Institute of Materials Research SAV Košice

Óbudai Egyetem

PROMATECH
RESEARCH CENTRE OF ADVANCED MATERIALS AND TECHNOLOGIES

Progress presentation of the 2nd semester (January 2024– July 2024)

Toughening of high-entropy ceramics by low-dimensional nanomaterials

PhD student: Sara Ines Moussaoui

Supervisors: Prof. Dr. Ján Dusza
Dr. Pinke Peter

Major current works of PhD study

- Experimental study at PROMATECH institute in Kosice
- Preparation of review paper about UHT-DPHECs
- Collaboration on AI in the field of High Entropy Carbide Ceramics
- YCN Scientific paper writing Workshop
- Poster Presentation at CMCEE Conference

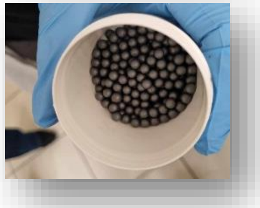
Powder Pre-Processing and Processing Technique

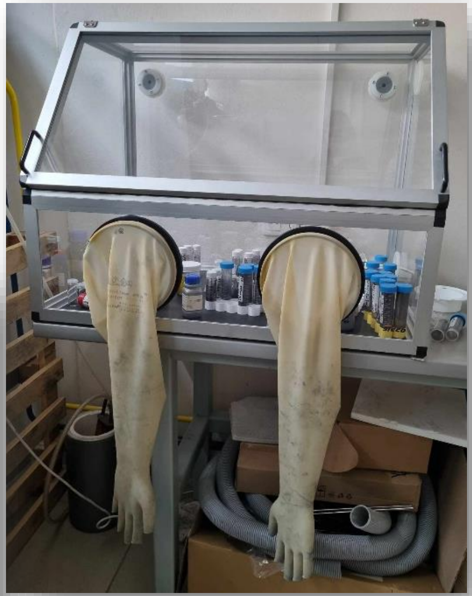
Powder Pre-Processing:

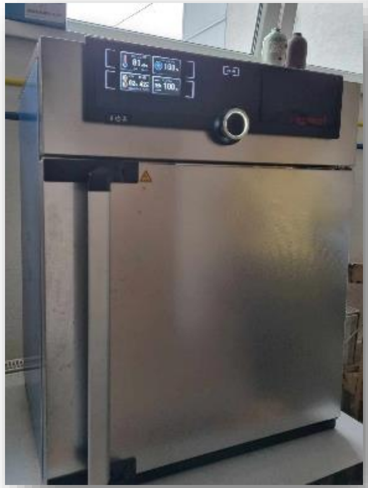
- **Storage in Argon Chamber**: The initial powder is stored in an argon chamber to prevent oxidation.
- **Milling**: The powder is milled with ethanol or isopropanol to achieve the desired particle size and composition. This step is crucial for ensuring the uniformity of the final product.
- **Drying**: After milling, the powder is thoroughly dried to remove any residual solvents.

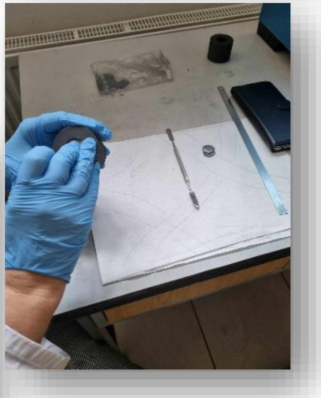
Powder Preparation for sintering:

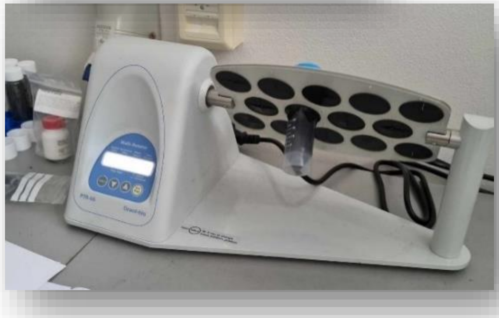
- **Weighing**: The required mass of the powder is weighed based on the sample volume and material density to ensure precise composition.
- **Mixing**: The powder is thoroughly mixed to ensure homogeneity. If enhancing with whiskers, nanoplatelets, and nanotubes, optimal dispersion within the composite is achieved using magnetic mixing. This step is essential for preserving the high-value nanostructures and enhancing the functionality and performance of the resulting dual-phase high entropy ceramics.
- **Molding**: The mixed powder is placed into a mold to form the desired shape.







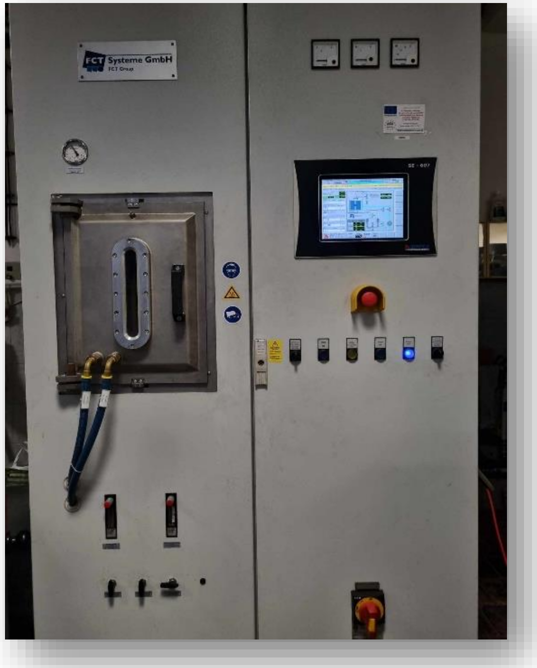


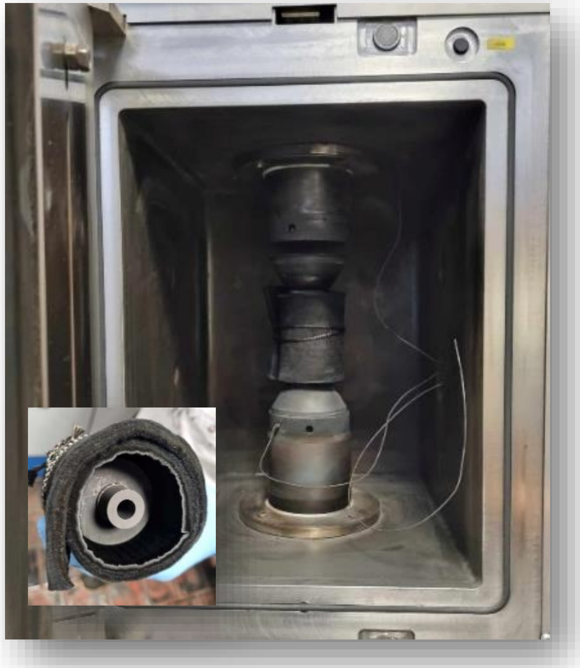


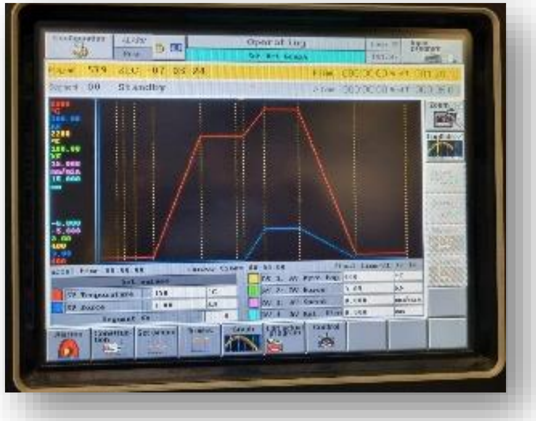
Processing Technique: Spark Plasma Sintering (SPS)

- Efficient technique for creating high-quality ceramics with improved mechanical properties.
- Short treatment times and highly compacted sinter bodies.
- Enables material transport and chemical reactions during sintering.

SPS settings for high entropy ceramics (HECs) involve controlling key parameters such as temperature gradient, current density, material conductivity, mold wall thickness, current pattern, heating rate, holding time, and sintering pressure. These parameters vary depending on the desired properties for each experiment.







Sample Preparation for Testing

Rigorous Grinding and Polishing Procedures:

- Preparation for mechanical testing and microstructural examination.
- Ensures accurate and reliable characterization.

The sintered sample:

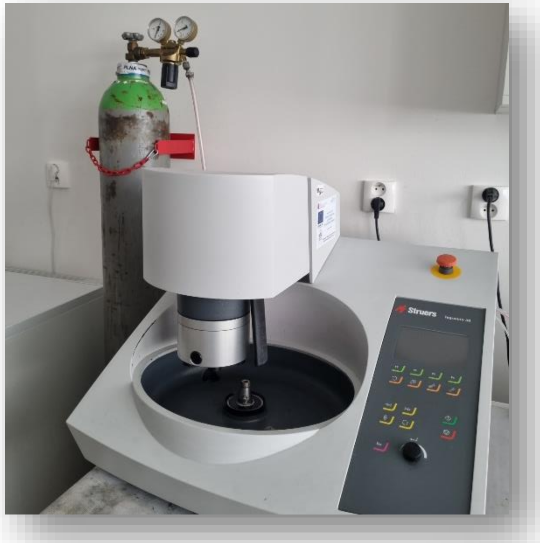
- Composition: ZrB_2, NbB_2, TaB_2, HfB_2 powders with 50 mol% TiC.
- Fabrication process via spark plasma sintering (SPS) with detailed parameters.

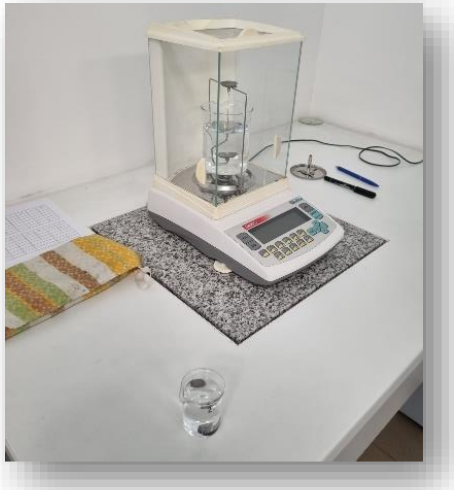
Ceramographic procedures

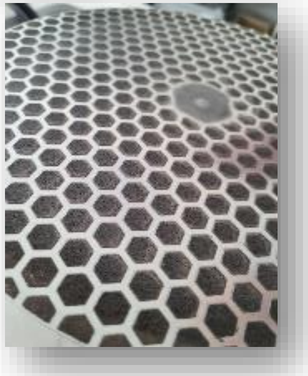
- Grinding and polishing steps using specific instruments and suspensions.
- Examination with an inverted light microscope for quality assurance.

Experimental Density Determination

- Archimedes method with distilled water immersion at room temperature.
- Triple weighing and theoretical density calculations.







Tribological and Mechanical Testing

Observation of Tribological Testing

- Overview of tribometers and their variants for measuring wear and friction.
- Description of sample mounting methods for room and high-temperature conditions.

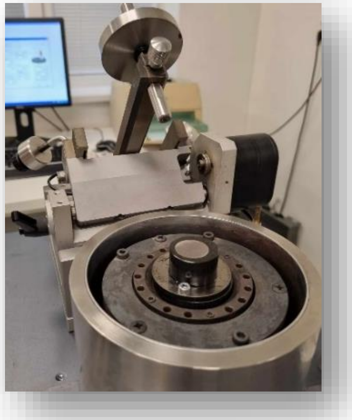
Insight into Nanoindentation:

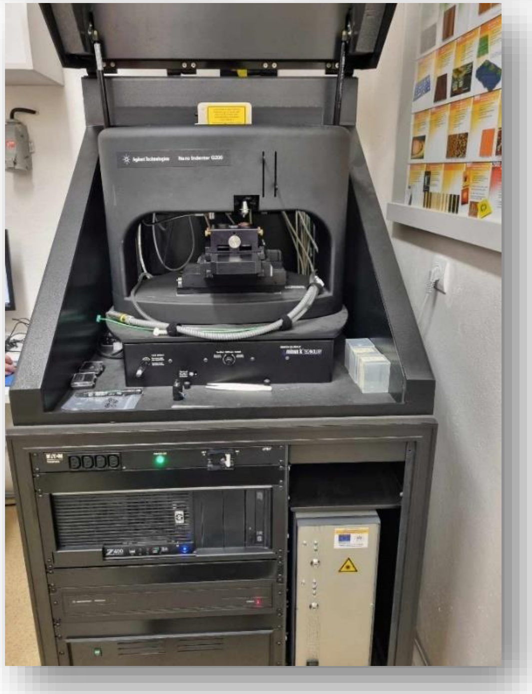
- Importance in material testing for modulus, hardness, and fracture toughness.
- Essential steps involved in the nanoindentation process.
- Applications and insights provided by nanoindentation.

Instrumentation of Atomic Force Microscope (AFM):

- Description of AFM operation and its effectiveness in measuring nanoscale properties.
- Detailed explanation of tip-sample interaction and data acquisition.

Conclusion: Highlighting the significance of tribological and mechanical testing in understanding material properties and performance.





Preparation of review paper about UHT-DPHECs

Title: **Development of dual-phase ultra-high temperature high entropy ceramics - A review**

Abstract:

- Introduction to the paper's focus on dual-phase ultra-high temperature high entropy ceramics (UHT-DPHECs).
- Highlighting the potential applications in extreme environments.
- Discussion on computational methodologies, processing techniques, and material properties.

Focus Areas:

- Design, optimization, and synthesis of UHT-DPHECs.
- Computational methodologies for predicting material properties.
- Various processing techniques and their effectiveness.
- Mechanical and tribological properties under demanding conditions.
- Current trends and future directions in HEC research.

References:

- Around 20 primary references utilized for UHT-DPHECs.
- Approximately 55 additional articles on high entropy carbide and boride ceramics.

Summary of Processing Methods:

- Reactive Spark Plasma Sintering (SPS)
- Boro/Carbothermal Reduction
- Sequential Carbothermal Reduction and Boride Conversion
- Reactive Sintering
- Ultra-Fast High-Temperature Sintering

First author	Paper title	Journal	Year
GILD	Reactive flash spark plasma sintering of high-entropy ultrahigh temperature ceramics	Scripta Materialia	2019
Mingde Qin	Dual-phase high-entropy ultra-high temperature ceramics	Journal of the European Ceramic Society	2020
Si-Chun Luo	Fine-grained dual-phase high-entropy ceramics derived from boro/ carbothermal reduction	Journal of the European Ceramic Society	2020
Annamaria Duszova	Nanohardness and indentation fracture resistance of dual-phase high-entropy ceramic	Ceramics International	2022
Lun Feng	Boro/carbothermal reduction co-synthesis of dual-phase high-entropy boride-carbide ceramics	Journal of the European Ceramic Society	2022
S. Huo	Reactive sintering of dual-phase high-entropy ceramics with superior mechanical properties	Journal of the European Ceramic Society	2022
Steven M. Smith II	High-entropy boride–carbide ceramics by sequential boro/carbothermal synthesis	Journal of the American Ceramic Society	2022
Annamaria Duszova	Dual-phase high-entropy carbide/boride ceramics with excellent tribological properties	Journal of the European Ceramic Society	2023
Annamaria Duszova	Highly wear resistant dual-phase (Ti-Zr-Nb-Hf-Ta)C/ (Ti-Zr-Nb-Hf-Ta) B2 high-entropy ceramics	Advances in Applied Ceramics	2023
Annamaria Duszova	On the phase and grain boundaries in dual phase carbide/boride ceramics from micro to atomic level	Journal of the European Ceramic Society	2023
Rui-Fen Guo	Ultra fast high temperature synthesis and densification of high entropy diborides and diboride-carbide ceramics	Journal of the European Ceramic Society	2023
Steven M. Smith II	Pressureless sintering of dual-phase, high-entropy boride–carbide ceramics	Journal of the American Ceramic Society	2023
Steven M. Smith II	thermodynamic analysis of metal segregation in dual phase high entropy ceramics	Journal of Materiomics	2023
Annamaria Duszova	Fracture strength of grains and grain boundaries in a dual-phase high-entropy ultra-high temperature ceramics	Journal of the European Ceramic Society	2024
Annamaria Duszova	Processing and microstructure development of reactive sintered (Ti-Zr-NbHf-Ta)C + (Ti-Zr-Nb-Hf-Ta)B2 high – etropy ceramics	Ceramics International	2024
Zi-Jian Huang	High-entropy boride-carbide based composite prepared by reactive spark plasma sintering	Journal of the European Ceramic Society	2024

Collaboration with Dr. Awais Qadir on High Entropy Carbide Ceramics

Overview:

- Collaboration focused on data collection for training AI models to predict properties of high entropy carbide ceramics.
- Objective: Create a comprehensive dataset from published experimental results for machine learning models.

Data Collection Parameters:

- Initial powders, pre-processing parameters (particle size, milling type, milling speed, milling time), sintering pressure and time.
- Obtained properties: configurational randomness, relative density, phase types.
- Mechanical properties: indentor load, Vickers hardness, nanohardness, fracture toughness, flexural strength.
- Tribological properties: sliding distance, dry/wet conditions, wear rate.

Importance:

- Fundamental for developing robust AI models.
- Advancement of high entropy carbide ceramics field.

YCN Writing Workshop Experience in Tampere

Title: YCN Writing Workshop Tampere Experience

Overview:

- Participation in Scientific Paper Writing Retreat organized by the Young Ceramic Network.
- Duration: 2nd to 5th of May in Tampere, Finland.
- Interaction with peers from Europe, UK, and Brazil.

Workshop Highlights:

- Warm-up activities, focused writing periods, breaks fostering discussions.
- Insightful conferences on enhancing writing skills:
  - "Optimize Your Writing Process" by Dr. Martina Michalikova.
  - "Scientific Writing from the Perspective of Editors/Reviewers and Attracting Readers" by Dr. Camilla Imarisio.
  - "AI and Ethics for Academic Writing" by Salla Westersrand.
  - "How to Write a Paper and Get It Published" by Prof. Pablo Colombo.

Benefits:

- Comprehensive understanding of scientific writing and publication process.
- Correction of mistakes, approach to writing and publishing research results.
- Enhancement of writing efficiency and enjoyment.
- Acknowledgment of significant responsibility as researchers in advancing science through dissemination of experimental data and analysis.

Planned Poster Presentation at CMCEE Conference in Budapest

Topic: Results on Dual-Phase High-Entropy Ultrahigh Temperature Ceramics

Synthesis Process:

- Two-step spark plasma sintering process at 2100°C for 5 minutes under 70 MPa.
- Composite consists of hexagonal high-entropy boride (HEB) and cubic high-entropy carbide (HEC) phases.

Analysis Techniques:

- SEM, EBSD, and EDX for microstructure, deformation, and fracture characteristics analysis.
- Nanoindentation with Berkovich diamond tip in CSM mode and Vickers hardness testing.

Results:

- Mean nanohardness values: 40.4 GPa (HEC) and 43.1 GPa (HEB).
- Microhardness: 21.73±1.36 GPa.
- Indentation fracture toughness: 6.04±0.93 MPa√m.

Conclusion:

- Enhances understanding of mechanical properties of high-entropy ultrahigh temperature ceramics.

Next Experimental Visit to Promatech Institute, Kosice

Objective: Incorporating micro and nano additives to enhance fracture toughness and mechanical performance of high-entropy ceramics.

Sample Preparation:

- Commercially available powders (Alfa Aesar) with molar ratios of 2TaC: 2HfC: ZrC: ZrB2: 2NbB2: 2TiB2 were used to achieve the target composition of a 50 mol.% (Ta-Hf-Zr-Nb-Ti)C - 50 mol.% (Ta-Hf-Zr-Nb-Ti)B2 dual-phase high-entropy ceramic. The raw powders were mixed and milled in a planetary ball mill to ensure homogenization.

Processing :

- The homogenized powder was sintered using a Spark Plasma Sintering (SPS) device (FCT HP D10-SD) at a temperature of 2100°C, a pressure of 70 MPa, and a sintering time of 5 minutes. In the subsequent step, the dual-phase high-entropy matrix (described system) will be prepared with the addition of 5, 10, 15, and 30 vol% of SiC whiskers, and sintered at 2100°C for 10 minutes.

Analysis Techniques:

- X-ray diffraction, SEM, STEM, EDS, and EELS for microstructure characteristics.

Purpose:Publish results in scientific articles

Thank you for your attention