of Microelectronics and Photonics, Warsaw, Poland; Warsaw University of Technology, Faculty of Physics, Warsaw, Poland |
| 10.35 | HPHT SINTERING BEHAVIOUR AND MECHANICAL PROPERTIES OF LOW-CONTENT cBN COMPOSITES WITH TiN AND TiN/SiCw BINDING PHASE                          | <i>Kinga Bednarczyk</i> | Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland   |
| 10.55 |   | <i>Coffee break</i>     |   |



**Session II: Advances in casting technology – chair Tomasz Dudziak**

11.10	USE OF SOFTWARE FOR THE DEVELOPMENT OF ADI WITH AUSTEMPERING IN HEATED AIR	<i>Leonardo Pereira</i>	Federal University of Rio Grande do Sul, Brazil
11.30	THE WEAR MECHANISM OF ZONE-QUENCH CAST STEEL	<i>Krzysztof Jaśkowiec</i>	Łukasiewicz Research Network – Krakow Institute of Technology, Poland
11.50	MACHINE LEARNING AND DCSP METHODS IN ADAPTIVE MULTIAGENT SYSTEM FOR PRODUCTION PROCESS MANAGEMENT	<i>Bartłomiej Śnieżyński</i>	AGH University of Science and Technology, Krakow, Poland
12.10	MICROSTRUCTURE AND MECHANICAL PROPERTIES OF THIN WALL CuNi-ALLOYED AUSTEMPERED DUCTILE IRON (ADI) CASTINGS OBTAINED IN FLOTRET PROCESS	<i>Adam Bitka</i>	Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland
12.30	EFFECT OF TITANIUM INCLUSIONS ON CORROSION OF LOW CARBON STEEL	<i>Ali Sheikh</i>	AGH University of Science and Technology, Faculty of Foundry, Krakow, Poland
12.50		<i>Coffee break</i>	

**Session III: High temperature and high entropy materials – chair Wojciech Polkowski**

13.05	Mo-Si-B ALLOYS AS ULTRA-HIGH MATERIALS BEYOND SUPERALLOYS: SELECTED ISSUES OF LIQUID STATE PROCESSING	<i>Grzegorz Bruzda</i>	Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland
13.25	EFFECT OF ALLOYING ELEMENTS ON SOLIDIFICATION, MICROSTRUCTURE AND OXIDATION RESISTANCE OF Fe-Cr-Mo-V-Ni-C HIGH ENTROPY WHITE CAST IRON	<i>Willian Martins Pasini</i>	Federal University of Rio Grande do Sul, Brazil
13.45	NUMERICAL AND EXPERIMENTAL MODELING OF RESISTANCE HEATING IN THE GLEEBLE 3800 THERMO-MECHANICAL SIMULATOR	<i>Thi Thu Trang Nguyen</i>	AGH University of Science and Technology, Krakow, Poland
14.05	MICROSTRUCTURE OF HIGH ENTROPY ALLOYS AlCoCuFeNiSi <sub>0.5</sub> X WITH CARBON ADDITION FABRICATED BY INDUCTION MELTING	<i>Konrad Chrzan</i>	Łukasiewicz Research Network – Krakow Institute of Technology, Poland

DAY 2

Session IV: Advances in coatings technologies – chair Daniel Tobola

9.00	COMPARISON OF EFFECT OF DIFFERENT INOCULANTS ON MICROSTRUCTURE OF MAGNESIUM ALLOY AZ91	<i>Dominik Mikusek</i>	Lodz University of Technology, Department of Materials Engineering and Production Systems, Łódź, Poland
9.20	IMPLEMENTATION OF THE INSTRUMENTAL INDENTATION TECHNIQUE TO DETERMINE THE MICROMECHANICAL AND TRIBOLOGICAL PROPERTIES OF HVOF COMPOSITE COATINGS BASED ON Cr <sub>3</sub> C <sub>2</sub> -NiCr	<i>Łukasz Boroń</i>	Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland
9.40	POSSIBILITIES OF USING SURFACE TEXTURE ANALYSIS AS AN ALTERNATIVE METHOD OF OPEN POROSITY MEASUREMENT	<i>Aneta Łętocha</i>	Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland
10.00	THE ANALYSIS OF THE MICROTEXTURED SUBSTRATE BOUNDARY LAYER IN THE CONDITIONS OF SUSPENSION PLASMA SPRAYING OF ZIRCONIA COATINGS	<i>Tomasz Kielczawa</i>	Wroclaw University of Science and Technology, Faculty of Mechanical Engineering, Wroclaw, Poland
10.20	AUSFERRITIC COMPACTED GRAPHITE IRON OBTAINED WITHOUT HEAT TREATMENT	<i>Barbara Kacprzyk</i>	Lodz University of Technology, Department of Materials Engineering and Production Systems, Łódź, Poland
10.40	INFLUENCE OF BATH COMPOSITION ON MORPHOLOGY, WETTABILITY AND CORROSION RESISTANCE OF ELECTRODEPOSITED Zn-Mn COATINGS	<i>Karolina Chat-Wilk</i>	AGH University of Science and Technology, Krakow, Poland; Łukasiewicz Research Network – Krakow Institute of Technology, Poland
11.00		<i>Coffee break</i>	

**Session V: Additive technologies – chair Grzegorz Skrabalak**

- |       |  |                          |  |
|-------|--|--------------------------|--|
| 11.15 | METAL ADDITIVE MANUFACTURING WITH FDM TECHNOLOGY: THE LATEST RESEARCH DIRECTIONS | <i>Martyna Adach</i>     | Wroclaw University of Science and Technology, Faculty of Mechanical Engineering, Wroclaw, Poland |
| 11.35 | DESIGNING AND EVALUATION OF CONFORMAL COOLING CHANNELS USED FOR HPDC TOOLING     | <i>Marcin Malysza</i>    | Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland                    |
| 11.55 | STRUCTURE OF MATERIAL Fe-C AFTER SPS (SPARK PLASMA SINTERING) PROCESS TECHNOLOGY | <i>Agnieszka Stanula</i> | Silesian University of Technology, Faculty of Mechanical Engineering, Gliwice, Poland            |
| 12.15 |  | <i>Coffee break</i>      |  |

**Session VI: Advances in biomedical and optical technologies – chair Katarzyna M. Marzec**

- |       |   |                        |   |
|-------|---|------------------------|---|
| 12.30 | THE USE OF BIOSPECTROSCOPIC METHODS FOR EVALUATION OF THE INFLUENCE OF KETOGENIC DIET USED IN PREGNANCY ON THE NERVOUS SYSTEM DEVELOPMENT IN OFFSPRING                    | <i>Marzena Rugiel</i>  | AGH University of Science and Technology, Faculty of Physics and Applied Computer Science, Krakow, Poland |
| 12.50 | FTIR MICROSPECTROSCOPY IN IDENTIFICATION OF BRAIN BIOCHEMICAL CHANGES ASSOCIATED WITH THE DEVELOPMENT OF GLIAL SCAR   | <i>Kamil Kawoń</i>     | AGH University of Science and Technology, Faculty of Physics and Applied Computer Science, Krakow, Poland |
| 13.10 | ENGINEERING OF Ag-TiO <sub>2</sub> NANOPLATFORMS FOR PHOTO-INDUCED ENHANCED RAMAN SPECTROSCOPY  | <i>Łukasz Pięta</i>    | Jagiellonian University in Krakow, Faculty of Chemistry, Krakow, Poland                                   |
| 13.30 | ASSESSMENT OF THE IMPACT OF THE MEASUREMENT MODE ON THE RESULTS OF SPECTRAL AND BIOCHEMICAL ANALYSIS CARRIED OUT BY MEANS OF FOURIER TRANSFORM INFRARED MICROSPECTROSCOPY | <i>Aleksandra Wilk</i> | AGH University of Science and Technology, Faculty of Physics and Applied Computer Science, Krakow, Poland |

13.50 NOISE REDUCTION IN OPTICAL SIGNAL DETECTION *Stanisław Karcz* Łukasiewicz Research Network – Krakow Institute of Technology, Krakow, Poland

14.10 SPARK SEPARATION TUBE (SST) – DEVICE FOR AUTOMATED LIQUID LAYERING FOR BLOOD SEPARATION BY DENSITY GRADIENT CENTRIFUGATION *Anita Molenda* Spark-Tech sp. z o.o., Krakow, Poland

14.30 **Closing session:** *Katarzyna M. Marzec, Tomasz Dudziak, Małgorzata Grudzień-Rakoczy*

## **SESSION I:**

# **Innovative materials and material characterization**

Chair: *Rafał Nowak*


*Thursday – 19 May 2022*

## SYNTHESIS AND CHARACTERIZATION OF $\text{MgAl}_2\text{O}_4$ SPINEL VIA MODIFIED SOL-GEL METHOD

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Magnesium Aluminate ( $\text{MgAl}_2\text{O}_4$ ) Spinel (MAS) presents a combination of excellent physical and chemical properties, such as high mechanical strength, refractoriness and resistance to chemical attack, which justify its wide use in refractory materials. Currently, there are several synthesis techniques, in which the sol-gel technique presented in this article has great potential for optimizing and reducing the energy cost in the production of this oxide.


This work aimed to obtain  $\text{MgAl}_2\text{O}_4$  spinel using a modified sol-gel method.  $\text{MgAl}_2\text{O}_4$  powders were synthesized using the precursors  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  and  $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  (hydrated nitrates) and  $\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$  (citrate hydrated). Afterwards, the powders were calcined at temperatures of 800°C, 900°C and 1000°C. One of the main advantages of the method, in addition to being a technically simple and easy-to-perform route, is that during the formation of the gel, the temperature is raised to 90°C for 1 h 30 min, after which the gel is dried. This results in a route of lower energy expenditure when compared to other works that use the technique of obtaining MAS via sol-gel. The  $\text{MgAl}_2\text{O}_4$  obtained were characterized by means of physicochemical techniques including X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy Dispersion X-ray Spectrometry (EDS), Diffraction Granulometry Laser, RAMAN Spectroscopy and Thermogravimetric Analysis (TG/DTA). The results showed that it was possible to form the spinel for the described calcination temperatures. Furthermore, the studied route proved to be viable, presenting a pure spinel, with particle size ranging from 37.53 to 45.99  $\mu\text{m}$ , crystallite size ranging from 14.43 to 23.80 nm, which presents a good potential for its use in refractories containing spinel.

**Keywords:** spinel,  $\text{MgAl}_2\text{O}_4$ , refractories, modified sol-gel method, physicochemical properties

## PASSIVE MAGNETIC METHODS AS NOVEL NON-DESTRUCTIVE TECHNIQUE OF DIAGNOSTIC FOR STEEL WIRE ROPES

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Steel wire ropes are used in every roped transport device, such as cableways, lifts, and hoists. Determining the exact technical condition of the tested wire rope depends on many factors. Although various nondestructive testing methods have been explored and investigated, the authors of the [1] suggest that electromagnetic inspection methods are still the most reliable and frequently used strategy. Still, a researcher should select each sensor and processing technique according to the application environment and defect characterization requirements. In [2], the magnetic concentrating sensor is comprehensively studied to inspect the steel wire ropes' technical condition. The researchers designed the magnetic concentrating sensor and a Hall array sensor and studied their damage detecting effect through simulation and experiment. In research [3], the traditional Hall sensor array was replaced by a magnetic concentrator. The magnetic concentrator can achieve a comprehensive collection of the magnetic flux leakage compared with separate Hall components. Hall components can be significantly reduced, which simplifies the subsequent signal processing.

Passive techniques, which do not require magnetization of the tested object with a strong external field, are becoming increasingly popular. The [4] investigates the influence of the Earth's magnetic field on the diagnostic result. The development status of several essential detection methods, including electromagnetic detection, optical detection, ultrasonic guided wave method, acoustic emission detection, eddy current detection, and ray detection, was reviewed, and their advantages and disadvantages were compared and summarized in [5]. The optical method has shown great potential for application, while other methods are still in the laboratory stage.

This work examines a rope in delivery state and then the same rope with artificially introduced discontinuities. An innovative sensor for detecting broken wires based on the magnetic concentrating principle is proposed to meet the requirements of damage detection of steel wire ropes in various working conditions. The author used the study of the optically pumped magnetometer with a laser pumped caesium module. Based on the research, the author discusses the magnetic passive technique's future use for nondestructive damage detection methods for steel wire ropes. An analysis of the passive technic's future on nondestructive damage detection methods for steel wire ropes was investigated. The obtained results show that based on the study of the distribution of the magnetic induction B along the measuring section of the rope, it is possible to locate the damaged area – the place with the most significant metallic loss. The challenges and difficulties of wire rope inspection lie in the sensor sensitivities and intelligent signal processing techniques and depend on the wire rope and defect types and the inspection conditions.

- [1] S. Liu, Y. Sun, X. Jiang, and Y. Kang, A Review of Wire Rope Detection Methods, Sensors and Signal Processing Techniques, *J. Nondestruct. Eval.* 39 (Nov. 2020) 85. doi: 10.1007/s10921-020-00732-y.
- [2] Y. Zhang, L. Jing, C. Chen, X. Bai, J. Tan, A comprehensive study of the magnetic concentrating sensor for the damage detection of steel wire ropes, *Mater. Res. Express* 7 (Sep. 2020) 096102. doi: 10.1088/2053-1591/abb69b.
- [3] Y. Zhang, L. Jing, W. Xu, W. Zhan, J. Tan, A Sensor for Broken Wire Detection of Steel Wire Ropes Based on the Magnetic Concentrating Principle, *Sensors* 19 (Jan. 2019) 17. doi: 10.3390/s19173763.
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- [5] P. Zhou, G. Zhou, Z. Zhu, Z. He, X. Ding, C. Tang, A Review of Non-Destructive Damage Detection Methods for Steel Wire Ropes, *Applied Sciences* 9 (Jan. 2019) 13. doi: 10.3390/app9132771.

## APPLICATION OF X-RAY COMPUTER TOMOGRAPHY IN RESEARCH AND QUALITY ASSESSMENT OF CERAMIC MOLDS FOR PRECISE CASTING OF RESPONSIBLE MACHINE PARTS

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The production of responsible machine parts in the process of precision casting requires a multilayer ceramic mold to meet a number of specific material and constructional features, which in turn makes the ceramic form one of the most important elements of the casting process. X-ray computed tomography (X-ray CT) is a non-invasive research technique that allows to map the internal structure of the examined object on the basis of its two-dimensional projections recorded at different angles. This technique allows not only qualitative 3D imaging, but also an accurate size analysis, quantification and localization of internal discontinuities.

The article presents the possibilities of using X-ray computed tomography to assess the macrostructure of multilayer ceramic forms used in the process of precise casting of machine parts from Ni superalloy (Fig. 1). The tomographic recording based on the analysis of 2D and 3D images made it possible to identify the type of internal discontinuities and areas of their deposition, i.e. porosity, along with the determination of its volume, microcracks, delamination between the mold layers and the different thickness of the first layer of the mold. Additionally, an analysis of the morphological features of porosity was performed in a quantitative and spatial (3D) way.

The research was carried out for ceramic casting molds, which were characterized by a different material configuration, i.e. matrix-based on quartz and molochite sand and water-dilutable binder – containing colloidal silica.

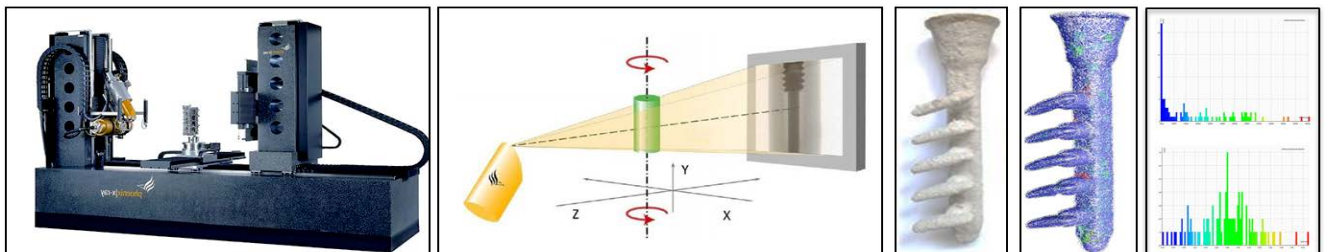


Fig. 1. Scheme of examinations of ceramic casting molds by X-ray computed tomography along with an exemplary result of a mold test using the VGStudioMAXg software (the vertical scale shows the values characterizing the dimensions of defects as a function of their color).

**Keywords:** X-ray computed tomography, ceramic moulds, Ni-base superalloys, precision casting

**Acknowledgments:** Work: “Application of X-ray computed tomography in research and quality assessment of ceramic molds for precise casting of responsible machine parts” (No. 1604/00) as part of the Development Fund – financed from the own funds of the Łukasiewicz Research Network – Krakow Institute of Technology.



## INSIGHT INTO THE DIFFUSION OF ELECTRICALLY ACTIVE AND INACTIVE IMPURITIES

Adrianna Wójcik<sup>1,2\*</sup>, Walery Kołkowski<sup>3</sup>, Andrzej Materna<sup>1,4</sup>, Emil Tymicki<sup>1,4</sup>, Włodzimierz Strupiński<sup>2,3</sup>  
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Precise and reliable determination of the spatial distribution of dopants in the structure as well as prediction of its temperature behavior is crucial for the practical applications of semiconductors. Standard dynamic Secondary Ion Mass Spectrometry (SIMS) provides depth profiles of impurity atoms without distinguishing electrically active and inactive parts. Whereas Electrochemical Capacitance Voltage (ECV) technique yields depth profiles of all carriers present in the structure. The observation of significant discrepancies between the profiles measured by these two techniques, which exceeds beyond their measurement uncertainty, sparks the discussion on the varying diffusion of electrically active and inactive impurities.

In our research, we aim to show a clear distinction between diffusion rates of total and electrically active impurities for various doped III-V structures. The initial ECV and SIMS experiments suggest that the total impurity distribution extends substantially deeper than its electrically active part. Furthermore, the results imply higher mobility of inactive impurities under thermal treatment, that presumably can move interstitially.

In the first experiment, we plan to investigate the Si-doped GaAs layer of 500 nm thickness embedded between undoped GaAs layers (GaAs/GaAs: Si/GaAs). Samples subjected to thermal treatment at various temperatures ranging between (800–1000)°C for 30 minutes will be tested using SIMS and ECV techniques, which will provide data for the quantitative determination of the diffusion parameters of the active and total impurity giving a starting point for a discussion on the diffusion mechanisms. The proposed complementary use of SIMS and ECV techniques shows a great promise to be a highly beneficial tool in the optimization of growth processes, determining the cause of electrical inactivity and controlling the level of dopant activation.

## HPHT SINTERING BEHAVIOUR AND MECHANICAL PROPERTIES OF LOW-CONTENT cBN COMPOSITES WITH TiN AND TiN/SiC<sub>w</sub> BINDING PHASE

Kinga Bednarczyk<sup>1\*</sup>, Piotr Klimczyk<sup>1</sup>, and Marcin Podsiadło<sup>1</sup>

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Cubic boron nitride composites belong to a group of a superhard materials used for cutting tools for the most demanding applications, especially for machining of hardened steel, cast iron and nickel superalloys. Due to the high hardness, fracture toughness, good thermal stability and properties control ability, the low-content cBN composites are currently being developed.

In this work, the effect of SiC whiskers addition on the sintering behaviour and properties of cBN composites was investigated. For this purpose, cBN-TiN and cBN-TiN/SiC<sub>w</sub> composites were fabricated. The binding phase for all composites was 35 vol.%, in which the SiC<sub>w</sub> content was 0 vol.% or 10 vol.%. Due to the technological aspects of whisker mixing, the ball milling (BM) and ultrasonic mixing (UM) method were applied to compare the effect of mixing parameters for microstructure homogeneity. It was found that the ball milling resulted in highly homogenous microstructure but caused numerous whiskers breakage as well as WC contamination that came from milling balls. Ultrasonic method did not lead to whiskers fracture and allowed to achieve satisfactory compound distribution. In addition, this method was much faster and less technologically advanced than ball milling.

All composites were sintered by High Pressure – High Temperature method for 60 seconds under 7.7 GPa. The sintering temperature ranged from 1700 to 2200°C. This sintering parameters allowed to achieve almost fully dense composites. For BM composites, a noticeable dependence was observed between density and sintering temperature, for both cBN-TiN and cBN-TiN/SiC<sub>w</sub> composites. For cBN-TiN composites, the density increased from 1700 to 1900°C and then stabilized. For cBN-TiN/SiC<sub>w</sub> composites, the density growth was observed up to 2100°C and then the density decreased slightly. Similar correlations exist for Young modulus. This is the typical behaviour of materials during the HPHT process. The increase of the density and the Young's modulus is related to the diffusion processes of mass transport and the plastic deformation under pressure and temperature. When the optimum temperature for a given material is exceeded, micro-cracking of the specimen occurs as a result of stress accumulation, which leads to a reduction in density, Young modulus and toughness. In the case of UM materials such a significant correlation has not been observed. Vickers hardness test revealed that for both composites manufactured by ball milling the hardness also increased with sintering temperature and reached its maximum in about 2000°C. SiC<sub>w</sub> addition resulted in higher HV1, but this effect was not very significant. Furthermore, the hardness increase was not noticed for composites manufactured by ultrasonic mixing. Scanning Electron Microscopy observations have shown that the microstructure remained homogenous after sintering but SiC whiskers were hardly visible, especially for BM composites. For UM composites, the whiskers were randomly distributed among the equally spaced TiN and cBN grains. Whiskers elongated shape and significant length (the length of whiskers was comparable to size of Vickers indentation mark) were the cause of large spreads of measured hardness in UM materials. Ball milling provided better results despite the whiskers breakages. The best sample achieved by ball milling had Young modulus of 600 GPa and HV1 about 25 GPa. Planned cutting tests will determine the suitability of these materials for cutting of Inconel 718.

**Acknowledgements:** This work was supported by Ministry of Science and Higher Education of Poland (project No. 3034).

**SESSION II:**  
**Advances in casting technology**

Chair: *Tomasz Dudziak*

*Thursday – 19 May 2022*

## USE OF SOFTWARE FOR THE DEVELOPMENT OF ADI WITH AUSTEMPERING IN HEATED AIR

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When submitted to a heat treatment cycle of austempering, nodular cast iron has its microstructure and mechanical properties improved and is now called Austempered Ductile Iron (ADI). The traditional means of austempering are mixtures of salt baths ( $\text{NaNO}_2$ ,  $\text{NaNO}_3$ ,  $\text{KNO}_3$ , etc.). The use of molten salts, in addition to making the heat treatment process more expensive, has environmental and operational restrictions. The use of Pb-based metallic baths faces severe environmental restrictions. Some studies have evaluated the use of metallic Sn and Zn baths, with research also related to obtaining ADI in mold. The use of gas flow to perform the cooling was successful on a laboratory scale. In this work, we seek the appropriate nodular iron alloy to obtain ADI in a CPy using heated air.

Six alloys are proposed, varying only in the nickel content, being 3.3% C, 2.7% Si, 1.0% Cu, 0.3% Mn, 0.2% Mo, 0.1% Cr, and 0, 0.4, 0.8, 1.2, 1.6 and 2% Ni. The thermodynamic equilibrium composition of austenite with graphite at the austenitization temperature of 900°C was simulated in FactSage. Using the equilibrium composition of austenite, the austemperability of nodular iron was estimated through TTT diagrams obtained using the MUCG83 software. CPy cooling curves were simulated in the Solidworks, with the airflow at a temperature of 340°C with velocities of 5, 10, and 15 m/s. In this way, the suitable alloy for cooling with heated air was determined. The chemical composition and the austempering cycle ( $T_\gamma=900^\circ\text{C}$ ,  $t_\gamma=120$  min;  $T_a=340^\circ\text{C}$ ,  $t_a=90$  min) were used to determine the mechanical properties of ADI.

The carbon content in the austenite in equilibrium with the graphite nodules at 900°C is illustrated in Figure 1A. Increasing the nickel content results in a reduction in the solubility of carbon in the austenite. The CPy cooling curves are superimposed on the TTTs diagrams and shown in Figure 1B.

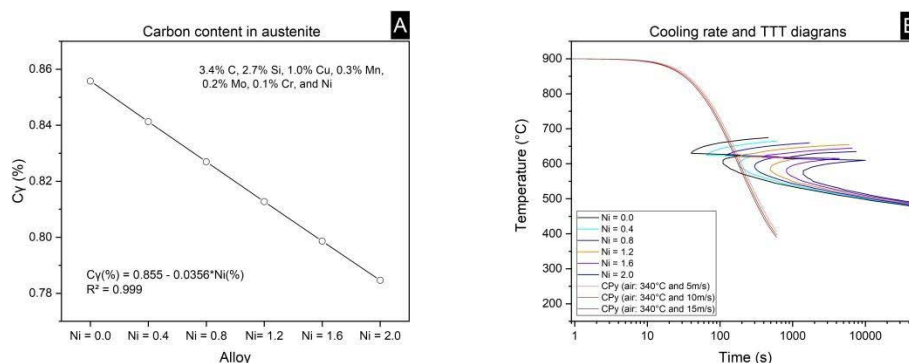


Fig. 1. Effect of nickel on the solubility of carbon (A). Cooling curves and TTT diagrams (B).

It was observed that the alloy containing 1.2% Ni is possibly suitable for obtaining pearlite-free ADI. The estimated mechanical properties for this alloy in the proposed austempering cycle are 1179 MPa of ultimate tensile strength, 923 MPa of yield strength, 7% of elongation, and 354 HV of hardness.

## THE WEAR MECHANISM OF ZONE-QUENCH CAST STEEL

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New cast steel mechanical properties can be obtained by modifications mechanisms related to the introduction of defects into the crystal lattice. Presented publication refers to zonal heat treatment. Indicated process increases the resistance of the dislocation movement through creating obstacles to their movement. Selected heat treatment process allows obtain greater hardness and strength in the selected area of the workpiece, which may be subject to intensive abrasive wear. In addition to modern zone hardening, the procedure for the production of test material and the method of preparing samples intended for abrasive resistance tests are also presented. The presented results refer to samples in which the boron content was modified. The tests were carried out at the pin-on-disc station with dry slide, the tests were carried out at different linear speeds, i.e.: 0.8 m/s and 0.4 m/s. During the tests, the pressure force was also modified. In addition to the coefficient of friction, a loss weight of the samples was also recorded during the test. An analysis of samples surfaces using scanning electron microscope (SEM) imaging was also carried out, and EDS studies of selected areas allowed to assess the mechanisms of wear of the tested samples. The samples were subjected to XRD tests. The influence of boron on abrasion resistance is an important practical issue, because it is an element whose effect in steel is noticeable at very small values compared to Cr, Mo or V.

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## MICROSTRUCTURE AND MECHANICAL PROPERTIES OF THIN WALL CuNi-ALLOYED AUSTEMPERED DUCTILE IRON (ADI) CASTINGS OBTAINED IN FLOTRET PROCESS

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Thin wall austempered ductile iron (ADI) castings may be considered as a low cost alternative for constructional parts made of steel or even aluminum alloys. The aim of this study was to determine the effect of the graphite nodules number in thin-walled ductile iron castings with the addition of Ni and Cu obtained in the Flotret process on the mechanical properties. The amount of graphite spheroids N in the castings as well as other graphite parameters were determined with the image analysis software. In the castings with the wall thickness of 2, 3, 5 and, for comparison, 13 mm number of graphite spheroids N was equal 500–560, 400–450, 380–420 and 230, respectively. Based on the dilatometric test results carried out with a quenching dilatometer, the optimal times for a given temperature of both austenitizing and austempering processes of castings heat treatment were obtained. The austempering step of the heat treatment (isothermal quenching in molten salt) was carried out at 270°C and 390°C in order to obtain the structure of lower and upper ausferrite, respectively. Tensile tests have shown that in the case of the lower as well as upper ausferrite, the number of graphite nodules does not significantly influence the tensile properties. For the higher austempering temperature the obtained tensile strength was between the range of 900–950 MPa with 6–9% elongation, and in the case of the lower austempering temperature the tensile strength above 1400 MPa was achieved with 2–4% elongation.

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## EFFECT OF TITANIUM INCLUSIONS ON CORROSION OF LOW CARBON STEEL

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This study investigates the effects of titanium on corrosion of low carbon steel to mark its validity of use in wet environments. The research is further connected to the kinetics and thermodynamics of steel casting as the next level of work. Two out of four samples provided by the casting industry have been prepared in metallography setup and quantitatively characterized by spectroscopy and have been subjected to corrosion experiments in the carbon dioxide environments electrochemically. An anomaly in the theoretical knowledge is observed during the experiments. Surface topography is consistent with the laboratory results however the microstructural evaluation is somewhat paradoxical. It is important to state that two samples with slightly different compositions with regard to titanium in steel have been worked on. The experiments have shown that there is a small effect of inclusions on corrosion behaviour of steel with slightly less or more amount of titanium. This is not in full compliance with the theoretical findings. This shows that the percentage of iron and significantly included elements have rather more effect on corrosion behaviour than titanium itself. The environment and the setup of experiments have been the same and only a slight difference has been noted in corrosion resistance which theoretically increases considerably with the titanium inclusions. Surface topography has shown similar features in the two cases studied. Pearlite has been observed predominantly in the optical microscopy defining the basic microstructure. The composition of elements in the samples and the graphical plots of corrosion parameters have been explicitly enunciated.

## **SESSION III:**

# **High temperature and high entropy materials**

Chair: *Wojciech Polkowski*

*Thursday – 19 May 2022*



## Mo-Si-B ALLOYS AS ULTRA-HIGH MATERIALS BEYOND SUPERALLOYS: SELECTED ISSUES OF LIQUID STATE PROCESSING

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In order to further increase an efficiency of energy related devices (e.g. internal combustion engines) new materials ensuring much higher working temperatures have to be introduced. In this regard a special attention is given to lightweight materials incorporating refractory metals. Among them, boron doped Molybdenum Silicides (Mo-Si-B alloys) have been recognized as particularly attractive candidates due to an excellent combination of low density and (ultra) high temperature strength. Although a high potential applicability has been proven at a lab scale, a bottleneck for wide commercialization of these materials is located within inefficient multi-stage and expensive fabrication methods.

Therefore, the main goal of our study is to develop new clean, efficient and cost-effective method for producing B-doped Mo silicides. We propose to use **pressure-less Reactive Melt Infiltration (RMI)** approach. Basically, in the RMI process a prepared preform or a near net-shaped template is infiltrated and densified by molten material under certain temperature/pressure/time conditions. We are adopting this concept for the Mo-Si-B system, i.e. a molybdenum preform is subjected to a direct contact with Si-B melt. The selected results of sessile drop experiments carried out under various temperature (up to 1550°C); and by using either bulk or porous Mo substrates, are discussed.

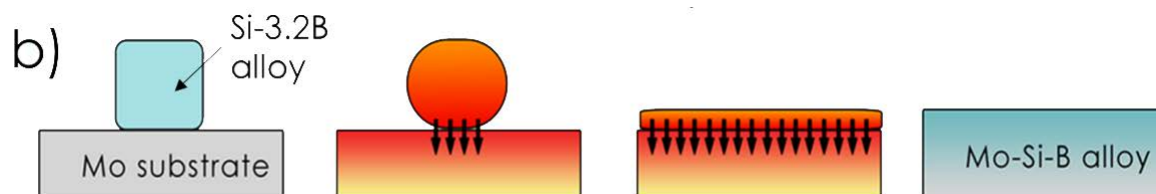


Fig. 1. A scheme of the explored RMI process for a fabrication of Mo-Si-B alloys.

**Keywords:** Mo-Si-B alloys, sessile drop method, liquid assisted processing, interfaces

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## EFFECT OF ALLOYING ELEMENTS ON SOLIDIFICATION, MICROSTRUCTURE AND OXIDATION RESISTANCE OF Fe-Cr-Mo-V-Ni-C HIGH ENTROPY WHITE CAST IRON

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Nowadays, the concept of high entropy alloys (HEAs) has expanded the limits of metallurgy and instigated many researchers to explore a variety of new alloys compositions to develop new materials for specific applications [1,2]. Considering Multi-Component White Cast Iron (MCWCI) systems, some boundaries conditions of high entropy alloys could be applied to the modeling of new alloys [3,4]. High Chromium Cast Irons and MCWCI were known for good erosive and abrasive wear resistance. However, many of the applications for these newly designed materials were thought for high temperatures applications, for this reason, the high-temperature behavior of these alloys needs to be investigated [5]. The present paper investigated the individual and interaction effects of the elements V, Mo, and Ni additions on the solidification phenomena and oxidation behavior. To fully understand the effects of the V, Mo, and Ni additions on the solidification sequence Continuous Cooling/Computer Aid (CC/CA) data were collected for the alloys, based on the 1st derivative and 2nd derivative analysis only the main effect of Mo has a strong influence in Tliquidus, Teutectic, and Tsolidus. Nickel and Vanadium do not have a significant effect on Liquidus temperature, however, influence the Teutectic and Tsolidus. The oxidation behavior tests of new alloys were carried out in air at temperatures of 973 K for 500 h. As result, the mass gain increases with an increase of the additions; the effects of Ni additions on the oxidation behavior are more significant at the first steps of exposure to the oxidation temperatures of 973 K. The addition of V and Mo starts to play a more significant effect on the oxidation behavior after the first steps of exposure and these additions present a multiply interaction effect to the mass gain.

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## NUMERICAL AND EXPERIMENTAL MODELING OF RESISTANCE HEATING IN THE GLEEBLE 3800 THERMO-MECHANICAL SIMULATOR

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In this work, the advanced numerical model of resistance heating [1–5] (including melting and free cooling) process of steel samples S355 are presented (Figure 1a). The experimental part was performed using Gleeble 3800 thermo-mechanical simulator (Figure 1b). Cylindrical samples ( $\varnothing 10$  mm and length 124 mm) and two kinds of copper grips were used. During experiments the temperature was determined in four different measuring points (e.g. copper grip, in the core of the sample, surface). The experimental and numerical procedure consists of four stages: in the first stage, the sample was heated at a rate of  $20^{\circ}\text{C}/\text{s}$  to the nominal temperature of  $1250^{\circ}\text{C}$ , and then  $1^{\circ}\text{C}/\text{s}$  to  $1300^{\circ}\text{C}$  (second stage). Before free cooling to the ambient temperature, the 30 seconds holding stage was applied in order to obtain a uniform temperature in the volume of the test sample. Finally, numerical model verification was performed based on the experimental results.

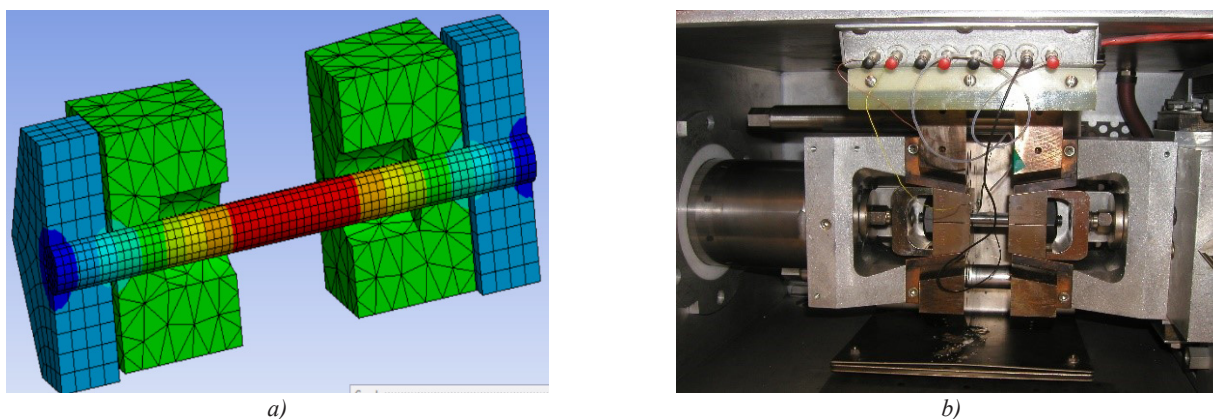


Fig. 1. Numerical model (a) and the view of chamber of Gleeble 3800 equipment (b)

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## MICROSTRUCTURE OF HIGH ENTROPY ALLOYS $\text{AlCoCuFeNiSi}_{0.5x}$ WITH CARBON ADDITION FABRICATED BY INDUCTION MELTING

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High entropy alloys are new group of materials, which are classified as multicomponent alloys. According to main rule, entropy of mixing for alloy in liquid state should be higher than 1.5 R, where entropy is calculated according to the following formula [1]:

$$\Delta S_{conf} = -R \sum_{i=1}^n X_i \ln X_i$$

Where:

R – gas constant,  $8.314 \frac{\text{J}}{\text{mol}\cdot\text{K}}$

$X_i$  – mole fraction

Due to occurrence large amount of chemical elements, in opposite to conventional alloys, all elements are in similar concentrations. It affects in untypical properties in some of HEA's. In presented results, alloy consisting of such elements as Al, Co, Cu, Fe, Ni, Si with small additions of C was tested. All of alloys were produced by induction melting with inert gas blow on metal bath surface, to reduce oxidation of input materials. Use of an induction furnace allowed to reduce chemical segregation. Magnetic field acting on input materials causing intensively mixing of liquid metal, decreasing inhomogeneity. Prepared alloy was casted into ceramic molds. Next, ingots was treated by homogenizing annealing in 980°C for 10 hours. After that, specimens from treated alloys was made. Microstructure images was prepared by using optical microscope and scanning electron microscope. Additionally EDS maps and hardness test was conducted. Research was showed that alloys possess complex structure, where are at least two phases. Alloy have high brittleness, so some changes in chemical compositions are required.

**Acknowledgements:** The author grateful acknowledge the funding of research by the Ministry of Education and Science (subsidy No. 3002/00).

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## **SESSION IV:**

# **Advances in coating technologies**

Chair: *Daniel Toboła*

*Friday – 20 May 2022*

## COMPARISON OF EFFECT OF DIFFERENT INOCULANTS ON MICROSTRUCTURE OF MAGNESIUM ALLOY AZ91

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Using inoculants to change the microstructure in magnesium alloys was a way of making magnesium casting with better properties with not very high cost. In this research as inoculants were used zirconium, yttrium, gadolinium, “mischmetal” and Emgesal Flux 5®. Castings were made with use ceramic mould. Each of the inoculant were added to magnesium alloy AZ91 in concentration from 0.1% to 0.6%.

Those five inoculants were chosen based on the literature because they make significant impact on the microstructure of magnesium alloy AZ91. In this research, were measured two main parameters: average difference of perimeters and average difference of diameters of two main phases –  $\alpha_{\text{Mg}}$  and  $\alpha_{\text{Mg}} + \gamma(\text{Mg}_{17}\text{Al}_{12})$  in inoculated samples in reference to AZ91. In every concertation of each inoculant, which was taken under the consideration, there were observed changes in microstructure. At first, changes of both parameters of microstructure were measured and compared in every inoculant to verify which concentration is optimal. At second part, analysed parameters of microstructure with different inoculants were compared to each other. The results made possible to evaluate every inoculant in obtaining microstructure with smaller grains which can lead to achieve higher mechanical properties without significant change of the chemical composition. That casting's production process could reduce cost of production parts from magnesium alloys with higher properties. Which is why, parts made out of that inoculated alloys could replace the same parts made from aluminium alloys, especially in automotive industry, aviation industry, space industry etc.

**Keywords:** theory of crystallization, magnesium alloy AZ91, DTA method, microstructure analysis

## IMPLEMENTATION OF THE INSTRUMENTAL INDENTATION TECHNIQUE TO DETERMINE THE MICROMECHANICAL AND TRIBOLOGICAL PROPERTIES OF HVOF COMPOSITE COATINGS BASED ON Cr<sub>3</sub>C<sub>2</sub>-NiCr

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The high-velocity oxy-fuel (HVOF) is widely used thermal spraying method due to the quality of produced coatings in term of porosity, hardness and fracture toughness. Coatings containing chromium carbide particles distributed in a nickel-chromium alloy matrix have been used in a wide range of applications, both industrially and in the aerospace sector, for instance.

The aim of the investigations was to compare the microstructure, mechanical and tribological properties of Cr<sub>3</sub>C<sub>2</sub>-NiCr+Co and Cr<sub>3</sub>C<sub>2</sub>-NiCr coatings deposited on ductile cast iron by HVOF. The effect of cobalt particles added to the chromium carbide coating on mechanical and tribological behavior in the system of Cr<sub>3</sub>C<sub>2</sub>-NiCr+Co/ductile cast iron was analysed in order to improve the lifetime of coated materials. The structure with particular emphasis of characteristic of the interface in the system of composite coating (Cr<sub>3</sub>C<sub>2</sub>-NiCr+Co)/ductile cast iron was studied using the light (LM) and scanning electron (SEM) microscopes, as well as the analysis of chemical and phase composition in microareas (EDS, XRD). Experimental results show that HVOF sprayed Cr<sub>3</sub>C<sub>2</sub>-NiCr+Co composite coating exhibits low porosity, high hardness, dense structure with large, partially molten Co particles and very fine Cr<sub>3</sub>C<sub>2</sub> and Cr<sub>7</sub>C<sub>3</sub> particles embedded in NiCr alloy matrix, coming to the size of nanocrystalline. The results were discussed in reference to examination of selected mechanical properties such as instrumented indentation and scratch testing, which also determine the adhesion of the produced coatings to the substrate.

It was found that the addition of Co particles was significantly increase resistance to cracking and wear behavior in the studied system.

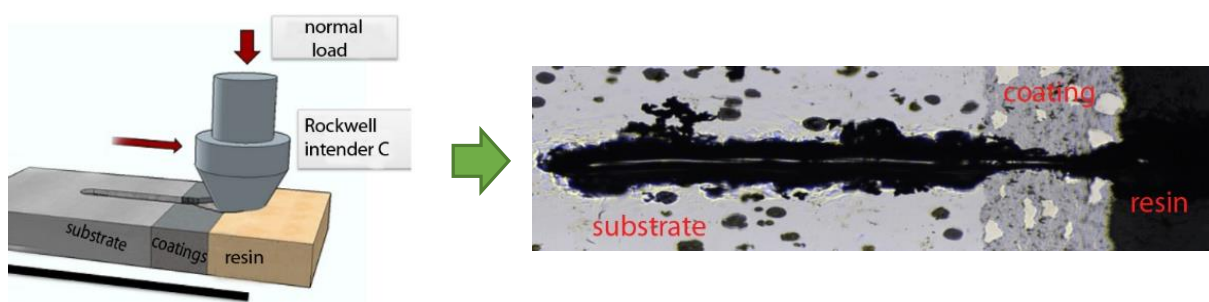


Fig. 1. A schematic of scratch adhesion test of thermal ceramics coatings.

**Keywords:** Cr<sub>3</sub>C<sub>2</sub>-NiCr coating, HVOF spraying, instrumented indentation, microstructure, scratch test, adhesion

**Acknowledgments:** Work: “Implementation of the instrumental indentation technique to determine the micromechanical and tribological properties of ceramic coatings” (No. 2045/00) financed from the funds for subsidies of the Ministry of Science and Higher Education.

## POSSIBILITIES OF USING SURFACE TEXTURE ANALYSIS AS AN ALTERNATIVE METHOD OF OPEN POROSITY MEASUREMENT

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Roughness parameters related with material ratio curve are atypical. They are calculated differently than the most commonly used height parameters of roughness. The diagram of material ratio curve, with the determined volume parameters of roughness is shown in the Fig. 1. There are studies in the literature that analyze volume parameters in order to obtain additional information related to the analysis of open porosity [2–3]. The aim of the study was to analyze the possibility of using surface texture analysis as an alternative method of open porosity measurement – for samples of a specific shape.

Ceramic ( $\text{Al}_2\text{O}_3$ ,  $\text{ZrO}_2$ ) and metallic (Zr) materials were selected in the research. The particle size range of the powders used was 0.0001–1 mm. The powders were sintered by Spark Plasma Sintering method using HP D5, FCT machine. Samples were sintered in the temperature range 1100–1650°C for 1–10 minutes and at the pressure of 10–48 MPa. Argon was used as the protective atmosphere for all samples. Surface texture tests were carried out using the confocal profiling method available in the Altisurf 520 optical measurement system. Some roughness measurements were also made with the TOPO 02 contact profilometer.

The research has shown that for the analyzed materials and measurement methods, it is not possible to unambiguously determine the open porosity.

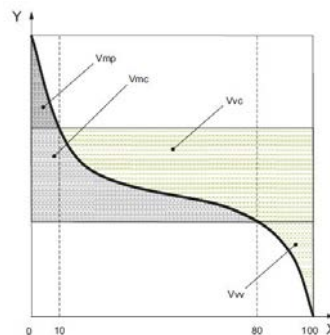


Fig. 1. The diagram of material ratio curve, with the determined volume parameters of roughness.

**Acknowledgements:** This work was supported by the Łukasiewicz – Krakow Institute of Technology, Poland.

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## THE ANALYSIS OF THE MICROTEXTURED SUBSTRATE BOUNDARY LAYER IN THE CONDITIONS OF SUSPENSION PLASMA SPRAYING OF ZIRCONIA COATINGS

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In this work, micro-scale laser processing is studied in order to structure bond coat and control the columnar Thermal Barrier Coating (TBC) build-up mechanism. This, in turn, opens the possibility to enhance the TBC performance for thermally stressed parts.

The investigation contain both numerical simulations and experimental trials. Firstly, metallic bond coat was sprayed on super alloy coupons by the means of Atmospheric Plasma Spraying (APS). Then, the metallic interlayer was microtextured prior to ceramic top coat spraying. After processing, as sprayed columnar TBCs were tested in terms of thermal performance. Both long term furnace oxidation test and furnace thermal fatigue cyclic tests (FCT) were performed to study the effect of laser pretreatment on plasma sprayed TBCs thermophysical properties.

In addition to the experimental approach the build-up mechanism was investigated numerically. The microtextured bond coat was modeled in a microscale with the geometry corresponding to the pattern of microtextured bond coat (Fig. 1). To estimate the plasma flow boundary conditions for microscale calculations, preliminary models were prepared and solved. The initial stage of numerical investigation considered entire spraying domain including interaction between plasma jet and the microtextured substrate as well as turbulent phenomena related to the plasma flow. Then, with plasma flow boundary conditions extracted from initial calculation results, advanced numerical models were introduced to the simulation including Discrete Phase Model (DPM) for molten particles tracking, and the heat transfer equations for modeling the heat flux to the substrate. Plasma flow mapping considered also the interaction between the continuous and discrete phase and the diffusion of plasma components with the use of single phase mixture model. Mentioned governing equations were solved under time dependent solver with PISO pressure-velocity coupling and fine time advancement to enhance the tracking resolution.

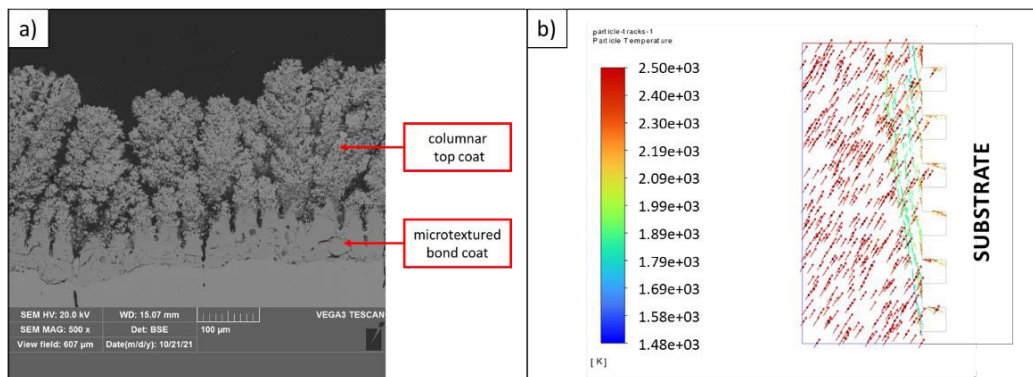


Fig. 1. Columnar TBC's interface cross-section, a) as sprayed, b) numerical model.

## AUSFERRITIC COMPACTED GRAPHITE IRON OBTAINED WITHOUT HEAT TREATMENT

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This article presents research concerning the possibility of ausferrite obtaining in cast iron with vermicular (compacted) graphite without the heat treatment of castings. Compacted graphite was obtained according to Inmold technology. The research on the effect of the cast iron chemical composition on the magnesium concentration in casting providing the compacted graphite was part of preliminary research only partially described in this article.

The effect of the applied alloying additives, i.e. molybdenum, nickel, and copper, on the temperature of phase transformations, both in the liquid and solid-state, was investigated. The possibility of registering a small thermal effect during the transformation of austenite into bainitic ferrite was demonstrated. The temperature of this transformation was determined. The impact of individually added the above-mentioned elements on the microstructure of compacted graphite iron. The results made it possible to evaluate the effect of molybdenum, nickel, and copper added in common on the possibility of obtaining ausferrite in cast iron with compacted graphite. Based on microstructural tests, the scope of alloying elements, providing a “pure” ausferritic matrix in castings with a wall thickness of 3–24 mm was determined. The mechanical properties of the newly developed ausferritic compacted graphite iron were determined. It has been shown that these properties are much more advantageous than the standard grades of cast iron with compacted graphite. The wear resistance of ausferritic compacted graphite iron vs. its chemical composition was also tested. Tested wear resistance was compared with different types of nodular cast iron, including ADI. Based on the results of these tests, it was unequivocally found a high abrasive wear resistance of ausferritic compacted graphite iron obtained without heat treatment of castings, which makes it suitable for various applications on machines and devices parts working in hard conditions, e.g. combustion engine blocks or brake discs.


**Keywords:** theory of crystallization, compacted graphite iron, DTA method, ausferritic compacted graphite iron

## INFLUENCE OF BATH COMPOSITION ON MORPHOLOGY, WETTABILITY AND CORROSION RESISTANCE OF ELECTRODEPOSITED Zn-Mn COATINGS

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Electrodeposited zinc-manganese alloys are known as anticorrosive coatings for steel substrates showing a protective ability much better than of pure zinc in many environments [1]. This improved protective ability was attributed to a formation of manganese oxide passive layer during or water exposure and/or sparingly soluble basic zinc salts [2] during corrosion in aqueous media. The best alloy characteristics was observed mostly for moderate element contents (up to 30% Mn), however, some authors stated that the corrosion resistance of the Zn-Mn alloys is more dependent on the coating's morphology than its composition [3]. The corrosion resistance of the zinc-manganese coatings can be enhanced by formation of the surfaces having high water repellency. It has been proved that superficial structure in a micro- and nanoscale help trapping a large amount of the air when metallic surface is exposed to the air atmosphere reducing attractive interactions between the solid surface and water droplets and thus hindering formation of the corrosion cells [4]. Surface morphology of the electrodeposited metallic layers is affected by many factors like current/potential conditions, bath composition and pH. Highly hydrophobic metallic deposits are produced predominantly by using various modes of the electrolysis carried out in aqueous solutions followed often by further modification of the surface with chemical compounds of low surface energy [5]. A literature review showed that there is no information on the influence of the surface morphology of the Zn-Mn electrodeposits on their wettability and, thus, corrosion resistance. Therefore, a study was conducted to determine the possibility of obtaining Zn-Ni alloy coatings characterized by low surface wettability. The effects of chloride and sulfate ions in gluconate baths and cathodic potential on the composition and surface properties of as-plated Zn-Mn alloys were investigated.

**Acknowledgements:** This work was funded by The National Science Centre of Poland under project 2018/31/N/ST8/00908. The authors would like to acknowledge Grzegorz Włoch, Ph.D. for SEM observations and Piotr Osuch, Ph.D. for assistance during WCA measurements.

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**SESSION V:**  
**Additive technologies**

Chair: *Grzegorz Skrabalak*

*Friday – 20 May 2022*

## METAL ADDITIVE MANUFACTURING WITH FDM TECHNOLOGY: THE LATEST RESEARCH DIRECTIONS

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The use of composite filaments in Fused Deposition Modeling (FDM) technology constitutes a new approach in additive manufacturing (AM) for production of metal parts. The metal-plastic composite filaments are characterized by producers as 80–90 wt.% metal content. The process consists of several stages (Figure 1). First, the 3D model is created and green part is printed. At that stage the structure is composed of metal powders distributed in the polymer matrix. Then, the debinding and sintering processes are performed, respectively. During debinding process, the polymers are removed and then, so-called brown part is subjected to sintering which allows obtaining of pure metal part. During that last process stage the shrinkage of about 16 vol.% takes place which should be considered when designing the part.

The topic of use FDM technology to produce metal parts is important in the field of additive manufacturing due to the cost reduction and the high potential to produce prototypes and final products.

This study aims to present the latest research directions for the production of 3D printed metal parts with FDM technology. The presentation shows the comparison of metal 3D printing technologies as well as advantages and disadvantages of using FDM technology to produce metal parts. The own research include the characteristic of the filament, the green part (after 3D printing), the brown part (after debinding) and the final, ready-to-use 3D print.

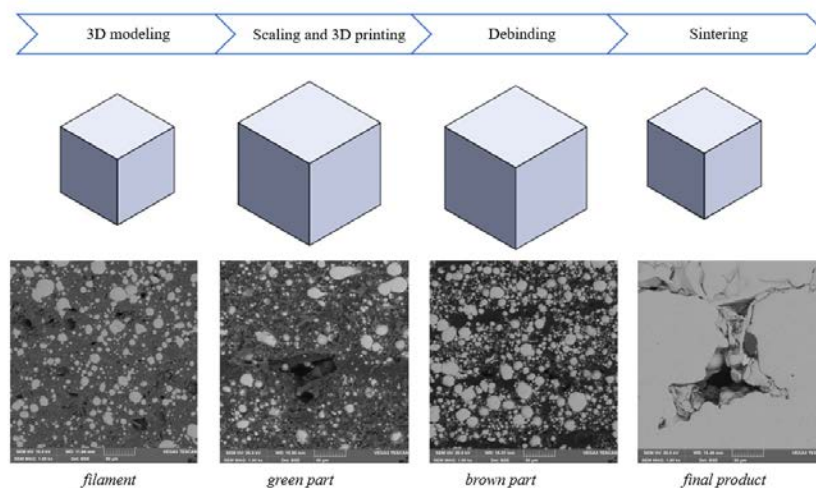



Fig. 1. Process of producing metal parts with FDM technology.

## DESIGNING AND EVALUATION OF CONFORMAL COOLING CHANNELS USED FOR HPDC TOOLING

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The development of 3D printing technology is very fast and more often used to support various production methods. This also applies to high pressure die casting, where liquid metal is moved at high speeds in a piston chamber and gating system. The specificity of this production method is its cyclical nature, which consists in repeating the steps from filling the shot sleeve, filling cast cavity, solidifying and cooling as well as opening the pressure die and casting ejection. The control over heat that arises during cyclic work is very important for the casting, but also for the tool which is the pressure die. The possibility of creating complex shapes of the cooling system, allows to control the heat transfer, which affects the operation life of the inserts, cores and other components of the pressure die. This paper presents the method of designing a conformal cooling system with the use of computer simulation. The influence of the cooling channel shape on the die temperature profile was assessed, as well as the influence on the method of filling and solidification of liquid metal in the volume of the die cavity. Virtual experiments were conducted in a comparative way, the initial shape and the revised versions. Additionally, the influence of changes on stresses in the area of elements stabilizing the die was compared. Finally the porosity prediction of different HPDC die designs is analysed.

**Keywords:** conformal cooling, design, HPDC, stress, aluminium castings

## STRUCTURE OF MATERIAL Fe-C AFTER SPS (SPARK PLASMA SINTERING) PROCESS TECHNOLOGY

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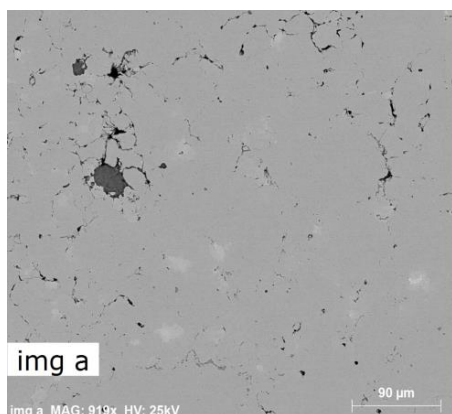
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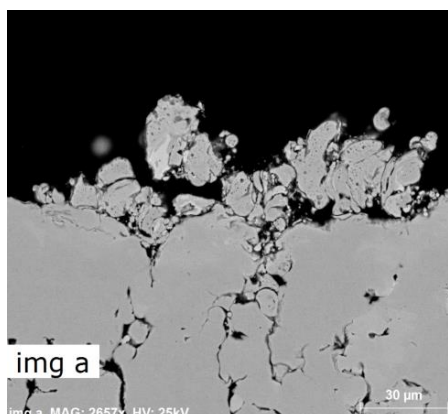
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The subject of this article is the element produced by SPS (Spark Plasma Sintering) process. The material used for this experiment is powder material Fe-C. The main aim of this paper was to present the special characteristics of the sinter metal element. To check, these items a Scanning Electron Microscope (SEM) analysis, Dispersive X-ray Spectroscopy (EDS) analysis, hardness test, density test, and combined carbon content were done. Based on this assessment it was found that this element's properties might be used in the automotive industry.



*Fig. 1. SEM of iron based material Fe-C reinforced by SPS. Magnification 919x*



*Fig. 2. SEM of iron based material Fe-C reinforced by SPS. Magnification 2667x*

**Keywords:** powder materials, SPS (Spark Plasma Sintering), iron based material Fe-C

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## **SESSION VI:**

# **Advances in biomedical and optical technologies**

Chair: *Katarzyna M. Marzec*

*Friday – 20 May 2022*



## THE USE OF BIOSPECTROSCOPIC METHODS FOR EVALUATION OF THE INFLUENCE OF KETOGENIC DIET USED IN PREGNANCY ON THE NERVOUS SYSTEM DEVELOPMENT IN OFFSPRING

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Most of antiepileptic drugs used in pregnancy may induce transient or long-lasting side effects in the nervous system of fetus. Therefore, there is a great need to look for the alternative therapies of epilepsy and the new antiepileptic drugs for pregnant women [1–3]. One of the approaches may be the use of the ketogenic diet, which is successfully applied for a treatment of drug-resistant epilepsy in children and adults [4]. However, due to the fact that the medical data of the influence of metabolic ketosis to the fetus is lacking, more studies is needed in this field [5]. In this study, the influence of ketogenic diet used during rat pregnancy on the developmental changes occurring in the brain of offspring was evaluated. Male Wistar rats at the 2, 6 and 14 days of postnatal life were the subject of the study. Two complementary methods of vibrational spectroscopy (Fourier transform infrared and Raman microspectroscopy) were used for the two-dimensional biochemical analysis of the samples and imaging of the morphological structures. The effect of the ketogenic diet used in pregnancy on the accumulation and distribution of proteins, lipids, cholesterol and compounds containing phosphate and carbonyl groups in the brains of the offspring were examined. Moreover, structural changes of the studied biomolecules were verified. Performed chemical mapping showed that the utilization of the ketogenic diet during pregnancy, in general, does not lead to the biomolecular hippocampal abnormalities. The exception from this rule were changes in the intensity of the absorption band associated with the compounds containing carbonyl groups observed for the group of the oldest animals. Such an effect is probably connected with the elevated exposition to ketone bodies during a fetus life.

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## FTIR MICROSPECTROSCOPY IN IDENTIFICATION OF BRAIN BIOCHEMICAL CHANGES ASSOCIATED WITH THE DEVELOPMENT OF GLIAL SCAR

Kamil Kawon<sup>1\*</sup>, Zuzanna Rauk<sup>2</sup>, Wojciech Kosiek<sup>2</sup>, Zuzanna Setkowicz<sup>2</sup>, and Joanna Chwiej<sup>1</sup>

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Traumatic brain injury (TBI), meaning functional or structural damage which appear as a result of the application of the external physical force, constitutes the main cause of death and disability of individuals and causes a great socioeconomic problem. TBI can be divided into two types, namely the primary one occurring directly after the impact of external forces and the secondary one which may manifest after hours, days or even years from it. The time gap between the primary and secondary injury constitutes an area for research of the new therapeutic strategies and, therefore, the better knowledge about posttraumatic pathological changes occurring in the brain during this period is necessary.

The Fourier transform infrared microspectroscopy (FTIR) was used in this study for topographic analysis of the injured rat brain. In our investigation we used the rat model of penetrating brain injury with blood-brain barrier disruption done at the 30th day of animal life. Rats were divided into four groups consisting of 6 individuals and their brains were examined after 2, 8, 16 and 30 days from the primary injury.

The chemical mapping of selected IR bands revealed that the site of injury is characterized by the significantly decreased accumulation of lipids as well as compounds containing phosphate and carbonyl groups as well as the elevated levels of proteins and cholesterol/cholesterol esters.

## ENGINEERING OF Ag-TiO<sub>2</sub> NANOPLATFOMRS FOR PHOTO-INDUCED ENHANCED RAMAN SPECTROSCOPY

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Surface-enhanced Raman Spectroscopy (SERS) one of the most powerful analytical techniques is widely used for life sciences, environmental protection, and biological analysis. A new technique called Photo-induced enhanced Raman spectroscopy (PIERS) is developed on the basis of traditional SERS but goes one step further. A suitable PIERS substrate can be constructed based on a semiconductor layer with noble metal nanostructures deposited thereon. After the process of its photoactivation with the use of light in the UV range, a significant amplification of the signal in relation to SERS is obtained. The enhancement mechanisms for PIERS are still under debate, thus designing a substrate that would exhibit a high and long-lasting signal enhancement after the photoactivation process remains a considerable challenge.

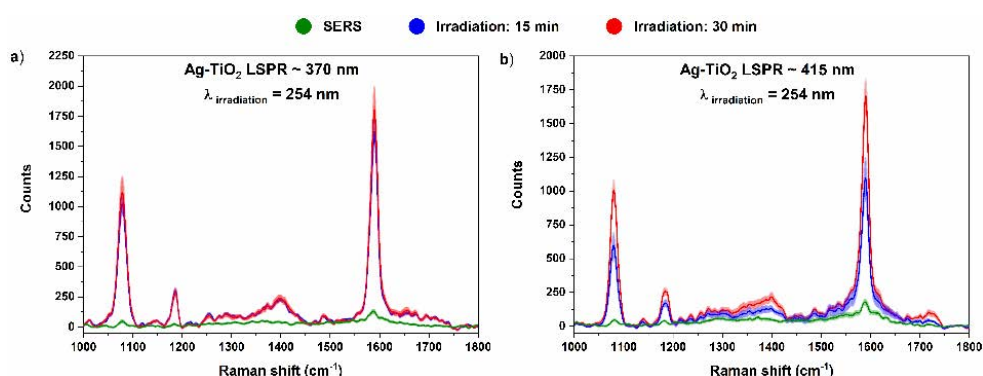


Fig. 1. PIERS (blue and red) and SERS (green) spectra of 4-mercaptobenzoic acid (10-5M). All spectra were acquired under identical measuring conditions.

The aim of this work was to broaden knowledge about the relationship between substrate morphology, its plasmonic features, and the amplification of the Raman signal due to the PIERS effect. Additionally, it was investigated how the wavelength of UV light and photoactivation time of the substrate affect the results.

Our nanoplatfoms were prepared in a two-step procedure [1]. First, a thin, 22-nanometer coating of TiO<sub>2</sub> was obtained on silicon wafers using the sol-gel method, and then Ag nanoparticles (NPs) were photo-grown from AgNO<sub>3</sub> solution under UV illumination. Ultimately, two different Ag-TiO<sub>2</sub> nanoplatfoms were fabricated with maxima of localized surface plasmon resonance (LSPR) at 370 nm and 415 nm. The enhancement of the Raman signal was probed with 4-mercaptobenzoic acid, with and without the photoactivation process using UV light with a wavelength of 254 nm and 365 nm.

The use of a substrate made of the nanometer-thick titanium dioxide film and silver nanoparticles, after activation with both 254 nm or 365 nm UV light allows even for 18-fold PIERS signal enhancement compared to SERS, and, importantly, it lasts up to 4 hours. The obtained results indicate that matching the plasmon resonance of the NPs and the wavelength of the UV light used for photoactivation negatively influences the signal amplification. Moreover, it has been shown that the optimal photoactivation time depends on the number of nanostructures present on the semiconductor surface.

[1] E. Wiercigroch et al., Journal of Raman Spectroscopy (2019) 1649–1660.

## ASSESSMENT OF THE IMPACT OF THE MEASUREMENT MODE ON THE RESULTS OF SPECTRAL AND BIOCHEMICAL ANALYSIS CARRIED OUT BY MEANS OF FOURIER TRANSFORM INFRARED MICROSPECTROSCOPY

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Fourier transform infrared (FTIR) microspectroscopy uses the absorption of mid-infrared radiation to study the distribution and structural changes of major biological macromolecules in a variety of samples, including tissues and cells [1,2]. The registration of IR spectra is usually carried out in the transmission or transmission-reflection (transflection) mode, which, although simpler, may result in the appearance of spectral artefacts resulting from the interfering effects, such as the electric field standing wave effect (EFSW). There is an alternative, the transmission mode which, however, requires the IR-transparent sample carriers and those are expensive and delicate [3–6]. This study addresses differences in the results of brain tissue measurements carried out using both modes. To achieve its goal, 7 samples of brain collected from rats implanted with human glioblastoma and control animals were examined. The samples placed on CaF<sub>2</sub> carriers, transparent for the tested infrared range, and silver coated MirrIR slides were measured, respectively, in transmission and transflection.

The work contains comparative analyses of the results obtained using both measurement modes. It includes spectral analysis and chemical mapping of the examined tissues. Statistical analysis of absolute and relative biomolecules content (Mann–Whitney U test) revealed consistent directions of changes in composition between different parts of brain tissue (striatum, white matter, cortex, glioblastoma) for both modes. What is more, it was concluded that typical characteristics of each mode, resulting from i.e. geometry, were clearly visible in chemical mapping. Transflection mode provided double the absorbance level than the transmission one, better resolution and contrast between adjacent structures. Moreover, in transflection, the 3012 cm<sup>-1</sup> spectral band was clearly visible, whereas it was not almost present in transmission. Additionally, the analysis of averaged spectra revealed that for samples less than 1-micron-thick, the expected EFSW occurrence was above the analytical range of wavenumber  $k \in (1000; 4000)$  [cm<sup>-1</sup>]. For theoretically determined wavenumbers of EFSW occurrence, there was no visible spectra shape disturbance in transflection obtained data. However, for such spectra an elevated baseline was observed.

The conducted analyses confirmed the suspected differences between the compared measurement modes. However, with the appropriate and accurate preparation of tissues, both modes can be used with similar effectiveness for differentiating brain tissue areas.

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## NOISE REDUCTION IN OPTICAL SIGNAL DETECTION

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In electronic circuits, when measuring and processing signals of low amplitude in the low frequency range, the main problems that occur are thermal and shot noises (popcorn noise). The average noise in a system results from the components that were used and the combination of noises generated by these components. During low frequency processing, the noise is inversely proportional to the frequency. Such noises are called 1/f noise or color noise. Color noise has different properties in the frequency domain. Thermal noise is generated by random collisions of carriers with the crystal lattice of a material. Materials and processes can provide different noise levels for similar components. In case of shot noise, the noise is caused by current flow in which the movement of individual carriers is mutually independent and random.

In the detection of optical signals, several techniques can be distinguished to minimize noise and improve the signal to noise (S/N) ratio. It is worth to boxcar average detection and phase detection method.

This paper presents the change of noise levels after reducing thermal and 1/f noises in a charge sensitive preamplifier (CSP) circuit. The circuit was developed to detect optical signals coming from particle counter designed for spheroidal particles. There are also presented methods of signal filtering to enable particle size measurements.

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## SPARK SEPARATION TUBE (SST) – DEVICE FOR AUTOMATED LIQUID LAYERING FOR BLOOD SEPARATION BY DENSITY GRADIENT CENTRIFUGATION

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Cell separation using density gradient centrifugation is a widely used technique in biology and medicine, in 2021 this market segment was valued at USD 1 555 Million. Crucial for this technique is manual procedure of liquid layering (i.e., blood) over density gradient medium.

Here, for the first time Spark Separation Tube (SST) is demonstrated. It is a pending patent device enabling regulation of the flow of liquids, which is essential for automation of liquid layering procedure.

SST comprises of two chambers and a piston type mechanism for liquid flow regulation. Chambers are connected by transfer channel and piston mechanism is movable axially. It is made of a highly clarified random polypropylene copolymer resin designed for injection molding of medical devices and for further sterilization by high energy radiation. Major challenges in prototyping for injection molding was firstly, design the transfer channel enabling continuous blood stream flow and secondly, compensation for the uneven deformation of the elements necessary to maintain the tightness of the piston during injection molding.

Prototype SST was validated in comparison to standard manual methods. Peripheral Blood Mononuclear Cells (PBMC) were isolated either by manual method of liquid layering or by SST. Results show that automation of liquid layering with SST is equal to manual procedure based on PBMC yield ( $5.3 \times 10^6/\text{ml}$  for SST;  $5.1 \times 10^6/\text{ml}$  manually; p value – non significant) and cell viability (>99% for SST; >99% manually; p value – non significant).

SST is a device suitable for automation of broadly applied laboratory technique of cell separation by density gradient centrifugation.

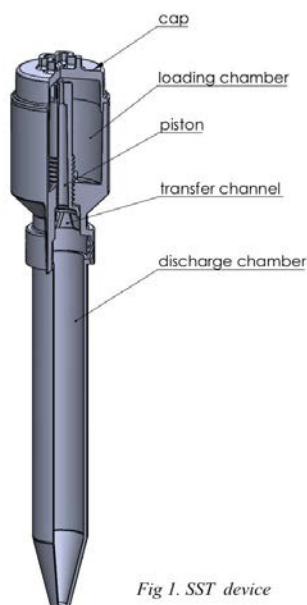


Fig 1. SST device

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## INDEX OF AUTHORS

<b>Name</b>	<b>Page</b>	<b>Name</b>	<b>Page</b>
Adach Martyna	36	Kołkowski Walery	16
Adamski Mateusz G.	45	Kosiek Wojciech	40, 41
Bednarczyk Kinga	17	Kudyba Artur	24
Bellé Matheus Roberto	19	Książek Marzanna	15, 24, 30
Bitka Adam	21, 25	Lech Sebastian	24
Boroń Łukasz	30	Łętocha Aneta	31
Bragança Saulo	13	Małek Kamilla	42
Bruzda Grzegorz	24	Małyszka Marcin	37
Chat-Wilk Karolina	34	Martins Pasini Willian	25
Chrzan Konrad	20, 25, 27	Materna Andrzej	16
Chwiej Joanna	40, 41, 43	Mazurek Paweł	14
Cichocki Kamil	27	Michałowski Paweł P.	16
Drozd Agnieszka	43	Mikusek Dominik	29
Dudek Piotr	37	Molenda Anita	45
Dudziak Tomasz	25	Nowak Rafał	24
Fujawa Tomasz	45	Nguyen Thi Thu Trang	26
Gajoch Grzegorz	44	Olbrich Karolina	43
Giuranno Donatella	24	Oleksy Waclaw	27
Głowacki Mirosław	20, 37	Oliveira Tiago	13
Górny Marcin	21	Pacyniak Tadeusz	29
Grudzień-Rakoczy Małgorzata	20, 27	Pereira Leonardo	19
Gumienny Grzegorz	33	Pięta Łukasz	42
Janik-Olchawa Natalia	43	Pilarczyk Wirginia	38
Jaśkowiec Krzysztof	20, 25	Pirowski Zenon	20
Jaworska Lucyna	31	Piwowarczyk Tomasz	36
Kacprzyk Barbara	33	Piwoński Ireneusz	42
Kalandyk Barbara	27	Podsiadło Marcin	17, 31
Karcz Stanisław	44	Polkowska Adelajda	24
Karczewski Krzysztof	24	Polkowski Wojciech	24
Karlinski de Barcellos Vinicius	19, 25	Ptak Waclaw	27
Kawoń Kamil	41	Putyra Piotr	31
Kiełczawa Tomasz	32	Pysz Stanisław	37
Kisielewska Aneta	42	Rapiejko Cezary	29
Klimczyk Piotr	17	Rauk Zuzanna	40, 41

<b>Name</b>	<b>Page</b>	<b>Name</b>	<b>Page</b>
Rudnik Ewa	34	Strupiński Włodzimierz	16
Rugieł Marzena	40	Tchórz Adam	15
Rząd Ewa	25	Tymicki Emil	16
Sheikh Ali	22	Wiercigroch Ewelina	42
Setkowicz Zuzanna	40, 41, 43	Wilk Aleksandra	43
Sokołowski Paweł	32, 36	Wilk-Kołodziejczyk Dorota	20, 37
Stanula Agnieszka	38	Wójcik Adrianna	16
Stojak Marta	45		





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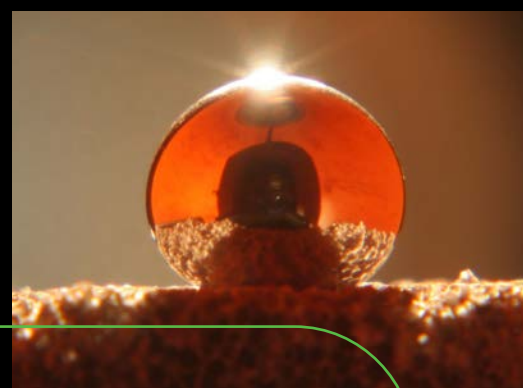
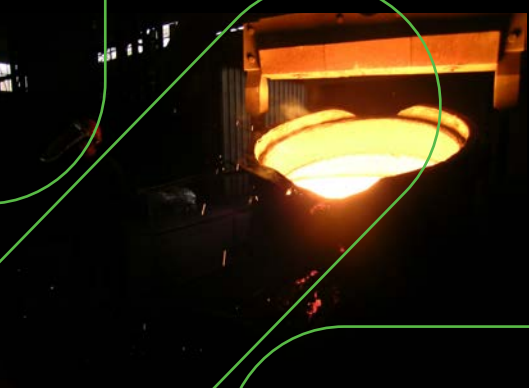
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