SHS 2024

Book of Abstracts

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Plenary Speakers

Alexander Mukasyan

Mukasyan Alexander, Professor of Chemical and Biomolecular Engineering, University of Notre Dame, Notre Dame, IN USA since 1996.

Ph.D. in physics and math (1986) Institute of Chemical Physics, Russian Academy of Sciences; Sci. Dc. (1994) Institute of Structural Macrokinetics, Russian Academy of Sciences. Academician of the International Informatization Academy (2001). Member of the Editorial boards for several International Journals; Member of International Advisory Boards of Ceramic World Academy. Medal of Exhibition of Achievements of National Economy (Moscow, 1993); Medal of Russian Academy of Sciences (2001); Medal of Kazakhstan Ministry of Science (2007).

Co-author of 5 books and 1 text-book, 12 Chapters in books, 24 invited reviews, more than 300 research publications in archive journals and 36 patents, including 14 US patents in the fields of engineering of advanced materials and combustion (see details: https://scholar.google.com/citations?hl=en&user=b8XySnEAAAAJ).

The main scientific interest relates to nanotechnology, high energy density materials, joining of refractory and dissimilar materials, as well as catalysis. The most cited publications: Solution combustion synthesis of nanoscale materials, Chemical Reviews 116 (23), 14493-14586, 2016; Combustion synthesis and nanomaterials, Current Opinion in Solid State and Materials Science 12 (3-4), 44-50, 2008; Combustion for Material Synthesis, CRC Press, Taylor and Francis, Boca Raton, London, New York, 2015, 398 p.

Mikhail Alymov

Prof. Mikhail I. Alymov is currently a director of the Merzhanov Institute of Structural Macrokinetics and Materials Science (ISMAN), Russian Academy of Sciences. He graduated from Moscow Engineering Physics Institute (MEPhI) in 1981, got a degree of Cand. Sci. (Phys.) in 1987 and that of Dr. Sci. (Phys.) in 1997. His research interests are and have always been in the field of powder metallurgy and composite materials. In 1997, he got an academic rank of professor in strength physics and in 2008 was selected a corresponding member of the Russian Academy of Sciences. Alymov authored about 300 publications, including 17 monographs and 7 patents.

Alymov suggested a number of highly efficient processes for producing structural and functional consolidated nanomaterials that can be implemented to reduce the risks of man-made accidents during production, storage, transportation, and processing of metallic nanopowders, including the process for fabrication of metallic nanomaterials with a hierarchically porous structure.

He is Editor-in-Chief of *Advanced Materials and Technologies* magazine; Deputy Editor-in-Chief of *Letters on Materials*; and an editorial board member in such journals as *Metals*, *Inorganic Materials*, *Inorganic Materials: Applied Research*, *Perspektivnye Materialy*, *Izv. Vyssh. Uchebn. Zaved.: Powder Metallurgy and Functional Coatings*, *Deformatsiya i Razrushenie Materialov*, *Composites and Nanostructures*, *International Journal of Self-Propagating High-Temperature Synthesis*.

Alymov holds a chair of the academic and qualification councils at ISMAN and is a member of the qualification council at IMET. He is a member of the following councils at the Russian Academy of Sciences for the following lines of research: materials and nanomaterials, metallurgy and materials science, inorganic chemistry, ceramic and glass materials, high-purity compounds, and combustion and flame.

Alymov is also active as an organizer of regular materials science forums. He is a recipient of multiple scientific achievement awards.

Dmitry Shtansky

Dmitry Shtansky is the Director of the Research Center of Inorganic Nanomaterials, Professor of the Department of Powder Metallurgy and Functional Coatings, and Principal Scientists of the Scientific-Educational Center of SHS at National University of Science and Technology “MISIS”. He graduated from the Physics Department of Moscow State University (1985), received his Ph.D. at the I.P. Bardin Iron and Steel Industry Institute (1992) and his Doctor of Science degree at the NUST “MISIS” (2002). He was awarded a Max - Planck - Society Postdoctoral Fellowship (Max-Planck-Institut für Eisenforschung Germany, 1995-1996), a Japan Society for the Promotion of Science (JSPS) Postdoctoral Fellowship (Ehime University, 1997-1999) and a JSPS Research Fellowship under the program “Research for the Future” (Tokyo University, 1999-2000). Under his supervision, 12 Ph.D and 1 Doctor of Science dissertations were defended.

Dmitry Shtansky is a so-author of 310 scientific papers published in referred journals and indexed in the Scopus database (including Int. Mater. Rev., Adv. Funct. Mater., Appl. Catal. B. Env., Chem. Eng. J., Biomaterials, Nano Research, J. Mater. Chem. A, Acta Mater, ACS Appl. Mater. Interfaces etc.) and has presented more than 330 scientific lecturers at international conferences, congresses, and seminars, including 25 invited and key-note lecturers. He is a co-author of 23 patents and 42 “know-how”. H-Index – 42 (Scopus) and 50 (Google Scholar), citation index: >6300 (Scopus) and >8200 (Google Scholar). He is an expert of the Russian Foundation of Basic Research, the European Commission, the Russian Scientific Foundation, and the Russian Ministry of Education and Science.

He was awarded a medal for his high contribution to the development of invention, a gold commemorative badge of NUST “MISIS”, a medal of NUST “MISiS” for Merits in Materials Science, and the honorary title of “Honored Worker of Science and High Technologies of Russian Federation”. He was elected Scientist of the Year at NUST “MISiS” in 2022. He is the chairman of the expert council of the MISIS Dissertation Council in the specialties “Biomechanics and Bioengineering” and “Biophysics”. His research interests include but are not limited to thin films and coatings (hard tribological coatings, self-lubricating coatings, biological coatings), surface engineering, metal matrix composites, antibacterial and fungicidal surfaces, nanostructures, heterostructures, nanoparticles, hexagonal boron nitride, catalysis and photocatalysis, plasma polymerization, nanoparticles-modified cotton fabrics, drug delivery.

Makis Angelakeris

Prof. Dr. Makis Angelakeris (M) is a professor at the School of Physics at the Aristotle University of Thessaloniki. His main scientific interest stems from the synthesis of magnetic thin films, multilayers and nanoparticles. Under this research scheme, he also works also works on structural, magnetic, electric, magneto-optic characterization of magnetic nanostructures. Within the last 10 years he is also involved in Biomagnetism applicability focusing on Magnetic Particle Hyperthermia and Magneto Mechanical Stress.

He has 136 publications in peer review International Journals, 126 abstracts in International Conference Proceedings and 98 abstracts in Local Conference Proceedings. He has delivered 22 invited presentations and has 3544 Citations by 2552 documents (excluding self-citations out of 2488 citations, h-index: 33, scopus:21/04/2022). He serves as regular referee in 10 International peer-review scientific journals (12-20 manuscripts annually).

His work is highlighted by two monographs: a review article on Magnetic nanoparticles: A multifunctional vehicle for modern theranostics, M. Angelakeris, Review Article (in Biochim Biophys Acta Gen Subj. 2017 Jun;1861(6):1642-1651) and a Book chapter on Magnetic Particle hyperthermia, in Vol. 8 of 21st Century Nanoscience, A handbook, (10 volumes), Taylor & Francis, in press (2020). He acted as Supervisor in 6 PhD theses (4 underway), in 17 MSc (2 underway) and 22 BSc theses (2 underway). He participated in 11 international or regional research projects. (Webpage: http://users.auth.gr/agelaker).

He is currently co-ordinating the MagnaCharta Group (Magnetic Nanostructure Characterization: Technology and Applications http://magnacharta.physics.auth.gr). The MagnaCharta group focuses on modern magnetic nanomaterials from systematic synthesis and robust investigation of physical properties together with technological applicability of nanomagnetism on diverse aspects such as information storage, biomedicine and sustainable growth. All members of MagnaCharta group may actively participate and provide services within the current proposal.

SHS 2024

Plenary Lectures

Combustion Synthesis and “Hot” Directions in Material Science

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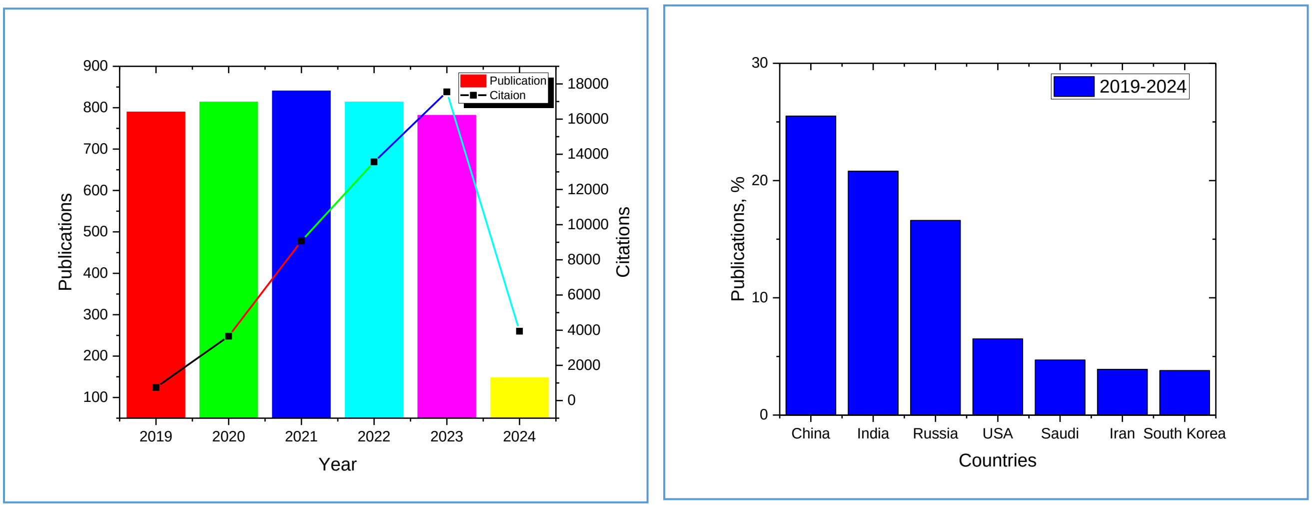
Combustion synthesis (CS) is an energy saving approach for fabrication of materials. The fundamental CS basis involves usage of self-sustained chemical reactions to develop optimum environment for formation of materials with desired structure and thus properties. In this work we outline and analyze several “hot” directions in material science, which attract most publication and citation attention during last 6 years (2019-2024). This task was accomplished based on the analysis of information acquired from the Web of Science, the world's leading platform for scientific research and citation data.

In the first stage, the following keywords were used: combustion & synthesis & materials and self-propagating & high-temperature & synthesis. The statistics on the publication and citation are shown in the Figure (data acquired on April 14, 2024). From the standpoint of publications, the CS/SHS field reached the saturation level (~850 per year), while citations linearly increased, approaching 18K per year. The geography of the publication is also presented in the Figure. In the second stage, all recent publications were ranked based on the number of citations. As expected, the higher citations belong to the reviews related to the different material science directions. The following “hot” directions were outlined: High Entropy Alloys; MAX phases and MXenes; Hydrogen Storage; Energy and Environment; Catalysis; Additive Manufacturing; Biomedicine. In the third stage, we briefly analyzed the advantages and disadvantages of CS/SHS approaches as compared to other available methods for fabrication materials in the outlined “hot” directions.

Figure: Publication and citation statistics on the CS/SHS; Publication distribution along the countries (7 top countries are shown; total 107 countries).



ISMAN: New results and achievements in SHS

M. Alymov

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Main scientific areas of ISMAN work:

- theoretical and experimental research in the field of structural macrokinetics;
- development of new methods and technologies for self-propagating high-temperature synthesis (SHS) of new generations of structural, heat-resistant, heat-resistant high-temperature materials based on refractory metals and their compounds, functional and high-purity materials;
- creation of SHS methods for direct production of products from powders of refractory compounds, combining the processes of combustion and high-temperature shear deformation of synthesis products, bypassing the stage of obtaining powders of the final product;
- development of new methods for experimental diagnostics of combustion processes: time-resolving radiography and high-temperature radiography to study the mechanisms of phase formation during SHS and study the dynamics of phase and structural transformations of materials.

The scientific and technological foundations for the production of nitride powders (AlN, Si_3N_4, ZrN, TiN) and compositions based on them ($Si_3N_4 \cdot MgO$, $AlN \cdot Y_2O_3$, AlN-VN-V_2N) by the SHS method have been created, scientific methods and principles for controlling morphology, chemical and phase composition of nitrides in SHS processes. Based on the results obtained, technologies for nitride powders with a wide range of characteristics in terms of chemical, phase and morphological composition have been developed. Using the developed technologies, ISMAN produces the above-mentioned nitride powders.

Methods have been developed for the direct production of products from powders of refractory compounds, combining the processes of combustion and high-temperature shear deformation of synthesis products, bypassing the stage of obtaining powders of the final product.

Technologies have been developed for the production of materials based on the MAX phases of ternary and quaternary compounds, as well as MXene based on them.

Size effects and critical phenomena of combustion and passivation of metal powder materials have been studied. A qualitative scheme for the ignition of a metal nanoparticle in a gaseous environment containing an oxidizer has been formulated. The obtained dependence of the critical size of nanoparticles on the defining parameters: oxidant content in the gas, initial temperature, and surface activity of nanoparticles after synthesis corresponds to known experimental data.

This work was supported by the Russian Science Foundation, project no. 22-19-00126.

Innovative approaches to creating bactericidal surfaces

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For centuries, humanity has been fighting infection. The foreign surface increases the risk of post-implantation infection and leads to the formation of a biofilm, which is difficult to remove with therapeutic methods due to the frequent bacteria multidrug resistance. Various modern approaches to creating bactericidal surfaces are considered (1-9):

(i) Controlled release of bactericidal ions,
(ii) X-ray and UV irradiation-induced generation of reactive oxygen species,
(iii) Formation of microgalvanic pairs on a heterogeneous surface,
(iv) Deactivation of bacteria during their direct physical contact with the surface,
(v) Loading of a therapeutic component into a specially formed surface relief,
(vi) Loading of a nanostructured surface with a therapeutic agent,
(vii) Surface immobilization of bactericidal agents,
(viii) Fabrication of anti-adhesive surfaces that prevent biofilm formation,
(ix) A combination of bactericidal and antifungal agents.

Various methods for obtaining antibacterial surfaces and factors affecting the bactericidal ion release (concentration of the bactericidal component, topography and surface roughness, the presence of various types of nanoparticles (cathodes and anodes), surface oxidation) are also considered. It has been shown that the simultaneous action of several mechanisms increases the therapeutic efficiency of bactericidal surfaces. Under optimal conditions, it is possible to achieve a high antibacterial and antifungal effect at a low dose of a therapeutic agent, which ensures the biological safety of medical devices.

The work was supported in part by the Russian Science Foundation (Agreement 20-10-00120-П).

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Synthetic controls for diverse biomedical applicability scenarios

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Synthetic controls refer to the precise manipulation of size, shape, composition, surface chemistry, and certain properties during fabrication. This level of control is crucial for tailoring nanostructures for specific applications in various fields, including biomedicine, catalysis, electronics, and environmental remediation. Magnetic nanostructures hold tremendous promise in the field of biomedicine due to their unique properties and versatile applications such as drug Delivery, Hyperthermia Therapy, Imaging, Biosensing, Cell Labeling and Tracking, Tissue Engineering and Detoxification.

Continued advancements in synthetic methodologies and characterization techniques are driving the development of increasingly sophisticated nanoparticle-based materials with enhanced performance and functionality. Specifically, for magnetic nanostructures, key aspects correspond to tuning their magnetism following approaches to:

1. Control Size: By controlling the size distribution of nanoparticles during synthesis, researchers can tune their magnetic properties.
2. Control Composition: Varying the composition of magnetic nanoparticles can significantly influence their magnetic behavior i.e. magnetic moment, magnetic anisotropy, and Curie temperature of nanoparticles.
3. Modify Surface: Can shield nanoparticles from agglomeration or specifically target biomedical probes.
4. Control Shape: The shape of nanoparticles also plays a critical role in determining their magnetic properties by adjusting the aspect ratio or geometry of the nanoparticles.
5. Multi-functionalize Nanocomposites: Incorporating magnetic nanoparticles into composite materials or hybrid structures with other functional materials (e.g., polymers, metals, ceramics) can lead to synergistic effects and tailored magnetic, electrical, and mechanical properties.
6. Engineer Nanostructures: Fabricating complex nanostructures (e.g., core-shell nanoparticles, heterostructures) allows for precise control over the magnetic interactions between different components, leading to enhanced magnetic properties and functionalities.

In this talk, I will present case studies of magnetic nanoparticle-based systems synthesized by Chemical Co-precipitation, Thermal Decomposition, Hydrothermal/Solvothermal Synthesis and Solid-State Pyrolysis and corresponding synthetic controls to produce nanostructures with controlled size, composition, morphology, and certain biomedical functionalities.

Keynote and Oral Presentations

Keynote Report

Historical Milestones and Recent Progress of Combustion Synthesis and Processing of Materials in Armenia

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Combustion synthesis, also known as self-propagating high-temperature synthesis (SHS), has a rich history and significant advancements in Armenia. Originating in the early 1970s, Armenian scientists have played a crucial role in developing and applying this technique, which involves exothermic reactions to prepare various advanced materials, including ceramics, intermetallics, and composites. This talk will review the historical milestones and recent progress in combustion synthesis research in Armenia, highlighting key contributions and technological advancements.

Historically, Armenian researchers have been at the forefront of pioneering SHS techniques, with notable work conducted at the Laboratory of Chemical Physics of the Academy of Sciences of Armenia. In 1975, the Laboratory was reorganized into the Institute of Chemical Physics of the Academy of Sciences, which included several research units working on solid-state combustion reactions and material synthesis technologies. Early research focused on understanding the fundamental principles of combustion reactions and exploring their potential for synthesizing refractory materials and high-temperature ceramics. Armenian scientists developed various methods for investigating rapid solid-phase reactions and combustion of solids in reactive gas environments.

Recent developments in Armenia have seen the application of combustion synthesis in creating nanomaterials, functional and structural ceramics, and complex alloys. Innovations in controlling reaction parameters and optimizing material properties have produced materials with enhanced mechanical, thermal, and electrical properties. Armenian researchers have also explored the influences of different factors, such as irradiation and external pressure, on exothermic reactions. Kinetic modeling of rapid exothermic processes and developing advanced instruments for investigating rapid reactions in condensed matter is another hallmark of the Armenian SHS community.

Collaborative efforts between Armenian universities, research institutions, and international partners have further propelled the field, fostering knowledge exchange and technological innovation. Integrating advanced characterization techniques, such as electron microscopy and spectroscopy, has provided deeper insights into SHS-produced materials' microstructural evolution and properties.

This presentation will underscore Armenian scientists' significant contributions to the combustion synthesis field, from its historical roots to contemporary advancements. The ongoing research and development efforts continue to position Armenia as a key player in the global materials science landscape, driving the potential of combustion synthesis for various industrial applications.

Oral Report

High entropy materials in the light of self-propagating high-temperature synthesis

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High-entropy materials defined in 2001 sparked renewed attention in multi-component materials that have been of interest for several decades due to their superior properties for various structural and functional applications. Since 2015, the high entropy concept has expanded further to ceramics, where four or more metals in equimolar proportions form high entropy structures with non-metals (C, N, B, O). Furthermore, transferring the high-entropy design concept to a MAX phases (MXenes) has opened additional pathways for the discovery of new materials and properties. The main design principle for these materials is the entropy stabilization effect endowing high phase stability to a system and disorder-enhanced properties significantly surpassing expected values from mixture rules.

Solid-state reaction methods reported in literature include mechanosynthesis or ball milling, metalothermic reduction, high-pressure torsion, sintering, and self-propagating high-temperature synthesis (SHS). The selection of an appropriate processing route is based on several aspects including phase stability, desired property and application from one hand, and availability of precursors, scale-up ability, efficiency of the process from another. Taking the advantage of the self-sustaining nature of the exothermic combustion reaction, SHS is getting popular for producing high-entropy materials with high efficiency and more environmentally friendly manner compared to traditional techniques. This method has appealing advantages for future commercialization due to time and energy efficiency, diversity of precursors, applicability to various types of ceramics. In this work, the progress regarding high-entropy materials prepared by SHS, solution combustion synthesis, mechanoactivated SHS, SHS assisted by spark plasma sintering is reviewed. Different aspects including the history, principles, compositions, crystal structure, theoretical/empirical design and properties are comparatively discussed. The research specifically attempts to answer how SHS will enhance the properties of these novel materials of remarkable structures for future applications.

Oral Report

Solution Combustion Synthesis of Ni_3CuN Complex Nitride: Reaction Mechanism

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Solution combustion synthesis (SCS) is a quick, energy-efficient method using exothermic reactions between metal nitrates and organic fuels to produce nanoscale materials [1-2]. It's used to create various oxides (garnets, spinels, perovskites), metals, intermetallics, high-entropy alloys, and nitrides, including complex antiperovskites.

The rapid nature of SCS complicates mechanistic studies, and the use of different fuels makes comparisons challenging. Further research is needed to understand the kinetics and tailor material properties for applications in catalysis and magnetic devices.

This study investigates the formation mechanism of Ni_3CuN complex nitride using SCS of nickel and copper nitrates with hexamethylenetetramine ($C_6H_{12}N_4$) as a fuel. By varying $C_6H_{12}N_4$ concentration, the impact on the combustion process and product formation was analyzed. Time-temperature measurements, X-ray diffraction (XRD), transmission electron microscopy (TEM), electron diffraction, and high-resolution element analysis revealed a multi-stage reaction: metal oxide formation, reduction to metals, alloy formation, and final nitridation.

Thermogravimetric analysis (TGA) identified key stages and showed that heating rate significantly influence reaction dynamics. Activation energy calculations indicated nickel nitrate decomposition as the rate-limiting step.

This study demonstrates SCS's potential for efficiently producing complex nitrides like Ni_3CuN, useful in catalysis, magnetic devices, and advanced materials.

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Oral Report

Advanced Analysis of Refractory Carbides Oxidation Using Ultra-Fast Scanning Electro-Thermography

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Here, we report the results of investigations of rapid exothermic solid-gas reactions for refractory tantalum carbide formation and carbide/metal oxidation applying Ultrafast Scanning Electro-thermography (USET) (*heating rate up to* 5×10^5 *K/s, cooling rates up to* 5×10^4 *K/s, data acquisition: 10 kHz*). For preparation of carbide/metal specimens, tantalum wires (99.7% purity, diameter: 0.1 mm) were annealed in methane medium yielding $TaC/Ta_2C/Ta$ specimens. By changing the temperature and exposure time, the thicknesses of the corresponding carbide layers were adjusted. Oxidation patterns of synthesized carbides were studied in air using USET power- and temperature- control heating modes.

In Power heating mode exothermic reactions exhibit two distinct modes of interaction: slow conversion with small self-heating and an ignition mode triggered above the critical power (P_c) and temperature (T_c) thresholds. The values T_c, P_c, and ΔT_{max} (self-heating due to exothermic reaction) to characterize oxidation under various experimental conditions were employed. As a simple case we studied the oxidation of a Ta wire in air under constant power, obtaining thermograms at varying air pressures and power levels. The $TaC/Ta_2C/Ta$ specimens were then oxidized under identical conditions. XRD analysis of quenched samples confirmed formation of high temperature triclinic form of Ta_2O_5. Surfaces and cross-sectional SEM imaging of reacted specimens reveal the formation of porous Ta_2O_5 scale on the underlying TaC surface. In the initial stages of the oxidation process carbide layers still contained well-packed grains. The accelerated oxidation led to the disintegration and cracking of the carbide layers, facilitating oxygen access to the deeper layers of the specimen and oxidizing the metallic core. The unreacted tantalum carbide grains were sandwiched between outer and inner layers formed by oxidation of the carbide layer and Ta. At this stage the weight gain far exceeded the expected value for complete TaC to Ta_2O_5 conversion. Notably, TEM analysis and Raman spectra revealed amorphous carbon only in the outer Ta_2O_5 layer, suggesting distinct oxidation pathways.

USET temperature - control heating mode was employed for the study of oxidation at comparatively low temperature region (1073-1773 K). Kinetic data for various heating rates and air pressures indicated that higher air pressure accelerates oxidation, while heating rate has minimal influence at a given pressure. XRD patterns and SEM images of intercepted and quenched specimens at different temperatures show the formation of highly porous orthorhombic tantalum pentoxide scale on the surface, significantly increasing the specimen diameter.

Oral Report

Influence of External Pressure on Solution Combustion Synthesis and Phase Evolution of the Iron Oxides

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Iron oxide nanoparticles, including magnetite, maghemite, and hematite, exhibit unique magnetic, catalytic, and optical properties that make them highly desirable for numerous technological advancements. In this work, we report the influence of the inert gas pressure on solution combustion synthesis of iron oxide nanoparticles in metal nitrite-glycine system. The thermodynamics and temperature – time history of the process was thoroughly investigated. The combustion products were characterized by various techniques including Mössbauer spectroscopy, X-ray diffraction, scanning electron microscopy, and vibrating sample magnetometer.

It is shown that variation of Nitrogen gas pressure in the reactor in the from 0.1 to 1.1 MPa influences the temperature profile of the process, decreasing maximum combustion temperature from 1200 °C to below 900 °C. Such changes of SCS conditions results in the gradual change of phase composition of the fabricated materials along the $FeO \rightarrow FeO{\cdot}Fe_2O_3 \rightarrow Fe_2O_3$ route. The size of the synthesized particles is also varied in the 50 – 400 nm range. The observed effects are explained by accounting for complex mechanisms showing in Figure, including gas phase combustion, followed by solid-gas reduction reactions.

An increase of ambient gas pressure leads to a significant slowing of the water evaporation during the pre-combustion stages. It means that much more water exists in the gel media after ignition. The latter results in decreasing of the reaction temperature and changing the flame composition. The latter influences the microstructure and composition of the combustion products.

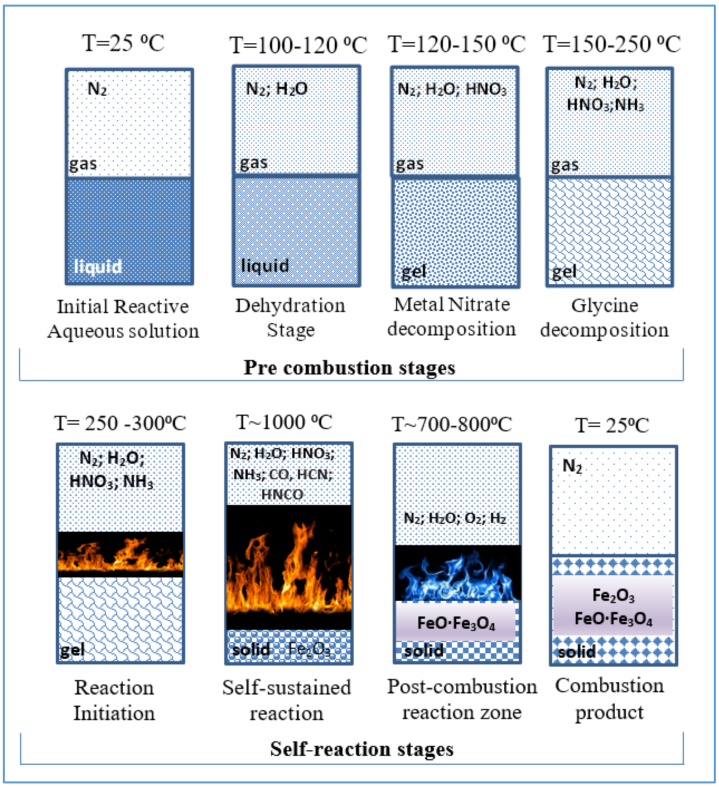

Figure: Schematics of different reaction stages during SCS in iron-nitrate-glycine system.

Keynote Report

SHS of the high-entropy alloys, compounds and ceramic-metal composites: an overview

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After the appearance of the first publications on high-entropy alloys in 2004 [1,2], the development of this scientific direction is proceeding at an accelerated pace. It is based on the scientific discovery of the possibility of obtaining disordered solid solutions of five or more metals, taken in comparable quantities, in the form of a single phase with a simple crystal structure. Materials based on high-entropy alloys have unique mechanical, electrical, magnetic and other properties. A new approach, based on the synthesis of multicomponent phases with a simple crystal structure, has been developed later for producing high-entropy ceramic materials [3,4]. New results have also appeared concerning high-entropy cermets, in which grains of the ceramic phase are located in a matrix of a high-entropy alloy [5,6].

The main methods for producing high-entropy materials (HEM) are casting and crystallization of multicomponent melts, mechanical alloying, and SHS. In this overview, we consider the current state of research and prospects for obtaining HEM using the SHS method. The possibility of direct synthesis from elements, the use of thermally coupled reactions, the combination of SHS with mechanical alloying, methods for consolidating HEMs and their properties are analyzed. It is shown that the SHS method makes it possible to quickly synthesize a wide range of new HEMs for scientific research, and is also promising for the industrial production of these new materials.

The work was supported by the Russian Science Foundation, Grant Number 20-13-00277P.

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Oral Report

Fabrication of high-entropy nitride ceramics by combustion in nitrogen of the mechanical alloying precursor TiZrHfTaNb

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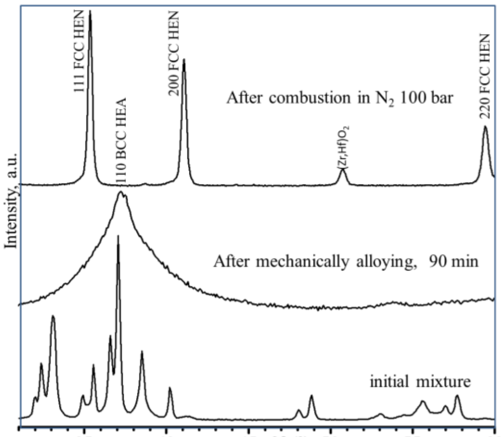
High-entropy ceramics (HECs) such as carbides, nitrides, borides, oxides, and silicides exhibit superior properties in terms of hardness, fracture strength, wear resistance, and corrosion resistance. The potential uses of this emerging group of materials range from structural to functional, including ultrahigh-temperature thermal protection, thermal and environmental barrier coatings, and wear-resistant coatings [1]. High-entropy nitrides (HENs) ceramics are particularly appealing for high-temperature and high-hardness applications. Simultaneously tough and hard HEN ceramics offer new opportunities for advanced applications that require high temperature and high hardness. The aim of the present study is to find a route for the synthesis of HEN (TiZrHfTaNb)N fine powders.

HEN was prepared by filtration combustion in nitrogen of mechanically alloying TiZrHfTaNb precursor powder. A mixture of initial powders Ti-Zr-Hf-Nb-Ta in equimolar ratio was subjected to high-energy ball milling (HEBM) in activator AGO-2 for 30, 60 and 90 minutes in argon atmosphere. The 90 min HEBM resulted in the mechanical alloying of the powders with the formation of single phase BCC solid solution TiZrHfNbTa (fig). The samples in the form of pressed hollow cylinders (D=7 mm, h=10 mm, hole 2-4 mm) were placed in a reactor which was filled with nitrogen at a pressure of 5 - 120 bar. After ignition of the sample from the tungsten coil, the propagation of the combustion wave was observed at a velocity of 1.5-4 mm/s depending on the nitrogen pressure. The dependence of the combustion velocity and temperature, the degree of dilution of the mixture, and the phase composition of the final product on the nitrogen pressure in the reactor and the sample geometry were determined. The optimal synthesis parameters for fabricating HEN (TiZrHfTaNb)N have been established.



Authors acknowledge funding from the Russian Science Foundation project 20–13-0277P.

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Oral Report

Combustion Synthesis of $(TiVCrMo)_4AlC_3$ High-Entropy MAX Phase and its Two Dimensional Derivative MXene

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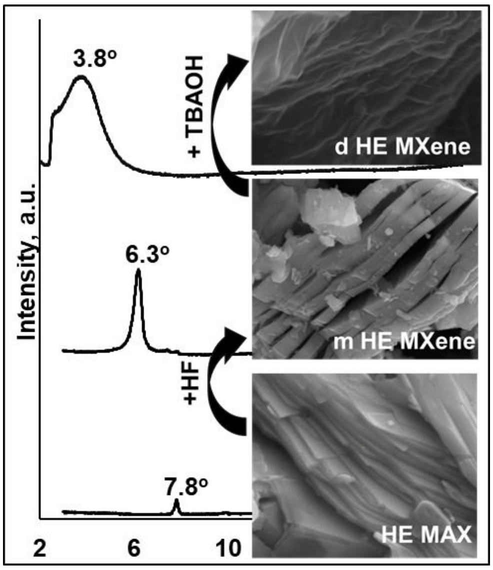
High-entropy (HE) MAX phases are a new emerging class of materials in which the five transition-metal [1] species are homogeneously dispersed into one MX slab due to their solid-solution feature, giving rise to a stable transition metal carbide in the atomic layers owing to the high configurational entropy.

In this thesis, the preparation of 413 high-entropy MAX phase from four transition metals (Ti/V/Cr/Mo) is reported.

$(TiVCrMo)_4AlC_3$ high-entropy MAX phase was prepared by single-stage self-propagating high-temperature synthesis (SHS) from mixture of elements in a constant pressure reactor in an Argon atmosphere. Polytetrafluoroethylene (PTFE) was used as a chemical activator to ensure sufficient heat release during the process and to achieve the necessary temperature conditions for MAX phase formation. The optimal conditions for the synthesis of the target material were determined depending on the ratio of raw materials, the dimensions of the sample (especially the diameter) and the pressure of the inert gas (Ar) environment. The obtained high-entropy MAX powder was subjected to characterization and selective etching process by hydrofluoric acid to preparemultilayer $(TiVCrMo)_4C_3T_x$ MXene by eliminating the Al layers. Tetrabutylammonium hydroxide (TBAOH) was used for the delamination of multilayered MXene into atomically thin nanosheets.



The microstructure evolution and phase transformation of MXenes from MAX phases is schematically illustrated in the figure.

The synthesized 413 HEMAX phase showed thermal and oxidation stability up to 500°C. At highest temperature, in the air flow, step-by-step oxidation of metals occurs, leading to the formation of TiO_2 and mixed oxides of constituent metals ($Cr_2Mo_3O_{12}$, $V_3Ti_6O_{17}$, $Al_2(MoO_4)_3$). In contrast to the MAX phase, HE_MXene shows instability to oxidation and undergoes complete oxidation after 300°C by a highly exothermic reaction.

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Oral Report

Synthesis of novel (Ti,Ta,V,Nb,Cr)$_2$AlC high-entropy MAX phase and its 2D derivative MXene

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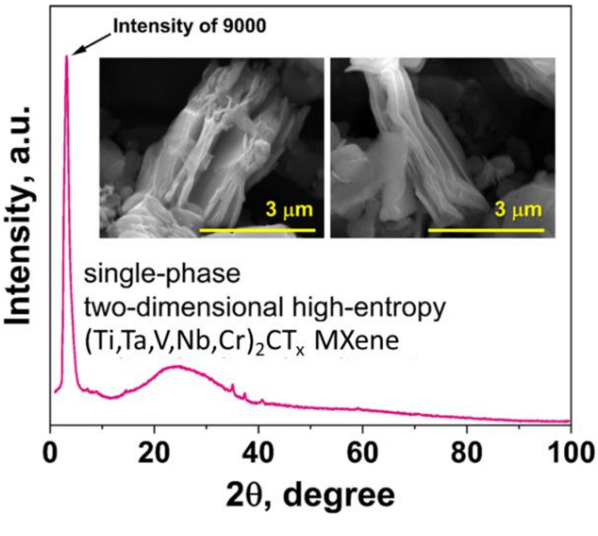
The concept of entropy-stabilized multicomponent systems involve incorporating multiple elements in equimolar or nearly equimolar proportions to contribute to the rise of configurational entropy, thereby stabilizing a single-phase solid solution across a wide temperature range. High-entropy design of MAX phases may open additional pathways for the discovery of novel materials with unique features. Moreover, in pursuit of durable electrode materials high-entropy 2D layered structures become of more interest due to their abundant electrochemically active sites, high surface area and excellent electrical conductivity.

Herein a novel (Ti,Ta,Nb,V,Cr)$_2$AlC high-entropy 211 MAX phase has been successfully synthesized from elemental powders using the self-propagation high temperature synthesis (SHS) method. The optimal combustion conditions and composition for the formation of this particular phase were revealed. Microstructural analysis results show that combustion products have layered structure, which is characteristic of the MAX phase. The (Ti,Ta,V,Nb,Cr)$_2$AlC HE MAX phase demonstrated oxidation stability in air flow up to 620°C.



The as synthesized (Ti,Ta,V,Nb,Cr)$_2$AlC high-entropy 211 MAX phase was subsequently treated with hydrofluoric acid to eliminate aluminum and obtain a 2D MXene nanosheets. Further delamination was carried using a sequential treatment involving organic solvents and deionized water. The XRD analysis results and distinct layered microstructure, shown in the figure, confirm that this method allowed to successfully synthesize pure, single-phase high-entropy (Ti,Ta,V,Nb,Cr)$_2$CT$_x$ MXene for the first time.

Keynote Report

Combustion Synthesis of High-Temperature Ceramics: From Solid Solutions to Heterophase Eutectic Compositions

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A review in the field of combustion synthesis of ultra high-temperature materials (UHTMs) is presented. Mechanism of chemical transformations in combustion wave for systems designed for the formation of solid solutions, including $(Zr_{1-x}Ta_x)B_2$, $(Hf_{1-x}Ta_x)B_2$, $(Zr_{1-x}Nb_x)B_2$, $(Hf_{1-x}Nb_x)B_2$, are discussed [1]-[4]. In the heating zone, a gas transport mechanism with chemosorbing of volatile boron oxides on Me^1, Me^2 particles surface and further formation of borides is realized. Main phases of Me^1B_2, Me^2B_2 appears in the combustion zone by the mechanism of dissolution-precipitation through the eutectic melt (L): $Me^1 + L\{Me^2\text{-}B\} \rightarrow Me^1B_2 + Me^2B_2$, where Me^1 is a more refractory metal. The $(Me^1,Me^2)B_2$ solid solution is structured finally in the post-combustion zone. Solid solutions of borides including example of high-entropy one with a unique combination of mechanical, thermophysical properties, oxidation and thermal resistance were obtained by hot pressing / SPS of SHS-powders.

The structure and properties of carbides (Ta,Zr)C, (Ta,Hf)C, (Nb,Zr)C, (Ta,Nb,Zr)C have been studied. Due to the formation on the surface of dense oxide-carbides layers that block the diffusion of oxygen, these carbides have high oxidative and thermal stability.

Using elemental synthesis of mechanically activated mixtures, the heterophase boride-carbide eutectic compositions were synthesized. The high combustion rate minimizes the evaporation of elements, ensuring the preservation of the composition specified by the stoichiometry, and the high cooling rate of combust products contributes to the formation of an ultrafine-grained (UFG) structures. The possibility of UFG UHTMs obtaining was demonstrated on example of HfB_2-HfC, ZrB_2-ZrC, $(Hf_{1-x}Zr_x)B_2$-$(Hf_{1-x}Zr_x)C$, $(Ti_{1-x}Zr_x)B_2$-$(Ti_{1-x}Zr_x)C$ systems.

Ceramics in systems Me^1B_2-SiC, $(Me^1,Me^2)B_2$-SiC, $(Me^1,Me^2)B_2$-$(Me^1,Me^2)C$-SiC with high thermal and oxidation resistance were studied. It was shown, that the Si-containing additives increases high-temperature resistance due to the formation of protective layers based on $xSiO_2 \cdot yB_2O_3$, $HfSiO_4$, $ZrSiO_4$, $(Hf,Zr)SiO_4$.

This work was supported by the Russian Science Foundation (project № 24-13-00085).

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Oral Report

High oxidation resistant coating based on self-propagating combustion synthesis and spark plasma sintering technique

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To achieve a high oxygen-blocking structure on coatings applied to carbon structural materials, ZrB_2-$MoSi_2$, HfB_2-$HfSi_2$-SiC, and ZrB_2-$MoSi_2$-$TaSi_2$ coatings were prepared by spark plasma sintering (SPS) method utilizing ZrB_2-$MoSi_2$, HfB_2-$HfSi_2$, and ZrB_2-$MoSi_2$-$TaSi_2$ composite powders synthesized by self-propagating high-temperature synthesis (SHS) technique as raw materials. Compared with commercial powders, the coatings prepared by SHS powders exhibited superior density and inferior oxidation activity, which significantly heightened the structural oxygen blocking ability of the coatings in the active oxidation stage, thus characterizing higher oxidation protection efficiency. Notably, the ZrB_2-40 vol%$MoSi_2$ coating sample prepared by SHS powders presented the lowest oxygen permeability of 0.3% and carbon loss rate of 0.29×10^{-6} $g\cdot cm^{-2}\cdot s^{-1}$. By alloying HfB_2 with $HfSi_2$ in the form of composite powders, the distribution of Hf oxides on the glass layer was homogenized, and the protective phase $HfSiO_4$ was formed, thereby enhancing the in situ repair of the oxidation-induced loosening of borides by silicon-based components. Compared to the $60HfB_2$-SiC coating, the $60(80HfB_2\text{-}20HfSi_2)_{SHS}$-SiC coating exhibited optimal oxygen blocking protection after oxidation at 1700 °C, with 87.96% and 92.33% reductions in oxygen permeability and carbon loss rate, respectively. Enhanced by the synergistic strengthening from dual transition metal silicides Mo and Ta, the destructive dynamic evolution of the coating's oxygen-blocking structure was suppressed. The increase in $MoSi_2$ content promoted the dispersion of transition metal oxide nanocrystals within the SiO_2 glass layer, while the strong complexing action of Ta^{5+} increased the overall viscosity of the glass film, thereby strengthening the inert oxygen-blocking ability of the composite glass layer during the inert oxidation stage. Compared to the $MoSi_2$-$50ZrB_2$ coating, the addition of 5 vol% $TaSi_2$ modified coating decreased oxygen permeability from 0.46% to 0.16%, a 65.22% reduction, elevating the cumulative protection efficiency to 99.70%.

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Oral Report

Self-Propagating High-Temperature Synthesis of Advanced Boride-Silicide Ceramics

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The results of the studies in the SHS field of a wide range of ceramic materials (CM's) based on hafnium, zirconium and molybdenum borides and silicides are presented. Designed CM's are promising for high-temperature applications for the fabrication of heat-loaded structures as well as powder precursors for the production of ceramic-matrix composites and cathodes for magnetron sputtering of functional coatings.

The features and mechanisms of combustion in multicomponent Mo/Zr-Si-B, Zr/Hf-Mo-Si-B, Zr-Si-Al-B and Zr/Hf-Si-B-C systems have been studied, as well as the mechanisms of phase and structure formation of heterophase SHS-products based on $MoSi_2$-MoB, ZrB_2-$ZrSi_2$/ZrSi, ZrB_2/HfB_2-$MoSi_2$-MoB, HfB_2/ZrB_2-$HfSi_2$/$ZrSi_2$-$MoSi_2$, and ZrB_2/HfB_2-SiC in the combustion wave [1-9]. Primary boride grains occur in a pre-combustion zone due to gas transportation of volatile boron oxides to the surface of metal particles. Further formation of borides proceeds in the combustion front through the Si-based melt via dissolution-precipitation mechanism. Silicides are also formed in the combustion zone due to the interaction of Si-based melt with refractory metal particles by reactive diffusion mechanism with the formation of "core-shell" structures. The later one disappear in the after combustion zone due to homogenization of the chemical composition.

Narrow-fraction heterophase powders of a wide range of boride-silicide compositions consisting of composite particles with an average size of 5-10 μm were obtained using various SHS schemes. They have been successfully applied to obtain structural ceramic-matrix composites with a unique combination of thermomechanical and thermophysical properties, thermal stability and high-temperature oxidation resistance.

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Oral Report

Azide SHS of compositions of highly dispersed nitride powders with carbides

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Recognized advanced ceramic materials include Si_3N_4, AlN, TiN nitrides, and SiC, TiC carbides. Products based on nitride-carbide compositions combine the excellent properties of two materials in one structure and are promising for the use of appropriate ceramic composites with increased properties, lower brittleness, good machinability, lower sintering temperatures compared with single-phase ceramics made of nitrides or carbides. However, the existing technologies for their production are energy-intensive and are implemented using complex, expensive equipment as well as expensive raw materials. In this regard, the application of a simple energy-efficient SHS process to obtain compositions of highly dispersed nitride-carbide powders from inexpensive reagents is of undoubted interest. The report presents the results of using such a type of SHS as azide SHS to obtain compositions of these nitrides Si_3N_4, AlN, TiN with carbides SiC, TiC.

In azide SHS, not gaseous nitrogen is used as a nitriding reagent, but sodium azide powder NaN_3, as well as halide salts – precursors of nitrided elements, which leads to relatively low combustion temperatures, the formation of a large number of intermediate vapor and gaseous reaction products, as well as final by-products of condensed and gaseous products separating the particles of the target powders. Ultimately, this makes it possible to synthesize highly dispersed (< 1 μm) powder compositions of Si_3N_4—SiC, AlN—SiC and TiN—SiC, with Si_3N_4 with a large proportion of α-modification [1]. However, in most cases, the amount of SiC phase synthesized in experiments turns out to be significantly lower than the expected theoretical amount, and SiC may even be completely absent in the target nitride-carbide composition. A similar pattern is observed in the synthesis of compositions with a phase of titanium carbide TiC. In this regard, polytetrafluoroethylene (PTFE) was included in the composition of the initial reagents, which was successfully used earlier in [2] to obtain a composition of Si_3N_4-SiC ceramic powders with a different set ratio of nitride and carbide phases by the SHS method without the use of sodium azide. The introduction of PTFE into the composition of the initial reagents of azide SHS in the preparation of nitride-carbide compositions made it possible to increase the amount of the carbide phase and bring the experimental composition closer to the theoretical composition.

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Oral Report

Medium- and High-Entropy Ultrahigh-Temperature Carbides: Fabrication and properties

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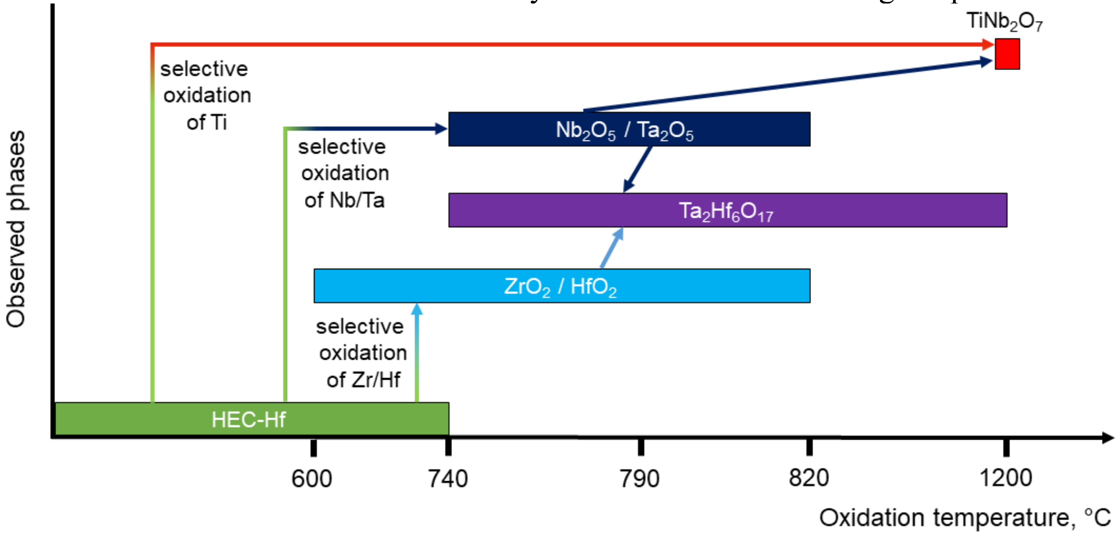
Medium- and high-entropy ultra high-temperature carbides are advanced materials that exhibit unique properties suitable for extreme environments. These carbides are composed of a combination of transition metals like titanium, tantalum, zirconium, niobium, hafnium, tungsten, and molybdenum. They are characterized by their exceptional hardness, high melting points exceeding 3000 °C, resistance to thermal shock, and chemical stability. These materials are crucial for applications in high-temperature nuclear reactors, engines, and vehicles due to their ability to withstand harsh conditions. The development of medium- and high-entropy ultra high-temperature carbides opens up new possibilities for creating materials with enhanced mechanical, physical, and chemical properties to meet the evolving demands of advanced technologies.

In this study, equimolar carbides (TaTiNbZr)C and (TaTiNbZrX)C (X = Hf, W, Mo) were synthesized by high-energy ball milling (HEBM), self-propagating high-temperature synthesis (SHS), and spark plasma sintering (SPS). It has been shown that Hf added provides the best heat oxidation resistance, increasing it by more than two times. This is due to the formation of complex oxides, such as $TiNb_2O_7$ and $Ta_2Hf_6O_{17}$, which improve the material's stability at high temperatures. The regularity of the formation of the high-entropy carbide structure and the oxidation mechanism of (TaTiNbZrHf)C at temperatures between 25 and 1200 °C is described. The evolution of the oxide layer structure with increasing temperature is as follows:



This study was conducted with the financial support of the Russian Science Foundation (Grant No. 23-79-10217).

Oral Report

Synthesis and characterization of ultra-high temperature HfCN and (Ta,Hf)CN ceramics

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The melting temperatures of hafnium carbide HfC and mixed tantalum-hafnium carbide (Hf,Ta)C are among the highest, ranging from 4232 ±84 K [1] and 4300±80 K [2], respectively. Recent theoretical studies have suggested that compositions in the Hf-C-N system may have even higher melting temperatures due to the high strength of covalent and ionic bonds in these compounds, as well as point defects and vacancies [3, 4].

Conventional methods for fabricating carbonitrides frequently demand prolonged exposures at elevated temperatures to yield a single-phase product. An alternative to conventional techniques may be a combination of self-propagating high-temperature synthesis (SHS) and spark plasma sintering (SPS). The fast combustion process and high heating rates during SPS allow for a significant reduction in the duration and energy consumption of materials production.

In this study, the aforementioned techniques were employed for the production of HfCN and (Ta,Hf)CN powders (SHS) as well as bulk ceramics (SPS). The synthesis of hafnium carbonitride was carried out in a laboratory reactor under various nitrogen pressures, utilizing both activated and non-activated mixtures of Hf + 0.5C. It was found that there is a dependence of combustion regimes on the nitrogen pressure in the reactor for non-activated mixtures, while the activated mixture burns in a stationary mode over the entire range of pressures studied. The conditions under which the synthesized hafnium carbonitride is single-phase were determined. Under these conditions, it was also possible to obtain single-phase powders of double carbonitrides (Ta,Hf)CN with different Hf/Ta ratios. The features of phase and structure formation during the combustion of reaction mixtures were established for both systems.

Studies of residual porosity dependence on consolidation parameters allowed the identification of a sintering regime at which dense ceramics in the Hf-C-N and Ta-Hf-C-N systems with a relative density of 98% were obtained. For the examined samples, data regarding hardness and fracture toughness were obtained, the correlation between specific heat capacity, thermal diffusivity, and thermal conductivity with temperature was measured, and the temperatures of the beginning and end of melting were determined.

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Keynote Report

Cermet Fabrication by SHS of Ceramic Skeleton with Subsequent Spontaneous Infiltration by Molten Metal

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Skeleton cermets are ceramic–metal composites with a high content of ceramic phase in the form of skeleton which possess unique properties and have recently been the subject of a significant amount of research [1]. However, existing technologies for their production are energy-intensive and are implemented with the use of sophisticated expensive equipment. The energy-efficient process of SHS has been employed in well-known technologies of SHS pressing and SHS casting to produce skeleton cermets with simultaneous use of the heat of the SHS reaction both for the synthesis of a ceramic skeleton and for metal melting for infiltration (impregnation) of the synthesized skeleton. But at the same time, only due to the heat of the SHS reaction, a small amount of metal can be melted, which limits the dimensions of the synthesized cermets, and for complete impregnation and production of non-porous cermets, the application of excessive pressure (SHS pressing) or centrifugal forces (SHS casting) is required, which greatly complicates the process. Recently, the authors of the report proposed a new method for producing skeleton cermets based on the performance of the SHS process in the simplest conditions of reactor-free combustion in air or in a sand fill to synthesize a porous ceramic skeleton followed by spontaneous infiltration of the synthesized hot skeleton with a metal melt prepared previously by heating from an external source, which allows using a melt mass sufficient for complete impregnation of the ceramic skeleton without applying excessive pressure or centrifugal forces [2].

The report presents the results of the development of this new method in the form of various experimental schemes that allow obtaining samples of cermets of various types and sizes. The conditions for the formation of the least defective SHS porous skeletons TiC, Ti_3AlC_2 and Ti_3SiC_2 for subsequent spontaneous infiltration are determined. The interactions of hot SHS skeletons with melts of metals Al, Cu, Sn have been studied: wetting, patterns of spontaneous infiltration, impregnation completeness, microstructure, phase composition depending on the temperatures of the skeleton and the melt, the delay time between the end of combustion and the beginning of impregnation, alloying of the skeleton and the melt. Samples of SHS cermets TiC-Al, Ti_3AlC_2-Al, Ti_3SiC_2-Cu, Ti_3SiC_2-Sn have been experimentally obtained, their physical and mechanical properties have been studied.

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Keynote Report

Environmental sustainability of SHS

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Several metrics exist for the quantification of the greenness of a particular synthesis, including mass-based metrics (e.g., Environmental-E factor, Atom Economy-AE, Mass Intensity-MI), environmental-metrics, and energy-based metrics. These metrics allow quantifying if and at which extent a particular synthesis meets one or more of the twelve principles of green chemistry and green engineering [1, 2]. The main limitation of the above-mentioned metrics is that they are typically focused on the sole chemical products or processes, thus neglecting (or only limitedly considering) further phases of their whole life cycle. This means that in order to quantify in a reliable and trustworthy way the environmental sustainability of a given product or process, a life cycle thinking (LCT) perspective is mandatory. At this latter regard Life Cycle Assessment (LCA) represents the prominent scientific methodology to assess resource use, emissions, and related potential environmental impacts associated with a given, product, process, service, or system throughout its complete life cycle [3].

Self-propagating High-temperature Synthesis (SHS), and more generally Combustion Synthesis (CS) is recognized worldwide as a greener synthetic strategy with respect to more conventional and well-established ones, mainly as a consequence of its peculiar characteristics (e.g., energy and time saving). Nevertheless, in most cases the environmental sustainability of SHS/CS is still to be considered as an act of belief, due to the surprising scarcity of scientific works aiming at its quantification [4, 5].

In this work the available metrics developed through the years to quantify the green characteristics and the environmental sustainability of a given product/process will be overviewed with a special emphasis on the LCA methodology, in order to highlight its potentialities in being applied to SHS/CS. Indeed, when applied to both SHS and solution combustion synthesis (SCS), LCA demonstrated the lower environmental impacts of these synthetic approaches with respect to the more conventionally employed ones. LCA therefore results fundamental in confirming the environmental sustainability of SHS/CS strategies, thus potentially contributing to further promote an always larger and larger utilization of this fascinating materials synthesis procedure.

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Oral Report

SHS metallurgy: New Alloys and Composite Materials for Science and Production

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Metallothermic SHS is a promising method for development of new materials and production of pilot batches of alloys and composite materials. Metallothermic SHS has a number of advantages, it eliminates heating equipment (furnaces), provides high performance, and allows to obtain a wide range of materials useful for practice [1–3]. In this work, we aimed at exploring the preparation of multicomponent high-temperature alloys based on nickel, titanium and their aluminides, tungsten and molybdenum, niobium, etc. and characterized by high strength, corrosion resistance and wear resistance at high temperatures under conditions of high loads and intense friction via metallothermic SHS. The following SHS reaction schemes were used:

$$\mathrm{NiO/Al + A \rightarrow NiAl/Cr,\ Co,\ Ti,\ Mo,\ W + Al_2O_3}, \quad (1)$$

$$\mathrm{CoO/Al + A \rightarrow CoO/Cr,\ Nb,\ W,\ Mo,\ C,\ Al + Al_2O_3}, \quad (2)$$

$$\mathrm{TiO_2/Al/Ca + A + E \rightarrow TiAl/Nb,\ Cr + Al_2O_3/CaO}, \quad (3)$$

$$\mathrm{MoO_3/Al/Si/B + A \rightarrow Mo–Si–B/Nb + Al_2O_3}, \quad (4)$$

where A is the alloying additive and E is the energy additive (CaO_2/Al).

Studies showed that the combustion temperature of mixtures is more than 2500–3000 K, which makes it possible to prepare cast alloys. Combustion of mixtures (1)–(4) in atmospheric conditions is accompanied by strong splashing of burning products. The forces of artificial gravity suppress it, so the synthesis of alloys was carried out in a centrifugal machine at the centrifugal acceleration $a = 1–300\ g$. The report presents the results of studying the combustion process, gravitational separation of the target and slag combustion products, and their phase composition and structure, as well as the influence of the scale factor on the processes and formation of cast products. The report also presents results on the development of integrated technologies for producing spherical powders from SHS alloys for additive technologies. Integral technology includes the production of a pilot batch of alloys, melting, and their subsequent gas atomization.

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Oral Report

The technology for producing cast high-purity chromium by centrifugal SHS metallurgy

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The metal chromium (Cr) is one of the most important alloying elements and has found its application in modern technologies. The use of Cr includes two main areas: (i) alloying steels and alloys; (ii) application of metal coatings.

The presence of chromium as an alloying element in modern steels and alloys can significantly improve the mechanical properties of the material. In addition, Cr belongs to the group of refractory metals and its introduction, in concentrations above 13%, significantly increases the operating temperatures of the alloys and corrosion resistance.

The application of Cr to the surface protects items from mechanical damage due to its hardness, as well as from exposure to aggressive environments due to its resistance to corrosion. Cr have good adhesion to many materials. Therefore, it has also found its application in microelectronics as an adhesion promoter for subsequent layers.

Modern industrial production of Cr includes two main technologies: (i) aluminothermal reduction of chromium (III) oxide (Cr_2O_3) using electric furnaces and (ii) electrolytic reduction of a solution of chromium anhydride (CrO_3) or chromium - potassium alum.

The technology of aluminothermic reduction is highly productive, but at the same time it has high energy consumption, an unecological process, low purity of metallothermic Cr (97.5-99.0% mass). The main impurities for this group of Cr are Al, Fe, Si, S, O and N.

Electrolytic reduction technology makes it possible to obtain high-purity Cr but is low-productive, technologically harmful production and, as the consequence has high Cr cost.

Therefore, the task of producing Cr based on new energy- and resource-saving technological solutions is relevant, especially taking into account the large-scale production of chromium.

Centrifugal SHS metallurgy for producing high-purity Cr, with a purity level close to electrolytic Cr, has prospects for industrial development. It should be noted that SHS metallurgy methods can significantly reduce the cost of chromium produced while simultaneously increasing productivity. This is of high practical interest.

This report will demonstrate the results of research on the production of high-purity cast chromium using SHS metallurgy methods. To achieve high levels of purity and depth of chemical reactions, all synthesis experiments were performed under conditions of centrifugal forces. The choice of chemical synthesis schemes and optimization of synthesis parameters made it possible to successfully solve the task. To obtain commercial chromium ingots and produce pilot batches of high-purity chromium, a centrifugal SHS installation, which has no analogues in the world, was specially designed and manufactured.

Oral Report

The pilot industrial centrifugal machine. Development, production and successful approbation for producing cast materials by centrifugal SHS metallurgy

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Technologies based on the SHS process to obtain practically in-demand materials have a number of technological advantages, among which are low energy consumption, high productivity, compliance with modern environmental requirements (low carbon footprint), etc. However, in order to move from laboratory research to the pilot industrial level and industrial technologies require the use of specialized equipment certified for use in industrial enterprises. Some of this equipment is produced industrially to solve problems in powder metallurgy (classifiers, dispensers, mills, etc.).

However, for safety and controlled SHS process, specially designed equipment is often required to allow the process to be scaled up and produce pilot batches of materials. In most cases, such specialized equipment is not available on the market and can only be produced according to individual requests.

As part of an agreement on long-term cooperation between LLC "Center for Research, Design and Technology" (Tula) and ISMAN (Chernogolovka), for the purpose of mutually beneficial development and use of high-tech production, a unique line of centrifugal machines was developed for the production of cast materials (pure metals, alloys, metal matrix composites) using centrifugal SHS metallurgy methods.

The design of the SHS installation is original [1] and includes such systems as: a laser initiation system, an automatic rotor speed control system to maintain a given level of overload (g), a water cooling system for the reaction (melting) unit and a video monitoring system with on-line capability recording the combustion process of the composition.

The installation has two reaction blocks located symmetrically on the rotor of the installation and allows for overloads of up to 400 g with a mass of burned samples of up to 10 kg. After selecting and optimizing synthesis parameters, a series of alloys Cr-W, Cr-Mo, Cr-Al and others were obtained with a mass of one ingot up to 5 kg.

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Keynote Report

Making Ultra-Tough Al_2O_3/ZrO_2 Nanoceramics through Columnar Submicrocrystals with Three-Level Micro-Nano Structures

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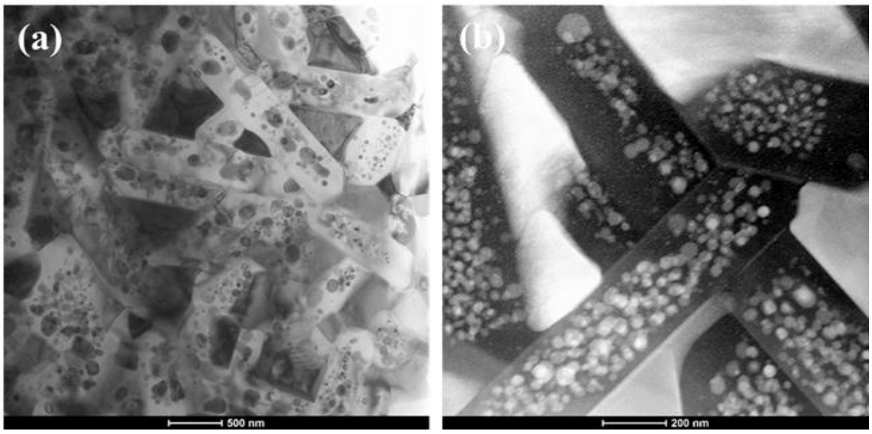
The limited fracture toughness exhibited by equiaxed nanocrystalline ceramics poses a significant obstacle to their widespread commercial applications. Herein, a novel approach is presented to address this limitation by producing ultra-tough nanoceramics using amorphous and supersaturated Al_2O_3/ZrO_2 solid solution micro powders. These powders are synthesized through Al-O_2 ultrahigh-temperature combustion synthesis, assisted by rapid water cooling[1]. The Al_2O_3/ZrO_2 micro powders containing amorphous and metastable dendritic solid solutions can induce a three-level micro-nano structure with a high content (up to 50–70%) of columnar submicro-crystals accompanied by high-density nanoprecipitation after sintering, as shown in the figure. Additionally, the precipitation mechanism of the nanoparticles is also studied by phase field simulation, and the results are well consistent with the experimental results[2,3]. This unique structure leads to a significant enhancement in the mechanical properties of Al_2O_3/ZrO_2 ceramics. The obtained ceramics exhibit exceptional bending strength of 1278 MPa and Vickers hardness of 20.26 GPa. In particular, the fracture toughness can reach a value of 15.59 $MPa\cdot m^{1/2}$, which is nearly twice as high as the toughness in standard ZTA ceramics[4-6]. This method offers several advantages, including simplicity, low cost, and high efficiency, making it an attractive option for the microstructural design of high-performance ceramics.



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Oral Report

The influence of mechanical activation on the physical and mechanical properties of AlN obtained by spark plasma sintering

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AlN-based materials are promising for use in various fields of technology, due to the combination of hardness like ceramics with thermal conductivity like metals. Due to the low sintering activity of AlN powders, high-density compacts are produced by methods involving the application of high temperatures and pressures, the addition of activating additives or nanopowders [1-4].

The goal of the work is to obtain materials from AlN powders with maximum density at relatively low temperatures and pressures.

AlN powders were obtained by carbothermal reduction of aluminum oxide powder AOA in N_2 and were subjected to mechanical activation (MA) in a planetary mill in N_2 with the isopropyl alcohol. For subsequent activation of sintering 7 wt.% of Al powder PA-4 Al or 3 wt. % of powder Y_2O_3 ItO-V was added to the AlN during MA. Consolidation of the powders was carried out by spark plasma sintering (SPS) of compacts in a vacuum at a temperature of 1750 °C, a pressure of 30 MPa, 5 min, or by pressing-sintering in a vacuum oven at a temperature of 1780 °C, 4 h.

After MA the average particle size of AlN powder with the addition of Al reached 7 µm, and with the addition of Y_2O_3 – 5 µm. The relative density of AlN samples after pressing-sintering was 73.2 % for samples without MA, mechanically activated: 82.1 % without additives, 84.4 % for the AlN-7Al system and 99.6 % for the AlN-3Y_2O_3 system; for MA materials after “pressing-SPS” - 99.4 %, 97.6 % and 99.7 % for the AlN, AlN-7Al, AlN-3Y_2O_3 systems, respectively.

After SPS of MA powders AlN, AlN-7Al and AlN-3Y_2O_3, the microhardness was 13.3, 17.0 and 9.3 GPa, and the compressive strength was 1479, 654 and 1688 MPa, respectively.

The lower values of density and strength of the material containing Al are due to the formation of films of aluminum oxynitride, which prevent the sintering of AlN particles, but help to increase the hardness of the composite.

Thus, the MA of AlN powders and the absence of impurities make it possible to obtain vacuum-tight ceramics using the SPS method at relatively low temperatures, pressure and sintering time.

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Oral Report

Calculation of the maximum gas pressure in a pore depending on the pore radius

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Porosity affects the properties of crystalline and amorphous materials. The pressure of the gas in the closed pores also affects the properties of the materials, which is especially important for additive technologies [1]. The aim of this work was to obtain an analytical expression for estimating the maximum possible gas pressure in closed pores.

The following assumptions were made: there are no liquefied gas in the pores, the gas in the pore do not dissolve in the material. The analytical expression was based on G. Lyame's solution of the problem of the stress–strain state of a material near a spherical pore [2]. The Laplace pressure P_L which acts from the material to the pore, is also taken into account:

$$P_L = \frac{2\alpha}{R}, \tag{1}$$

where α is the surface tension of the material and R is the pore radius. Pressure in the pore is equal to the difference between the Laplace pressure and the gas pressure in the pore.

Plastic deformation of the material near pore begins when the stresses reach a certain critical value. According to the fourth theory of strength, plastic deformation in a material begins when the equivalent stresses σ_{eq} exceed the specified permissible stresses [σ]:

$$\sigma_{\text{eq}} = \frac{3|P_L - P_G + P_0|}{2} \le [\sigma]. \tag{2}$$

The permissible stress [σ] is the yield strength of the material $\sigma_{0,2}$. If we consider the isothermal case and convert (2) taking into account (1), we can obtain the expression for calculating the maximum possible pressure in the pore $P_{G\,\max}$ with radius R:

$$P_{G\,\max} = \frac{2\alpha}{R} + \frac{2}{3}\sigma_{0,2}. \tag{3}$$

Expression (3) is a criterion for the onset of plastic deformation of the pore at a pressure above $P_{G\,\max}$. Note that expression (3) must be used for pores larger than 1 nm since the classical approach to surface tension does not work for smaller pores.

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Oral Report

Assessment of the formability of powder-polymer mixtures with the main types of binders for the production of metal parts using PIM technology

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One of the technologies of thixotropic metallurgy that uses a change in the rheological state of the processed material is the Powder injection molding (PIM) [1]. The purpose of this work is to compare the powder-polymer mixtures (feedstocks) used in PIM technology with the main types of binders: based on polyoxymethylene and based on a mixture of waxes with polyolefins. The types of feedstocks being compared contain powder identical in volumetric filling and granulometric composition.

The process of forming feedstocks can be divided into 2 stages: filling and compaction. For the compaction stage, it has been established [2] that feedstocks with a wax-polyolefin binder are preferable for molding semi-finished products with large-sized elements in which there is a risk of shrinkage defects. This is explained by the fact that the isochoric coefficient of thermal elasticity of feedstocks with a polyoxymethylene-based binder is 1.4 times greater than that of feedstocks with a wax-polyolefin binder, i.e. to compensate for changes in the volume of such feedstock, 1.4 times greater pressure will be required. At the same time, at the stage of removal from the mold, polyoxymethylene-based feedstocks reduce the likelihood of damage to the casting due to better thermomechanical properties [3]. For the mold filling stage, it has been established [4,5] that feedstocks with a wax-polyolefin binder have a significantly lower viscosity, which is an advantage when molding thin-walled products, but the 1.5 times greater sensitivity to the shear rate gradient makes them less resistant to jets and segregation of components. At the same time, feedstocks based on polyoxymethylene are more technologically advanced in their thermophysical properties, since the kinetic effects in the thermal processes of their processing are less significant, and the rate of equalization of the temperature field in them is significantly higher [5]. These competing differences lead to the need to compare complex parameters characterizing the moldability and formability of feedstocks.

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Oral Report

Gas pressure in the critical size pore in the compacts obtained by gas extrusion of nickel nanopowders

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When obtaining products from powders and powder mixtures, the problem arises about the effect of porosity and pressure in pores on mechanical properties. It is known that high gas pressure in the pores increases the rigidity of the product and compressive strength, but reduces the tensile strength. The aim of this work is to estimate the pressure inside a material by the minimum pore size in nickel rods obtained by hot gas extrusion of nickel nanopowders.

Hot gas extrusion was chosen for the consolidation of the nickel nanopowders because this method uses local heating in the deformation region, and the workpiece is in the heating region for the least amount of time, which can be critical for nanopowders because during their consolidation, it is necessary to slow grain growth. During gas extrusion, the workpiece is extruded through a forming die of a set diameter due to a combination of local heating and high pressure of an inert gas (Ar) [1].

Nickel nanopowder with an average particle size of 72 nm was used as the initial material. Compacts were made from nanopowders by pressing them in an elastic shell and heat treatment. The compact density was 93.5 % of the theoretical density and all the pores were closed [2]. The compacts were extruded by hot gas extrusion with a deformation degree of 93% to a wire with a diameter of 2 mm. The argon gas pressure was 400 MPa, and the extrusion temperature was 910 °C. The density increased to 98.5 % of the theoretical density.

To determine the critical pore radius, the microstructure of the wire cross-sectional view was studied using an electron microscope. Studies have shown that the maximum pore diameter reaches 670 nm, and the minimum is 220 nm. To estimate the gas pressure in the pore, an expression was used to determine the minimum pore radius R_{cr} [3]:

$$P_G = \frac{2\alpha}{R_{cr}} + P_0 - \frac{2}{3}\sigma_{0,2},$$

where P_G is the gas pressure in the pore, α is the surface tension coefficient of the material, P_0 is the external pressure, and $\sigma_{0,2}$ is the yield strength of the material. At the extrusion temperature, α for Ni is 1.86 N/m, $\sigma_{0,2}$ is 60 MPa, and the pressure P_0 is 400 MPa. R_{cr} = 110 nm (D = 220 nm). Then, the gas pressure in the pore will be 390 MPa. If we assume that the pore size does not change when the material is cooled from 900 to 20 °C, then from Gay-Lussac's law states that the pressure in a 110 nm pore for this material can reach 98 MPa.

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Keynote Report

Current and Future of SHS for MAX Phases and 2D MXenes

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MXenes are potentially the largest class of 2D materials discovered so far. With a general formula of $M_{n+1}X_nT_x$, M is an early transition metal (Ti, V, Nb, Ta, etc.), X is C and/or N, T_x represents the surface groups (-O, -OH, -F, -Cl), and n = 1–4, over 30 stoichiometric phases have already been discovered, with many more predicted computationally. This class of materials has been widely studied owing to their exceptional properties, including hydrophilicity, scalability, mechanical strength, thermal stability, redox capability, and ease of processing. Owing to these properties, MXenes are widely used in electrochemical energy storage, biomedicine, printable electronics, electromagnetic interference shielding, and more. Because MXenes inherit their structure from $M_{n+1}AX_n$ (MAX) phase precursors, understanding MAX phase synthesis leads to control over flake size, defect density, and chemical composition of the resultant MXene. Historically, SHS has been widely used for the synthesis of MAX phases, but very few studies have topochemically synthesized MXenes from these MAX phases. Herein, the benefits and limitations of SHS-synthesized MAX will be described, focusing on their use for MXene synthesis.

Keynote Report

Nanoenergetic Materials: Recent trends and Emerging applications

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Nanoenergetic materials (NMs) are experiencing rapid growth and development with emerging applications in diverse fields, including fire extinguishing device, high-power linear actuators, solid fuels for micropropulsion systems, and biocidal agent defeat formulations [1, 2]. These materials are typically produced in heterogeneous forms and exhibit highly exothermic systems based on thermite composites, which are a combination of metallic fuel (usually aluminum powder) and various metal or nonmetal oxides powders such as Fe_2O_3, Co_3O_4, MoO_3, and highly powerful oxidizers include I_2O_5, Bi_2O_3 and $Bi(OH)_3$. Both bismuth- and iodine-based oxidizers have shown excellent performance for nanoenergetic gas generators reaching a maximum pressure × volume per mass (PV/m) value up to 14.8 $kPam^3/g$ [3]. The reason for the exceptional performance of bismuth- and iodine-based NMs is because the boiling temperature of the reaction final product, such as iodine or bismuth, is significantly lower than the combustion adiabatic temperature, which contributes to gaseous products during combustion and improves the pressure discharge ability. The use of nanostructured particles, as opposed to microparticles, enhances intimate contact between the fuel and oxidizer, reducing mass transport limitations and increasing reaction rates and reactivity. Thermodynamic calculations of nanoenergetic reactions have shown maximum adiabatic temperatures of up to about 4000 K. This presentation explores the modeling and experimental development of novel nanoenergetic systems, emphasizing their high pressure-volume values and energy densities for emerging applications.

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Oral Report

Scalable in-situ solution combustion synthesis for the preparation of multichannel ceramic structured catalysts

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Solution combustion synthesis (SCS) is a preparation technique that can be used to synthesize a variety of structured catalysts. The self-propagating exothermic redox reaction between organic salts and a fuel allows the direct coating of supports as monoliths and open cell foams. As a results, a thin film of the desired catalyst is directly formed on the support for the final application. The methodology can be applied for the scale-up of fuel processors for the production of syngas and, further, hydrogen [1]. Reactions of interest are the steam reforming of biogas and dodecane [2].

Details on the scale-up of SCS and related hydrogen productivity results will be presented. Figure 1 shows the scale-up of in-situ SCS for coated monoliths and coated open cell foams and the performance obtained on the multichannel reactors.

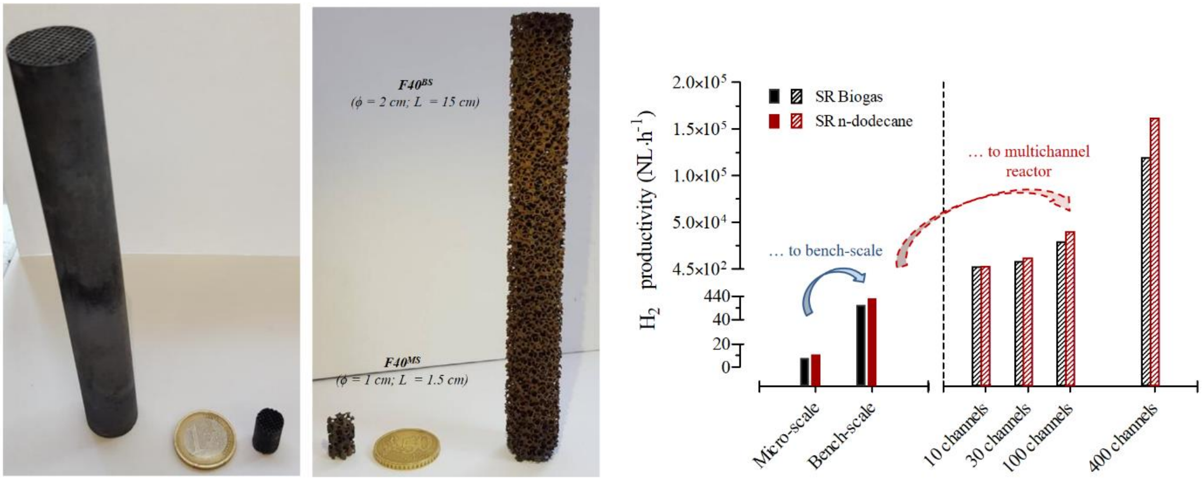

Figure 1. Scale-up of in siity SCS coated monoliths and open cell foems and related H_2 productivity by steam reforming of biogas and dodecane.

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Oral Report

Combustion Synthesis of YBCO Superconducting Composites

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The technological ways of sample preparation and synthesis methods are significantly impact on the electrophysical properties of high-temperature superconducting composites, particularly YBCO based composites. Nowadays, most leading scientists from all over the world are focusing their main efforts on reducing the cost and time of obtaining superconducting materials and products based on them with specified properties. The main task in this scientific work is to obtain high-temperature superconducting composites based on cuprates and to study their electrophysical properties. A series of experiments were conducted and based on them the optimal parameters of sample synthesis process were selected. Laboratory samples were obtained by burning in two consequently stages: in the atmosphere of a muffle furnace and in the oxygen environment of a tubular furnace, respectively. As a green component of sample mixture were used the powders of copper oxide, yttrium oxide, barium oxide. All green components were thoroughly mixed, grounded, and calcined in air at high temperature with long holding time in a muffle furnace in a normal atmosphere. After that, the obtained powders were then pressed into the form of granules. To obtain samples fully saturated with oxygen, the samples were annealed in an oxygen flow in an oxygen tubular furnace, followed by slow cooling to room temperature. It has been established that the initial ratio of green components, the annealing temperature, and the holding time have a direct effect on the qualitative and quantitative formation of the superconducting phase, which particularly effect on the J_c parameter [1-2].

Acknowledgement: This research has been funded by the Science Committee of the Ministry of Science and Higher Education of the Republic of Kazakhstan (Grant No. AP19677755).

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Oral Report

Magnetic Heating Properties of Iron-oxide Nanoparticles Obtained by Solution Combustion Synthesis

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Iron-based magnetic nanoparticles have attracted considerable attention for their wide range of applications in biomedicine, environmental cleanup, and data storage. [1]. Solution combustion synthesis (SCS), provides a versatile method for the large-scale production of these nanoparticles (NPs), utilizing self-sustained chemical reactions in solutions containing oxidizers and fuels [2]. One key advantage of SCS is its ability to precisely tailor the size, composition, and shape of the nanoparticles [3]. In this study, we focused on exploring the magnetic properties of iron-oxide nanoparticles produced through SCS using the iron-nitrate-glycine system for application in *magnetic hyperthermia*.

Our investigation involved a thorough analysis using various techniques such as X-ray diffraction, scanning electron microscopy, and vibrating sample magnetometry to understand the structural and magnetic characteristics of the synthesized nanoparticles. We also delved into the impact of synthesis variables like precursor concentration, combustion temperature, and *inert gas pressure* on the magnetic behavior of the nanoparticles.

Our findings highlighted a clear relationship between the synthesis parameters and the magnetic properties of the nanoparticles, indicating that factors like morphology, size, and composition significantly influence their performance. Notably, we discovered that inert gas pressure serves as a critical factor in controlling the structure and magnetic properties of the nanoparticles.

Furthermore, our research explored the potential applications of these nanoparticles in magnetic hyperthermia, showcasing their potential for advancements in this field. Specifically, we observed a significant maximum specific loss power (SLP) of 174 W/g under optimal magnetic hyperthermia conditions (magnetic field: 33.5 mT, frequency: 259.6 kHz), as illustrated in the Figure.

The research is conducted within the ADVANCE Research Grants provided by the Foundation for Armenian Science and Technology.

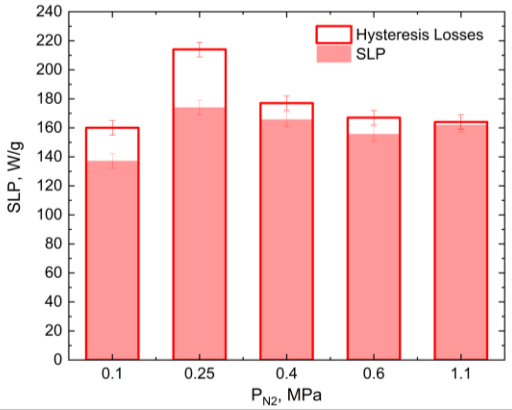

Figure: SLP and hysteresis losses, as a function of inert gas (N_2) pressure.

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Oral Report

$(Mo_{2/3}Y_{1/3})_2AlC$ i-MAX Phase Through Self-Propagating High-Temperature Synthesis

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The application of MXenes in industrial contexts is often challenged by their complex and energy-intensive production processes, which lack cost and energy efficiency. Moreover, the scalability of such processes is often at fault. By and large, a significant factor contributing to the efficacy of the MXene synthesis process lies in the approach taken towards synthesis of the parent MAX phase.

In this study, we employed Self-Propagating High-Temperature Synthesis (SHS) to prepare the *i*-MAX phase $(Mo_{2/3}Y_{1/3})_2AlC$. This method involves the utilization of Poly(tetrafluoroethylene) - $(C_2F_4)_n$ as the promoter to instigate an exothermic reaction, with the adiabatic temperature carefully regulated by controlling the molar ratio of the fuel, aluminum, and yttrium. Furthermore, an alloy of aluminum and yttrium with the chemical formula of $AlY_{2.3}$ is employed to compare *i*-MAX phase yield. The resulting *i*-MAX phase exhibits a morphology characterized by plate-like structures scattered in irregular closely packed porous grains, intermixed with impurity phases and side products such as AlF_3 and YF_3. This work underscores the remarkable potential of the employed method in facilitating the straightforward and efficient synthesis of various MAX and *i*-MAX phases.

Keywords: MXene, SHS, combustion synthesis, MAX phase, *i*-MAX.

Oral Report

Low-Pt Nanoparticles-decorated Micro-nanoporous CuOx Coniferous Electrocatalysts via Thermal Explosion Reaction and Dealloying

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Realizing the efficient and stable bifunctional electrocatalyst for hydrogen evolution reaction (HER) and oxygen evolution reaction (OER) represents a pivotal milestone in the realm of sustainable hydrogen production yet challenging because the availability of resources such as Pt, Ir or Ru, commonly employed in HER/OER, is limited. Cu-based electrocatalyst is low cost and environmental but has been little-explored on water splitting on account of the fragile H adsorption and instability of Cu complexes. Herein, a novel micro-nanoporous CuO_x coniferous composite decorated with low-Pt nanoparticles (Pt@MNPC) is synthesized, through a combination of thermal explosion reaction and dealloying strategy. The bicontinuous ligament/micro-nanoporous structure, featuring a coniferous internal surface, enhances electron transfer and electrolyte accessibility, further increasing the exposure of active sites and channels crucial for efficient mass transport. Besides, the uniform distribution of ultrasmall Pt nanoparticles homogeneously micro-dispersed on the CuO_x matrix further facilitate the catalytic activity due to the inherent activity. Accordingly, the optimized MNPC@Pt-10 exhibits remarkable performance towards both HER and OER, achieving low-overpotentials of 150 and 300 mV@10 mA cm^{-2} in 1M KOH electrolyte, respectively. By the way, when applied to photocatalytic degradation of methyl orange, Pt@MNPC-10 demonstrates a remarkable degradation rate of 81.71% within a mere 5 minutes.

Keywords: thermal explosion reaction; dealloying; micro-nanoporous Pt@CuO_x; hydrogen evolution reaction; oxygen evolution reaction

Acknowledgments: This work was supported by the National Natural Science Foundation of China (No. 52020105011) and the Advanced Analysis & Computation Center of China University of Mining and Technology.

Oral Report

Formation of a ternary alloy based on Cu-Mn-Al system under high-temperature synthesis conditions: combustion, structure and phase formation and thermoelectric properties

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This work is devoted to the production of promising ternary alloys based on the Cu-Mn-Al system [1] using the method of self-propagating high-temperature synthesis and the study of their thermoelectric properties in a wide temperature range, such as electrical resistance and Seebeck coefficient.

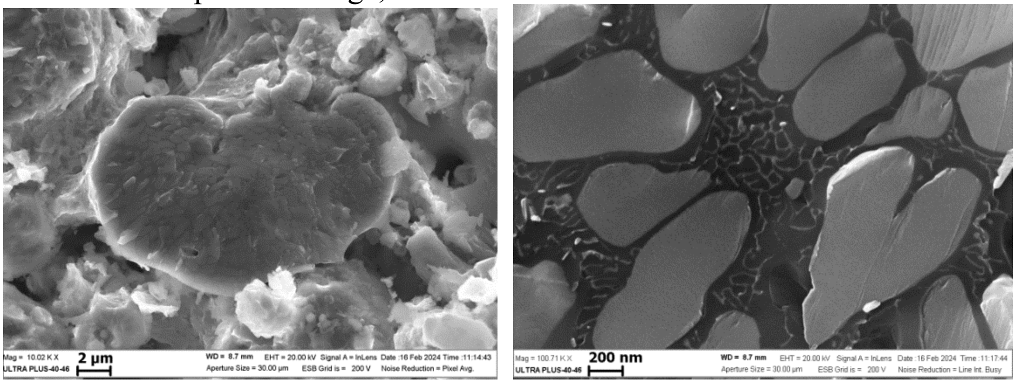

Fig.1 *SEM of the synthesized alloy based on the composition Cu2MnAl*

The synthesized alloys contain a triple Heusler phase (L21) Cu_2MnAl with a cubic crystal lattice (space group Fm-3m) and double phases Al_2Cu, Cu_3Al and AlCu. The phase formation of intermediate double compounds may be due to the fact that in the case of different compositions the reaction took place with an excess of aluminum or manganese. Studies of the thermoelectric properties of alloys based on the ternary phase Cu_2MnAl showed a Seebeck coefficient value of 12 µV/K. The electrical resistivity of the studied alloys at room temperature is in the range of 244÷258 µOhm·cm. The value of the Seebeck coefficient was obtained using a laboratory setup for measuring the Seebeck coefficient of thermoelectric materials in the temperature range of 300–800 K [2].

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Oral Report

Application of TiH_2 powder produced by the SHS method to the preparation of porous titanium and study of its mechanical properties

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Porous titanium is unique material that combines low density and high functional properties, making it attractive for industrial applications such as chemical filters, gas diffusion layers, energy absorbers, medical implants, catalyst supports, etc. [1].

In the course of the study, porous titanium with high porosity (more than 59 %) was prepared by powder metallurgy using a pore-forming agent - ammonium bicarbonate (NH_4HCO_3) and titanium hydride (TiH_2). Titanium hydride was synthesized by the method of SHS hydrogenation of a titanium sponge. The influence of the proportion of pore-forming agent (0, 10, 20, 30 and 60 %) in the mixture, temperature (1150-1200 °C) and sintering medium (vacuum, argon) on the porosity and mechanical properties of porous titanium was investigated.

It is observed that the porosity of the samples increases with the increase in the porous agent content. The strength and Young's modulus of the porous samples decrease with an increase in the porosity content and vary with the medium and sintering temperature. A change in sintering temperature does not significantly affect the compressive mechanical properties of the porous samples. It was found that the porosity value for samples with 0 % porosity agent sintered in vacuum and in argon is different. Samples sintered in vacuum have a porosity of 3 %. Samples sintered in argon have a porosity of 7.9-12.6 %. For samples with 60 % porosity, the porosity of vacuum sintered and argon sintered samples is almost the same and varies from 56.1-59.3 %. It is shown that the strength of porous samples depends on the medium and the sintering temperature. The strength of samples sintered in vacuum is usually higher and can reach 1450 MPa. Changing the sintering temperature from 1150 to 1200 °C does not significantly affect the compressive strength characteristics of porous samples.

The research was carried out at the expense of a grant from the Russian Science Foundation (project No. 22-19-00126). https://rscf.ru/project/22-19-00126/.

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Oral Report

Reaction mechanism during combustion of energetic materials W/PTFE/Al

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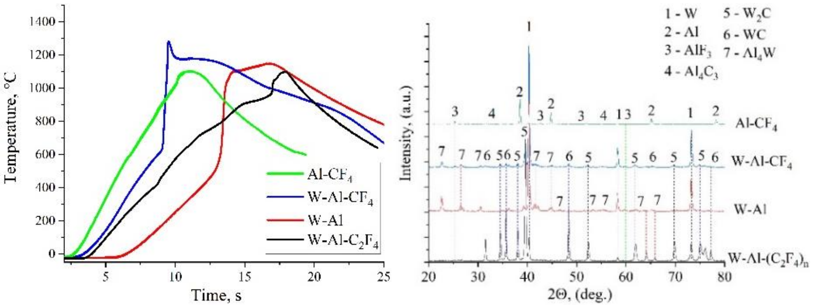
Reactive materials (RM) are inert mixtures of solid components under normal conditions, and under strong mechanical or thermal stress they are capable of intense exothermic reactions [1]. The purpose of this work is to optimize the composition of a reactive material based on tungsten and polytetrafluoroethylene (PTFE) for obtaining high-density combustion products; study of the reaction mechanism of a composition based on tungsten and PTFE. The first explosion experiments showed that the double W/PTFE systems is difficult to be initiated by shock-wave loading. An increase in the reactivity can be achieved by preliminary by activating additives such as aluminum powders. Thermodynamic calculations were performed in the W/PTFE/Al system with different Al contents. The maximum adiabatic combustion temperature corresponds to composition with Al 10% and is equal to 2866 °C with a high proportion of condensed products.

It is known that under normal conditions PTFE is an inert powder. But when PTFE is heated, TFE monomer is formed, which is a colorless gas. The final decomposition products are C and tetrafluoromethane CF_4. Therefore, we assume that aluminum and tungsten react with PTFE decomposition products:

$$\text{-}(CF_2\text{-}CF_2)n\text{-} \rightarrow nCF_2{=}CF_2;\ CF_2{=}CF_2 \rightarrow 2C + CF_4$$

$$4Al + 3CF_4 \rightarrow 4AlF_3 + 3C;\ W + C \rightarrow WC_2$$

Fig. 1 shows thermograms of the mixtures under study and analysis results. It can be seen that when replacing PTFE with TFM, the combustion intensity increases. This is manifested in an increase in the slopes and heights of the peaks of the curves. The lower combustion intensity of the mixture with PTFE is associated with the process of polymer decomposition and shows that Al and W react with the decomposition products of PTFE.



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Oral Report

Properties of biocompatible iron oxide 'Core-Shell' type nanoparticles obtained with Camellia Sinensis extracts

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Leaves of *C. sinensis* contain many secondary metabolites with a wide range of activities and are also a unique source of various biologically active substances, especially from the group of polyphenols. Tea is one of the most consumed beverages in the world, and tea waste can be used for biocompatible nanoparticles production with potential for various applications [1]. Among magnetic metal nanoparticles, iron oxide nanoparticles are of great interest because of their superparamagnetic properties, low toxicity, high bioavailability, and biocompatibility.

The goal of this research was the synthesis of Fe_3O_4 NPs with a biocompatible method using various *Camellia sinensis* extracts, as well as the determination of nanoparticle's physio-chemical properties. For determination of the total content of flavonoids (TCF) was used colorimetric method. The characterization of the samples structure was carried out by the X-ray diffraction analysis. Magnetic characteristics of the composites were studied using a vibrating sample magnetometer in magnetic fields [2].

The determination of TCF revealed that among all studied extracts the highest TCF was observed in the 25% ethanolic extract of *C. sinensis* (53.0±3.98 mkg/ml) which was selected for further synthesis. As a result of the "green" synthesis, a black precipitate was formed. Nanoparticles exhibit superparamagnetic properties and the zeta potential other than zero indicates the stability of the particles in an aqueous solution. The X-ray analysis of obtained NPs showed that the peaks associated with iron oxides correspond to pure Fe_3O_4, and the broadening of the peaks is associated with the particle's nanosize fraction.

Thus, from the above results, synthesized biocompatible iron oxide magnetic nanoparticles can be obtained in a short time, using an environmentally friendly, economical method. Their physicochemical properties can enable a broad range of applications, including visualization, magnetic hyperthermia, optical physics, and more.

The Science Committee of RA, in the frames of the research project № 21T-1F243, supported the work.

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Keynote Report

Powders Based on Titanium Diboride and Carbide for Wear-Resistant Thermal Spray Coatings Obtained by Mechanically Activated Self-Propagating High-Temperature Synthesis

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Wear of machine parts in friction pairs is an important problem of modern mechanical engineering. Creation of antifriction composite materials and coatings, which combine ductile metal matrices and high-strength refractory fillers, is becoming relevant. With this combination of phases, a significant increase in the load-bearing capacity of materials, high wear resistance and scuff resistance are achieved.

When developing composite materials, researchers' attention is drawn to oxygen-free refractory compounds: transition metal borides and carbides. High-temperature sintering, as a method of producing composite materials, requires the introduction of ceramic reinforcing phases directly into the metal bond. However, often due to poor wetting between the components, the resulting composite material is destroyed. To overcome this disadvantage, the technology of self-propagating high-temperature synthesis (SHS) is well suited, where the reinforcing component is synthesized in the process of obtaining a composite. [1]

The aim of this work is to study the possibility of obtaining fine-grained composite powders of the Fe-Mo-Ti-B_4C system with uniform distribution of strengthening phases.

The synthesis of the composite powder in the Fe-Mo-Ti-B_4C system was carried out by combining the methods of mechanical activation, which ensures the occurrence of less thermodynamically preferable reactions of Fe and Mo borides formation, which additionally strengthen the bond, and SHS, which is carried out in a laboratory reactor in an inert atmosphere. The synthesized powders contain TiB_2 inclusions of 1 – 1.5 µm in size and TiC inclusions of up to 1 µm in size, which are fairly well-distributed in the powder particles (Figure 1). The phase composition of the synthesized powder includes TiB_2 and TiC, a solid solution based on α-Fe, and small amounts of Fe_2B and MoB_2. The average micro hardness of the powder is in the range from 15 to 19 GPa. Thermal spray coatings with a thickness of up to 300 microns were obtained from the synthesized composite powder. The coating-substrate boundary is without visible defects. There are no cracks or delamination on the surface of the coating. The average micro hardness of the coating is 19.5-20.4 GPa. The coating inherits the structure of synthesized powders and additionally contains spinel-type oxides (Ti_2O_3 and $FeTiO_3$). The obtained coatings demonstrate high antifriction properties (sliding friction coefficient – 0.21) and resistance to abrasive wear.

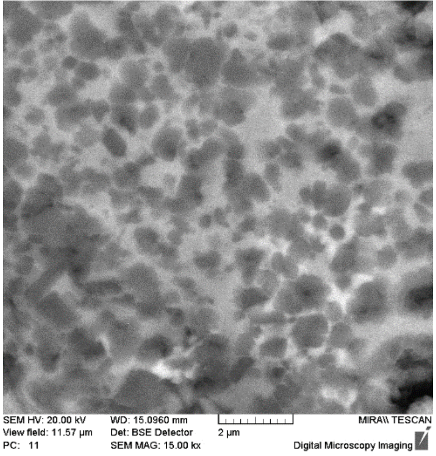

Figure 1. SEM image of the cross-section of a composite powder particle obtained in the Fe-Mo-Ti-B_4C system, magnification × 15000

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Oral Report

Features of Mechanical Activation of Al + CuO Thermite Mixture

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Research was carried out to find optimal conditions for the production of mechanically activated energy composites based on Al+CuO mixtures in the «Activator-2SL» planetary ball mill. New data were obtained on X-ray phase analysis of changes in the elemental composition of the mixture depending on the activation time. With an increase in activation time $t_a > 2$ min, a loss of oxygen content in the mixture and the appearance of free copper occurs, and at $t_a > 4$ min the crystalline phase γ-Al_2O_3 appears. An unexpected result was obtained - during the activation of the Al+CuO mixture in a hexane environment, copper oxide Cu_4O_3 (Paramelaconite) is formed, which is absent in the initial components. During mechanical activation of pure CuO, the formation of Cu_4O_3 does not occur.

Based on the results of X-ray analysis, compositions with the lowest content of reaction products were selected, for which measurements of the ignition temperature and combustion rate were carried out.

Based on experimental data, the optimal mechanical activation conditions for producing mechanically activated composites in a planetary mill were determined. For an initial mixture of aluminum pyrotechnic powder PP-2 and CuO particles with a size of 40-50 microns, the optimal activation $t_a = 100$ s.

Fast-burning compositions have been obtained that are comparable to compositions based on nano-sized components in terms of combustion rates and laser initiation parameters. The combustion speed of the mechanically activated Al/CuO porous composition exceeds 200 m/s, the critical energy density of the laser pulse is <1.7 J/cm^2, the ignition delay is <2.3 ms

An analysis of the process of mechanical activation of thermite compositions based on mixtures of aluminum powders with solid oxidizing agents was carried out. During mechanical processing of thermite mixtures, an increase in reactivity occurs both due to an increase in the oxidizer-fuel contact surface and due to the activation of oxidizers. According to [1], for most solid oxides, mechanochemical activation leads to the formation of “weakly bound” oxygen, which facilitates the metal oxidation reaction.

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Oral Report

Advanced SHS technology for the production of a novel promising class of MAB phase based materials

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Ceramics with a layered crystal structure are a promising material for numerous practical applications and a distinctive model object for fundamental research. The most well-known atomic-layered compounds are the MAX phases [1]. Another group of compounds that may potentially exhibit comparable properties are metal borides. The nanolayered MAB phases, comprising M being a transition metal (such as Mo, W, Cr, Fe, etc.), A being an element of group IIIA-VIA (most commonly Al), and B being boron, also belong to this group [2]. They represent a younger class of oxygen-free ternary compounds having an orthorhombic or hexagonal crystal structure. Materials based on the MAB and MAX phases exhibit excellent properties of both metal and ceramics, including good electrical conductivity, thermal conductivity, high fracture toughness and excellent resistance to high-temperature oxidation. The majority of publications to date have focused on the preparation, identification, and investigation of MAB phases of the following compositions: MAlB (M = Mo, W), M_2AlB_2 (M = Cr, Mn, Fe), Cr_3AlB_4 and Cr_4AlB_6 with *Cmcm*, *Cmmm*, *Pmmm*, and *Cmmm* space groups, respectively [2]. New prospects for obtaining MAB phases arise when employing self-propagating high-temperature synthesis (SHS) [3]-[6].

This work is devoted to investigating the kinetics and mechanism of combustion, analysis the phase and structure formation stages of synthesis products, as well as the preparation of ceramics based on the MoAlB, Fe_2AlB_2 and Mn_2AlB_2 MAB phases with the study of their structure, phase composition, mechanical and thermophysical properties.

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Oral Report

Mechanically activated SHS of high entropy silicides: mechanisms of combustion and structure formation

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The aim of this work was to study the possibility of obtaining ceramics based on high-entropy silicide via SHS on the example of $(Mo_{0,2}Zr_{0,2}Hf_{0,2}Ta_{0,2}Nb_{0,2})Si_2$ composition. The reactive mixtures were prepared by two schemes: 1 – High-energy mechanical processing (HEMP) of all reagents simultaneously using a planetary ball mill; 2 – HEMP of metal (Mo, Zr, Hf, Ta, Nb) powders to obtain multicomponent granules followed by silicon admixing. The HEMP duration effect on the metal granules (MG) structure has been studied. The HEMP conditions under which the MG has a homogeneous structure were determined. The increasing in HEMP duration leads to a transition from the MG nanolayered microstructure to a homogeneous solid solution.

The macrokinetic parameters of combustion of reactive mixtures have been studied. The combustion temperature (T_c) does not change significantly (within 100 K) with an increase in the initial temperature (T_0) from 298 K to 720 K and is close to the Si melting point. At the same time the combustion rate (U_c) increased by 2.5 times. This is typical for liquid-phase combustion mechanism.

The evolution of the phase composition during the combustion process has been studied. Firstly, silicon melts in the combustion front and interact with MG by reactive diffusion mechanism yield an intermediate layer consisted of silicides grains less than 1 μm in size of on MG's surface. In the post-combustion zone coalescence of multicomponent silicide $(Mo,Zr,Hf,Ta,Nb)Si_2$ grains occurs and oxide particles $(Hf,Zr)O_2$ are precipitated along their boundaries.

The SHS-powders based on hexagonal and orthorhombic $MeSi_2$ with an average particle size of 10.7 μm were obtained. They were subjected to hot-pressing yield ceramics with a relative density higher than 98%. The phase composition, structure and properties of HP ceramics has been studied.

This work was financially supported by the Ministry of Science and High Education of the Russian Federation (project 0718-2020-0034).

Oral Report

Time Resolved Photoemission Spectrometer

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Recently an advanced Radio Frequency Timing (RFT) technique has been developed [1, 2]. By converting the time of arrival of incident keV electrons to a hit position on a circle, ellipse or spiral by means of radio frequency electromagnetic fields lying in the range 500-1000 MHz, this device achieves extremely precise timing. Test studies with the RF synchronized femtosecond laser beam demonstrated ~10 ps time resolution and 0.2 ps/hour stability. The RFT has potential applications in many fields of science and industry [2, 3]. We report the RFT based photoelectron spectrometer (Figure 1). RF synchronized photons (3) directed to the sample target (8). The emitted electrons are accelerated by a voltage V ~2.5 kV applied between the target (8) and an accelerating electrode (7). The accelerated electrons are deflected through 90 deg. by the permanent magnet (4) and pass through a collimator (5) before entering the electrostatic lens (9). This focuses the electrons on the position-sensitive detector (PSD) consisting of a dual chevron microchannel plate (MCP) (11) and delay-line anode (12). However, before reaching the PSD, the electrons pass through the RF deflection system (10), which performs circular sweeps of keV electrons by means of a 500 MHz radio frequency electromagnetic field. By converting the time of arrival of incident electrons to a hit position on a circle, this device achieves picosecond precise timing and can be used for time resolved photoemission studies [4]. Results of experimental studies from Tantalum, Gold and Graphen will be presented.

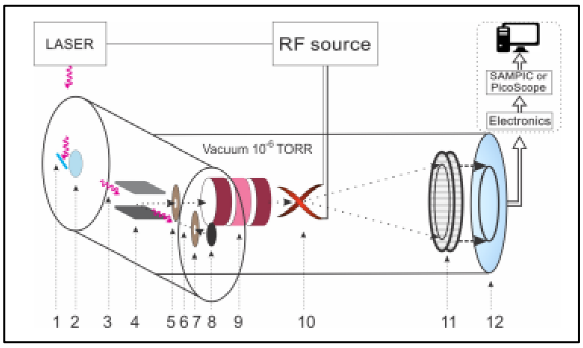

Figure 1: Schematic of the spectrometer RF. 1 – mirror; 2 – quartz window; 3 – photons; 4 – magnet; 5 – collimator; 6 – photoelectron; 7 – accelerating electrode; 8 – sample target; 9 – electrostatic lens; 10 – RF deflector; 11 – MCPs; 12 – delay line anode.

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Oral Report

SHS synthesis of micro- and nanocrystals exposed with facets: The formation mechanism and application notes

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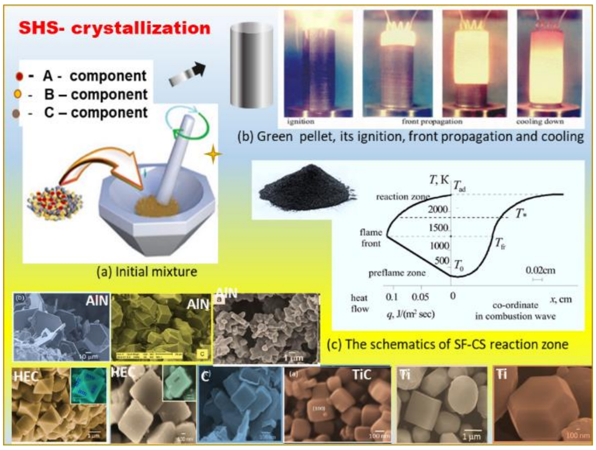
Nanocrystals with exposed distinct facets constitute a crucial category of materials due to their continuous, uniform, and highly ordered structure, which imparts them with unique properties. Crystal facet engineering can efficiently modify the kinetic parameters responsible for improved charge pair transfer, separation parameters, and reaction rates in an electrocatalytic or photocatalytic reaction. In brief, morphologies with different exposed facets undergo distinct atomic rearrangements and coordination, leading to variations in surface electronic structures (band gap, band edge potential, etc.), electrical conductivity, surface built-in electric field, reaction centres, and reactant adsorption sites. By simply altering the exposed plane, a drastic change in catalytic activity and selectivity of a catalyst can be achieved, thus sparking increased interest in morphology tuning and crystal facet engineering.

Today, SHS has emerged as a highly popular approach for the preparation of micro- and nanostructures of high-temperature materials. In a remarkably short period, this method has garnered significant attention and popularity due to its remarkable advantages, including its speed, low cost, and efficiency in terms of energy and time savings. The main challenge associated with SHS is the uncontrollability of phase and morphology; however, recent years have seen some progress. The current presentation aims to compile existing literature data on SHS-synthesized inorganic micro- and nanocrystals with exposed facets. The following scopes are planned to be included in the presentation:



(a) Experimental methodology for synthesizing faceted nanocrystals using the SHS method, including considerations of process thermodynamics (adiabatic temperature and equilibrium reaction phases), types of precursors used in the experiments, material classes (nitrides, carbides, metals, and non-metals), reaction environments (solid, liquid, and gas), types of dopants (seeds and fluxes), and synthesis regimes, including process temperatures and combustion velocities.

(b) The nucleation, growth, and faceting mechanisms observed during the combustion process. Theoretical insights into growth mechanisms using Density Functional Theory (DFT) calculations and Wulff construction methods.

(c) Performance testing of nanocrystals in energy storage devices and perspectives for further applications. Figure above demonstrates a schematic with different micro- and nanocrystals (e.g., AlN) synthesized by the SHS process.

Acknowledgement

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Oral Report

Fast Reaction Propagation in Nanothermites

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The speed of combustion propagation in highly porous nanothermites (NT) can reach more than 1000 m/s, while the released thermal energy is comparable to or exceeds the heat of explosion using conventional explosives [1].

The propagation of a combustion reaction in an NT mixture placed in a shell is a complex and multiscale process. In accordance with pyrometric measurements during the combustion of thermite mixtures based on nanosized Al, temperatures reaching 3100–3500 K are observed [2]. At such temperatures, a substantial quantity of gaseous products are formed, including a number of gaseous suboxides AlO, Al_2O, and Al_2O_2 [3]. When using the optical methods for recording of propagation of the glow front through the substance and assuming that only a small part of the NT mixture reacted behind it, it can be stated with high level of confidence that this rate is not the result of fast kinetics, but is a consequence of high-speed injection of hot intermediate products through the macropores. In this case, the average rate of this process, other things being equal, is mainly dependent on the steady-state pressure gradient and porosity (macroscopic and microscopic) of the initial NT mixture. To explain the mechanism of propagation of combustion reaction (PCR) in such system, a model is proposed. This model is based on assumption of strong influence of pressure gradient created during the combustion of HT (Darcy's law) on the PCR velocity. In this model, the injection of high-temperature jets that ignite the initial composition is considered as a process of the nonisothermal filtration of a hot substance through an immobile matrix of the initial substance with micrometer-sized pores.

Model experiments with inert barriers in tubes with NT (microspheres, air gap) revealed the characteristic features of the studied mechanism and made it possible to assess the permeability of the NT mixture and the pressure at the border of ignition and combustion zones. The calculation results are consistent with the experimental data, which, in turn, demonstrates the reliability of the proposed PCR mechanism. Moreover, the ability to reinitiate the reaction in NT through inert barriers (glass microspheres, viscose) of up to 30 mm in length was discovered experimentally.

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Keynote Report

Fundamentals and Applications of Solution Combustion Synthesis

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Solution combustion synthesis (SCS) is a well-established technique in materials science, renowned for its efficiency in producing a diverse array of advanced materials through exothermic chemical reactions in a liquid medium. This review explores the fundamental principles and wide-ranging applications of SCS, emphasizing its role in synthesizing nanostructured oxides, metals, intermetallics, and composite materials with exceptional structural and functional properties.

At its core, SCS involves selecting fuel and oxidizer precursors that react exothermically to sustain high-temperature reactions, leading to the rapid and uniform formation of desired materials. The process is governed by thermodynamic and kinetic parameters and controlled to achieve specific morphologies, phase compositions, and structures. Detailed investigations into precursor chemistry, reaction mechanisms, and thermal profiles provide a comprehensive understanding of the SCS process and its capabilities.

The applications of SCS are extensive and impactful. SCS-derived materials are employed as catalysts, battery electrodes, and fuel cell components, contributing to advancements in sustainable energy technologies. Environmental applications include using these materials for pollution control, water purification, carbon capture, and addressing critical environmental challenges. In the biomedical field, SCS produces biocompatible materials crucial for drug delivery systems, medical imaging, and tissue engineering.

In conclusion, solution combustion synthesis is a versatile and robust method for producing advanced materials with tailored properties. Its fundamental principles, rooted in thermodynamics and kinetics, and its diverse applications across multiple fields underscore the significance of SCS in advancing materials science and technology. This review will highlight recent developments and anticipate future innovations, demonstrating the ongoing contributions of SCS to various technological domains.

Keynote Report

Production of metal oxides by solution combustion method

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The production of metal oxides by solution combustion has currently been the subject of intensive research due to its potential advantages. Comparatively to other synthesis methods, the liquid phase combustion method allows reducing the synthesis temperature and is both practical and scalable [1-3]. In this concern, solution combustion synthesis of metal oxides such as Co_3O_4, Fe_3O_4, WO_3 and SnO_2 opens new perspectives for the controlled preparation of nanostructured materials with defined phase compositions and properties. These oxides are of considerable relevance in various fields including electronics, catalysis and magnetism [4, 5].

In this thesis the specified metal oxides obtained by solution combustion method are reviewed, their features, possibilities and prospects of their application in various fields of science and technology are investigated, especially for solving urgent environmental issues by water purification and application of highly sensitive gas sensors.

Nanoparticles of metal oxides were obtained by heating at 250-500 ^{0}C. SEM, TEM, and XRD analyses were performed to investigate the morphological properties of the obtained nanoparticles. According to SEM analysis Co_3O_4 nanoparticles have a uniform spherical shape, whereas Fe_3O_4 nanoparticles have a homogeneous polygonal shape. Both Co_3O_4 and Fe_3O_4 nanoparticles have some tendency to aggregation due to their distinctive magnetic properties. Agglomerates can be broken up by mechanical mixing with another compound for further application of these materials in a composite. The SEM and TEM figures show that the WO_3 nanoparticles are finely dispersed spherical in shape, while SnO_2 has irregular shape and heterogeneous structure. The average particle size of Co_3O_4 is 106.1 nm, Fe_3O_4 is 102.1 nm, WO_3 is 37.5 nm, and SnO_2 is 24.8 nm. The EDS analysis results show high purity of Co_3O_4 (70.2% (Co) and 23.7% (O)), Fe_3O_4 (66.3% (Fe) and 30.2% (O)), WO_3 (73.7% (W) and 17.1% (O)), and SnO_2 (73.7% (Sn), 17.1% (O)), with some chlorine content present in the reaction. The X-ray diffraction patterns obtained as a result of the XRD analysis of samples of cobalt, tin, magnetite and tungsten oxide nanoparticles correspond to the image in PDF format. For cobalt oxide, the average crystallite size was 12.59 nm (PDF: 01 - 078 - 1969), for magnetite - 20.4 nm (PDF: 82 - 1533), for tungsten oxide - 22.4 nm (PDF:04 - 006 - 7123), for tin oxide - 18.0 nm (PDF:29 - 1484).

The obtained results show that the solution combustion method allows manipulating the properties and morphological features of the product for their effective application in various fields. The synthesized metal oxide nanoparticles have a high potential for further use in gas sensors (SnO_2, WO_3), in water purification in a composite with porous carbon materials (Fe_3O_4), and electronic devices (Co_3O_4, Fe_3O_4, WO_3).

Acknowledgements

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Oral Report

Solution Combustion Synthesis of Ni-Containing Catalysts for Decomposition of Methane

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The catalytic decomposition of methane (CDM) is a novel COx-free method for the production of hydrogen. The second product of the reaction is carbon nanomaterial. The search for a catalyst for the CDM process is an important task since the catalyst determines the yield of products and the conversion of methane. There are many methods for the preparation of catalysts for methane decomposition. Solution combustion synthesis has several advantages. It is fast, uses solid feedstock, and is a single-step process.

This work is dedicated to the solution combustion synthesis (SCS) of Ni-containing catalysts (Ni/Al_2O_3). Various fuels will be used in the research, such as oxalic acid, hexamethylenetetramine, citric acid, etc. The mixture of Ni and Al nitrates with fuel was taken for synthesis. The problem of finding the optimal conditions for SCS will be addressed based on regression analysis, establishing relations between synthesis conditions and the yields of the formed products. Regression analysis has shown that the yields of carbon and hydrogen are most strongly influenced by temperature compared to other synthesis parameters.

There are three types of ignition approaches of mixture of nitrates and fuel, e.g. microwave radiation, heated coil, furnace heating. It was found that each of methods led to formation of certain particle size distribution and forms the different yield of carbon and hydrogen in CDM.

The work was carried out within a scope of State Task of Ministry of Science and Higher Education of Russian Federation (FSUN-2023-0008).

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Oral Report

Water gas shift activity of Pt/CeO₂ prepared by solution combustion synthesis: influence of the synthesis parameters

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Steam reforming of natural gas (NG), followed by a clean-up process is the most common industrial process to produce hydrogen (H_2). Water gas shift (WGS) is the first step of the CO clean-up process, aiming at increasing H_2 concentration and reducing carbon monoxide (CO) concentration. WGS is an exothermic equilibrium reaction ($\Delta H298 = –41.1$ kJ mol^{-1}), thermodynamically limited at high temperature and kinetically limited at low temperature. Thus, highly active catalysts are required.

In the present study we studied the operative parameters influencing the preparation of 1 wt% Pt/CeO_2 catalyst by combustion solution synthesis to optimize the WGS reaction in a fuel processor [1]. Several catalysts were synthesized, via one- and multi-shot solution combustion synthesis (SCS). The main results (Figure 1) show that the catalyst prepared by one-shot SCS without calcination is the most promising and lasting material, able to keep a reaction rate of ~ 2 mol CO gPt^{-1} h^{-1} after 125 h of time-on-stream.

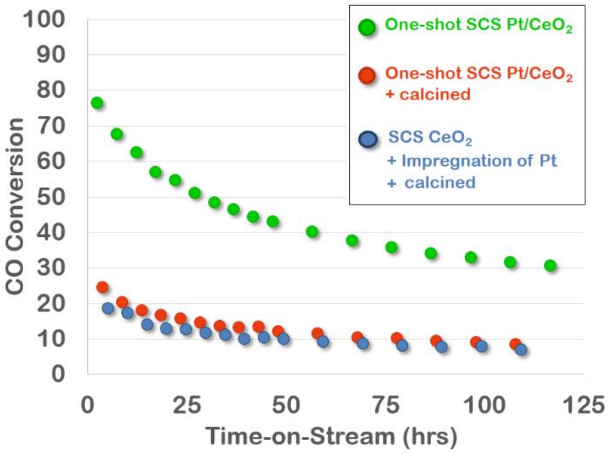

Figure 1. WGS activity of 1 wt.% Pt/CeO_2.

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Oral Report

IR-transparent MgO-RE2O3 (RE = Y,Gd,Lu,Sc) ceramic composites

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$MgO-RE_2O_3$ (where RE = Y, Gd, Lu, Sc) composites have favorable optical, mechanical, and thermal properties, making them promising candidates for various applications such as infrared windows, luminescent materials, and laser media. The fundamental requirement for ensuring high light transmission of the composite ceramics in the IR spectral region is the formation of a pore-free structure while maintaining submicron grain sizes. This work is devoted to compare the structure, mechanical and optical properties of $MgO-RE_2O_3$ ceramics fabricated by hot pressing (HP) of the glycine-nitrate combustion synthesized nanopowders.

The selected methods allow the sintering of ceramics with a density of more than 99% and a fine grain size of 150-300 nm. The Sc_2O_3 and Gd_2O_3-based ceramics have a shallow short-wave transmission limit; the highest transmittance is exhibited by samples sintered at temperatures of 1300 °C and 1350 °C, respectively. The $MgO-Lu_2O_3$ and $MgO-Y_2O_3$ composites have improved optical properties over the entire measurement range compared to the previous ones. The transmission in the range of 3-6 μm of the samples obtained at 1400 °C corresponds to single crystals of Lutetia and yttrium.

Characterization of defects in $MgO-RE_2O_3$ composites was performed by optical microscopy using infrared cameras in the NIR (0.7-1.0 μm) and SWIR (0.9-1.9 μm) wavelength ranges. The developed technique makes it possible to detect in the volume of visually opaque samples both small-sized closed pores and micro inclusions of non-isometric shape with a crystal-like cut.

The study was funded by the Russian Science Foundation (RSF) Project No. 22-73-10084.

Oral Report

The modification of the ZnO structure during the spray solution combustion process for later use in the fabrication of materials with thermoelectric properties

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Spray solution combustion synthesis (SSCS) could offer some advantages for the fabrication of thermoelectric materials. It facilitates the intermolecular mixing of the initial constituents. The subsequent calcination phase following the reaction permits the annealing of each individual particle [1]. The final product usually has a spherical shape with a structure ranging from solid to hollow, with either a porous or rigid external surface [2]. With the appropriate consolidation and sintering regime, such input characteristics may be used to control the porosity of the final thermoelectric material (TE) [3].

In this study, we examine the impact of the fuel used at SSCS on the formation, sintering, and thermoelectric properties of the ZnO-based material. For the refinement of a structure of bulk material, a slight amount of In was added, and spark plasma sintering was used.

The powders produced by SSCS exhibited a diverse morphology, ranging from hollow spheres with a smooth, rigid framework to a frame composed of smaller, spherical aggregates when glycine or urea were employed. Urotropine-assisted synthesis produced a product with a broad particle size and shape distribution. Citric acid caused numerous flakes to form. It was found that a triple excess of fuel had additional effects on the morphology. The utilization of glycine results in an enhancement of the porosity of the particles, whereas citric acid compromises the integrity of the spheres, and urea leads to the compaction of the particles.

The product after the glycine assisted synthesis was selected for further investigation of the sintering process and study of the thermoelectric properties. The objective was to regulate the introduction of pores into the bulk TE in order to diminish its thermal conductivity. It was shown that a change in the fuel concentration results in different porosity, improved sintering, leads to the texturing of the bulk material, and changes in thermoelectric properties.

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Oral Report

Influence of the combustion mode on the microstructure and properties of the Ni-based materials

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Ni-based nanocomposites have tremendous potential as materials for electrochemical energy storage, particularly in supercapacitors. Therefore, it is crucial to develop efficient, controllable, and reliable methods for synthesizing these materials [1]. In this study, we employed the solution combustion method (SCS) to synthesize Ni/NiO nanocomposites, utilizing nickel nitrate as an oxidant and glycine as a fuel. The advantage of SCS is that it provides precise and uniform formulation of the desired composition by thoroughly mixing the reagents at the molecular level in a liquid solution. In addition, SCS allows the manipulation of the phase composition and microstructure of the resulting products by adjusting the fuel-to-oxidizer ratio [2]. The most commonly used approach is the volume combustion synthesis (VCS) mode, which involves uniformly preheating the reactive media to the self-ignition temperature. However, the self-propagating high-temperature synthesis (SHS) mode offers greater control over the material properties. With SHS, the solution is locally preheated, and a reaction front gradually propagates along the media, forming the desired products. In turn, cellulose-assisted combustion synthesis (CACS) involves the impregnation of the reactive solution into a cellulose solid skeleton followed by the initiation of self-sustained reactions in such heterogeneous media, allowing one-step synthesis of the powders with high surface area. In this study, we investigated and compared the VCS, SHS, and CACS modes. We obtained time-temperature profiles for both combustion modes and conducted thermodynamic calculations. Furthermore, we investigated the phase, structure, microstructure, morphology, and magnetic features of the fabricated powders. Our investigations demonstrate the effective ways of using different combustion modes to fabricate nanomaterials with tailored morphologies, which is particularly advantageous for the production of electrodes for supercapacitors.

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Keynote Report

Preparation and 1000-1600 °C high-temperature anti-oxidation mechanism of MoSi2-based composite coating by doping ZrB2-SiC on Nb substrate

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$MoSi_2$ are synthesis via SHS route, and the industrial $MoSi_2$ heating elements are fabricated by powder metallurgy technology, and then spent $MoSi_2$, ZrB_2, and SiC were used as raw materials to prepare $MoSi_2$-ZrB_2-SiC composite coating on niobium substrate by spark plasma sintering (SPS), and the high-temperature oxidation resistance of the coating was studied at 1273K to 1873K. The results showed that the surface of $MoSi_2$-ZrB_2-SiC composite coating was dense without any defects after sintering. The coating surface mainly consisted of $MoSi_2$, ZrB_2 and SiC, phases with uniform distribution. After high-temperature sintering, Nb_5Si_3 diffusion layer that strengthens the layer bonding formed between the coating and substrate, indicating good metallurgical bonding generated during SPS process. During oxidation process, the oxidation of ZrB_2 particles led to the continuous formation of ZrO_2 and $ZrSiO_4$ phases, and the SiO_2 oxide scale thickness gradually increased, indicating that the high temperature promoted the oxidation of $MoSi_2$, ZrB_2, and SiC to form protective Zr-Si-O composite oxide scale. In addition, due to the diffusion of Si with Nb that promoted by higher temperature, the Nb_5Si_3 diffusion layer thickness gradually increased with increasing temperature. After oxidation at 1873K for 100 h, the Zr-Si-O composite oxide scale of the composite coating still maintained a complete and dense morphology and exhibited good bonding to the coating without any gaps or debonding, thus demonstrating good high-temperature oxidation resistance.

Key words: SHS; MoSi2; anti-oxidation coating; recourse recycling

Oral Report

New Generation of SHS-Materials for High Power Impulse Magnetron Sputtering and Spark Plasma Sintering of Oxidation Resistant Coatings

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The development of protective coatings with high wear and oxidation resistance is one of the priorities of modern industry. Promising coating materials from the point of view of thermal stability and heat resistance are silicides and borides of transition metals [1, 2], as wear-resistant compounds of the MAX-phase type are of great interest [3].

Present report is devoted to the production of protective hard oxidation-resistant coatings by High Power Impulse Magnetron Sputtering (HIPIMS) and Spark Plasma Sintering (SPS) technologies using ceramic cathodes and powders, respectively. Compositions of $HfSi_2$-HfB_2-$MoSi_2$, $ZrSi_2$-ZrB_2-$MoSi_2$, $MoSi_2$-MoB-HfB_2, $MoSi_2$-MoB-ZrB_2, Ti_2AlC, and Cr_2AlC were obtained by self-propagating high-temperature synthesis (SHS) and hot pressing (HP). Complex studies of coatings have been carried out using transmission and scanning electron microscopy, X-ray photoelectron spectroscopy, X-ray diffraction, energy dispersive analysis, glow discharge optical emission spectroscopy, micro- and nanoindentation, tribological and scratch testing. To estimate the oxidation resistance, high-temperature annealing in air was performed.

The results showed that the coatings, depending on the method of preparation and composition, have hardness in range 15 - 35 GPa. Due to the high mechanical properties, coatings greatly increased the wear resistance of the substrate material. The best oxidation resistance in air up to 1500°C demonstrates $MoSi_2$-MoB-HfB_2 and $HfSi_2$-HfB_2-$MoSi_2$ coatings. The high protective properties were due to the formation of Si:B:O, HfO_2, and $HfSiO_4$ surface films.

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Oral Report

Enhanced oxidation resistance of ZrB_2-$MoSi_2$ coating through $MoSi_2$-$TaSi_2$ double-silicide alloying modifying

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To inhibit the destructive dynamic diffraction induced by high-temperature oxidation-induced loosening of ZrB_2 based coatings, self-propagating high-temperature synthesis was used to prepare the ZrB_2-$MoSi_2$-$TaSi_2$ non-equilibrium alloyed boride composite powders modified by dual transition metal silicides, and the effect of different volume ratios of $MoSi_2$-$TaSi_2$ bi-silicide alloying on the oxygen resistance mechanism of the ZrB_2-$MoSi_2$-$TaSi_2$ composite coatings was investigated. The results indicated that the oxidative self-separation effect of $TaSi_2$ and $MoSi_2$ accelerated the dispersion of dual transition metal Zr/Ta oxide nanocrystalline particles within the glass layer, forming a Zr-B-Ta-Si-O complex-phase glass film, which resulted in an optimal oxygen resistance performance for the $50ZrB_2$-($MoSi_2$-$10TaSi_2$) coating (ZMT10). Compared to the ZrB_2-$MoSi_2$, the ZMT10 coating showed reductions of 28.87% in weight gain and 48.21% in oxygen penetration, increasing protective efficiency to 99.71 %. Excessive $TaSi_2$ led to Ta^{5+} dissolution, oxide aggregation, and a dendritic structure, facilitating oxygen diffusion and oxidation, raising the oxygen permeability in $50ZrB_2$-($MoSi_2$-$30TaSi_2$) coating to 0.73 %, while lowering protective efficiency to 99.18 %.

Oral Report

In situ phase formation at electric spark treatment of metallic alloys

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Electric spark treatment (EST) is widely used for surface hardening, as well as repair and restoration of worn metal parts. The advantages of such treatment include high energy efficiency, rapid local heating and cooling, which reduces the heat affected zone and ensures the production of coatings with fine-grained, nanocrystalline and even amorphous structures [1]. Two approaches to the selection of consumable electrodes are known in the technology. Traditionally, an increase in the wear resistance of coatings formed during EST is achieved by transferring the hard phases from electrode to workpiece. An alternative approach is *in-situ* synthesis of phases in the coating that were not present in either the electrode or the substrate. For the first time, the idea of initiating an exothermic chemical reaction for synthesis of nickel aluminides using the energy of an electric discharge between an electrode (anode) and a substrate (cathode) was implemented late 1990s [2]. Unlike classical SHS a phase formation during EST caused by spark discharge is interrupted, but with multiple overlapping spots of local synthesis, it results in continuous coatings containing a dense intermetallic layer with a thickness of more than 10 μm [3].

The aim of the study is to examine the conditions for the formation of protective coatings at EST of metallic alloys based on Ti, Ni and Nb using near-eutectic Al-Ca-Me electrodes.

The formation of coatings at EST in an inert atmosphere corresponds to the mechanism of gas-free combustion, which includes three stages [5]: reagents transport (i), primary (ii) and secondary (iii) structure formation. Studying EST of Nb substrate with Al-Ca-Si-Y electrode, using SEM, EDS, and TEM and taking into account the results [4], the authors suggest that at stage (i) the eutectic Al-Ca-Me melt spreads over the surface of the Nb substrate, (ii) Nb diffuse in the melt, (iii) when quenching the melt, an intermetallic compound Al_3Nb crystallizes. So, drops of melt formed at EST react with substrate and then solidify into intermetallic phases, giving wear and corrosion resistant coatings.

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Oral Report

Influence of Composition on the Structure and Properties of HEA-Si-B-C-N Coatings Deposited by DCMS and HIPIMS Methods Using SHS-targets

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Currently, significant attention is being given to the development of oxidation-resistant coatings for protecting critical products exploited at extreme conditions High entropy alloy (HEA) coatings are promising materials for high temperature applications due to their good hardness and thermal stability, wear- and oxidation resistance [1,2]. The purpose of this work is to study the influence of the chemical composition of coatings in the Mo-Ta-Nb-Zr-Hf-Si-B-C-N system on their structure and properties.

The coatings were deposited by direct current magnetron sputtering (DCMS) and high-power impulse magnetron sputtering (HIPIMS). (Mo,Ta,Nb,Zr,Hf)SiB target was manufactured using self-propagating high-temperature synthesis (SHS) and hot pressing technologies. To study the effect of nitrogen addition, sputtering was carried out in an Ar + N_2 environment with an increase in N_2 flow rate from 0 to 37.5 sccm. In order to produce coatings with an increased silicon concentration, co-sputtering of (Mo,Ta,Nb,Zr,Hf)SiB and SiBC SHS targets was carried out. The coatings were studied by SEM, EDS, TEM, XRD, nanoindentation, scratch- and the “pin-on-disk” testing. To determine the oxidation resistance of the coatings, annealing in air was carried out.

The results showed that the all coatings were characterized by an amorphous structure with a uniform distribution of elements throughout the thickness. For MoTaNbZrHfSiB coatings, it was found that the transition from DCMS to HIPIMS mode contributed to a 2.5-fold decrease in the thickness of the oxide layer at a temperature of 1300 °C. The introduction of nitrogen in the optimal concentration into the coatings led to a reduction in the friction coefficient by 1.5 times and the wear rate by an order of magnitude, while the oxidation resistance decreased as a result of intense evaporation of nitrogen oxide. The coatings with a high silicon content had record oxidation resistance up to 1500-1600 ºC, which is associated with the formation of a protective layer with a high volume fraction of the a-SiBO phase.

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SHS 2024

Posters

Poster Report

Nacre-like (V,Zr,Ta,Nb,Mo)$_2$AlC/Al$_2$O$_3$ high-entropy ceramic composite by self-propagating high-temperature synthesis

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The MAX phases are ceramics with a characteristic hexagonal layered structure characterized by good thermal shock resistance, corrosion resistance, high processability, biocompatibility and thermoelectrical conductivity [1]; however, there is an urgent need to improve their mechanical properties and resistance to high temperature oxidation. High-entropy MAX phases constitute a new branch of MAX materials with targeted properties. Current studies mainly consider the preparation of dense bodies by sintering. To the best of our knowledge, there are no studies related to the multi-element MAX phase powder preparation for further exfoliation process. The synthesis and properties of high-entropy MAX phases are still in their nascent stage of exploration. Main focus is currently given onto self-propagating high-temperature synthesis of alumina reinforced high entropy MAX phase composite ((V,Zr,Ta,Nb,Mo)$_2$AlC/Al$_2$O$_3$) by using mixture of elements and oxides of corresponding constituents.

Before the combustion experiments, a thermodynamic analysis was performed to assess the thermodynamic feasibility of self-sustaining interaction in the initial multicomponent system aimed at obtaining the 211 structure of a high-entropy ceramic composite. Relatively moderate combustion conditions (1400-1700ºC) were chosen for the specific brick-mortar (nacre-like) structure preservation.

The microstructure and phase composition of the samples synthesized from various compositions and at different pressures have been characterized by using SEM, XRD, EDS analyses methods. The optimum conditions for the final (V,Zr,Ta,Nb,Mo)$_2$AlC/Al$_2$O$_3$ composite preparation were revealed. It is considered as an ideal biomaterial for implant applications, especially for joint replacements. 2D MXene composites derived from the resulting high-entropy MAX phase composite are currently also of great interest owing to their electrochemical and electromagnetic shielding performance.

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Poster Report

Design and combustion synthesis of NiCoCrMnAlB & NiCoCrMnAlC systems

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Compounds with a layered structure have a potential to act as nanolaminated materials with electrochemical, photocatalytic and electrocatalytic properties [1], [2]. By comparing the formation energies of the MAB and MAX phases with those of their available competing binary M-B and M-Al, M-C and ternary M-Al-B, M-Al-C phases, it was found out that some of the Ni-, Co-, V-, Cr-, Mo-, W-, Mn-, and Fe-based MAB and MAX phases could be favorably synthesized under appropriate experimental conditions [3].

The aim of this work is to study the design and synthesis of NiCoCrMnAlB, and NiCoCrMnAlC systems. A novel 321-type $Ni_{1.2}Co_{1.2}Cr_{0.3}Mn_{0.3}AlB_2$ and $Ni_{1.2}Co_{1.2}Cr_{0.3}Mn_{0.3}AlC_2$ MAX phases have been designed and synthesized by the self-propagating high temperature synthesis (SHS) and solution combustion synthesis methods. Preliminary thermodynamic calculations have shown that using a mixture of metals and boron or carbon powders, the adiabatic temperature exceeds 1800 K. Using metal oxides and nitrates as precursors for MAX/MAB synthesis, the combustion temperature we controlled in a desired range. The phase composition of the synthesized MAX/MAB phases was studied by X-ray diffraction analysis and Rietveld refinement method. The microstructure of MAX/MAB phases was investigated by the SEM/EDS analysis. The synthesized and characterized powders are adapted for future catalytic applications.

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Poster Report

Preparation of ceramic materials based on Ti-B-C-N system by SHS-compaction and influence of mechanoactivation on synthesis parameters and physical-mechanical and operational properties

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The progress of science and technology is closely related to the use of new ceramic and composite materials. However, the widespread use of these materials is limited due to the lack of effective technologies for their production. Due to their physical and mechanical properties, ceramic compositions have been found to be the most effective for protection against damaging elements with increased penetration. This led to a sharp increase in demand for much more expensive but at the same time lighter ceramics.

The purpose of this work is to develop self-propagating high-temperature synthesis - compaction, ceramic consolidated materials and innovative technologies for their production.

The work is devoted to the actual problem of obtaining multifunctional materials based on the Ti-B-C-N system Ti-BN, Ti-BN-C, Ti-B_4C compositions, which will have qualitatively improved properties, such as ductility, flexural strength, wear resistance, and will also be able to work under radiation conditions and They will be able to protect objects from both radiation and dynamic loads with high intensity and energy.

In addition, scarce, expensive, limited materials and complex technologies will not be used to obtain ceramic material. The resulting product will be competitive, cheap, easy to manufacture both technologically and economically. They will not have a high cost, will not require a large expenditure of material, energy and hardware resources, and will be characterized by high viability.

The materials developed by the AR 22-955 project can be successfully used as materials for protecting various objects, including space objects, from damaging elements and radiation with increased penetration.

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Poster Report

Specific of Plastic Deformation Localisation in Cylindrical Bronze Samples under Impact Loading

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Shock-wave loading is a common method of high-energy impact on metals and alloys. The effect of shock waves on a material creates a significant increase in its strength. When explosives are detonated, the shock waves propagate towards and at angles to each other creating high pressures. The structure of metals and alloys under shock-wave loading inevitably undergoes changes, and this is influenced by several factors at once: high pressure, high-speed deformation, tensile stresses during unloading and temperature rise. However, shock-wave loading is often accompanied by material fracture.

This paper examines the formation of spall damage and plastic deformation localisation in cylindrical bronze samples under shock wave loading via explosives. Two loading schemes were used: plate-impact throwing and compression by a sliding detonation wave. The plate-impact scheme is widely used to determine the dynamic strength of materials. Sliding detonation wave compression is used to generate high pressures over the entire contact area between the explosive and the sample. The explosive used was ammonite with detonation velocity D = 3600-4800 m/s. The samples had a cylindrical shape with height h = 40 mm and diameter d = 25 mm. The pressure exerted on the sample at this detonation velocity is 15 GPa, which significantly exceeds the fracture toughness of bronze, which is 5 GPa, resulting in a fracture of the material.

The specificity of plastic deformation localisation in cylindrical samples is the presence of free surfaces that contribute to the dissipation of converging shock waves. Fracture evolution occurs according to the plastic deformation model. As a result of the interaction of the forward and backward unloading waves, fracture occurs when the stress exceeds the dynamic strength of the material at the point of their interaction. When the plate impacts the samples, layer-by-layer delamination of the material is observed at the impact surface, as well as the formation of microcracks to a depth of up to 10mm. In the rest of the material, the interaction of the shock waves leads to the formation of micropores and small microfractures. During compression by a sliding detonation wave, microcracks are formed throughout the depth of the material from scattered microfractures, which in turn develop from micropores. The micropores thus give rise to microcrack propagation by creating additional free surfaces in the material. The microcracks observed all propagate along the axis of the sample and are predominantly located in the centre between the free surfaces, forming a spall fracture zone.

Poster Report

SHS of advanced ceramics based on MoAlB

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In the last decade, there has been increased research interest in materials with an atomic layered structure. *One example of this* class of materials are MAB phases, *which consists of boride blocks separated by one or more layers of aluminium*. The most promising of the family of MAB phases is MoAlB, which is characterized by substantial hardness, high thermal and electrical conductivity [1], oxidation resistance [2], and high temperature ablation resistance [3].

This study focuses on the production of MAB phase MoAlB based materials by various SHS techniques: force SHS pressing technology, layer-by-layer combustion regime and termal explosion mode.

The compact ceramics obtained by force SHS pressing technology was characterized by porosity of 9.6 % with the content of MAB phase reaching 97 %. The main structural component of these ceramics was lamellar grains of MoAlB up to 15 μm in size with discrete inclusions of Mo_3Al_8 intermetallic, MoB boride and Al_2O_3.

Synthesis products produced by layer-by-layer combustion regime and termal explosion mode were porous agglomerates with the MAB phase content of 97 % and 92 %, respectively, which were subjected to mechanical grinding to a fraction with an average particle size of 10-15 μm.

The studies of the phase formation process in combustion wave were carried out *in situ*. It was found that in layer-by-layer combustion regime the MoAlB phase is formed in single stage [2], whereas in the case of thermal explosion mode, it forms in two stages via Mo_3Al_8 intermetallic and MoB boride [4].

Bulk ceramics produced by hot pressing of the SHS powder were characterized by residual porosity of 1.5 %, hardness of 11.3 GPa, bending strength of 397 MPa, and crack resistance of 4.7 MPa·m$^{1/2}$ [2]
This work was supported by the Russian Science Foundation (project no. 21–79-10103).

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Poster Report

Structure and composition of materials synthesized from mechanoactivated chasms of Ti-BN, Ti-BN-C, Ti-B_4C composition by SHS compaction

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Using the original technological scheme of SHS-compaction by the group of the Institute of Metallurgy and Materials Science with the support of the AR-22-955 grant project, synthetic composite ceramic materials "constructed" from hard ceramic refractory compounds were obtained on the basis of the Ti-B-C-N system Ti-BN composition SCCM-1, Ti- BN-C composition based on SCCM-2 and Ti-B4C composition based on SCCM-3.

These parameters are time and pressure characteristics and are determined experimentally. These parameters are time and pressure characteristics and are determined experimentally. As a result of the correct selection of these characteristics and the influence of mechanoactivation in the Ti-B-N-C system, compacted with a porosity of 2% and 3.5% and 3% respectively, with a smoothness of 92.3 HRA and 93.2 HRA and 93.5 HRA with a density of -4.5 respectively g/cm3 and 4.7g/cm3 and 4.73g/cm3 materials. The study of the phase structure and microstructure of the material allows to specifically determine the areas of possible use of the material and to make conclusions about its operational properties under extreme conditions.

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Poster Report

Structure and Properties of SHS- Solid Solutions (Hf1-xTax)B_2

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This paper presents an in-depth study on the combustion synthesis in Hf-Ta-B, $(Hf_{1-x}Ta_x)B_2$ solid-solution formation and characterization of ceramics aiming to explore their potential applications.

Boride ceramics combine high values of hardness, corrosion resistance and thermal conductivity, which makes them promising for application in composite materials. Solid solutions $(Hf_{1-x}Ta_x)B_2$ were synthesized from elements and powders with particles size d_{50} <10μm were produced. [1]. Dense ceramis were hot pressed from single-phase solid solutions with various stoichiometry $(Hf_{1-x}Ta_xB)_2$ and their structure and properties were studied [2-4]. The dependences of the unit cell parameter on the “x” shown that they obey Vegard's law. Analysis of ceramics structure by SEM, TEM, EDS confirmed the existence of a region of single-phases solutions between HfB_2 and TaB_2 phases. It has been established that an increase in the tantalum content in the solid solution $(Hf_{0.8}Ta_{0.2})B_2$ and $(Hf_{0.6}Ta_{0.4})B_2$ leads to a decrease in the size of subgrains to 0.2-1 μm, which correlates with the size of coherent scattering regions.

Ceramics $(Hf_{0.8}Ta_{0.2})B_2$ and $(Hf_{0.6}Ta_{0.4})B_2$ have a record value of hardness (63-70 GPa) and elastic modulus (550-587 GPa). The temperature dependences of the electrical resistance, heat capacity, thermal diffusivity, and thermal conductivity were determined in over a wide range of solid solutions stoichiometry x = 0 – 0,9.

This work was financially supported by the Ministry of Science and Higher Education of the Russian Federation (project 0718-2020-0034).

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Poster Report

Use of Surface-localized Self-propogating High-temperature Synthesis (SHS) to Produce a Porous Permeable Composite Material Based on Titanium Sponge Powder

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It is known that porous permeable materials (PPMs) are used as: 1) catalyst carriers, including those used in steam-gas reforming of hydrocarbons; 2) electrodes of fuel electrochemical elements of hydrogen energy systems; 3) filter elements of complex purification and water treatment systems.

Such PPMs should be resistant to corrosion, in which the oxidizing agent may be oxygen, or alkaline or acidic solutions containing oxygen of weak and medium concentration. At the same time, the PPMs-catalyst carriers should be resistant to high-temperature corrosion, and the PPM electrodes or filters should be resistant to alkaline and acid corrosion. One of possible solutions to this problem can be a powder composition based on titanium sponge powder "Ti-Al-Nb+[Mo, Ta, V, W and so on]", from which porous samples are pressed using the technology developed at O.V.Roman Powder Metallurgy Institute (Minsk, BY), then they are sintered in vacuum or one-stage electric pulse sintering (EPS) is performed. Then, the porous samples sintered by one or the other method are subjected to a localized process of self-propagating high-temperature synthesis (O-N-SHS) on the surface of the PPMs in a controlled oxygen-nitrogen atmosphere. This process is the final operation in the developed technology. A special feature of conducting localized O-N-SHS on the surface of PPMs is the creation of contact compression with the help of electrodes, transmission from an adjustable source of an alternating current of low or medium frequency (up to 1 kHz) discharge, at which electrothermal heating up to the temperatures of SHS initialization is possible, forming a dense ceramic oxide-nitride shell, both on the outer and inner pore surface of PPMs. The temperature is controlled by a pyrometric sensor. At the same time, high-temperature reactions of chemical synthesis of intermetallides containing an orthorhombic phase occur in the formed PPM matrix.

The technology developed at O.V.Roman Powder Metallurgy Institute allow to produce PPMs in the form of a layered composite, the ceramic shell of which is represented by oxide-nitride ceramics, and the core – by a titanium alloy matrix disperse–strengthened with intermetallides, or by an intermetallic matrix containing an orthorhombic phase. The thickness of the ceramic shell of the composite and the ratio of the oxide-nitride phase contained in it are affected by the regulated concentration of oxygen and nitrogen in the composition of oxidizing atmosphere, and the content of intermetallic and orthorhombic phases in the metal matrix of the composite is affected by the quantitative and dispersed composition of base additives (powder: Al and Nb), as well as additional additives (powder: Mo, Ta, V, W and so on) introduced into the base.

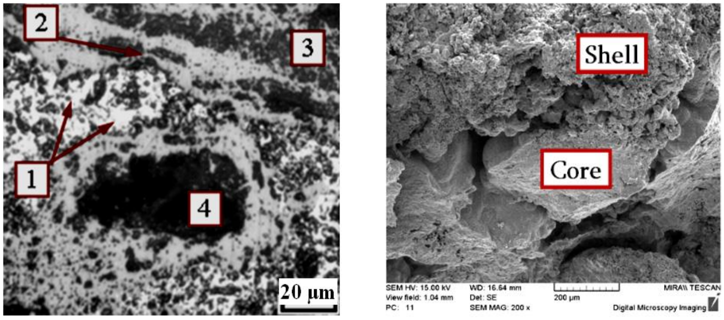

Fig: PPM structure after localized on the surface SHS: optical microscope image; (b) SEM image
1– matrix; 2 –interface between matrix and ceramics; 3 – ceramic shell; 4 – pore.

The developed technology for the production of PPMs from compositions based on titanium sponge powder is implemented at O. V. Roman Powder Metallurgy Institute, when producing pilot batches of samples of catalyst carriers and fuel cell electrodes.

Poster Report

Self-Propagating High-Temperature Synthesis for producing 2D Bi, Sb-chalcogenide based materials and their solid solution for functional applications

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Layered 2D materials constitute a crucial group of substances with potential applications spanning various technological sectors. Nevertheless, there exist several obstacles associated with their development, manufacturing, and practical implementation. The intricate chemical composition and structural complexity of certain layered 2D materials render them challenging to produce on an industrial scale. The properties of layered 2D materials exhibit a wide spectrum, contingent upon their chemical composition, structure, and layer count. This variability can pose challenges in comprehending and effectively utilizing these materials for specific applications [1].

For an extended period, Bi_2Te_3, Sb_2Te_3 and solid solutions based on them ($Bi_2Te_{3-x}Se_x$, $Bi_xSb_{1-x}Te_3$, etc.) were primarily recognized for their thermoelectric characteristics and subsequent utilization in thermoelectric cooling systems upon their discovery [2]. These material categories share a common structure, featuring layers consisting of covalently bonded X (Se and Te)-M (Bi and Sb)-X-M-X sheets. These quintuple layers, with a thickness of approximately 1 nm, are interconnected by weak van der Waals forces. However, recent investigations have revealed that Bi_2Te_3-based materials also exhibit topological insulator properties, prompting their exploration and application across various electronic domains [3]. The examination of Bi_2Te_3-based photosensors has been conducted through diverse studies, utilizing epitaxial layers or individual nanostructures such as nanowires or nanoplates [4]. Consequently, substantial interest has arisen in the nanoarchitectures of Bi,Sb chalcogenide-based materials.

In this study, certain aspects of a conventional and straightforward method for synthesizing Bi,Sb-based chalcogenides using Self-Propagating High-Temperature Synthesis are presented. Additionally, potential applications of these materials are discussed.

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Poster Report

Effect of High Heating Rate on the Formation of TaC

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Tantalum carbide is characterized by excellent physical properties in melting point, hardness, elastic modulus, thermal conductivity, thermal shock resistance, and chemical stability, which makes it a desirable material for aircraft and rockets in aerospace industries [1]. The tantalum–carbon reactive system possesses a high energy of reaction with an adiabatic combustion temperature of 2743 K, which is significantly below the melting points of the reactants, as well as any intermediate phases and final products.

Given that the heating rate can have a significant effect on the mechanism of chemical reaction, in this work the mechanism and kinetics of interaction in Ta-C system was investigated by High-Speed Temperature Scanner (HSTS-3) technique (V_h=100–4800 K/min).

According to the results obtained, depending on the heating rate, two different ranges of heating rate can be clearly distinguished, which differ from each other by the absence (I) and presence (II) of the exothermic peak on the thermogram. In the first range, at relatively low heating rates (100-150 K/min), carbidization process occurs without noticeable self-heating, as a result of which the sample cooled immediately after the temperature of 1423 K exhibited a mixture of Ta, Ta_2C, and TaC, with further temperature escalation predominantly leading to the formation of TaC. In the second range at high heating rates (200-4800 K/min), the process was accompanied by a strong self-heating ($\Delta T>250K$), which is clearly seen as an exothermic peak on the thermogram and as a result of increasing the heating rate, it shifts to a higher temperature region**.** The tantalum carbidization mechanism observed across all heating rates followed a consistent sequence of Ta→Ta_2C→TaC. For instance, at heating rate of 1200 K/min and almost at 1523K the sample predominantly consisted of TaC. The investigated range of the heating rate (100-4800 K/min) is significantly lower than the heating rate of the material during the Ta+C reaction in the combustion mode, which can reach tens of thousands of degrees per minute. The latter is accompanied by a fairly high combustion temperature of >2273K and leads to the formation of coarse-grained tantalum carbide. Note that due to the comparatively low heating rates and especially low temperature compared to SHS, there was notable impact on the particle size of the synthesized TaC and as a result of which the TaC grains were obtained finer (for example V_h=100 K/min, T=1973K, particle size <100 nm). In this work, using the shift of the exothermic peak depending on the heating rate, the effective activation energy of the Ta+C reaction, calculated by the Kissinger method, was 232 ± 28 kJ/mol.

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Poster Report

The Spark Plasma Sintering of Magnetic (CoZnFeMnNi)O High Entropy Oxide Synthesized by SCS Method

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High-entropy oxides (HEO) are usually single-phase oxide systems containing 4 or more cations, the configuration entropy of which is higher than 1.5R (R is the universal gas constant) [1]. The random distribution of cations increases the configurational entropy and contributes to phase stability. The properties of these oxides depend on the choice of component metals, which can be changed depending on the required applications. Currently, HEOs with the following types of structures are known: rock salt, fluorite, bixbyite, pyrochlore, magneto plumbite, perovskite, spinel. Recently, soft magnetic materials have become widely used due to the constant magnetic properties under the influence of a strong external magnetic field. Compared with the commonly used air gap, the high entropy material nanocomposites can effectively increase the effective permeability of the material cores. It is assumed that HEO of the composition (CoZnFeMnNi)O will be characterized by clearly expressed magnetic properties, since the elements Fe, Ni and Co included in its composition are ferromagnetics. In this work, the preparation pathway of (CoZnFeMnNi)O HEO with a rock-salt structure (R-HEO) is demonstrated by a facile solution combustion synthesis (SCS) method. Metal nitrates in equiatomic ratio and glycine were used as precursors. It was shown that the parameters of the combustion process, and especially the phase composition of the combustion products, significantly depend on the amount of glycine and acidity of the solution. It has been shown, that to obtain a single-phase (CoZnFeMnNi)O R-HEO, the optimum amount of glycine should be 6 mol in a highly acidic (pH=2) solution. When the SCS obtained R-HEO is heated in air, a rock-salt to spinel phase transition begins at about 600°C and the formation of $(FeCoNiMnZn)_3O_4$ HEO oxide is observed, but after a temperature of 900°C a reverse spinel to rock-salt transition occurs with the formation of R-HEO. A similar transition occurs, when the nanopowders synthesized by the SCS method were compacted using the spark plasma sintering technique. In particular, at 1000°C, a partial rock-salt to spinel phase transition occurs. But at 1100°C a high entropy alloy with an FCC structure was formed and reverse phase transformation spinel to rock salt occur. Due to this transition, a significant change in the magnetic properties of the specimen took place. The bulk sample after SPS independent of the sintering temperature (1000 & 1100°C) is characterized by higher magnetization (Ms=17.9 & 12.6 emu/g), but lower coercivtiy (37.5 & 35.2 Oe) and lower energy product (12.9 & 5.85 Oe emu/g), than R-HEO powder obtained as a result of combustion synthesis (Ms=3.2 emu/g, Hc=215 Oe, E=18.7 Oe emu/g).

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Poster Report

Composite rods by method combining SHS and hot gas extrusion of reactive powder mixture Ni-Al and Mg-B

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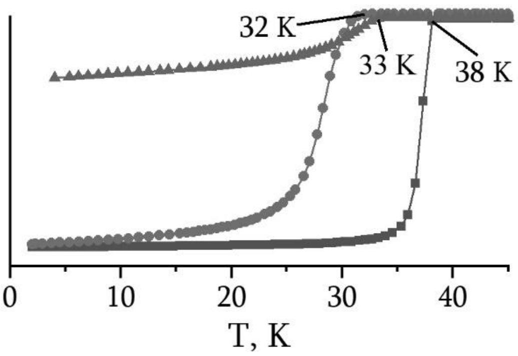
To obtain rods and wires from metal-intermetallics and metal-ceramics, rolling of elemental or synthesized powders in a pipe with next long-term heat treatment is used. In this paper, the production of intermetallics of the Ni-Al system and an MgB_2-based superconductor in a steel shell in one technological stage is investigated. For this purpose, the SHS was combined with hot gas extrusion, which uses high inert gas pressure and simultaneous local heating in the deformation region. [1]. The aim of the work is to establish the relationship between the parameters of hot gas extrusion (gas pressure and extrusion start temperature) and the phase composition and properties of the rods.

Elemental powders were used as sources for Ni-Al and Mg-B powder mixtures. A mild steel rod was used as a shell. As a result of HGE with a degree of deformation of 75 %, steel rods with a core of Ni-Al intermetallics or MgB_2 with impurities were obtained. Ni-Al-based rods were obtained at pressures of 200, 300 and 400 MPa and an extrusion start temperature of 750, 650 and 600 ° C, respectively. Mg-B-based rods were obtained at 200, 250 and 300 MPa and temperatures of 670, 650 and 600 °C.

In Ni-Al-based rods, the highest content of the target phase NiAl was obtained at a pressure of 200 MPa and a temperature of 750 °C (70 wt.%). The yield strength is higher in rods with the lowest NiAl content due to the presence of ductile Ni and Al. By quenching the reaction products, it was found that the reaction in the core proceeds before the deformation begins. Areas of Al accumulation were found in the rods obtained at 300 and 400 MPa.



The highest MgB_2 content was found at 200 MPa and 670 °C (65 wt.%). The diameter of some rods has increased due to the accumulation of impurity gases between the core and the shell. The superconducting transition temperature (T_c) of the rods is shown in the figure. Rods obtained at 200 MPa have the highest T_c (T_c for monocrystals is 39 K). An accumulation of Mg was found in the rods obtained at 300 MPa.

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Poster Report

Modification by Aluminum Nitride Nanosized Particles of Materials Based on the Ti–B–Fe System Produced by SHS Extrusion

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In the presented work, using the SHS-extrusion method, composite electrode materials based on the (Ti-B-Fe)xAlN system with the addition of nanosized AlN particles at x = 0, 5, 10, 15 wt. % were obtained. These materials can be used as electrodes for applying wear-resistant coatings. The influence of additives on the combustion characteristics of the studied system was investigated. It was shown that adding 5 - 15 wt. % AlN leads to a reduction in combustion characteristics, and the measurement results are presented in the table below.

Composition (Ti-B-Fe)xAlN, wt %	x = 0	x=5	x=10	x=15
Combustion velocity, mm/s	16	9	7.5	2.5
Temperature, °C	1790 - 1900	1650 - 1750	1600 - 1700	1470 - 1520

SEM and XRD of the extruded materials revealed that the primary strengthening phase for unmodified SHS electrode materials consists of TiB2 grains (dark gray areas) with sizes of 1.5-2 µm, located in the FeTi intermetallic binder. The addition of nanosized AlN particles to the initial mixture results in the formation of a new strengthening TiN phase (rounded gray grains) with a size of about 2 µm due to SHS. As the content of AlN nanoparticles increases, a significant decrease in the grain size of TiB2 from 0.9-2 to 0.2-1.8 µm is observed. This reduction in TiB2 grain size is attributed to the fact that the introduction of a nanomodifier in the form of dispersed AlN particles lowers the combustion temperature, as some of the heat generated during the chemical reaction between titanium and boron is used for AlN decomposition and further low exothermic or endothermic reactions with initial components and synthesis products. These factors restrict the growth of TiB2 grains. The reduction in TiB2 grain size, combined with the formation of TiN, leads to an increase in the hardness of extruded materials (HV10/30, GPa) from 10.7 to 14.7 with the addition of 5 wt. % AlN. Increasing the amount of AlN to 10 and 15 wt. % results in hardness increasing to 12.5 GPa[1,2].

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Poster Report

Enhanced Methane Reduction of Nickel Oxide through Hydrogen Pretreatment: Kinetic and Morphological Insights

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Reduction of nickel oxide (NiO) with methane (CH_4) or hydrogen (H_2) is an essential process in extractive metallurgy and catalysis. In Ni metallurgy CH_4 reduction of Ni is preferred over carbon due to lower operating temperatures and reduced pollutant emissions.

In this work the results of investigation of the initial stages of hydrogen-enhanced methane reduction of nickel oxide, based on *in situ* measurements of reaction rate, as well as on periodic measurements of sample weight loss during reduction are presented. The effects of hydrogen (H_2) pretreatment on NiO and the reduction with H_2+CH_4 mixtures with a low H_2 content are investigated. The experiments were carried out on high-speed scanning electrotermography setup [1,2] under reduced gas pressure (below 15 kPa) at 1050°C by using Ni wires 100 micron in diameter (NP-2 trademark, purity 99.5 %). The latters were preliminary oxidized in air (P=0.1 MPa, T=1300°C, t=150 s) with formation of NiO layer on metal surface with ~6 micron in thickness.

The experiments highlighted that both H_2 pretreatment or its minimal content in H_2+CH_4 mixtures crucially affect the nucleation and dynamics of CH_4 reduction. Without H_2, NiO undergoes distinct nucleation, forming Ni nuclei in specific areas during the CH_4 reduction. Microscopic techniques, confirmed that H_2 promotes quick Ni nucleation across the NiO crystal surfaces without altering the growth mechanism. This ongoing reduction process significantly transforms Ni morphology, forming nanoscale grains and porous structures, which, in turn, influence CH_4 decomposition. The above is supported by independent measurements which have shown that the decomposition of methane on pure metallic nickel occurs approximately 20-30 times faster than on NiO.

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Poster Report

Salt assisted solution combustion synthesis of Ni/NiO nanopowders with enhanced specific surface area

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Due to environmental friendliness and low cost, nickel oxide (NiO) and Ni/NiO composites are promising candidates for electrochemical energy storage applications, particularly for supercapacitors [1]. Therefore, it is of great importance to develop efficient, controllable, and reliable methods for synthesizing these materials [1]. In this study, we employed the solution combustion method (SCS) to synthesize Ni/NiO nanocomposites, utilizing nickel nitrate as an oxidant and glycine as a fuel. Among various synthesis methods, solution combustion synthesis is a simple, time- and energy-saving process that produces homogeneous and fine powders. It provides a precise and uniform formulation of the desired composition by thoroughly mixing the reagents at the molecular level in a liquid solution. In addition, SCS allows the tailoring of the phase composition and microstructure of the resulting products by adjusting the fuel-to-oxidizer ratio [2]. Adding gasifying oxidizers such as ammonium nitrate will enhance the release of gaseous products during the combustion reaction, enhancing the porosity and specific surface area of output products. Ammonium nitrate is an additional heat source, leading to elevated combustion temperature. The high combustion temperatures during SCS result in particle sintering and growth. Halide salt, such as KCl, was used during solution combustion synthesis to prevent the agglomeration and sintering of the particles. The salt is melted during combustion synthesis and then forms the coating on the combusted products, preventing them from further agglomeration, thus obtaining fine particles. We investigated the fabricated powders' phase, structure, microstructure, morphology, and magnetic features. Our findings show the effectiveness of applying SCS with the addition of ammonium nitrate and KCl to reach Ni/NiO fine powders with a high specific surface area, which is advantageous for producing electrodes for supercapacitors.

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Poster Report

SHS of ultra-high temperature boride-carbide ceramics in the HfB2-HfC system

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The pseudobinary eutectic HfB_2-HfC composites belong to the class of ultra-high temperature ceramics. Their lowest melting point is 3140 °C [1] and due to ultrafine grain structure they have superior hardness, crack resistance, creep resistance and excellent thermal stability under oxidation at extremely high temperatures. Such ceramics exhibits microplasticity, which is not typical for the individual compounds HfB_2 and HfC.

This study is devoted to the production of heterophase submicron sized powders of eutectic HfB_2-HfC composition via SHS, including the use of high-energy mechanical processing (HEMO). These SHS-powders have been successfully applied for hot pressing of ultrafine-grained structural ceramics [2, 3].

The HfB_2–34%HfC (at.%) composite products were obtained via layer-by-layer combustion synthesis. They consist of highly porous agglomerates sized up to 100 µm, which can be easily milled into a powder consisting of composite particles of 1-10 µm in size. Each particle consists of HfB_2 grains of 0.5-2 µm in size and HfC grains sized 0.3-1 µm. An extremely low oxygen impurity content below 0.29 wt.% was reached.

The consolidation process of powders was studied. The HfB_2-HfC ceramics with grain size less than 5 µm, hardness up to 20 GPa, crack resistance up to 10 $MPa \cdot m^{0.5}$, bending strength up to 400 MPa and thermal conductivity up to 60 W/(m·K) were obtained. Si-containing dopants markedly improved the oxidation resistance. The kinetics and mechanism of oxidation at 1650°C were studied. The oxidation process is described by a step law function $q=at^n$ and is limited by the formation of a multilayer oxide film based on HfO_2, $HfSiO_4$ and $SiO_2 \cdot B_2O_3$.

A complex study of mechanochemical synthesis of eutectic HfB_2-HfC composition has been carried out. The nonlinear dependence of the chemical conversion degree on the HEMO duration is obtained.

This work was supported by the Ministry of Science and Higher Education of the Russian Federation (project 0718-2020-0034).

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Poster Report

The kinetics and mechanism of solution combustion synthesis in $Ni(NO_3)_2$ + hexamethylenetetramine and $Co(NO_3)_2$ + hexamethylenetetramine systems

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Solution combustion synthesis (SCS) utilizes exothermic self-propagating reactions to prepare nanoscale materials that can be widely used in electronics, biomedical, energy-saving and conversion technologies, and other applications. The thermodynamics, kinetics, and mechanisms of SCS reactions are not well studied. In this work, a systematic study of the mechanism and kinetics of SCS reactions involving $Ni(NO_3)_2$ and $Co(NO_3)_2$ as oxidizers and hexamethylenetetramine ($C_6H_{12}N_4$) as a fuel was carried out.

The prepared solutions were placed in paper boats, dried at a temperature of 100°C for 24 hours, and then combustion was initiated in a constant pressure reactor at a nitrogen pressure of 3 atm. These gels also were investigated by the thermal analysis method and from the obtained data the activation energy was calculated for both systems

Detailed investigations using combustion diagnostic methods, thermal analysis, and mass spectroscopy measurements allow us to propose that the process includes multistage decomposition of the oxidizer and sublimation of the fuel.

In both systems, the gas phase consists of nitrogen (N_2), hydrazine (N_2H_4), and methane (CH_4), released during the decomposition of hexamethylenetetramine.

When nickel nitrate hexahydrate decomposed, the intermediate composition $Ni(NO_3)_2 \cdot 2Ni(OH)_2 \cdot 4H_2O$ is formed. The decomposition of $Ni(NO_3)_2 \cdot 2Ni(OH)_2 \cdot 4H_2O$ produces nitrogen oxides (NO, NO_2, N_2O) and nickel oxide (NiO). Excessive amounts of N_2H_4 and CH_4 in a fuel-rich system reduce NiO to nanoscale Ni. Synthesized Ni is readily consolidated into samples with a relative density above 90% even at 773 K during fast processing.

The decomposition of cobalt nitrate hexahydrate leads to the formation of cobalt oxide (CoO), which is then reduced to Co by methane (CH_4) and hydrazine (N_2H_4). The combustion synthesized Co was subjected to rapid pressureless sintering at a temperature of 1275 K, and consolidated into compact samples with a relative density of about 90% and micrometer-size grains (from 1 to 2 µm). High-resolution electron microscopy investigation shows the increase in lattice dislocation due to the $\varepsilon \rightarrow \gamma$ transition at high-temperature processing. The prepared γ-Co exhibits a higher nanohardness and Young's modulus than ε-Co prepared by slow melt solidification and rolling methods.

Poster Report

The influence of technological parameters of magnetron sputtering on the structure and properties of films of a high-entropy alloy of the CoCrFeNiTi

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The rapid development of electronic devices leads to increased demands both in terms of improved performance and resistance to extreme external conditions, especially when using electronic components in research on earth and in space, automotive and medicine. [1]. One of the most popular electronic devices is the thin film resistor or chip resistor [2]. The chip resistor must have a low temperature coefficient of resistance (TCR) close to 0 ppm/°C, otherwise a high TCR will lead to resistance drift and, as a result, failure of the device based on it.

Factors that influence TCR: technological sputtering parameters (power, time), annealing temperature and film composition, where the last factor plays a huge role [3]. The application method is the main one for obtaining a TCR close to zero. High-entropy alloys (HEA), due to their unique properties, such as high resistance to oxidation and corrosion, radiation resistance, diffusion-barrier resistance, stability of properties when exposed to high temperatures, are a contender for displacing traditional materials used in the production of thin-film resistors [4].

The HEA films studied were sputtered onto a silicon substrate with thermal oxide. The work outlines the influence of sputtering power and time on the morphology and structure, TCR, magnetic properties and microhardness. The films turned out to be homogeneous in composition with an amorphous structure and nanocrystalline inclusions. The work was supported by the Russian Science Foundation grant No. 20-13-00277 P.

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Poster Report

Preparation of NiAl-Steel Graded Composite During Self-Propagating High-Temperature Synthesis

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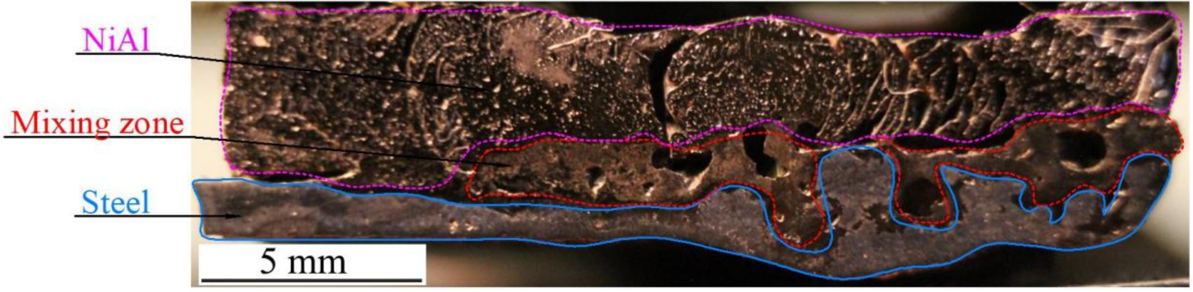
Self-propagating high-temperature synthesis (SHS) can be used to produce both porous and non-porous NiAl intermetallic, which is a promising material in many industries because of its high melting point and hardness, good thermal conductivity and oxidation resistance at elevated temperatures as well as small specific gravity [1,2,3]. It is known that a layered composite consisting of a NiAl layer and a metal layer can be obtained using SHS [1-4]. However, it does require the use of an external source of heat and pressure. The aim of this work was to obtain a functionally graded NiAl low-carbon steel composite by SHS using the pressure of the gaseous reaction products (impurity gases) formed during the synthesis of the starting powders in the reactor.



In this work, a composite of NiAl and low carbon steel was produced by the exothermic synthesis of TiC and NiAl and the pressure generated by the released impurity gases. Metallographic studies revealed that in the central part of the sample there was melting of the steel surface at the NiAl interface and formation of a mixing zone. Elongated pores were found in the NiAl layer, formed when impurity gases passed through NiAl crystallization. As a result of the research, due to the pressure and heat generated during the synthesis of the starting powders, SHS modes were established for the production of the NiAl composite with low-carbon steel.

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Poster Report

Obtaining products from tungsten-free hard alloys using the SHS method with compaction

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Self-propagating high-temperature synthesis received extensive development, in particular, ISMAN has developed more than 20 tungsten-free grades of hard alloys based on titanium carbide instrumental and structural purposes of the STIM group. LLC "SVS-Composite" has chosen the direction of manufacturing tungsten-free hard alloys by SHS synthesis of titanium carbide with obtaining titanium-nickel or titanium-copper intermetallic compounds as a binder followed by force compaction of the SHS reaction products for forming drawing tool blanks.

It is known that intermetallic compounds titanium-nickel and titanium-copper have higher physical and mechanical properties than nickel or copper respectively. Based on these prerequisites, the composition of SHS mixtures was formed according to special created calculation algorithm, when the ratio of components ensured the production of 50-65% (wt.) titanium carbide in the finished product and the rest are titanium-nickel or titanium-copper intermetallic compounds. In SHS processes the following was used: titanium powder, nickel powder or copper, soot The technology included mixing the initial mixture, initiation reactions, SHS reaction, hot pressing obtained SHS products, cooling, processing of the workpiece to the required sizes - sandblasting, grinding, electrical discharge processing, finishing with a diamond wheel. According to this scheme, finished products were obtained - die blanks with dimensionsin agreement with potential consumers. The report provides photos of the received products. The hardness of the product material is: with intermetallic compounds titanium - nickel 68-70 HRC (85.5-86.5 HRA), with titanium-copper intermetallic compounds 62-64 HRC (82.5-83.5 HRA).

Wear resistance tests of these two types of alloys according to the method abrasion in comparison with standards (metals) of similar hardness showed a twofold increase in the wear resistance of the resulting alloys by based on titanium carbide with binders of titanium-nickel intermetallic compounds or titanium-copper. On the composition of the charge and the SHS technology for producing tungsten-free hard alloy with a binder of titanium cuprids received RF patent No. 2691656 with priority from 01/22/2018 Experimental samples obtained at the existing pilot industrial site Draw blanks are tested: products with nickel - during drawing pipes at specialized enterprises of the Ural region, and products with copper - at the Ural factories for processing non-ferrous metals in relation to drawing profiles from copper, bronze, brass. The main disadvantage of the products identified during testing is the presence of microporosity on the working surface of the portage. Paths have been outlined improvements in technology that will help level out existing drawbacks. Productivity of the existing site for the production of commodity the number of draws is about 20 pieces per shift, due to which it is possible to cover a significant share of the needs of Ural factories for these products.

Therefore, we invite SHS specialists to cooperate for fine-tuning proven technology, as well as potential interested consumers of such products.

Poster Report

Synthesis of iron nitride powder in the mode of thermally coupled SHS process

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Iron nitrides are of interest because of their high saturation magnetization, corrosion resistance, and wear resistance. They can be used as promising magnetic materials, coatings for structural steel, and as ligatures for the production of special purpose nitrogenous steel. Iron nitrides are mainly produced in flow furnaces in an ammonia environment [1]. However, this method has disadvantages, such as the duration of the process and the complex and energy-intensive production. The self-propagating high-temperature synthesis (SHS) method is proposed as an alternative for synthesizing iron nitrides. However, direct synthesis of iron nitride powders in the classic SHS mode is not possible due to the low heat release during the reaction of iron with nitrogen. This heat release is insufficient for chemical reactions to occur in the SHS mode. To overcome this, a thermally coupled SHS process, known as synthesis with a chemical furnace, is used to carry out weakly exothermic reactions [2].

In this work we used VS-1 spherical carbonyl iron powder (3-8 microns) and urea (0, 9, 15, 30 wt. %) as a nitrogen source. A chemical furnace consisting of a mixture of 5Ti+3Si (T_{ad}=2402 K) was used. The synthesis was carried out in a high-pressure bomb in a nitrogen medium (6.5 MPa).

Nitride products in the form of Fe-Fe_3C-Fe_xN compositions (x=2-4) were synthesized. The Fe_xN fraction, determined by X-ray analysis, was up to 50 % by weight. Increasing in urea concentration leads to a higher fraction of nitrided products and an increased carbon concentration. At high temperatures, the carbon interacts with iron powder to form Fe3C. The microstructure study revealed that nitrided iron particles have a spherical core-shell composition, with the iron core and the Fe_xN iron nitrides (x=2-4) shell. The shell of the spheres is made up of submicron-sized needle-shaped crystals.

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Poster Report

Structure and Properties of Zr-B-C, Ti-B-C, and Zr-Ti-B-C Hard Films Deposited by Magnetron Sputtering Using Composite SHS-Targets Based on Boride-Carbide Eutectic Compositions

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Thin films in Me-B-C systems (where Me is a refractory metal) are promising for use as functional layers to improve the characteristics of critical engineering products. Such films have a combination of high mechanical properties, wear resistance, corrosion resistance with high thermal stability and oxidation resistance. Moreover these films are interesting in terms of specific electrophysical and optical characteristics [1,2]. The aim of work was to obtain and comprehensively study Me-B-C (Me: Zr, Ti) films.

The films were manufactured by direct current magnetron sputtering (DCMS) and pulsed magnetron sputtering (PMS) using the targets of ZrB_2-ZrC, TiB_2-TiC, $(Ti,Zr)B_2$-(Ti,Zr)C compositions fabricated by the force SHS-pressing and hot pressing of SHS-powders technologies. During the deposition, the following parameters varied: pulse frequency and duration, power, flow rate of the working and reactive gas (nitrogen), operating pressure, and bias voltage. Complex studies of films have been carried out using TEM, SEM, XRD, EDS, GDOES, micro- and nanoindentation, tribological and scratch testing. To estimate the oxidation resistance/thermal stability, high-temperature annealing in air/vacuum was performed. Optical transmission and reflection coefficients, refractive index were determined by spectrophotometry. The electrical resistance was measured using a four-probe method over a wide temperature range.

The results showed that the films deposited at the optimal deposition parameters demonstrated hardness up to 40 GPa. Due to the high mechanical properties, films greatly increased the wear resistance of the substrate material. Films with high zirconium content showed a best oxidation resistance during heating in air up to 700°C. The high protective properties were due to the formation of ZrO_2 surface layers. Films with high thermally stable showed the absolute value of the temperature coefficient of resistivity a value of 2×10^{-5} K^{-1} at 600 °C. It was found that the optical transmission coefficient increases in proportion to the nitrogen content in the films. Films with the highest concentration of zirconium had the highest optical characteristics.

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Poster Report

Investigation of the effect of silicon content on the characteristics of SHS compositions Si_3N_4-Yb_2O_3

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The use of composite powders (CP) based on silicon nitride obtained by self-propagating high-temperature synthesis (SHS) is a promising direction for increasing the high-temperature strength of silicon nitride ceramics [1]. Previously, SHS composite powders of the Si_3N_4-MgO composition and the corresponding ceramics were studied in the works, it was revealed that there is a great potential to increase the level of high-temperature strength of ceramics, the realization of which is possible when using sintering activators at the SHS stage and carrying out complex work to optimize the technology. Ytterbium oxide is a promising sintering additive that allows increasing the high-temperature strength of ceramic materials based on silicon nitride [2, 3]. The purpose of this work was to study the effect of silicon content on the process of obtaining SHS compositions Si_3N_4-Yb_2O_3, phase composition and morphology of particles of synthesis products.

A 30-liter SHS reactor was used for the synthesis of composite powders. The working pressure of the process was 4 MPa. The composition of the initial charge included the following components: Si (21, 23, 25 and 27 wt.%), Si_3N_4, Yb_2O_3. Ytterbium oxide was calculated so that its amount in the secondary phase was 16 wt.%.

The effect of the silicon content in the initial charge on the phase and morphological composition of the synthesis products was studied, it was found that with an increase in the silicon content, the synthesis temperature increases from 1640 °C to 1966 °C, as a result of which the alpha phase content of silicon nitride decreases from 85% to 0%. The composition of the secondary phases of the composition changes with an increase in the combustion temperature. At a temperature of 1810 °C, only quaternary ytterbium-silicon oxynitride ($Yb_4Si_2N_2O_7$) is present as the oxide phase. A further increase in temperature is characterized by a decrease in the peaks of the oxynitride phase in the composition, which may be due to the formation of a beta phase, which has a higher peak intensity compared to the intensity of the peaks of the oxide phases. At a lower temperature of about 1650 °C, the composition of the secondary phases is represented by the phases of oxynitride ($Yb_4Si_2N_2O_7$), disilicate ($Yb_2Si_2O_7$) and silicon monosilicate (Yb_2SiO_5).

The influence of the synthesis temperature on the morphology of the composition particles has been studied. It was found that at a temperature of 1640 °C, the particles of the composition have an equiaxed shape with a particle size of 1-2 μm. With an increase in the combustion temperature, the number of equiaxed particles decreases due to a decrease in the content of the alpha phase of silicon nitride and an increase in the formation of the beta phase of silicon nitride, which has a characteristic structure represented by elongated columnar grains.

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Poster Report

Investigation of the influence of mechanical activation and impurity gas release on macrokinetic combustion patterns of the Ti-C-B system for granular and powder mixtures

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The study of combustion processes of condensed heterogeneous systems is an important task. In previous studies, it was established that impurity gas evolution significantly affects the combustion process and the properties of the final product. The aim of the study is to determine the influence of mechanical activation (MA) and impurity gases (IG) on the combustion laws of granulated mixtures (granule size 0.6-1.6 mm) with bulk density and traditional pressed samples (relative density 0.53-0.6) of composition 100-x(Ti+C)+x(Ti+2B), where x = 0, 20, 40, 60, 80, 90, 100 mass %.

The study found that MA of powders leads to a gradual decrease in the combustion rate of pressed samples with increasing Ti+2B content in the mixtures – a decreasing dependency (from 25 to 12.5 mm/s for compositions of 100%(Ti+C) and 20%(Ti+C)+80%(Ti+2B), respectively). However, the combustion rate of compacts made from unactivated powders increases – an increasing dependency (from 13 to 37 mm/s for compositions of 100%(Ti+C) and 100%(Ti+2B), respectively). These results contradict the literature's view on the influence of MA on the combustion process, which suggests an increase in combustion rate.

Transitioning to granulated samples leads to a substantial increase in the gas permeability of the charge, thereby reducing the influence of IG. The combustion rates of granulated samples are higher than those of pressed ones for all investigated compositions. Granulated mixtures from MA powders have combustion rates 2-5 times higher compared to granules from unactivated powder (110 and 22 mm/s for compositions of 100%(Ti+C) and 185 and 80 mm/s for compositions of 20%(Ti+C)+80%(Ti+2B), respectively). MA and granulation do not affect the phase composition of the combustion products, which correspond to the phases TiC and TiB_2.

This work shows that the transition from pressed to pelletized samples significantly changes the combustion rate of 100-x(Ti+C)+x(Ti+2B) mixtures and allows us to separate the effects of impurity gases and chemical activity on the combustion process of the mixtures after MA.

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Poster Report

Synthesis of cast refractory high-entropy alloys using centrifugal SHS metallurgy and production of oxide-fibre/alloy matrix composites based on them.

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The potential of the Ni-based heat-resistant alloys is limited by an operating temperature of about 1100 °C, for physical reasons and no longer meets the requirements for new generation turbine-type power engine units. A further increase in the operating temperatures of the units is associated with the need to improve the characteristics of gas turbine engines (GTE) and gas turbine units (), reduce fuel consumption of GTE at the flight stage, and GTU during energy generation and gas transportation, reducing the load on the environment by reducing harmful emissions into the atmosphere, etc.

A current direction in the development of new materials for the manufacture of high-temperature structural parts are refractory metals Nb, Mo, Cr and alloys based on them. However, during their development, intractable problems arise related to the conflict between strength – crack resistance. These difficulties can be overcome by combining fiber composites and a metal matrix.

A relatively new approach to the development of high-temperature materials is their formation on the new concept, which excludes the simultaneous use of several metals with relatively close concentrations. Such alloys are called high-entropy alloys (HEAs). In this regard, the refractory high entropy alloys (RHEAs), with potentially high physical and mechanical characteristics under high temperature conditions are greatest interest. Such alloys are formed from refractory elements (Nb, Mo, Cr, Ta, V, W, Hf, Zr, Ti etc.), and usually have a single-phase solid solution structure with a bcc crystal lattice.

This research includes two main areas of research:

(i) Development of effective methods for producing cast RHEAs based on refractory metals by centrifugal SHS metallurgy, using initial oxide raw materials (metal oxides);

(ii) Development of physical and technological methods, using liquid-phase and solid-phase technologies for producing the oxide-fibre/alloy matrix composite family based on synthesized RHEAs and comprehensive structural-phase analysis of the resulting composite materials and analysis of physical and mechanical properties.

This report will present the main synthesis regularities of the RHEAs based on the Mo-Nb-Ta system with the introduction of various components (V, Zr, Hf, Cr, Ti etc.) and some results for formation of the oxide-fibre/alloy matrix composites based on them.

The research was funded by the Russian Science Foundation (project No. 24-13-00065).

Poster Report

Combustion Synthesis of Cermets from Granular Mixtures Ti+C–NiCr

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Materials based on titanium carbide with metal binders are increasingly used as a replacement for hard alloys based on tungsten and chromium carbides [1]. To reduce fragility and increase adhesion when spraying protective coatings, a metal binder is introduced into powders based on titanium carbide - Ni, Mo, NiCr, Cu, etc. One of the methods for producing such powders is self-propagating high-temperature synthesis (SHS) [2]. The purpose of this work is to study the macrokinetic laws of the synthesis of granular mixtures of TiC+NiCr.

At work for the first time, the synthesis of the TiC+NiCr composite from a granular mixture with titanium of different dispersion, containing different amounts of impurity gases, was carried out. The features of the combustion process of a granular charge are explained by its structure – the presence of physically separated cells (granules) with a powder mixture, which can ignite because of conductive heat transfer from granule to granule or convective heating by gas released from the charge. The combustion front in the powder and granular charge based on titanium with a smaller characteristic size of titanium particles propagated at a higher rate, despite the higher content of impurity gases in it. The effect of impurity gas release on the combustion rate of powder mixtures is explained using a convective-conductive combustion model. When using a granular mixture containing finely dispersed titanium powder, synthesis products were obtained without side phases of intermetallic compounds, which were easily crushed to micron sizes and could be used for plasma spraying of wearresistant coatings.

A method for determining the critical conditions for the transition of combustion of granular mixtures to the convective mode is proposed and tested. The content of impurity hydrogen in the mixtures TiC+NiCr is estimated, $X = 0–30\%$. It is established that in all studied mixtures of granules 0.6 mm in size, a conductive combustion mode is realized. It is shown that for a mixture of granules 1.7 mm in size, the flow of impurity gas provides a convective combustion mode in the mixtures with $X = 0$ and 10%, a conductive combustion mode for $X = 30\%$, and a transition from conductive to convective mode at $X = 20\%$. XRD analysis of the synthesis products indicates no intermetallic phases and no dependence of the phase composition on the granule size.

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Poster Report

Self-propagating high-temperature synthesis of thermoelectric material based on Cu2Se with SiC doping

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Research into thermoelectric materials for commercial applications is receiving increasing attention. [1]. However, their effectiveness remains limited. Thermoelectric materials Cu_2Se-based are currently the most widely used. It has been demonstrated by recent studies that the inclusion of SiC in the Cu_2Se system can significantly enhance the thermoelectric performance of this material. [2]. Enhancing the characteristics and technology for acquiring such materials is a pressing matter that requires immediate attention.

The aim of this study is to assess the viability of manufacturing thermoelectric material using Cu_2Se with SiC addition (0, 0.5, 5 wt.%) via self-propagating high-temperature synthesis (SHS) in a single step. Furthermore, the research will examine the phase composition and electrophysical properties.

Thermoelectric material based on Cu_2Se was obtained in combustion mode from powder mixture of Cu and Se mixed in stoichiometric ratio 2:1 with addition of SiC in the ratio of components 0, 0.5, 5 wt.%.

The product's phase composition and electrophysical properties were determined using X-ray phase analysis (XRF) and measurements of the temperature dependence of electrical conductivity and Seebeck coefficient.

The analysis of electrophysical properties suggests that the addition of SiC leads to an increase in power factor, as a result of the increase in electrical conductivity and Seebeck coefficient. The Cu_2Se product, with a 5 wt.% SiC additive, exhibits a power factor of 14.4 $\mu W\cdot cm^{-1}\cdot K^{-2}$ at 900 K, which is 250% higher than that of Cu_2Se without additives.

The research was supported by the UMNIK-21 competition Contract 17415GU/2022.

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Poster Report

Influence of relative density of charge billets on temperature and burning rate of Ti-C-NiCrAlY and Ti-C-NiCrAl compositions and comparison with Ti-C-NiCr

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The key parameter of charge billets combustion in the self-propagating high-temperature synthesis (SHS) mode is relative density. The value of relative density directly depends on the applied pressing pressure of the initial powders. The study of the effect of relative density on the temperature and burning rate will give insight into the "survivability" of the material capable of plastic deformation by SHS extrusion and SHS compression methods. Earlier in [1] it was shown that the content of 30 wt.% NiCr bond is optimal. The purpose of the present work is to investigate the effect of relative density on the temperature and burning rate of Ti-C-NiCrAl and Ti-C-NiCrAlAlY compositions and to compare them with Ti-C-NiCr.

In this work, commercial powders of Ti, C, NiCr, NiCrAl and NiCrAlY were selected as objects of study. The pressing pressure of the charge blanks was 30-120 kN.

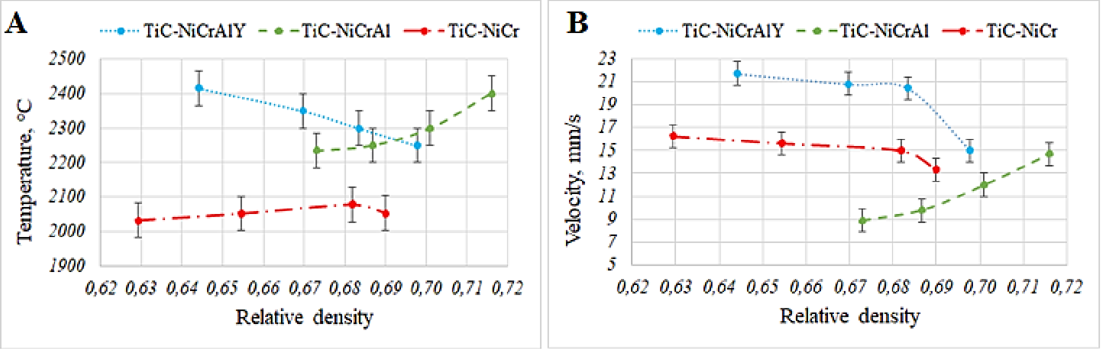

Fig. 1. Dependences of relative density on: a - on temperature, b - on combustion rate

Studies have shown that with increasing relative density for the composition Ti-C-NiCrAlY the temperature and combustion rate decreases (Fig. 1). For the composition Ti-C-NiCrAl with increasing relative density, an increase in both temperature and combustion rate is observed. Comparing these values with the composition Ti-C-NiCr it can be observed that there is a maximum value of both temperature and combustion rate, but after passing the extremum there is a decrease in these parameters due to the large contact of particles.

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Poster Report

Development of energy-efficient technology for zirconium powder production using SHS method

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Zirconium has a high melting point, good plasticity, excellent corrosion resistance and low neutron absorption cross-section, due to which it is widely used in metallurgy, nuclear power engineering, electronics, ceramics and refractories [1]. In particular, zirconium alloys are used for the production of nuclear reactor elements by powder metallurgy methods. The production of zirconium powder by hydrogenation-dehydrogenation (HDH) from zirconium sponge has become widespread due to its simple process flow, low production cost and good productivity. A typical HDH process consists of three steps: hydrogenation, grinding and dehydrogenation. The hydrogenation of zirconia sponge is usually carried out in a vacuum furnace, which is a long and energy-consuming process. A promising method to produce zirconium hydride is self-propagating high-temperature synthesis (SHS). The synthesis proceeds without external energy consumption, only due to the heat of its own chemical reaction $\mathrm{Zr} + \mathrm{H_2} \rightarrow \mathrm{ZrH_2} + Q$ (40 kcal/mol).

In this work, zirconium hydride was obtained by SHS hydrogenation of zirconium sponge. The hydrogenation of the sponge was carried out in a sealed 2 L reactor. Before synthesis, the reactor was sealed and filled with hydrogen to a pressure of 2 MPa. The hydrogenated zirconia sponge was mechanically milled in a drum-ball mill. As a result of SHS hydrogenation of zirconium sponge, zirconium hydride with hydrogen content of 2.04 wt.% was obtained. According to X-ray phase analysis, the SHS hydrogenation product is a single-phase ε-zirconium hydride with tetragonal lattice. To obtain zirconium powder, the zirconium hydride was dehydrogenated in a vacuum furnace at about 750 °C. As a result of dehydrogenation, zirconium powder with particle size ~ 80 microns and purity of 99.3 wt.% was obtained. According to XRF data it contains α-Zr and traces of ZrH2. Further studies are needed to improve the quality of the final product.

Thus, a new technology for obtaining zirconium powder from zirconium sponge by energy-efficient SHS-hydrogenation and thermal dehydrogenation method was developed. The developed technology is very promising, because it allows to reduce the time of zirconium powder production, to reduce energy and labor costs, to obtain high-quality powders, and at the same time, the process is harmless to the environment and humans.

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Poster Report

SHS Metallurgy Of Cast Molybdenum Carbides Under Gas Pressure

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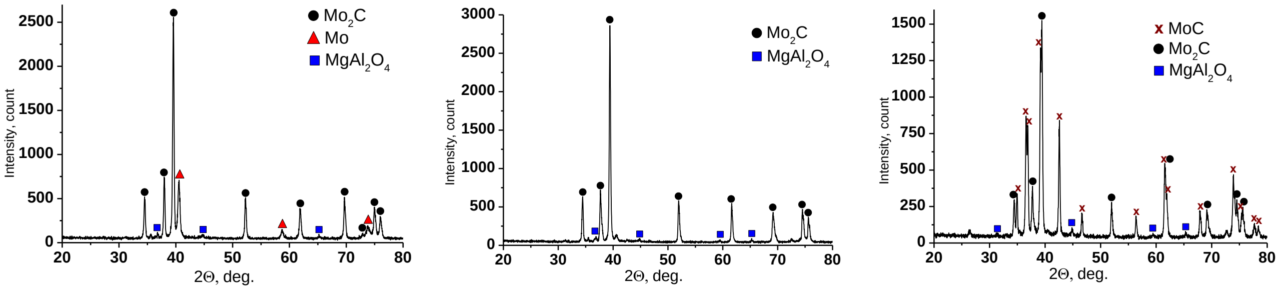
Molybdenum carbides are characterized by a set of properties important for science and technology: high hardness, wear resistance, corrosion resistance, relatively high electrical and thermal conductivity, catalytic activity comparable to that of platinum group metals [1,2]. The complex properties of molybdenum carbides make it possible to replace platinum group metals with them in some technologies [3], in particular in hydrogen production processes, namely in the composition of catalysts [4]. Powder materials based on molybdenum carbide are produced using various techniques, including powder metallurgy methods. In the presented study, the following chemical scheme was used for the synthesis of cast molybdenum carbides by SHS metallurgy in a reactor under argon pressure:

$$MoO_3/Al/Mg + \alpha C \rightarrow Mo\text{-}C + Al_2O_3\ x\ MgO$$

MoO_3, Al, Mg and C powders of technical purity were used in the experiments. The ratio of Al and Mg in the base mixture (MoO_3/Al/Mg) was 60/40% wt. The carbon content in the initial mixture was varied (α) from 0 to 7% wt.

Calculations using the Thermo program showed that with increasing carbon content in the mixture, the calculated combustion temperature decreases from 4100 to 2700 K. Combustion products contain molten Mo-C, Al_2O_3, MgO and gases (metal vapors, suboxides, CO and CO_2). Experiments have established that in the range $0 \leq \alpha\ C \leq 7\%$ wt. mixtures burn, combustion products have the form of a two-layer ingot (Mo-C and Al_2O_3 – MgO) and are easily separated.). The report presents the results of studying the influence of αC on combustion processes, separation of melts of target and slag combustion products, formation of the composition and structure of alloys. Figure 1 shows the effect of αC on the phase composition of the target product (Mo-C).



αC=4% wt. αC=5% wt. αC=7% wt.

Fig. 1. The effect of carbon content in the mixture on the phase composition of cast Mo-C

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Poster Report

The mechanism of formation of a "finger" during the combustion of a granular mixture of Zr+0.5C in the argon stream

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Zirconium carbonitride (ZrC0.5N0.5) is a promising powder material due to its physical and mechanical properties. Zirconium carbonitrides can be obtained by the SHS (self-propagating high-temperature synthesis) method in a flow reactor using a granular mixture, which eliminates the need for synthesis at high pressure [1]. To gain a deeper understanding of the process, it is important to distinguish between the effects of zirconium's reaction with nitrogen and those caused by heat and mass transfer due to the gas flow. This will enable us to establish the macrokinetic patterns of the combustion process when using granular mixtures of Zr+0.5C. The objective of this study is to experimentally determine the macrokinetics of combustion in granular mixtures of Zr+0.5C and analyze the results using established theoretical models for the propagation of the combustion front in these mixtures.

The experiments showed that the impurity gas did not affect the combustion rate of Zr+0.5C mixtures when an external argon flow was not present. The transfer of combustion between granules was facilitated by the movement of molten zirconium. A new mode of combustion for granular mixtures was observed in the presence of an argon flow. This mode is characterized by a fast propagation of the combustion front, accompanied by longitudinal and transverse shrinkage of the burnt samples, forming a 'finger'. The observed phenomena can be explained by the significant longitudinal shrinkage of the samples, which decreases the permeability of the reaction products. The gas flow is prevented from passing through the products and is instead directed towards the reactor walls, forming a finger. Increasing the gas flow through a narrow channel on the side of the reactor results in higher gas velocity and temperature of the reaction mixture at the sample's edge. It is estimated that the decrease in sample shrinkage is a result of the pressure drop in the longitudinal section and the surface tension forces caused by the temperature gradient in the transverse section.

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Poster Report

Thermal Stability of Oxidation-Resistant MoTaNbHfZr-Si-B Coatings under In Situ TEM Heating

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Currently, considerable attention is being paid to the creation of oxidation-resistant coatings to protect critical products exposed to high temperatures and aggressive environments, as well as various types of wear. High-entropy coatings based on silicides of refractory metals such as Mo, Ta, Nb, Hf, Zr, W, due to their high melting point, oxidation resistance and thermal stability [1, 2], can be used for appropriate applications. The purpose of this work was to study the thermal stability of MoTaNbZrHf-Si-B coatings by In-situ heating inside column of transmission electron microscope (TEM).

The coatings were deposited by high-power impulse magnetron sputtering (HIPIMS) with varying argon consumption from 25 to 125 ml/min. The MoTaNbZrHf-Si-B target was manufactured via self-propagating high-temperature synthesis and hot pressing. The fine structure of the coatings was investigated in situ using a measuring complex consisting of a TEM JEM 2100 JEOL and a Gatan 652 holder. The lamellae of coatings were obtained by the focused ion bombardment method. The selected area electron diffractions (SAED) and bright field (BF) images of coating structure were recorded in its initial state, as well as after isothermal exposure for 15-30 minutes at temperatures of 200-1000 °C in increments of 200°C. The processing of the SAED and BF images was carried out using ImageJ and Olympus Radius software. To determine the oxidation resistance, the coatings were annealed in a muffle furnace at temperatures of 1100, 1300, and 1500 °C.

All HIPIMS coatings were amorphous in the as-deposited state. The coatings obtained at high argon consumption had a columnar structure. The analysis of coatings during heating in a TEM column showed that an increase in argon consumption contributes to an increase in the thermal stability of the coating: when Ar 125 ml/min, crystallization occurs at a temperature of 700 °C only during prolonged exposure (15 min), while for coatings obtained at Ar 25 ml/min, crystallization begins instantly. The coatings obtained at argon flow rates of 25 and 125 ml/min at 700 °C were dominated by phase components: h-$MoSi_2$ and h-Ta_5Si_3+t-Nb_5Si_3, respectively. All coatings at a temperature of 1000°C contain phases h-Ta_5Si_3, t-Nb_5Si_3, h-$MoSi_2$, o-$HfSi_2$ and o-$ZrSi_2$. At the same time, the possibility of formation of solid solutions, for example, $(Hf,Zr)Si_2$, cannot be excluded. High-temperature annealing showed that the coating deposited at an argon consumption of 25 ml/min was characterized by better oxidation resistance at a temperature of 1300 °C, which is due to its denser structure and lower oxygen content.

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Poster Report

SHS of MAX phases based on $(Cr_{1-x}Mn_x)_2AlC$ and a capillary interaction with copper melts

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MAX phases combine the properties of ceramic and metals, which makes them promising for use in conditions of high temperatures and oxidizing environments. One of the methods for producing such materials is SHS (SHS-metallurgy). In this work, manganese-doped MAX phases $(Cr_{1-x}Mn_x)_2AlC$ were obtained. For the synthesis, mixtures were used, the composition of which was calculated using the following chemical scheme: Cr_2O_3 / MnO_2 / CaO_2 + Al + C→ $(Cr_{1-x}Mn_x)_2AlC+Al_2O_3$ + CaO_2, where $x = 0.05$, 0.15 and 0.30. The experiments were carried out in a SHS reactor with a volume of 3 liters at an initial argon pressure of $P = 5$ MPa.

The resulting materials consist of solid solutions of the MAX phase Cr_2AlC doped with Mn, as well as carbides and aluminides of chromium and manganese. The reflections of the MAX phase are shifted relative to the position of the Cr_2AlC reflections, which indicates the replacement of some chromium atoms by manganese atoms. Scanning electron microscopy showed that the phases have a layered nanolaminant structure, characteristic of MAX phases with layer thicknesses of several nanometers (from 3 to 20 nm).

Pressed powders of MAX phases were impregnated with copper melts at temperatures of 1100 – 1250°C in vacuum. Wetting kinetics were studied using high-speed imaging. Good wetting of the surface of the MAX phase with a Cu melt (0.8 at. % Cr) at a temperature of ≥1150 °C was found. The contact angle of 35° was established within 0.003 seconds. The capillary interaction is reactive: wetting was accompanied by the active formation of a Cu-Al-based liquid phase inside the “mother” Cr_2AlC phase with the preservation of a solid chromium carbide frame.

In the zone of contact with the copper melt, the MAX phase decomposes into chromium carbide and aluminum, which leaves the phase into the copper melt. Manganese in the MAX phase leads to an increase in the contact angle, while under the contact surface the MAX phase also decomposes and the formation of liquid occurs. The formation of the abovedescribed compositional structure leads to an increase in microhardness several times.

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Poster Report

SHS of advanced high-strength titanium alloy for laser powder bed fusion

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Multicomponent high-temperature titanium alloys are of significant interest for technical applications in mechanical engineering. They exhibit excellent oxidation, corrosion, and creep resistance. The low density of high-temperature titanium alloys (5 g/cm^3) makes them superior in specific strength to contemporary nickel superalloys for similar applications (9 g/cm^3).

Currently, titanium alloys are widely used in additive manufacturing to produce parts with complex shapes. Powders for this technology are obtained either through gas atomization or the plasma rotating electrode process. However, these methods require large capital costs and extensive experimental work to set up the powder production process.

In this work, the technology for producing spherical powders from multicomponent titanium alloys is based on the self-propagating high-temperature synthesis (SHS) method with subsequent plasma spheroidization. The SHS method allowed for high microstructure homogeneity and a minimal amount of non-metallic impurities in powder precursors. The spheroidized SHS powder obtained in this work was characterized by a uniform distribution of components, a narrow particle size distribution, and high surface quality.

Compact samples obtained from spherical powders of multicomponent titanium alloys by L-PBF method were characterized by a minimal number of defects and a high level of mechanical properties under compression in the temperature range of 800-1100 °C: : at 800 °C – E=115.2 GPa, $\sigma_{0.2}$ = 528 MPa, σ_c= 1148 MPa, and at 1100 °C – E=48.2 GPa, $\sigma_{0.2}$ = 98 MPa, σ_c = 149 MPa.

This work was carried out with financial support from the Ministry of Education and Science of the Russian Federation (project no. 0718-2020-0034 of State Assignment).

Poster Report

Synthesis of titanium nitride by combustion in a reactor with a controlled feed of reagents

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TiN can be obtained by burning of porous titanium samples in a nitrogen gas. To obtain the complete conversion to the nitride, the reaction wave temperature must be decreased to diminish the effect of melting of the titanium. Experimentally this can be achieved by addition of diluent, typically in the form of the product of combustion or ammonium chloride. Thus, the sample retains open porosity, which ensures the filtration flow of nitrogen to the combustion front. Without dilution it is not possible to obtain single-phase TiN by combustion synthesis.

The purpose of this work is to demonstrate the fundamental possibility of obtaining of titanium nitride from titanium powder without dilution, using a new way of organization of the combustion process in a nitrogen gas, different from that ones used previously in laboratory practice. For this purpose, a reactor with a controlled feed of Ti powder [1] was used. In this reactor, a continuous flow of Ti powder freely falls in a gaseous nitrogen onto a top layer of a product with high temperature, where it is involved into an exothermic reaction with nitrogen. The advantage of this method of organizing the process is a possibility to generate such a thermal regime in which the heat release will be sufficient to maintain the titanium nitriding reaction, but not sufficient to melt both the titanium itself and the resulting nitrides. The thermal regime of the process is regulated by changing the mass flow rate of titanium powder and nitrogen gas upon its blow. The absence of filtration difficulties eliminates the need to use high nitrogen pressures, as well as to add an inert TiN diluent or a nitrogen-containing additive NH_4Cl to the initial mixture.

We obtained the single-phase titanium nitride by this method. According to chemical analysis by Kjeldahl method the nitrogen content in the product is 19.5 wt. %, it corresponds to the $TiN_{0.86}$ composition.

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Poster Report

Biological and physical properties of newly synthesized Ni-Fe plasmonic nanoparticles

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In recent years, the study of plasmonic nanoparticles has gained significant attention due to their potential applications. They are becoming increasingly attractive for early disease diagnosis and hold potential as therapeutic agents, including for antibacterial and antitumor purposes. Additionally, they offer possibilities for targeted drug delivery.

The aim of this study was to investigate the cytotoxic properties of Ni-Fe plasmonic nanoparticles synthesized using chemical and 'green' methods. The biocompatibility, magnetic, and thermal properties of the synthesized nanoparticles were evaluated. Cytotoxicity was assessed using the disk diffusion method and the RBC test [1]. temperature-modulated calorimetry (TMC) studies were conducted to evaluate the heating efficiency of plasmonic NiFe nanoparticles over time. These studies were carried out under magnetic field conditions of 220 Oe and a frequency of 350 kHz.

Our findings revealed that the average size of the nanoparticles under study, depending on the synthesis method, ranged from 420 to 520 nm, including the stabilizing shell. Antibacterial activity studies indicated that chemical Ni-Fe plasmonic nanoparticles at doses ranging from 1 mg/ml to 2 mg/ml did not inhibit the growth of the gram-negative bacterium *Escherichia coli* K-12. Biogenic NiFe nanoparticles did not exhibit significant cytotoxicity. Chemically synthesized plasmonic nanoparticles demonstrated dose-dependent high magnetic and thermal properties, suggesting their potential for theranostics and hyperthermic therapy in oncological diseases.

Based on the results obtained, it can be concluded that the newly synthesized biogenic Ni-Fe plasmonic nanoparticles exhibit high biocompatibility and hold promise as a drug delivery system. Additionally, chemical nanoparticles demonstrate potential applications in hyperthermia and theranostics for tumor diseases.

The work was supported by the Science Committee of RA, in the frames of the research project № 21T-1F243.

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Poster Report

Combustion synthesis and sintering of single-phase aluminum oxynitride AlON

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Aluminum oxynitride (AlON) ceramics is well known due to its excellent optical [**Error! Reference source not found.**,**Error! Reference source not found.**,**Error! Reference source not found.**] and mechanical [**Error! Reference source not found.**] properties Due to combination of its thermomechanical properties and transparency in UV, VIS, and IR, aluminum oxynitride (AlON) is regarded as a challenge to tempered glass and sapphire single crystals [**Error! Reference source not found.**].

Synthesis of pure single-phase AlON powder is complicated by the low heat release of mixtures caused by the low aluminum content. Increasing aluminum content results in a multiphase product [2, 5, 6, 7].

So, in our experiments we coupled two reactions: Al oxidation and nitriding. Extra Al and Mg(ClO4)2 were introduced into basic Al/Al2O3 mixture.A method for producing single-phase aluminum oxynitride has been developed, which consists of synthesizing the powder in filtration combustion mode in a high-pressure nitrogen atmosphere, which makes it possible to develop a technology for producing γ-AlON powders on a semi-industrial scale.

Research has been carried out on the possibility of regulating the microstructure of aluminum oxynitride (AlON) powders in order to obtain fine powders. The importance of aluminum dispersion in the formation of the AlON microstructure has been established. High values of the initial nitrogen pressure make it possible to increase the nitrogen content in AlON, and, at the same time, increase the heat removal rate, which has a positive effect on the particle size (the higher the cooling rate, the smaller the particle size). Using a mixture of aluminum of different grades as initial reagents makes it possible to obtain a bimodal distribution of particles.

The processes of sintering of oxynitride ceramics have been studied. Sintering modes and technology have been selected to eliminate contamination of samples during sintering in furnaces with a graphite heater.

The influence of sintering agents such as yttrium oxide and a mixture of oxides of yttrium, magnesium and lanthanum and their amount on the sintering ability and optical properties of ceramics have been studied. Thus, a mixture of yttrium, magnesium and lanthanum oxides made it possible to obtain ceramics with light transmission up to 52%, while ceramics using only yttrium oxide only up to 22%.

Ceramics obtained from SHS powders have light transmittance up to 52%, hardness 17.7 ± 2.0 GPa, Young's modulus E = 320 ± 29 GPa, elastic recovery 62.8%. The band gap of the first ceramic samples was also assessed by two different methods, and it is 5.85 - 6.0 eV.

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Poster Report

Self-propagating high-temperature synthesis of light alloys in the Ti-Al-Mg system

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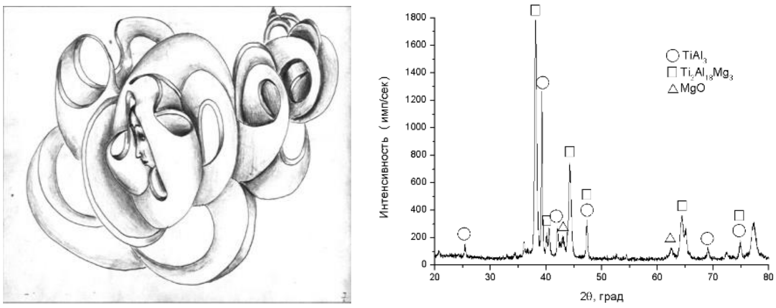
Because of their low weight, Ti-Al-Mg alloys are excellent for applications requiring a low material density. These alloys may be used to make strong, lightweight, and heat-resistant components, which will increase their efficiency and save fuel. Ti-Al-Mg alloys have the potential to displace steel as the primary structural component in the transportation sector in the future. Generally, a 10 % weight loss can result in a 3÷7 % reduction in specific fuel consumption [1].



(a) (b)

Fig.1 *(a) - SEM, (b) – X-Ray patterns of the synthesized Ti-Al-Mg alloy.*

Using the method of self-propagating high-temperature synthesis, an alloy with a ternary intermetallic phase, $Ti_2Al_{18}Mg_3$, was produced and investigated for the first time from a mixture with a composition of 15Ti-74Al-11Mg (wt.%). The initiation temperature of the SHS reaction was 630 °C at a sample heating rate of 3.7 °C/sec, and the maximum temperature of combustion wave propagation was 850 °C. Combustion proceeded in the frontal mode at a speed of 6.6 mm/sec. As the temperature rised to the melting point of the Mg-Al alloy, aluminum and magnesium melt, and Ti particles started to dissolve in the Mg-Al melt. Dissolved titanium reacts with the Al-Mg melt to form intermetallic Ti-Al particles on the surface and around the Ti particles. Ti-Al intermetallic particles in the Mg-Al melt are gradually dispersed as a result of capillary forces. After cooling, a multiphase composite based on Mg-Ti-Al was formed. The microstructure of the synthesized alloy is presented in the form of $TiAl_3$ grains 5 μm in size located in a $Ti_2Al_{18}Mg_3$ matrix. The microhardness of the alloy is in the range of 1140-2040 MPa.

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Poster Report

Investigation of MgAlON synthesis processes in the mode of self-propagating high-temperature synthesis

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MgAlON is a compound from the ternary system of MgO, AlN and Al_2O_3. Optical ceramics made of MgAlON is a promising material that has a wide range of applications: missile fairings, laser technology, IR windows and domes, transparent armor, etc. MgAlON ceramics has high strength, hardness, excellent corrosion resistance. In addition, the material has a high transparency range from the infrared spectrum to the ultraviolet spectrum.

From literature data for synthesis of MgAlON powder it is known about application of such techniques as solid-phase synthesis [1], carbothermal reduction [2] and aluminothermal reduction [3].

The listed methods have a number of significant disadvantages. Long holding times at high temperatures lead to high energy costs, also large-sized and difficult to operate equipment.

The method of self-propagating high-temperature synthesis is devoid of these disadvantages. The equipment in this case is small and easy to operate. The synthesis process takes from a few seconds to a few minutes. And, also with the use of this method a high purity of the product is achieved. All these advantages make the SHS method an attractive alternative for the synthesis of MgAlON powder.

This report presents the results of a combustion study of a mixture of Al, Al_2O_3, MgO and $Mg(ClO_4)_2$. Experiments were carried out with different proportions of components in the initial mixture. The effect of initial gas pressure on the final product was studied. The effect of nitriding and aluminum oxidation reaction on the rate of combustion processes was also studied. The optimum compositions of combustion products were determined, and tests were carried out for firing and densification of ceramic materials for the production of dense ceramics.

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Poster Report

SHS of composite material of 3Ni-Al mechanically activated particles and Ti+2B mixture

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Cermet composites, characterized by excellent strength, hardness and abrasion resistance, chemical stability, oxidation resistance, combine properties which are attractive for various applications [1]. It seems promising to obtain porous composites with interpenetrating phases (IPC) of metal/ceramics, intermetallic/ceramics by self-propagating high-temperature synthesis (SHS); these materials can be used as filters, membranes, catalyst carriers, porous preforms, etc. [1-3]. SHS, which proceeds in the combustion mode, is distinguished by a high velocity and low need for additional energy, allows controlling the combustion product characteristics, synthesizing ceramics and, at the same time, intermetallic, the melt of which penetrates into the ceramics pores [2-3]. The purpose of this work is to obtain a porous metal-ceramic composite TiB_2–Ni_3Al with a controlled structure characteristic of IPC by the SHS method.

3Ni–Al–Ti–2B powder system was used to obtain the composite. It consisted of mechanically activated composite particles-granules (3Ni + Al) and a mixture of fine boron and titanium powder. The SHS was carried out in the combustion mode without heating. In the combustion front, chemical reactions occurred both in the composite granules (between aluminum and nickel) and in the mixture around them (between titanium and boron) [3]. As a result of the combustion a porous frame of titanium diboride was formed with nickel aluminide melt penetrating into its pores. The SHS product had a composite structure with diboride and intermetallic phases, similar to IPC [1, 3]. In place of the granules there are pores that repeat their shape. In TiB_2–Ni_3Al, the porous structure and the structure of interpenetrating ceramic and intermetallic frameworks significantly depend on the quantitative ratio between the granules and the Ti + 2B mixture as well as on the dispersion of the components.

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Poster Report

Pulsed Electrospark Deposition of protective coatings using V2AlC-based SHS-electrode

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The MAX phases represent a family of the ternary carbides or nitrides, with the general formula $M_{n+1}AX_n$, where n = 1, 2, 3; M is a transition metal; A is mostly a IIIA and IVA-group element; and X is C and/or N. These materials are being considered for various applications required remarkable thermal stability and oxidation resistance. In order to obtain the MAX-phase based coatings it is necessary to eliminate the factors of chemical and thermal destruction of the MAX-phase during the pulsed electric spark deposition (PED): no interaction with the substrate and gas media, as well as decomposition in the process masstransfer [1].

PED onto Ti-substrate using the electrode with 75 vol. % of V_2AlC was studied. The electrode has been obtained by SHS [2]. Coatings deposited onto sublayer of TiC in an inert atmosphere were found to consist of VC, V_2AlC, TiC, V(Ti,Al) and have thickness till 30 μm. The nanocrystalline structure of the coatings was studied in-situ by heated up to 900 °C directly in column of high resolution TEM. Nanoparticles of the MAX phase V_2AlC with an elongated morphology measuring 100 nm in length and 30 nm in width were found in coating structure. The mean values of coatings hardness (H) and elastic modulus (E) were detected by using matrix indentation: H=15.4 GPa, E=288 GPa. In case of Ti alloy H=4.9 GPa, E=129 GPa. Electrochemical and tribo-corrosion studies were carried out in artificial seawater (ASTM D 1141-98). Compared to titanium alloy, the coating had a lower coefficient of friction (0.35) and increased wear resistance. Moreover the coating has enhanced corrosion resistance, exhibiting a corrosion current density an three order of magnitude lower than that of the titanium substrate.

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Poster Report

Synthesis of V2AlC ceramics by SHS using VO2 as a starting material

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The V_2AlC MAX phase is being considered for a wide range of applications. Its high stiffness, thermal stability, and resistance to thermal shock and oxidation make it suitable for use in harsh environments [1]. While the current synthesis methods for V_2AlC have demonstrated success, they often rely on the use of expensive V powder as a raw component and require high sintering temperatures along with the application of pressure. This can limit the scalability and cost-effectiveness of the synthesis process. The objective of this study was to establish a synthesis technique for the large scale production of V_2AlC ceramics, utilizing non-toxic and low-cost VO_2 powder.

We demonstrate a low-cost and rapid magnesium-thermic combustion synthesis of V_2AlC phases from an inexpensive oxide precursor. The V_2AlC powder was successfully prepared using VO_2/Mg/Al/C as starting materials through self-propagating high-temperature synthesis in molten NaCl, followed by leaching in dilute hydrochloric acid of the as-synthesized product. The samples were characterized by powder X-ray diffraction, Rietveld refinements and scanning electron microscopy. The phase composition (fig.1) of the as-prepared product is influenced by the magnesium and carbon content in the mixture. The optimal ratios of the components and acid leaching conditions for obtaining a powder containing 93% V_2AlC have been determined.

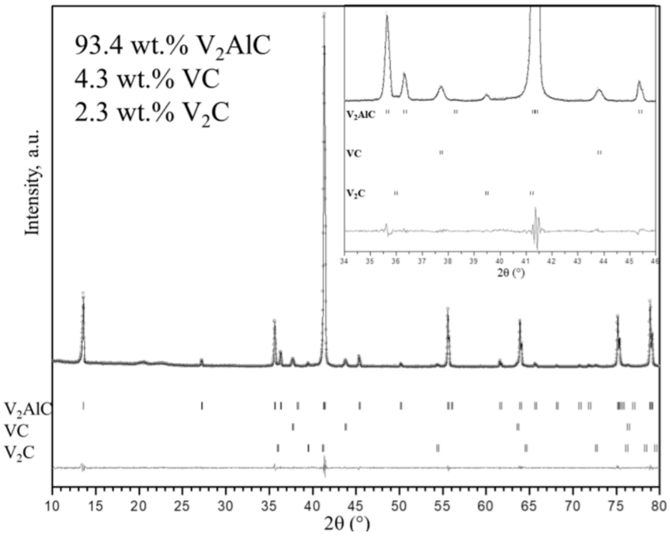

Figure 1. XRD pattern of the combustion product after acid leaching.

Therefore, the choice of non-toxic and low-cost raw materials and utilizing the rapid and energy-efficient SHS technique allow for a drastic reduction in production cost of V_2AlC.

Authors acknowledge funding from the Russian Science Foundation project 20–13-0277P.

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Poster Report

Thermoelectric Properties of Heusler Alloys Co2TiAl Synthesized by SHS Metallurgy

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The progress of modern technology is closely related to the search for new sources energy, primarily electrical energy as universal for practical use. Thermoelectricity is a promising creative method of direct conversion of thermal energy into electrical energy meeting the requirements for the cleanliness of its production and renewability. From this point of view, alloys of the Co-Ti-Al ternary system consist of relatively inexpensive, non-toxic and earth-abundant chemical elements, with high mechanical strength and heat resistance, which makes them suitable candidates as promising thermoelectric materials.

The traditional ways for 2Co-Al-Ti compounds production are arc melting and spark plasma sintering. One of the alternative methods of obtaining intermetallic compounds is self-propagating high-temperature synthesis (SHS) that is widely studied. In the present work a compact, pore-free intermetallic alloy Co_2TiAl (Heusler alloy) has been synthesized for the first time using SHS metallurgy method. The structure, electrophysical and magnetic properties of the synthesized product have been investigated.

X-ray phase analysis showed the formation of Co_2TiAl phase (Heusler phase), the mass content of which equals to 99 mass. %. The unit cell parameters of Co_2TiAl amounted to $a=5.8291\pm0.0002$ Å. The compound has a face-centered cubic lattice (Fm3m), a structural prototype of $AlCu_2Mn$.

The maximum value of the Seebeck coefficient equal to – 36.5 μV/K is observed at a temperature of ˜ 800K. The value of the Seebeck coefficient at room temperature 300 K was -29.5 μV/K, which is of the same order that the value obtained for the Co_2TiAl alloy in [1]. The relatively large Seebeck coefficient in Co_2TiAl is possibly associated with the special ferromagnetic state of Heusler alloys and the contribution of spin fluctuations to the thermoEDS. The synthesized material is weakly ferromagnetic. The maximum value of specific saturation magnetization at room temperature was 3.64 Am^2/kg. The results the electrical resistivity, magnetic properties, thermoelectric measurements at room temperature show high reproducibility and correlation with the results of other authors. [1,2]

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Poster Report

MA-SHS consolidation of (Ti,Zr)C solid solutions

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Titanium and zirconium carbides have a high melting point (3693 K and 3343 K respectively), low density, high hardness, strength, electrical conductivity and chemical resistance, NaCl type face centered cubic structure and form a continuous series of the $Ti_xZr_{1-x}C$ mixed alloys [1].

To date, (Ti,Zr)C solid solutions are prepared by spark plasma sintering (SPS) and hot pressing (HP) methods. Without detracting from the advantages of these methods, we note that the shortcomings are a multi-stage and duration of the technological processes. ISMAN has been developed an energy-efficient single-stage process for the preparation of a UHTC, including self-propagating high-temperature synthesis (SHS) and hot product consolidation (Fig. 1). Later, a mechanically activated (MA) blend of a metal and non-metal powder was used for the synthesis.

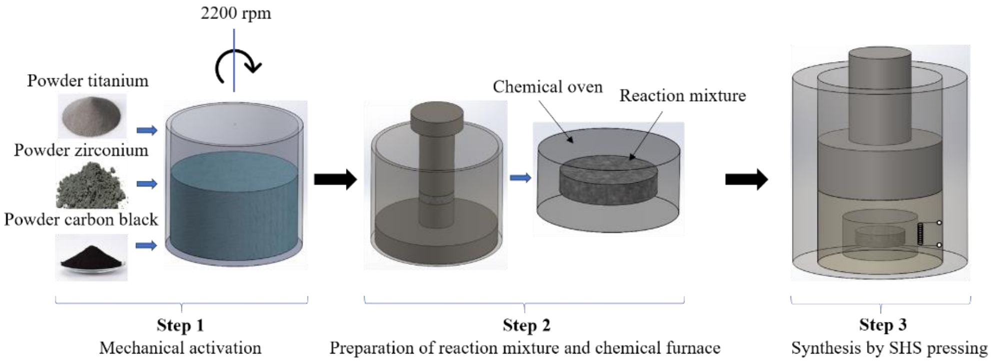

Figure 1 – Schematic diagram of (Ti,Zr)C solid solution preparation
using the MA-SHS method.

As a result, high-density $Ti_xZr_{1-x}C$ (x = 0.15; 0.24; 0.49; 0.88; 0.92) solid solutions with a submicron structure were successfully produced by MA-SHS under pressure. The effect of mechanical activation (MA) on the properties of reaction blends containing titanium, zirconium and carbon black powders, the formation of phase composition and microstructure, and the physical and mechanical properties of the alloys were investigated by means of X-ray diffraction (XRD) and backscattered electron mode (BSE). It is shown that the MA of the blends in hexane for 40-60 min resulted in the amorphization of the metal particles and the formation of the carbide phase crystallites in them. The resulting TixZr1-xC mixed alloys are characterized by grain sizes of 0.1 μm, relative density of 99.2%, microhardness (HV) of 20.9 GPa.

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Poster Report

The Influence of Stretching and Compression of the Reaction Zone on the Stability of the Filtration Front

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In the present work, we consider the systems with high gas permeability and thermal conductivity at a low thickness of prolonged solid reagent with special emphases on the combustion wave structure. Changes in the mass of the products and in the thermophysical parameters of the condensed component during combustion taken into account. We study theoretical the influence of the compression and stretching of combustion surface on the propagation and stability of the filtration front, as well as the loss of stability of the combustion wave in a linear approximation with respect to the curvature of the reaction zone. The initiation and propagation of cellular combustion of titanium powder layer whose transverse size varies in the front propagation direction experimentally study.

We conclude from our analysis that the instability of the quasi-isobaric filtration front leads to the formation and propagation of inhomogeneous combustion regimes, the evolution of which is determined not only by kinetic parameters, filtration transport of the gas reagent into the active zone of the chemical reaction, heat transfer mechanisms, but also by the geometric characteristics of the layer. Compression and stretch of the front significantly shift the flammability limits and stability boundary of planar infiltration front and also lead to new phenomena, such as extinction without any external heat loss, caused by deformation of the flame. The compression of the front provides an increase in the temperature in the reaction zone and a stabilization of unstable combustion. The stretching of the front leads to the loss of stability of the flat front and the initiation of cellular wave structures propagating in a self-oscillatory regime. Cellular waves are formed as the combustion moves away into the unstable region with an increase in the transverse size of the layer towards the direction of the front movement.

Poster Report

Synthesis of cast chromium-containing master alloys with Mo, W and Al by centrifugal SHS metallurgy.

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Alloying technologies for modern industrial alloys have become almost universally widespread in various industries. The introduction of master alloys of a given composition makes it possible to precisely control the concentration of alloying elements and to introduce easily oxidizing or volatile components into the alloy at high melting temperatures without significant losses due to their introduction in a bound state (fused).

In most modern high-tech metallurgical industries, to produce alloys, they use chromium-containing master alloys produced primarily on the basis of high-purity electrolytic chromium refined in hydrogen or aluminothermic chromium, but after refining melts using electroslag or vacuum-arc remelting methods to remove impurity components. This is due to continuously increasing requirements for the purity of alloying materials, which is due to increased requirements for the performance properties of modern industrial alloys used in the creation of new types of equipment.

The problem of increasing the energy efficiency of the master alloys production based on refractory metals (W, Mo, Cr, etc.) with various chemical compositions and required impurity level is an important and actual task for providing leading high-tech industries by advanced materials.

Taking into account the possibility of centrifugal SHS metallurgy [1,2] technique for synthesis of cast alloys, in the work we focused on developing alternative methods for the master alloy production for such practically popular Cr-W(Al), Cr-Mo(Al) and Cr-Al systems, which will lead to a reduction cost while maintaining the properties and quality of such materials.

The chemical composition analyze of the obtained cast master alloys under optimized conditions (optimized mutual ratio of initial component and centrifugal acceleration level value, introducing functional additives) by inductively coupled plasma mass spectrometry (ICP-MS), for main and trace elements, indicate that the obtained alloys satisfy all basic requirements for permissible concentrations as the target alloy elements so impurity components. Particularly noteworthy is the low content of harmful impurities such as sulfur and hydrogen.

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Poster Report

Synthesis of experimental small-tonnage batches of nickel β-alloys by centrifugal SHS casting and influence of the scale factor on the structure and properties

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Nickel aluminide based alloys are an alternative to standard heat-resistant superalloys as applied to engine construction. One of the promising methods for producing the cast β-alloys is centrifugal SHS-casting [1-5]. In previous studies by the authors, the optimal balance of high-temperature strength and heat-resistant was obtained by alloying the base composition CompoNiAl-M5-3 [1] with additional elements: Mo, Re, Ta and Ti. Mechanical properties were achieved: σ_{ucs} = 1644 ± 56 MPa, σ_{ys} = 1518 ± 29 MPa, and heat resistance: weight increase in 30 hours at 1150 °C – 52 g/m^2. After studying laboratory samples weighing up to 1 kg [4], it was required to optimize the centrifugal SHS-casting parameters in order to produce the large-sized ingots up to 5 kg. Authors found an optimal parameters for production of homogeneous ingots and studied their properties in comparison with laboratory one.

Ingot weighing 4.7 kg was produced on novel unique equipment for producing cast materials by centrifugal SHS-casting[5]. The productivity of the installation is 45.0 ± 2.0 kg/hour. The chemical composition coincides with the calculated values, the structural and phase homogeneity of the ingot has been established. The content of gas impurities decreased from 0.022 wt. % up to 0.002 ± 0.0005 wt. %. Samples prepared from different parts of the ingot had a similar structure and properties. Heat resistance has improved to 19 g/m^2. The large ingot retained its mechanical properties in comparison with the laboratory sample: σ_{ucs} = 1654 ± 10 MPa, σ_{ys} = 1526 ± 25 MPa.

This work was financially supported by the Ministry of Science and Higher Education of the Russian Federation (project 0718-2020-0034).

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Poster Report

Obtaining Magnetic – Abrasive Powder Materials Based on Ti-C-Fe System by SHS – Grinding Method

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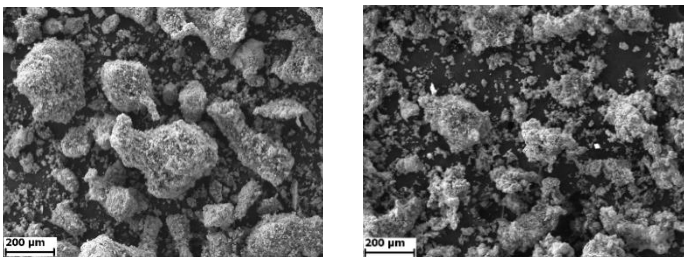
To obtain high surface cleanliness of simple and complex profile parts, the method of magnetic abrasive finishing (MAFO) has become widespread. This method based on the use of magnetic abrasive materials (MAM), which are a composition of abrasive and magnetic components. Among traditional MAMs, composites based on TiC - Fe are balanced in terms of properties and cost. Such materials can be obtained by the SHS grinding method, combining SHS and grinding of products to a powder state, in one technological cycle and in one installation. The advantages of the chosen method lie in the ability to vary deformation parameters (strain rate, external pressure, type of deformation device), which have a significant impact on the structure formation of the material, while eliminating a number of difficulties associated with grinding strong, difficult-to-deform synthesis products to a powder state when cooled to room temperature condition.

As a result of the work carried out, prototypes of TiC-based powder materials containing 20 and 30 wt. % Fe. Analysis of the microstructure showed that during the synthesis process, iron melts and wets the surface of the resulting titanium carbide particles. It is also shown that by changing the type of deforming device (cone, cutter), it is possible to change the qualitative characteristics of the resulting powder: grain size, its shape and morphology. The figure shows micrographs of the characteristic morphology of the resulting powders. The particles obtained using a cone (Fig. on the left) predominantly have an irregular round shape. When using a deforming device in the form of a cutter (Fig. on the right), the morphology of the particles changes significantly: splinter-shaped particles predominate. It is assumed that this is due to an increase in the intensity of deformation when using a cutter. Another characteristic feature is the particle size reduction [1, 2].



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Poster Report

Structure and properties of protective electrospark coatings obtained using $ZrSi_2$-$MoSi_2$-ZrB_2 and $HfSi_2$-$MoSi_2$-HfB_2 SHS-electrodes

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To increase the service life of critical parts made of metallic alloys on base of refractory metals, the most effective way is using protective coatings based on oxidation-resistant ceramic materials. In this work, coatings were applied using electrospark deposition (ESD) technology on substrates made of low-alloyed (Cr base; V 1.5%; Y 0.5%; B 0.05%) VKh1-17A alloy. ESD technology was carried out in an argon environment (99.995 %) on "Alier-Metal 303" installation (E=0.05 J). $ZrSi_2$-$MoSi_2$-ZrB_2 and $HfSi_2$-$MoSi_2$-HfB_2 alloys were used as consumable electrodes. Powders obtained by self-propagating high-temperature synthesis (SHS), were subjected sintering under hot pressing (HP) to produce electrodes.

Type of interaction between material of the consumable electrode and the that of substrate was determined using Palatnik's coefficient [1]. It has been established that the coatings form as an alloy of elements belonging both the anode and cathode (substrate), which ensures high adhesion of the coatings to substrate. For both electrodes a maximum value of the total gain of the cathode ($\sum\Delta Ki$) are observed in the 1st minute of treatment, and as the treatment continues, decrease in the mass of the cathode occurs. The formed coatings have 100% continuity and a thickness of 15-20 microns. The $ZrSi_2$-$MoSi_2$-ZrB_2 coatings are characterized by a hardness (H) of 18.2 GPa and an elastic modulus (E) of 274 GPa, and the $HfSi_2$-$MoSi_2$-HfB_2 coatings have H=16.9 GPa, E=332 GPa. The use of ESD technology increases the hardness of the chromium alloy by more than 4 times. The roughness (parameter Ra) of the $ZrSi_2$-$MoSi_2$-ZrB_2 coating is 2.09 μm, and that of the $HfSi_2$-$MoSi_2$-HfB_2 coating is 4.12 μm. When treated with a Zr-containing electrode, the transition diffusion zone of 5-7 μm thickness is formed, where except Cr, Si, Zr, Mo, V were found. The maximum specific weight gain at t = 1000 °C for 30 hours at air was observed for the uncoated BX1-17A substrate - 1.11 mg/cm^2, which is 1.6 times more than that of the $ZrSi_2$-$MoSi_2$-ZrB_2 coating and 1.7 times more than that of the $HfSi_2$-$MoSi_2$-HfB_2 coating.

This work was supported by the Russian Science Foundation (project No. 23-49-00141).

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Poster Report

High-entropy coatings for protection of steel structures of marine and coastal infrastructure

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Steel structures of marine and coastal infrastructure are susceptible to premature failure due to the intense corrosive action of seawater. Seawater is a complex solution of various salts and suspended organic and inorganic particles. Components that are simultaneously subjected to wear and corrosion experience an effect known as tribocorrosion. This leads to accelerated degradation of steel structures, resulting in increased economic losses [1]. Another challenge is a microbially influenced corrosion (MIC), which results from the interaction between microorganisms and submerged metal surfaces, particularly following biofilm formation. One promising method for protecting the surface of metal components from wear and corrosion is the deposition of coatings [2].

In this study, we propose Fe-Cr-Ni-Co-(Cu)x and Fe-Cr-Ni-Co-(Mo)x high-entropy coatings (HECs) for the protection of hardened steels. These coatings were produced by the electrospark deposition method in vacuum or protective Ar atmosphere. The effect of Cu and Mo content on resistance of HECs to corrosion, tribocorrosion, and MIC was studied in detail.

The coatings were characterized in terms of their structure, morphology, elemental composition, mechanical properties, and resistance to corrosion, tribocorrosion, and MIC in artificial seawater. The validity of the chosen approach was confirmed by tests in the marine environment of Black Sea.

This work was supported by the Russian Science Foundation (project no. 20-79-10104-П)

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Poster Report

Structure and Properties of Ti-Al-C Coatings Deposited by DCMS and HIPIMS Using a Ti2AlC Target

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It is known that transition metal carbides (TiC, Cr_3C_2, etc.) can be successfully used as protective coatings due to their high hardness, wear and corrosion resistance [1,2]. The application of materials based on $M_{n+1}AX_n$ phases (where M = Ti or Cr, A = Si or Al and X = C or N), combining the unique properties of metals and ceramics, is promising [3]. The purpose of present work was to study the structure and properties of coatings deposited by magnetron sputtering of a target based on the Ti_2AlC MAX-phase.

Magnetron sputtering was carried out in direct current (DCMS) and high-power pulsed (HIPIMS) modes. To increase the concentration of the MAX phase in coatings, the following approaches were used: increasing peak power up to 100 kW, applying a high bias voltage of -500 V, and annealing in a vacuum and in carbon-containing environment. The structure, mechanical and tribological characteristics were studied by SEM, EDS, XRD, GDOES, nanoindentation, “pin-on-disk” and impact tests. To determine the oxidation resistance of the coatings, annealing was carried out in air at temperatures of 800, 900 and 1000°C with expositions of 1 h.

The results showed that, depending on the deposition mode, coatings contained the fcc-TiC phase or a mixture of fcc-TiC and h-Ti_3Al phases. The coating obtained in the DCMS mode had the best mechanical properties: hardness of 31 GPa, elastic modulus of 294 GPa, as well, as a minimal friction coefficient of ~0.2 and wear rate of $8\cdot10^{-6}$ $mm^3N^{-1}m^{-1}$. When determining oxidation resistance, it was found that the HIPIMS coating deposited at a peak power of 100 kW had a minimal thickness of oxide layers in the range of 250-510 nm at 800-1000°C.

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Poster Report

SHS of high-entropy alloys for the production of multifunctional catalysts

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High-entropy alloys (HEAs) are gaining immense popularity due to their unique properties [1]. They also find application in the field of catalysis [2]. Centrifugal self-propagating high temperature synthesis (SHS) opens up wide possibilities for obtaining HEAs in a wide range of compositions from metal oxides and aluminum. Using this method, FeCoNiCuCrAl alloys with a Cr content of 4 and 9 wt.%, and near equiatomic concentrations of other metals, were synthesized. On their basis by leaching with 20% NaOH were obtained catalysts, preserving the structure of HEAs. The initial HEAs and catalysts were characterized by XRD, SEM, EDA, and BET methods. Specific surface area of the catalysts is 4.5 and 2.4 m^2/g.

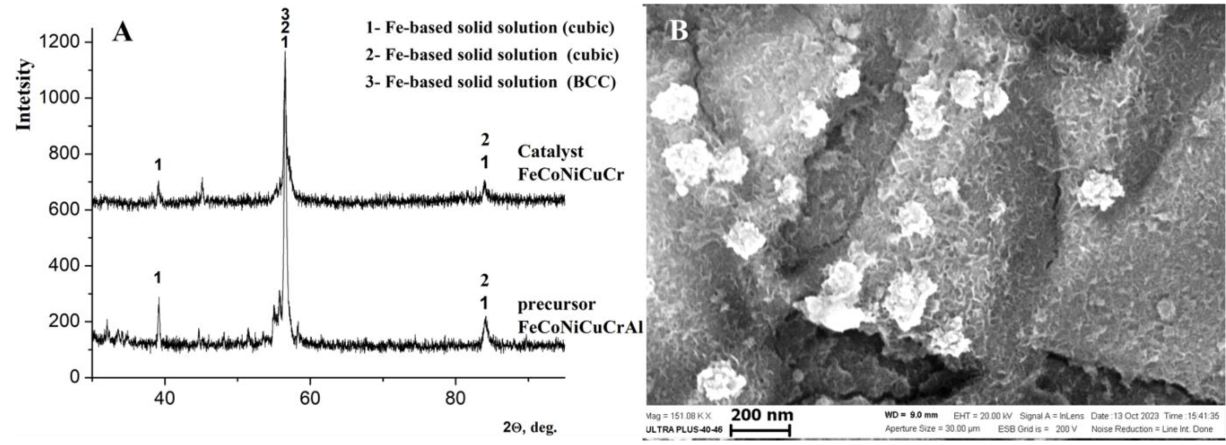

Fig. 1. A) diffraction patterns of FeCoNiCuCr catalyst and FeCoNiCuCrAl precursor, B) surface FeCoNiCuCr catalyst microphotograph

The catalytic properties were studied in the processes of hydrogenation of CO_2 on a gas mixture consists of (% vol.): $5CO_2$+$20H_2$+75He, GHSV = 3600 h^{-1}, and deep oxidation of CO and propane in a gas mixture consists of (% vol.): $0.15C_3H_8$+0.6 CO+$2.0O_2$+N_2 (up to 100%), GHSV=120,000 h^{-1}. The complete oxidation of CO occurs at a temperature of 300°C, propane conversion is 90% at 400°C. Main products of the CO_2 methanation reaction are methane and CO. The conversion of CO_2 is 39,5% at 400°C with 66% selectivity for methane. Thus, SHS-HEA open up great prospects for the creation of highly active multifunctional catalysts.

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Poster Report

Opoka supported Co–Ni and Co-Mn catalysts produced by low-temperature combustion

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Low-temperature combustion in porous media is a promising approach to synthesizing supported catalysts [1, 2]. In this work natural opoka, a silica-calcite sedimentary rock, is used as a support. Opoka was washed, calcined at 500°C and then impregnated with mix solution of Co nitrate with Ni or Mn nitrate as an oxidant and urea as a fuel in order to prepare catalyst containing 5 wt % Co +5 wt %Ni or 5 wt % Co +5 wt %Mn. After drying, a reaction that occurs in a combustion wave is initiated in the sample by heating. Temperature in the combustion wave does not exceed 325°C. The combustion products were additionally washed with 10% H_2O_2 solution to stabilize active phases. The initial opoka and catalysts were characterized by XRD, SEM, EDA, and BET methods. Specific surface area of the opoka is 21 m^2/g, after calcination 42 m^2/g and 29-40 m^2/g for catalysts.

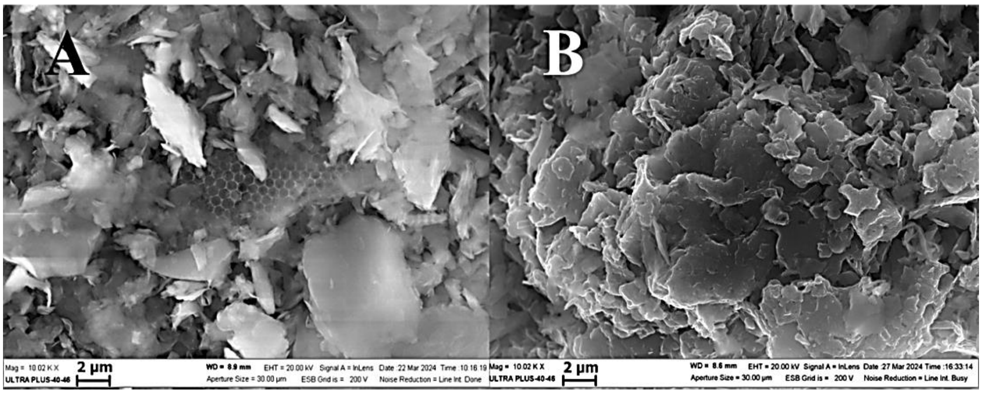

Fig. 1. SEM microphotographs: A) calcined opoka B) Co-Mn/opoka catalyst

The catalytic properties were studied in CO_2 methanation on a gas mixture consists of (% vol.): 5 CO_2+20 H_2+75 He, GHSV = 3600 h^{-1} on catalyst with Co-Ni active phase, and deep oxidation of CO and propane in a gas mixture consists of (% vol.): 0.15 C_3H_8+0.6 CO+2.0 O_2+N_2 (up to 100%), GHSV=120,000 h^{-1} on Co-Mn catalyst. The complete oxidation of CO occurs at a temperature of 540°C, propane conversion is 97%. In the CO_2 methanation reaction, the conversion of CO_2 is 49.4% at 450°C with 90% selectivity for methane.

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Poster Report

Synthesis and study of catalysts on TiB2 and TiCB supports with Ni, NiCo and CoCu active phase

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The CO_2 methanation reaction is of great interest as a promising way of CO_2 utilization. Polymetallic catalysts on carbide and boride supports can be effective in this process[1]. SHS of granular mixtures makes it possible to obtain composite materials containing titanium boride or carboboride and a metal binder. In this way, precursors for catalysts of the composition TiB_2-NiAl, TiB_2-(Ni,Co)Al, TiB_2-(Co,Cu)Al, TiCB-NiAl, TiCB-(Ni,Co)Al, TiCB-(Co,Cu)Al were synthesized. Methanation catalysts with 20 wt. % active phase were then produced by removing aluminum by leaching. The catalysts were characterized by XRD, SEM, EDS and BET methods. The specific surface area of catalysts on a TiB_2 substrate is 5-12 m^2/g, and on a TiCB substrate 3-8 m^2/g.

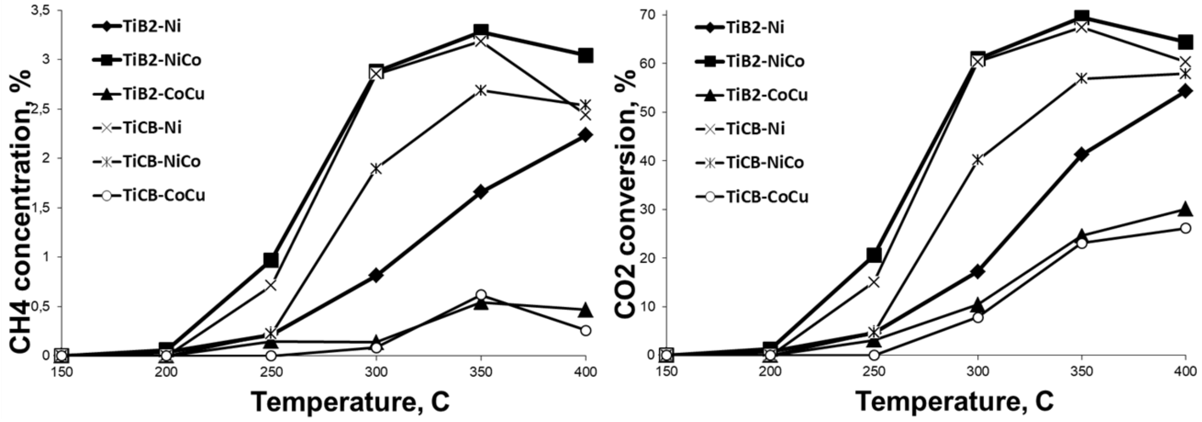

Fig.1. Dependencies of CO_2 conversion and methane yield on temperature on various catalysts.

The catalytic properties were studied in CO_2 methanation on a gas mixture consists of % vol.: $5CO_2+20H_2+75He$, GHSV = 3600 h^{-1}. The maximum conversion of CO_2 reaches 65-69% at temperature 350°C on catalysts with Ni and NiCo active phase, while the methane selectivity was 85 and 100%, respectively.

The results of catalytic tests indicate that the synthesized catalysts are promising for use in the CO2 methanation reaction.

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Poster Report

Formation, structure and phase composition layered composite materials based on TiB-Ti and TNM alloy

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Materials based on titanium aluminides are widely used as intermetallic compounds, which have a high melting point, low density, high elastic module, increasing yield strength (for TiAl) with increasing temperature, resistance to oxidation and fire, high strength/density ratio, heat resistance. But because of their poor formability, low fracture toughness, and low elongation at failure below 700 °C, their practical applicability is severely constrained. Titanium aluminides are alloyed with transition metals (Nb, Mo) and boron to enhance their mechanical and physical properties, including creep resistance, wear and crack resistance, strength, ductility, and formability. It is proposed to approach the problem of increasing the physical and mechanical characteristics of TNM alloys by creating fundamentally new layered composite materials (LCM) based on intermetallic/ceramics with improved physical and mechanical characteristics compared to world analogues.

To obtain LCM, the free SHS compression method was used. This method combines combustion processes in the self-propagating high-temperature synthesis (SHS) mode and high-temperature shear deformation [1]. The essence of this method is the compaction and molding of the synthesized material under the influence of constant low pressure (10-50 MPa) without the use of special molds, or using molds with a movable side border. This is an important advantage of the free SHS compression method when producing large-sized slabs and plates.

The goal of this work was to investigate the formability of materials with the following compositions: 100 vol % TiB-(20–40) wt % Ti, 100 vol % TNM, 30TiB/70TNM, 50TiB/50TNM, and 70TiB/30TNM under circumstances that combine shear deformation with combustion in the mode of self-propagating high-temperature synthesis, and investigate the phase composition and structural characteristics of the resultant layer composite materials.

This work was supported by the Russian Science Foundation, grant no.: 22-19-00040, https://rscf.ru/project/22-19-00040.

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Poster Report

SHS of Pyrochlore Type Ceramic Matrices for Immobilization of Actinide-Containing Nuclear Waste

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The reprocessing of spent nuclear fuel leads to the accumulation of large quantities of high-level waste the safe and long-term storage of which is represents a complex scientific and technical problem. The high toxicity of high-level waste is due to the presence in its composition of isotopes with half-lives ranging from hundreds (241Am) to tens of thousands (239Pu) and millions of years (237Np). For a long-term burial of high-level waste in the geological environment, ceramics based on analogs of rock-forming minerals with high chemical, thermal and radiation stability are being developed. One of these it is the titanate mineral pyrochlore $Y_2Ti_2O_7$. The ceramics based on $Y_2Ti_2O_7$ were synthesized by SHS densification according to the following scheme:

$Ti + Fe_2O_3 + Y_2O_3 + CaO + ZrO_2 + TiO_2 + HLW \rightarrow (Y,Ca,La,Ce)_2(TixZr1\text{-}x)_2O_7 + Fe$,

wherein HLW is a mixture of oxides simulating the behavior of the actinide-zirconium rare earth fraction of spent fuel reprocessing (wt. %): CeO_2 – 25.0; La_2O_3 – 50.7; ZrO_2 – 19.6; MnO_2 – 3.8; Fe_2O_3 – 0.9.

The microstructure of the synthesized composites based on $Y_2Ti_2O_7$ compacts was studied using a Zeiss Ultra Plus ultra-high resolution auto-emission scanning electron microscope based on the Ultra 55 with an INCA 350 Oxford Instruments X-ray microanalyser. X-ray phase analysis of the SHS products was carried out on a DRON-3 diffractometer with a monochromator on the secondary beam. The XRD patterns were recorded in step scan mode on CuK_α radiation in the range of angles $2\theta = 20° - 80°$ with a step of 0.02° and exposure of 4 seconds at one point.

It was shown that the dense matrices were produced on the basis of a phase with a pyrochlore structure of the composition $Y_2(Ti_{0.7}Zr_{0.3})_2O_7$ and Fe. The elements of the waste simulants entered the pyrochlore structure as isomorphic impurities and did not form independent phases. The resulting matrices have a cast structure and an open porosity of less than 1%. Chemical resistance tests of the resulting ceramics by leaching the matrix elements in distilled water showed good water resistance of the matrices with respect to the elements Y, Ti, Zr [1].

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Poster Report

Structural Macrokinetics of Combustion of Granular SHS Mixtures Based on Titanium

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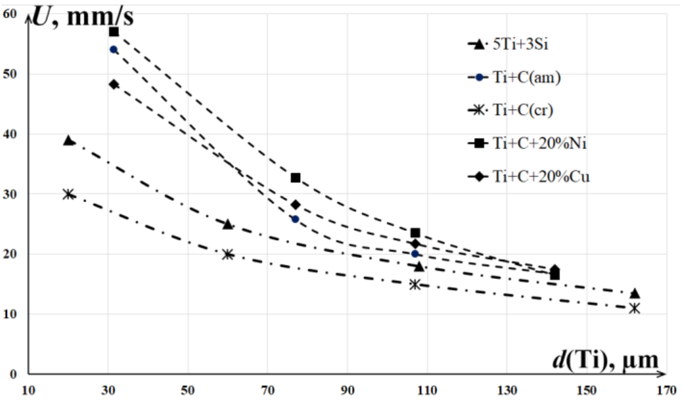
Most theoretical combustion models developed since the discovery of self-propagating high-temperature synthesis (combustion synthesis) do not take into account the release of impurity gases observed in combustion experiments [1]. A method for consideration of the influence of impurity gases on the combustion velocity of powder mixtures is proposed in the convective-conductive combustion model. Within the framework of this model, the velocity of movement of a melt layer under the action of capillary forces and the pressure difference of impurity gases in front of and behind the burning front is the observed combustion velocity [2]. This work shows how changing the structure of the initial powder mixtures 5Ti+3Si, Ti+C^{cr}, Ti+C^{am}, (Ti+C^{am})+20%Ni и (Ti+C^{am})+20%Cu (where C^{cr} is graphite and C^{am} is carbon black) by granulating them led to a change in the combustion velocity depending on the size of titanium.



The dependences of the combustion velocities of powder mixtures U on the Ti particle size behave differently and are not monotonic. The burning velocities of the studied granular mixtures, in contrast to powder mixtures, are described by the dependence $U \sim d^{-y}$, where d is a characteristic size of titanium particle, y = 0.5-0.6 for mixtures of titanium with non-metallic crystalline components, and y = 0.7-0.8 for mixtures with amorphous carbon. This is in qualitative agreement with the dependence $U \sim d^{-1}$, predicted by the convective-conductive combustion model. Predictability of the combustion mode of mixtures is critical for fast-paced scale-up synthesis processes.

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Poster Report

The Rheological Behavior of SHS Powder Compounds with Thixotropic Properties During Cold Uniaxial Pressing

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Solid-phase technology is a type of resource-saving technology used in modern materials science. It allows a significant savings in material and energy resources during the production of blanks and parts for various functional purposes. One example of this technology is the method of uniaxial cold pressing in special molds. To successfully produce high-quality compact blanks and products it is essential to study the dependence of particle packing density on applied pressure. This involves investigating the rheological characteristics of materials under dynamic loading conditions [1].

The aim of this study was to investigate the optimal pressing conditions and rheological properties of materials based on MIM-4140 steel according to ISO 22068 (an international analogue of Russian steel 38KhMA according to GOST (State Standard) 4543, which contains a wax-polyolefin polymer blend and titanium carbide, obtained using the SHS (self-propagating high-temperature synthesis) method. This type of alloy is a widely used powder material for producing feedstock for manufacturing parts using MIM (metal injection molding) technology [2].

The rheological characteristics of the sample were studied using an Instron testing machine in a uniaxial compression mode. The sample was placed in a steel mold and was pressed at a constant loading rate of 2 mm/minute. The maximum force applied to the plunger was 10 kN and the maximum pressure reached 127 MPa. After 30 seconds of loading, the sample was removed from the mold. The tests were conducted at different temperatures, ranging from room temperature to 200 °C. As a result, thixotropic behavior was observed in the material, which is a nonlinear viscoelastic response to increasing pressure. Based on the experiments, kinetic curves were obtained to determine the rheological properties of the material. These curves show the relationship between stress and strain $\sigma = f(\varepsilon)$ and the relationship between pressure on relative density $P = f(\rho_{rel})$. Finding such dependencies makes it possible to determine the optimal pressing conditions for the process of producing blanks and products from them.

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Poster Report

Self-propagating high-temperature synthesis of a Co_2B-based composite material

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According to physical properties, cobalt boride Co_2B is a refractory material. It increases the service life of metal parts when used in coatings, increases surface resistance to corrosion and wear, and is used as a catalyst for remanufacturing technologies and fuel cells [1]. Recently, cobalt boride has been considered as a promising multi-electron transfer anode material for rechargeable batteries. [2]. As a rule, composite materials based on borides are obtained by long-term exposure of precursor elements at high temperatures [3]. Currently, an alternative method for producing boride materials is the method of self-propagating high-temperature synthesis [4]. The purpose of this work is to obtain a composite material based on Co_2B by the SHS method.

The synthesis is carried out from elemental powders of cobalt, boron and titanium with molar ratios of the initial components Co:B:Ti = 15:8.75:1, theoretically calculated to obtain a material composition of 90 at.% Co_2B – 10 at.% TiB_2. Since SHS occurs at high temperatures and high rates of the process, the formation of nonequilibrium phases is possible during the synthesis. As a result of the reaction a τ-phase ($Ti_3Co_{20}B_6$) was formed in addition to the target products. The composition of the final product was calculated by the corundum number method based on the results of X-ray phase analysis: 91 at.% Co_2B – 5 at.% TiB_2 – 4 at.% $Ti_3Co_{20}B_6$.

Thus, a Co_2B-based composite material with 91 at.% of cobalt boride was obtained using the method of self-propagating high-temperature synthesis in the Co-B-Ti elemental system.

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Poster Report

Preparation of W-Cu pseudoalloy by combining of self-propagating high-temperature synthesis and infiltration

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Tungsten-copper pseudoalloy has found wide application in many industries due to its combination of high strength, high thermal conductivity, resistance to arc erosion and minimal coefficient of thermal expansion. It is known that W-Cu pseudo-alloys are produced by powder metallurgy methods - by impregnating a porous tungsten frame with copper or sintering a mixture of tungsten and copper powders at a temperature of 1250°C–1400°C in a hydrogen atmosphere or vacuum [1]. In this work, we investigated the possibility of producing a tungsten-copper pseudoalloy by infiltration using a highly exotremic combustion reaction of silicon in nitrogen as a heating source. Due to the heat released as a result of combustion, the porous tungsten frame was impregnated with molten copper. Previously [2], using this heating method, we obtained compact samples of the pseudoalloy under consideration by liquid-phase sintering. A tungsten frame with a diameter of 12 mm and a height of 6 mm was pressed to a relative density of 0.6, after which it was sintered in vacuum at a temperature of 1200 °C for 5 hours. A billet pressed from copper powder to a relative density of 0.5 was placed on a tungsten frame. Next, the entire assembly was covered with silicon powder, placed in a quartz tube and burned in a nitrogen atmosphere at a pressure of 7 MPa.

The resulting sample of the W-Cu composite is homogeneous, with a uniform distribution of tungsten particles throughout the volume of the material, defects and pores not filled with copper are practically absent and contains only two phases - tungsten and copper. The relative density of the resulting pseudoalloy was 0.9.

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Poster Report

Synthesis and investigation of novel hafnium-zirconium carbonitride ceramics: Effect of metal ratios and nitrogen on mechanical properties and oxidation behavior

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Single-phase bulk hafnium-zirconium carbides and carbonitrides with different hafnium-to-zirconium ratios (4:1, 1:1, and 1:4) were obtained by a combination of mechanical activation (MA), self-propagating high-temperature synthesis (SHS), and spark plasma sintering (SPS) methods. Analysis of the phase composition and microstructure evolution during low-energy mechanical treatment allowed for the establishment of the optimal MA regime (350 rpm, 90 min). After the treatment, nanostructured layered Hf/Zr/C composite particles with an average size of 10 to 30 μm (depending on composition) formed in the entire volume of the powder mixture. The resulting SHS-products (Hf,Zr)(C,N) consisted of particles with a “spongy” structure and rounded grains.

The study aimed to investigate the influence of the ratio of metal components and nitrogen on the mechanical properties [1] and oxidation resistance [2] of hafnium-zirconium ceramics. The incorporation of nitrogen into the crystal lattice of carbides resulted in an enhancement of mechanical properties by an average of 13%, and oxidation resistance by a factor of 2 to 3.5. The oxidation resistance of hafnium-zirconium carbides and carbonitrides decreased with the increase in Zr concentrations.

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Poster Report

Preparation of silicon carbide from silicon powders and soot

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As is known, ceramic materials based on silicon carbide have such properties as increased mechanical strength, wear resistance, inertness to radiation and chemical influences, as well as oxidation resistance [1,2]. Thanks to these characteristics, the resulting powders can be used both as individual components of structural ceramics and as composites in aggressive environments as parts of technological devices, in areas such as nuclear energy and the electronics industry [3,4]. Now progress in the development of technology for producing SiC does not stand still, due to the rapid growth in the needs of the defense, aerospace and automotive industries. Therefore the urgent problem remains the search for the most energy-efficient method for producing silicon carbide. The purpose of this work is to study the effect of different types of carbon on the phase formation of SiC.

The work used powders of taunite, carbon fiber, colloidal graphite preparation C-1, soot and silicon with a particle size of up to 40 microns. Previously the initial powders of silicon and carbon were mixed in a ball mill in a ratio of 1:1. The compacts were obtained by pressing the mixture in a cylindrical mold at a pressure of 8 MPa. Then the cylindrical samples were sintered in a vacuum furnace at a temperature of 1400 °C. The phase composition was determined on a Dron-3M diffractometer with CuKα radiation (λ = 1.54158 Å) in the angle range 20÷80 with continuous scanning.

Taunite, carbon microfiber and colloidal graphite preparation C-1 were used as the most chemically active type of carbon. As X-ray phase analysis showed all samples contain the SiC phase, however, free silicon and carbon remained in the synthesized compacts, which indicates the incompleteness of the process, although the theoretical calculation showed the possibility of 100% formation of silicon carbide. For samples where taunite, carbon fiber and preparation C-1 were taken as carbon, the average content of the β-SiC phase was calculated using the corundum number method, and it was 74.3%, 86.5% and 76.7%, respectively.

In subsequent experiments P-803 soot was used as carbon. X-ray phase analysis revealed the presence of only the SiC phase. It has been determined that the use of soot can significantly reduce the amount of free silicon and carbon and increase the SiC content to 100%.

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