



HTC 2022

10th International Conference
on High Temperature Capillarity



BOOK OF ABSTRACTS

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COTI
CONFERENCE

September 12–16, 2022, Kraków, Poland

This book contains the abstracts submitted by the participants for the **10th International Conference on High Temperature Capillarity HTC 2022**.

The professional quality of the abstracts is the authors' responsibility.

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The HTC 2022 Logo (designed by Natalia Sobczak) refers to the sessile drop method, widely used in investigations of high temperature capillarity phenomena, and to the Kraków costume (KraKowiak), as one of the most popular and recognizable Polish folk costumes.

Editors: Natalia Sobczak, Simeon Agathopoulos

Computer typesetting: Magdalena Jasińska

Cover design: Magdalena Jasińska

Publisher: Polish Foundrymen's Association

ul. Dukatów, 31-431 Kraków, Poland

www.stowarzyszenie-stop.pl

Book of Abstracts, HTC 2022

September 12–16, 2022

ISBN 978-83-963247-1-9

© 10th International Conference on High Temperature Capillarity HTC 2022, Kraków, POLAND, 2022

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WELCOME TO THE HTC 2022



Dear HTC2022 Participants,

On behalf of the Local Organizing Committee, it is my great honour, privilege, and happiness to welcome you to the 10th International Conference on HIGH TEMPERATURE CAPILLARITY (HTC2022). I am proud that this jubilee conference takes place in Krakow, one of the most important scientific, historical, and cultural centres in Poland, where we also hosted the 2nd HTC conference 25 years ago. I would also like to express my personal satisfaction that the organization of this prestigious scientific meeting has put the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences in charge.

As previous well-established HTC conferences, HTC 2022 focuses on experimental, theoretical, and applied aspects of high-temperature liquid-state materials science related to surfaces and interfaces, wetting and capillarity in materials themselves as well as in materials processing and operating at elevated temperatures.

The HTC 2022 conference brings together academic scientists, researchers, students, and industrial experts in the domain of interest worldwide. It hosts 73 participants representing 32 organizations from 15 countries (alphabetically): Bulgaria (3), China (6), France (3), Germany (5), Greece (2), Hungary (2), Israel (3), Italy (1), Republic of Korea (4), Poland (30), Spain (1), Sweden (1), Switzerland (3), Ukraine (6), and the USA (3).

The 3.5-day Program of HTC2022 is split into 12 sessions, including 11 Thematic Sessions and a Poster Session dedicated to Processing (P1-P5), Surfaces and Interfaces (S1, S2), and Wetting (W1, W2). In addition, following the tradition of HTC conferences, a special subject has been selected for a wider debate at HTC2022. This time it is about Micro- and Nanojoining (N1, N2). Among 81 submitted contributions, there are 8 invited lectures, 33 oral presentations and 40 posters.

I hope that the HTC2022 conference will provide a fruitful forum for exchanging knowledge, experience, and ideas on the latest developments and trends in the field of liquid metal engineering and liquid metal assisted processes as well as to promote cooperation between academia, research institutions, and industry.

I would like to express my deepest gratitude to all participants who shear their knowledge, expertise, and best practices as the key factors for contributing to significant discoveries in materials science and engineering. I hope you will enjoy Krakow and Poland, while your participation in the HTC 2022 will contribute to productive discussions and networking. Special thanks are directed to the Ministry of Education and Science of Poland, the World Foundry Organization, the Polish Materials Science Society, the Krakow Municipality, and industrial partners, the Energy Industries of Ohio (USA) and Netzsch (Poland), for their support to the HTC 2022 as well as to the members of the International Scientific Committee and the Local Organizing Committee for their efforts in arranging this event.

Prof. Natalia Sobczak

Corresponding member of the Polish Academy of Sciences
Chairperson of the HTC2022 conference

GENERAL INFORMATION

VENUE

NOVOTEL KRAKOW CITY WEST
11 Armii Krajowej Street, 30-150 Kraków, Poland

DATE

September 12–16, 2022

COMMITTEES

International Scientific Committee

Simeon Agathopoulos, Greece (Chairperson)
Joergen Brillo, Germany
George Kaptay, Hungary
Joonho Lee, South Korea
Taishi Matsushita, Sweden
Javier Narciso, Spain
Yuriy Plevachuk, Ukraine
Eugen Rabkin, Israel
Ping Shen, China
Natalia Sobczak, Poland
Boris Straumal, Uzbekistan
Toshihiro Tanaka, Japan
Fabrizio Valenza, Italy

Local Organizing Scientific Committee

Natalia Sobczak (Chairperson)
Przemysław Fima (Vice-chairperson)
Joanna Wojewoda-Budka (Vice-chairperson)
Agnieszka Bigos
Monika Bugajska
Marta Janusz-Skuza
Izabella Kwiecień
Maciej Szczerba
Anna Sypień
Sylwia Terlicka
Anna Wierzbicka-Miernik
Katarzyna Liszka
Anna Inglot

GENERAL INFORMATION

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Ministry of Education and Science of Poland –
financial support within Excellent Science Program
(Project no DNK/SP/515231/2021).



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within Kraków Scientific Conferences Program



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P.O. Box 31274, Independence, OH 44131, USA

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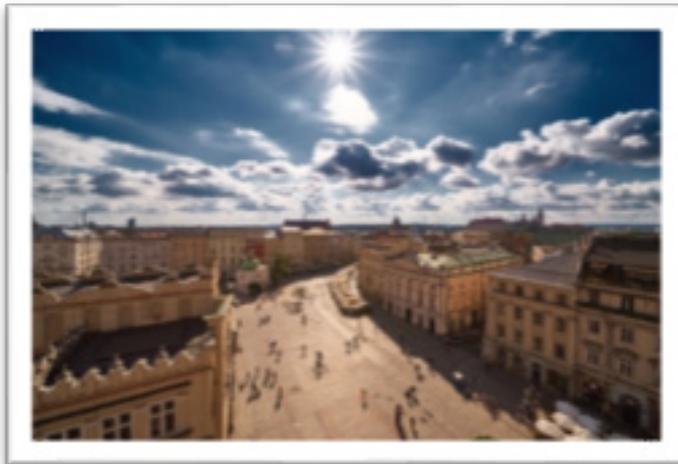
POLISH MATERIALS
SCIENCE SOCIETY

Polish Materials Science Society
8 Krasynskiego St., 40-019 Katowice, POLAND

GENERAL INFORMATION

ABOUT KRAKÓW

Due to its demographic, economic, social and scientific-cultural strength – ranks second in Poland among cities. It has unique values that are the basis of its economic development and an increase in the quality of life. It has high-quality human capital at its disposal. It is a city people consciously choose as a place to live, work, study, spend free time in a variety of ways. Sustainable development and the ability to meet specific challenges with the skillful use of own resources are the main priorities.



Fot. Piotr Krochmal



Fot. Piotr Krochmal

GENERAL INFORMATION

The academic center, with its 650 year old University, is permanently connected with the city and builds an unrepeated resource of knowledge in a unique way. It is the key to competitiveness and innovation not only of Kraków, but also of the entire region. The intensively developing economy based on knowledge is a completely new process in the economic life of the City, which makes it part of the modern economies of the world.

The overriding goal for Kraków is not only to be a modern city but also to be proud of its historical heritage. It aspires to be an open, rich, friendly and safe metropolis, vibrant with culture. Smart management and strengthening the sphere of modern services and the research and development sector are the foundations for the development of Kraków – a city where innovation and effective cooperation between science and business are the focus.

We invite you to visit our website and learn about the possibilities offered by magical Kraków – rooted in tradition, sensitive to everyday life and open to development: <https://business.krakow.pl/>.



HISTORY OF HTC CONFERENCES

The International Conferences on High Temperature Capillarity (HTC) have a long tradition. The first HTC Conference was organized in May 1994 at Smolenice castle (Slovakia) thanks to great efforts of world-class leaders in the field of high temperature capillarity, Professor Y. Naidich from Ukrainian Academy of Sciences (Kiev, Ukraine), Dr. N. Eustathopoulos from Institut National Polytechnique de Grenoble (France), and Dr. R. Sangiorgi from Istituto di Chimica Fisica Applicata dei Materiali, CNR (Genova, Italy). During the first conference, the HTC International Scientific Committee was established, consisted of seven members: N. Eustathopoulos (chairperson), Y. Naidich (Ukraine), K. Nogi (Japan), A. Mortensen (Switzerland), M. Nicholas (UK), A. Passerone (Italy), and N. Sobczak (Poland).

Traditionally, the HTC Conferences take place every 3 years around the globe (Table 1). They aim at issues related to surfaces and interfaces, wetting and capillarity in materials themselves as well as in materials processing and operating at elevated temperatures, including the following topics:

- Liquid surfaces of metals, glasses and salts,
- Surface energy and adsorption,
- Metal/metal, metal/ceramic, metal/glass and ceramic/ceramic interfaces: wettability, adhesion, interfacial reactions, segregation, grain boundary wetting and intergranular films,
- Capillarity in microgravity,
- Capillarity in nano-science and nano-technology,
- Marangoni phenomena,
- Corrosion and embrittlement by liquid metals,
- Advances in measurement techniques,
- Modelling and simulation,
- Materials processing by liquid-assisted techniques: crystal growth, metallurgical and metalcasting processes, synthesis of metal-matrix and ceramic-matrix composites, additive manufacturing, joining dissimilar materials,
- Joining dissimilar materials by liquid-assisted techniques.

Nowadays, HTC Conferences serve as a unique venue for scientists and experts to exchange their knowledge, ideas, and experience in various areas of high-temperature liquid-state materials science, particularly related to scientific, methodological, and practical aspects of liquid metal engineering.

HISTORY OF HTC CONFERENCES

Table 1. List of the International Conferences on High Temperature Capillarity

No	Chairpersons	Place	Year
HTC-1	Yu. Naidich – Institute of Materials Science Problems, Ukrainian Academy of Science; N. Eustathopoulos – Institut National Polytechnique de Grenoble	Smolenice, Slovakia	1994
HTC-2	N. Sobczak – Foundry Research Institute, Krakow	Kraków, Poland	1997
HTC-3	K. Nogi – Joining and Welding Research Institute, Osaka University	Kurashiki, Japan	2000
HTC-4	A. Passerone – Institute for Energetics and Interphases, IENI-Dept. of Genoa, National Research Council	San Remo, Italy	2004
HTC-5	E. Louis – Instituto Universitario de Materiales de Alicante (IUMA), Universidad de Alicante, Spain	Alicante, Spain	2007
HTC-6	P. Nikolopoulos – University of Patras	Athens, Greece	2009
HTC-7	W.D. Kaplan – Technion-Israel Institute of Technology	Eilat, Israel	2012
HTC-8	B. Straumal – Karlsruhe Institute for Technology; Institute of Solid State Physics, Russian Academy of Sciences	Bad Herrenalb, Germany	2015
eHTC-9	G. Kaptay, University of Miskolc	Hungary	2020
HTC-10	N. Sobczak – Institute of Metallurgy and Materials Science, Polish Academy of Sciences	Kraków, Poland	2022

Former Members of the HTC International Scientific Committee (alphabetically): Craig Carter (USA), Dominique Chatain (France), Ivan Egry (Germany), Nicolas Eustathopoulos (France), Hidetoshi Fujii (Japan), Enrique Louis (Spain), Andreas Mortensen (Switzerland), Yury V. Naidich (Ukraine), Panayiotis Nikolopoulos (Greece), Kiyoshi Nogi (Japan), Alberto Passerone (Italy), Antoni Tomsia (USA).

PROGRAM TIMETABLE



10th International Conference on HIGH TEMPERATURE CAPILLARITY HTC-2022, Kraków, POLAND September 12-16, 2022

Monday - 12 September	Tuesday - 13 September	Wednesday - 14 September	Thursday - 15 September	Friday - 16 September
	Registration: 8:00-9:00 Chairs: N. Sobczak/J. Wojewoda-Budka	Registration: 8:00-9:00 Chairs: J. Janczak-Rusch/J. Lee	Registration: 8:00-9:00 Chairs: M. Storozenko/V. Turilo	Registration: 8:00-9:00 Chairs: Yu. Plevachuk/J. Morgiel
	09:00-10:40 N1 Welcome remarks J. Janczak-Rusch J. Zimmerman M. Trybula	09:00-10:40 N2 V. Turilo Q. Zhao (on-line) O. Belhaddad A. Druzhinin (on-line)	09:00-10:40 S2 O. Dezellus B. Reiplinger M. Levi I. Jastrzębska	09:00-10:40 P4 L. Xi (on-line) U. Klotz B. Wang (on-line) S. Agathopoulos
	Coffee: 10:40-11:10 Chairs: J. Narciso/G. Kaptay	Coffee: 10:40-11:10 Chairs: T. Matsushita/A. Sypień	Coffee: 10:40-11:10 Chairs: E. Rabkin/M. Satermus	Coffee: 10:40-11:10 Chairs: S. Agathopoulos/P. Czaja
	11:10-12:30 P1 M. Caccia P. Shen (on-line) D. Geleta	11:00-12:30 W1 M. Storozenko E. Rabkin J. Lee	11:10-12:30 W2 B. Straumal F. Valenza Y. Plevachuk A. Bigos	11:10-12:10 P5 R-F. Guo (on-line) T-S. Liu (on-line) Closing remarks
	Lunch: 12:30-14:30 Chairs: F. Valenza/J. Wojewoda-Budka	Lunch: 12:30-14:30 Chairs: J. Brillo/A. Bigos	Lunch: 12:30-14:30 Chairs: J. Brillo/A. Bigos	Lunch: 12:30-14:30
	14:30-16:10 S1 J. Morgiel X. Xiao (on-line) G. Kaptay P. Czaja	14:30-16:10 P2 T. Matsushita M. Satermus J. Narciso D. Migas A. Kuś		
	Coffee: 16:10-16:40	Excursion & Conference Dinner 15:50-22:00		Coffee: 16:10-16:40
	POSTER Session and Entertainment 16:30-19:00 Meeting of International Scientific Committee	16:40-18:40 P3 R. Asthana (on-line) A. Sypień B. Yanachkov K. Janus D. Wilk-Kołodziejczyk		
Visit to IMMMS-PAS (Optional)				
Registration: 17:00-20:00 Opening: 18:00-18:20 Get together: 18:20-20:00				

INVITED SPEAKERS

JOINING OF SILICON-BASE CERAMICS AND COMPOSITES TO HIGH-TEMPERATURE ALLOYS VIA LIQUID-PHASE BONDING

Rajiv ASTHANA

*University of Wisconsin-Stout, Menomonie, Wisconsin, USA
*Currently, Faculty Fellow, NASA Glenn Research Center, Cleveland, Ohio, USA
Email: asthanar@uwstout.edu*

SYNTHESIS AND PROPERTIES OF CERMETS FOR HIGH-TEMPERATURE APPLICATIONS PRODUCED VIA SPONTANEOUS MELT INFILTRATION

Mario CACCIA

*Montana Technological University, Butte, MT, USA
Email: mcaccia@mttech.edu*

SURFACE TENSION, INTERFACIAL SEGREGATION, AND GRAPHITE SHAPE IN CAST IRONS

Olivier DEZELLUS

*LMI UMR CNRS, Villeurbanne, France
Email: olivier.dezellus@univlyon1.fr*

MATERIALS DEVELOPMENT FOR MICRO- AND NANOJOINING TECHNOLOGIES

Jolanta JANCZAK-RUSCH

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Laboratory for Joining Technologies & Corrosion
Duebendorf, Switzerland
Email: jolanta.janczak-rusch@empa.ch*

EFFECT OF LOCAL CONDITIONS ON PHASE TRANSFORMATIONS DURING REACTIVE WETTING OF MgO SUBSTRATE WITH LIQUID ALUMINIUM

Jerzy MORGIEL

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Email: j.morgiel@imim.pl*

WETTING AND INTERFACIAL REACTIVITY IN FeNiCrBSiMoC-MeB₂ SYSTEMS

Maryna STOROZHENKO

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National Academy of Science of Ukraine, Kiev, Ukraine
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ATOMISTIC MODELING OF IMMISCIBLE NANO-MULTILAYERS FOR NANOJOINING APPLICATIONS BY LIQUID-ASSISTED PROCESSES

Vladyslav TURLO

*Empa – Swiss Federal Laboratories for Materials Science and Technology
Advanced Materials Processing Team, Thun, Switzerland
Email: vladyslav.turlo@empa.ch*

CIRCUMVENTING SOLIDIFICATION CRACKING SUSCEPTIBILITY IN Al-Cu ALLOYS PREPARED BY LASER POWDER BED FUSION

Lixia X. XI

*Jiangsu Provincial Engineering Laboratory for Laser Additive Manufacturing of High-Performance Metallic Components, College of Materials Science and Technology
Nanjing University of Aeronautics and Astronautics, Nanjing, China
Email: xilixia880@nuaa.edu.cn*

SCIENTIFIC PROGRAM

Tuesday – 13 September

Micro- and Nanojoining (N1)		Chairs: S. Agathopoulos/N. Sobczak
	09:00–09:20	Welcome remarks
J. Janczak-Rusch	09:20–10:00	MATERIALS DEVELOPMENT FOR MICRO- AND NANO-JOINING TECHNOLOGIES
J. Zimmerman	10:00–10:20	THERMAL RIDGES – ‚NANOMOUNTAINS‘ IN DEFORMED NICKEL
M. Trybula	10:20–10:40	BEHAVIOUR OF GRAPHENE ON COPPER SURFACE UPON WETTING WITH LIQUID TIN
Coffee: 10:40–11:10		
Processing (P1)		Chairs: J. Narciso/G. Kaptay
M. Caccia	11:10–11:50	SYNTHESIS AND PROPERTIES OF CERMETS FOR HIGH-TEMPERATURE APPLICATIONS PRODUCED VIA SPONTANEOUS MELT INFILTRATION
P. Shen (on-line)	11:50–12:10	CONSTRUCTION OF HIGH-PERFORMANCE NACRE-INSPIRED METAL-CERAMIC COMPOSITES VIA ICE TEMPLATING AND MELT INFILTRATION
D. Geleta	12:10–12:30	EFFECT OF PROCESS PARAMETERS ON FLUIDIZATION AND AGGLOMERATION BEHAVIOR OF IRON ORE FINES
Lunch: 12:30–14:30		
Surfaces and Interfaces (S1)		Chairs: F. Valenza/J. Wojewoda-Budka
J. Morgiel	14:30–15:10	EFFECT OF LOCAL CONDITIONS ON PHASE TRANSFORMATIONS DURING REACTIVE WETTING OF MgO SUBSTRATE WITH LIQUID ALUMINIUM
X. Xiao (on-line)	15:10–15:30	STATISTICAL MODELING OF THE INFLUENCE OF OXYGEN ON THE SURFACE TENSION OF BINARY ALLOY
G. Kaptay	15:30–15:50	A COHERENT SET OF MODEL EQUATIONS FOR VARIOUS SURFACE AND INTERFACE ENERGIES IN SYSTEMS WITH LIQUID AND SOLID METALS AND ALLOYS
P. Czaja	15:50–16:10	PHASE EVOLUTION AT THE LIQUID SOLDER Sn–Zn–Ag–Al/Cu INTERFACE
Coffee: 16:10–16:30		
POSTER Session and Entertainment		
16:30–19:00		

SCIENTIFIC PROGRAM

Tuesday – 13 September

Poster Session: 16:30–19:00

Wetting

- W_01** MICROSTRUCTURAL PHENOMENA AT INTERFACES OF LIQUID Sn-Zn BASED SOLDERS WITH Ni SUBSTRATE
Aleksandra Dybel
- W_02** WETTING AND INTERFACIAL INTERACTIONS OF TiAl-X (X = Fe, Ni) ALLOYS ON ALUMINA
Fabrizio Valenza
- W_03** GRAIN BOUNDARY WETTING PHENOMENA IN THE EZ33A MAGNESIUM-BASED ALLOY
Boris Straumal
- W_04** INVESTIGATION OF WETTING PROCESSES AND INTERPHASE INTERACTION IN THE SYSTEM OF Sn - NiO POWDER
Yuriy Plevachuk
- W_05** EFFECT OF hBN COATING ON WETTABILITY AND REACTIVITY BETWEEN LIQUID MAGNESIUM AND COPPER SUBSTRATE
Jerzy J. Sobczak
- W_06** HIGH-TEMPERATURE INTERACTION OF LIQUID MAGNESIUM WITH STEEL SUBSTRATES
Paweł Darlak
- W_07** PULSED CURRENT-DRIVEN WETTING OF 3YSZ BY LIQUID COPPER AND ULTRAFAST JOINING WITH NICKEL
Ping Shen
- W_08** WETTING AND PECULIARITIES OF CONTACT INTERACTION IN THE NiTi-Cr(-Ti, Zr)B₂ SYSTEMS
Olena Poliarus
- W_09** PECULIARITIES OF CONTACT INTERACTION AND STRUCTURE FORMATION DURING WETTING OF ZIRCONIUM AND CHROMIUM DIBORIDES WITH NiAl AND NiTi INTERMETALLICS
Olena Poliarus
- W_10** IN SITU SYNTHESIS OF MeC TYPE CERAMIC PHASES IN THE ASPECT OF HIGH-TEMPERATURE WETTABILITY EXAMINATIONS
Łukasz Szymański
- W_11** WETTABILITY AND INTERFACIAL MICROSTRUCTURE OF NEW GENERATION LEAD-FREE SOLDER ALLOYS ON COPPER SUBSTRATE
Maciej Sobolewski
- W_12** WETTING OF NANOCOATINGS ON SILICON OXIDE BY METAL SOLDERS
Vitaliy Krasovskyy
- W_13** WETTING BEHAVIOUR AND REACTIVITY OF LIQUID COPPER ON 304 AND 316L STAINLESS STEELS SURFACES
Anna Wierzbicka-Miernik

SCIENTIFIC PROGRAM

- W_14** INVESTIGATION OF THE EFFECT OF HEAT EXPOSURE ON THE WETTING OF LASER BEAM TREATED ALUMINIUM SHEETS
Ferenc Tajti
- W_15** GRAIN BOUNDARY WETTING IN THE W-Ni ALLOYS
Brigitte Baretzky
- W_16** HIGH-TEMPERATURE INTERACTION OF LIQUID MAGNESIUM WITH A PURE SILVER SUBSTRATE
Sylvia Terlicka
- W_17** NEW SOLDER, BASED ON Sn-Zn EUTECTIC WITH ADDITION OF Ag, Al AND Li
Janusz Pstruś

Surfaces and Interfaces

- S_01** EXTENSION OF THE GIBBS-DUHEM EQUATION TO PARTIAL MOLAR SURFACE THERMODYNAMIC PROPERTIES OF SOLUTIONS
George Kaptay
- S_02** INVESTIGATION OF INTERFACIAL TENSION BETWEEN SILICON STEEL AND ALUMINA
Jiwoo Park
- S_03** A DIP DIVE INTO THE THERMAL AND PHYSICOCHEMICAL PROPERTIES OF COPPER SLAGS
Aleksandra Kalęba
- S_04** STRUCTURAL CHARACTERIZATION OF INTERFACES FORMED BETWEEN MOLTEN Al-Si ALLOY AND TiB₂ SUBSTRATES
Halyna Prykhodko
- S_05** REACTIVITY BETWEEN SAC305 ALLOY AND ELECTROLESS Ni-P AND Ni-P-Re COATINGS ELECTROLESS PLATED ON COPPER
Izabela Kwiecień
- S_06** COPPER/STEEL INTERACTION IN EXPLOSIVE WELDING PROCESS – IN-DEPTH ANALYSIS OF REMELTING AREAS THROUGH THE WETTABILITY TESTS
Marta Janusz-Skuza
- S_07** INTERFACIAL PHENOMENA BETWEEN GRAPHENE ON Cu SUBSTRATE COATED BY Ni, Cu, W LAYER, AND LIQUID Ga-Sn-Zn ALLOY
Tomasz Gancarz

Processing

- P_01** GRAIN BOUNDARY OXIDE LAYERS IN THE NdFeB-BASED PERMANENT MAGNETS
Brigitte Baretzky
- P_02** DETERMINATION OF HIGH-TEMPERATURE CORROSION MECHANISM OF NOVEL Cr-FREE REFRACTORY MATERIAL FOR THE COPPER INDUSTRY BY EXPERIMENTAL COUPLED WITH SIMULATION APPROACH
Ilona Jastrzębska
- P_03** MECHANICAL MIXING OF METALS DURING HIGH-PRESSURE TORSION
Brigitte Baretzky
- P_04** METAL-CERAMIC PROSTHETIC RESTORATIONS PRODUCED BY SELECTIVE LASER MELTING
Simeon Agathopoulos

SCIENTIFIC PROGRAM

- P_05** MANUFACTURE OF BRASS COMPONENT USING ADDITIVE TECHNOLOGY
Javier Narciso
- P_06** PRODUCTION OF PURE IRON STRIP SPECIMENS BY ELECTRODEPOSITION FROM A CHLORIDE BATH FOR USE IN TENSILE TESTS
Krasimir Kolev
- P_07** PHENOMENA OBSERVED AT A1050/S355J2N INTERFACE FORMED DUE TO EXPLOSIVE WELDING SUPPORTED BY AN IN-DEPTH ANALYSIS OF LIQUID ALUMINIUM WETTABILITY TEST ON S355J2N CARBON STEEL
Monika Bugajska
- P_08** FORMATION OF AN INTERFACIAL BOUNDARY WHEN BRAZING DISSIMILAR MATERIALS KOVAR - Ti(Mo)
Svitlana Maksymova
- P_09** PHASE COMPOSITION, MICROSTRUCTURE, AND SURFACE HARDNESS OF SURFACE PLASMA GAS NITRIDED Ti-10V-2Fe-3Al WITH INDIRECT PLASMATRON
Deyan Veselinov
- P_10** A STUDY ON THE STICKING PHENOMENON DURING IRON ORE REDUCTION BY HYDROUS GAS
Eunju Kim
- P_11** THE INFLUENCE OF TiC AND TiB₂ PARTICLES ON THE MICROSEGREGATION AND MICROSTRUCTURE EVOLUTION OF CAST IN718 ALLOY
Qinglong Zhao
- P_12** STUDY ON THE PHASE COMPOSITION AND MICROSTRUCTURE OF Ti-8Al-1Mo-1V AFTER SURFACE PLASMA GAS NITRIDING WITH INDIRECT PLASMATRON
Deyan Veselinov
- P_13** HIGH-TEMPERATURE SURFACE TENSION PHENOMENA IN VISCOUS GLASSES TO PRODUCE CERAMIC FOAMS
Sevasteia Maria Gkiouzel
- P_14** REACTIONS BETWEEN Al₂O₃-SiO₂ REFRACTORY CERAMICS DOPED WITH ALKALINE-EARTHS AND MOLTEN Al
Simeon Agathopoulos
- P_15** REACTIONS BETWEEN MOLTEN ARTIFICIAL SLAGS OF OXIDE GLASSES AND REFRACTORY CERAMICS OF MgO-C DOPED WITH NANO-Al₂O₃ AND TiO₂
Sevasteia Maria Gkiouzel
- P_16** REFRACTORY BEHAVIOR OF NOVEL MgO-C CERAMICS, DOPED WITH NANO-MgO, TiO₂ AND ZrO₂, TOWARDS ARTIFICIAL OXIDE SLAGS
Simeon Agathopoulos
- P_17** THE CELLULAR PRECIPITATION OF A SUPERSATURATED SOLID SOLUTION IN THE Co-32 wt.%W ALLOY AT DIFFERENT ANNEALING TEMPERATURES
Yu. O. Vronska

SCIENTIFIC PROGRAM

Wednesday – 14 September

Micro- and Nanojoining (N2)		Chairs: J. Janczak-Rusch/J. Lee
V. Turlo	09:00–09:40	ATOMISTIC MODELING OF IMMISCIBLE NANO-MULTILAYERS FOR NANOJOINING APPLICATIONS BY LIQUID-ASSISTED PROCESSES
Qinglong Zhao (on-line)	09:40–10:00	THE MICROSTRUCTURE AND TENSILE PROPERTIES OF CAST Ti64 ALLOY CONTAINING TRACE TiC NANOPARTICLES
O. Belhaddad	10:00–10:20	STUDY OF INTERFACIAL INTERACTIONS BETWEEN INDIUM BUMPS AND GOLD WETTING LAYERS FOR PHOTONIC PACKAGING APPLICATIONS
A. Druzhinin (on-line)	10:20–10:40	THE EFFECT OF INTERFACE STRESS ON THE GRAIN BOUNDARY GROOVING IN NANOMATERIALS: APPLICATION TO THE THERMAL DEGRADATION OF Cu/W NANO-MULTILAYERS
Coffee: 10:40–11:10		
Wetting (W1)		Chairs: T. Matsushita/A. Sypień
M. Storozhenko	11:10–11:50	WETTING AND INTERFACIAL REACTIVITY IN FeNiCrBSi-MoC-MeB ₂ SYSTEMS
E. Rabkin	11:50–12:10	SOLID-STATE DEWETTING OF THIN METAL FILMS ON ROUGH SUBSTRATES
J. Lee	12:10–12:30	IN SITU OBSERVATION OF DYNAMIC WETTING OF LIQUID Cu ON AN ULTRA-LOW CARBON STEEL PLATE
Lunch: 12:30–14:30		
15:50–19:00	Bus guided tour of Krakow's Old Town (the meeting point-in front of Novotel Kraków City West Hotel; return way on your own)	
19:00–22:00	Conference dinner (Szara Kazimierz Restaurant, 39 Szeroka Street, Kraków)	

SCIENTIFIC PROGRAM

Thursday – 15 September

Surfaces and Interfaces (S2)		Chairs: M. Storozhenko/V. Turlo
O. Dezellus	09:00–09:40	SURFACE TENSION, INTERFACIAL SEGREGATION, AND GRAPHITE SHAPE IN CAST IRONS
B. Reiplinger	09:40–10:00	SURFACE TENSION OF LIQUID Ti, V AND THEIR BINARY ALLOYS MEASURED BY ELECTROMAGNETIC LEVITATION
M. Levi	10:00–10:20	DIFFUSION-INDUCED RECRYSTALLIZATION DURING THE EARLY STAGES OF SOLID-STATE DEWETTING OF Ni-Pt BILAYERS
I. Jastrzębska	10:20–10:40	HIGH-TEMPERATURE INTERACTIONS BETWEEN BASIC REFRACTORY AND COPPER SLAGS USING HOT STAGE MICROSCOPY AND OTHER EXPERIMENTAL METHODS
Coffee: 10:40–11:10		
Wetting (W2)		Chairs: E. Rabkin/M. Saternus
B. Straumal	11:10–11:30	GRAIN BOUNDARY WETTING PHENOMENA IN THE HIGH ENTROPY ALLOYS
F. Valenza	11:30–11:50	ADVANCING AND RECEDING CONTACT ANGLES OF LIQUID METALS ON A TILTING PLATE
Y. Plevachuk	11:50–12:10	EFFECT OF METAL DEPOSITED NANOPARTICLES AND THE NANOPARTICLE DOPED FLUX ON SOLDER JOINTS BETWEEN LEAD-FREE SOLDERS AND METAL SUBSTRATES
A. Bigos	12:10–12:30	WETTING BEHAVIOUR AND INTERFACIAL REACTIVITY OF TIN SOLDER WITH NANOCRYSTALLINE Ni AND Ni-Mo COATINGS
Lunch: 12:30–14:30		
Processing (P2)		Chairs: F. Hodaj/A. Bigos
T. Matsushita	14:30–14:50	A MODIFIED CAPILLARY MODEL FOR THE PENETRATION OF THE NON-WETTING POROUS MEDIUM SYSTEM
M. Saternus	14:50–15:10	BENEFITS AND LIMITATIONS OF THE USE OF Pb, Sn AND Bi ALLOYING ELEMENTS IN THE HOT DIP GALVANIZING BATH
J. Narciso	15:10–15:30	REACTIVE INFILTRATION OF MoO ₃ TO PRODUCE MESOPOUROUS Mo ₂ C
D. Migas	15:30–15:50	INDUCTION VACUUM MELTING AND MICROSEGREGATION OF γ - γ' Co-BASED SUPERALLOYS
A. Kuś	15:50–16:10	PRELIMINARY STUDY OF SELECTIVE LASER MELTING PARAMETERS FOR PROCESSING CO-BASED ALLOY WITH HIGH GLASS FORMING ABILITY
Coffee: 16:10-16:40		

SCIENTIFIC PROGRAM

Processing (P3)

Chairs: B. Straumal/S. Terlicka

R. Asthana (on-line)	16:40–17:20	JOINING OF SILICON-BASE CERAMICS AND COMPOSITES TO HIGH-TEMPERATURE ALLOYS VIA LIQUID-PHASE BONDING
A. Sypień	17:20–17:40	MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF THE INTERFACIAL PHENOMENA OCCURRING IN BRAZING Ti-6Al-4V ALLOY USING TiZrCuPd AMORPHOUS RIBBONS
B. Yanachkov	17:40–18:00	SAMPLE PREPARATION FOR EXPERIMENTAL STUDIES OF THE MECHANICAL RELAXATION OF PURE IRON IN THE PRESENCE OF HYDROGEN
K. Janus	18:00–18:20	MICROSTRUCTURAL EVOLUTION STUDY DURING TEMPERING OF A HIGH-CARBON NANOBAINITIC CAST STEEL AFTER CONTROLLED THERMOMECHANICAL TREATMENT
D. Wilk-Kołodziejczyk	18:20–18:40	SELECTION OF THE CHEMICAL COMPOSITION OF INCONEL 740 CASTINGS WITH THE USE OF THE CLASSIFICATION AND NEURAL-FUZZY ALGORITHM

Friday – 16 September

Processing (P4)

Chairs: Yu. Plevachuk/J. Morgiel

L. Xi (on-line)	09:00–09:40	CIRCUMVENTING SOLIDIFICATION CRACKING SUSCEPTIBILITY IN Al-Cu ALLOYS PREPARED BY LASER POWDER BED FUSION
U. Klotz	09:40–10:00	DEVELOPMENT OF NEW ACTIVE BRAZING ALLOYS USING HIGH-THROUGHPUT THERMODYNAMIC CALCULATIONS
Bao Wang (on-line)	10:00–10:20	EFFECT OF SUB-RAPID SOLIDIFICATION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al-Mg-Si ALLOY FABRICATED BY TWIN-ROLL CASTING
S. Agathopoulos	10:20–10:40	NOVEL EFFECTIVE METHODS TO PRODUCE AlN/Al AND AlN/Cu JOINTS

Coffee: 10:40–11:10

Processing (P5)

Chairs: J. Brillo/P. Czaja

R-F. Guo (on-line)	11:10–11:30	CONSTRUCTION OF NACRE-MIMETIC Al/Al ₂ O ₃ CERAMIC-METAL COMPOSITES WITH A “BRICK-MUD” STRUCTURE VIA BIDIRECTIONAL FREEZE CASTING AND PRESSURE INFILTRATION
T-S. Liu (on-line)	11:30–11:50	ACHIEVING ULTRA-HIGH STRENGTH IN Al-Zn-Mg-Cu Alloy BY TRACE IN SITU NANOPARTICLES IMPROVED MULTISTAGE MICROSTRUCTURES

11:50–12:10 Closing remarks

12:30–14.30 Lunch

SOCIAL PROGRAM

Get together

Monday, 12th September, 2022

Novotel Krakow City West, 11 Armii Krajowej Street, Krakow, Poland

Time: 18:00

Bus guided Tour of Kraków's Old Town

Wednesday, 14th of September, 2022

Time: 15:50–19:00

Conference dinner

Wednesday, 14th of September, 2022

Szara Kazimierz Restaurant, 39 Szeroka Street, Kraków

Time: 19:00–22:00

HTC 2022 BEST POSTER AWARD

The HTC 2022 Best Poster Award of 2000 PLN (brutto) is sponsored by the Polish Materials Science Society (PTM). The winner will be selected by the HTC International Scientific Committee through evaluation of posters and voting. The winner will be announced during Conference Dinner on September 14, 2022.

POST-CONFERENCE PUBLICATIONS

Full-length cost-free post-conference papers will be published in Special Issues of:

1. Journal of Materials Engineering and Performance (Springer)
2. Archives of Metallurgy and Materials Science (Polish Academy of Sciences)
– with additional option of cost-free Open Access

Both journals are indexed in JCR. All submitted manuscripts will be reviewed following regular reviewing procedure of the selected journal.

Contact: Natalia Sobczak, n.sobczak@imim.pl

LIST OF LAUREATES

of HTC2022 scholarship for financial support to participate in HTC2022 conference within “Excellent Science Program” of the Ministry of Education and Science of Poland (Project no DNK/SP/515231/2021)

No	Participants	Affiliation	Country
1	BELHADDAD Oualid	CEA LETI	France
2	BUGAJSKA Monika	Institute of Metallurgy and Materials Science Polish Academy of Sciences	Poland
3	DARLAK Pawel	AGH University of Science and Technology	Poland
4	DRUZHININ Aleksandr	Swiss Federal Laboratories for Materials Science and Technology EMPA	Switzerland
5	DYBEŁ Aleksandra	Institute of Metallurgy and Materials Science Polish Academy of Sciences	Poland
6	GKIOUZEL Sevasteia Maria	University of Ioannina	Greece
7	JANUS Karol	Institute of Metallurgy and Materials Science Polish Academy of Sciences	Poland
8	KALEBA Aleksandra	AGH University of Science and Technology	Poland
9	KIM Eunju	Korea University	Korea
10	KOLEV Krasimir	Institute of Metal Science Equipment and Technologies with Hydro- and Aerodynamics Centre Acad. A. Balevski Bulgarian Academy of Sciences	Bulgaria
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13	LEVI Mor	Technion - Israel Institute of Technology	Israel
14	MIGAS Damian	Silesian University of Technology	Poland
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20	ZIMMERMAN Jonathan	Technion - Israel Institute of Technology	Israel

HTC 2022



10th International Conference
on High Temperature Capillarity

ABSTRACTS
THEMATIC SESSIONS

MICRO- AND NANOJOINING (N1)



HTC 2022 10th International Conference on HIGH TEMPERATURE CAPILLARITY
12 -16 September, 2022, Krakow, Poland

MATERIALS DEVELOPMENT FOR MICRO- AND NANOJOINING TECHNOLOGIES

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With the ongoing miniaturization, functionalization and diversification of microelectronic devices and sensor components, micro- and nano-joining have become key technologies for the large-scale production of integrated components. The development of nanostructured materials for these technologies and an in-depth understanding of their behaviour, in particular of surface and interface phenomena, are crucial for the progress in this field. Two classes of materials are emerging as the main joining materials for the nanojoining technology: nanoparticle-based pastes ("nanopastes") and nanomultilayers.

Non-reactive nanomultilayers (NMLs) were proposed in 2008 by Empa, Swiss Federal Laboratories for Material Science and Technology as a new class of nanojoining materials and have been intensively studied in our laboratories since then. NML fillers typically consist of alternating nanolayers (NLS; individual thickness < 10 nm) of a brazing metal or alloy (e.g. Ag, Cu, Ag-Cu) and a chemically-inert barrier material (e.g. carbon, nitride, oxide, refractory metal) and are deposited by conventional magnetron sputtering techniques. We have exploited short and fast diffusion paths along surfaces and interfaces and pre-melting phenomena in these nanostructured materials and will give an overview of our main findings [1-11].

Additionally, a brief preview of our new activities on nanopastes will be given. We have established our full-equipped nanopaste laboratory and started our research towards understanding the phenomena in metallic nanoparticle at elevated temperatures towards design of tailored nanopastes for specific joining applications.

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MICRO- AND NANOJOINING (N1)



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THERMAL RIDGES – 'NANOMOUNTAINS' IN DEFORMED NICKEL

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The surface topography of polycrystalline metals and ceramics provides a wealth of valuable information about the underlying material. This notion resulted in widespread interest in topographical characterization of surfaces in the second half of the last century. While this interest has somewhat waned in recent years, the surface of a polycrystal still has ways to surprise us. We tracked the surface topography of cold-rolled nickel during a thermal anneal at the temperature close to the recrystallization onset. While we expected to see the evolution of thermal grain boundary grooves, we found a new surface feature instead. This feature, which we name 'thermal ridge', appears as mountain range, reaching up to several hundred nanometres in height and extending along an entire grain boundary. We fully characterized these thermal ridges and studied the effects of strain and annealing parameters on their formation, and eventual decay. We devised a model, based on Mullins' classical moving grain boundary model, to mathematically describe the phenomenon. The stark similarity between thermal ridges and hillocks in thin films implies hillock formation can be partially attributed to the same factors that are responsible for the formation of thermal ridges. The primary among these factors are the grain boundary migration and strong surface anisotropy of the metal.

MICRO- AND NANOJOINING (N1)



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BEHAVIOUR OF GRAPHENE ON COPPER SURFACE UPON WETTING WITH LIQUID TIN

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Graphene is a 2D material with extraordinary properties, which make it interesting for application in broad areas [1]. Among all properties, mechanical properties are unique due to carbon atoms arranged in honeycomb lattice structure and sp^2 hybridization. The π electrons, highly delocalized in the plane of graphene, are the cause of excellent electronic properties. The presence of structural defects violates symmetry of one-atom-thick carbon layer, which influences its properties [2]. The structural defects are essential in chemical and electrochemical studies, where they act as preferential bonding sites for adsorption of atoms and molecules. Vacancy, impurity and topological defects are the most commonly detected in graphene. Up to date, there is no way to obtain defect-free graphene in larger areas through experiment, and this phenomenon influences its properties [3]. The experimental techniques used to obtain graphene on a metal surface, usually allow it to deposit more than a single layer [4].

In line with our recent discoveries [5], we consider herein a semi-flake structure of graphene with varying numbers deposited onto a pure Cu surface in contact with liquid tin. First, we present a temperature-dependent behavior of structure of semi-flake structure of graphene on copper by a combination of computational techniques. Secondly, the most stable configuration of graphene on copper surface was considered for wetting with liquid tin. Finally, behaviour of flakes during wetting has been analyzed together with their structure. Chemical and topological analyses have also been performed for both interfaces and compounds formed.

Acknowledgement

Financial support from the National Science Centre Poland, project No 2018/29/B/ST8/02558. The Authors would like to thank the Academic Computer Centre CYFRONET AGH, Poland, for providing computer resources and technical assistance.

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SYNTHESIS AND PROPERTIES OF CERMETS FOR HIGH-TEMPERATURE APPLICATIONS PRODUCED VIA SPONTANEOUS MELT INFILTRATION

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One of the most challenging environments for materials can be encountered in high-temperature heat exchangers (HEXs). HEXs are widely utilized in power generation such as concentrated solar power (CSP) or nuclear power, among other industries. In HEXs, materials are subjected to high stresses (differential pressure across the HEX), high temperatures, high temperature gradients, and often exposure to potentially corrosive fluid flow. In order to drive the efficiency of power cycles up, the working temperature of the turbines needs to be increased. This in turn, raises the operational temperature of heat exchangers, thus increasing the demands on heat exchanger materials. Metal alloys, typically used in HEXs, experience a drastic decrease in their thermomechanical properties at temperatures above 550°C [1]. In order to significantly increase power cycle efficiencies, operational temperatures must be increased beyond that and thus there is a need to develop HEXs with less conventional materials to meet these demands. Composite materials, particularly ceramic/metal composites, pose an attractive alternative to metal alloys in such applications.

Cermets, or ceramic/metal composites, combine the high toughness and high thermal conductivity of metals with the high-temperature hardness and stiffness of ceramics. Such properties make them ideal for structural applications in high-temperature environments such as power generation, friction stir welding tools, machining tools, rocket nozzles, etc. One of the main challenges for cermets in high-temperature applications is the difference in thermal expansion between the ceramic and metal phases. Especial care needs to be taken in the selection of these phases to avoid residual stresses and early failure of the material. Due to the elevated hardness of these materials, near net shape manufacturing via liquid-assisted techniques is especially attractive to avoid extensive costly machining operations.

In this work, the manufacture of several cermet systems is explored utilizing spontaneous melt infiltration (non-reactive and reactive). Thermomechanical properties as well as shape retention during processing are analyzed and related to the material's microstructure.

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PROCESSING (P1)



HTC 2022

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CONSTRUCTION OF HIGH-PERFORMANCE NACRE-INSPIRED METAL-CERAMIC COMPOSITES VIA ICE TEMPLATING AND MELT INFILTRATION

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Inspired by the fantastic “brick-and-mortar” structure and fabulous properties exhibited in nacles, we have synthesized a series of biomimetic metal-ceramic composites using a combined ice-templating and melt-infiltration technique. The morphologies and structures of the composites can be tailored by changing some intrinsic and extrinsic factors during the directional freezing of ceramic slurry and the subsequent melt infiltration. Several strategies were proposed to facilitate pressure or pressureless infiltration of molten metals into porous ceramic architectures based on wetting principle. As a consequence, defects were greatly reduced and the mechanical properties of the composites were substantially improved. The fracture behavior and toughening mechanisms of the composites were elucidated by in-situ observation of crack propagation. This study provides a good paradigm for the preparation of high-performance nacre-inspired structural materials in a simple, economical and scalable manner.



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EFFECT OF PROCESS PARAMETERS ON FLUIDIZATION AND AGGLOMERATION BEHAVIOR OF IRON ORE FINES

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Direct reduction ironmaking(DRI), on the basis of fluidized bed has gained interest of many scholars for decades due to its numerous advantages. However, the agglomeration of ore particles caused by iron whiskers created during gas-based reduction will result in de-fluidization which may stop plant operation. Besides, process parameters such as composition of reducing gas, viscosity, density, flow rate, operating temperature, particle shape and size play a great role in minimizing de-fluidization. Therefore, the effect of process parameters on de-fluidization and fluidization characteristic of iron oxide particles should be investigated. In this work, a coupled DEM–CFD (discrete element method–computational fluid dynamics) was implemented to investigate the effect of agglomeration, de-fluidization and fluidization characteristic of iron oxide particles in the fluidized bed ironmaking technology. Different reducing gas compositions and flow velocities were employed. The details of the results will be presented at the conference.

KEYWORDS: agglomeration, DEM-CFD, de-fluidization, fluidization, multiphase flow, reducing gas

SURFACES AND INTERFACES (S1)



HTC 2022 10th International Conference on HIGH TEMPERATURE CAPILLARITY
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EFFECT OF LOCAL CONDITIONS ON PHASE TRANSFORMATIONS DURING REACTIVE WETTING OF MgO SUBSTRATE WITH LIQUID ALUMINIUM

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Multicomponent ceramic materials used as shielding coating or crucibles during casting aluminium and aluminium-rich alloys often contain large share of MgO oxide. Therefore, it is the reactivity of these two dominating phases, which decide the phases durability of such protection. It was shown that the contact of the liquid aluminium with MgO substrate causes at first nucleation of the MgAl₂O₄ spinel crystallites at the oxide surface, which subsequently form a tight layer of columnar grains growing into the aluminium drop [1]. This work presents new data concerning subsequent stages taking place during reactive wetting of MgO single crystals with liquid aluminium in a vacuum.

Exceeding certain thickness, the reactively formed MgAl₂O₄ layer starts cracking enabling penetration of the liquid aluminium below original interface with numerous interpenetrating channels. They are filled with molten metal but still separated from the substrate with an instantly forming layer of spinel phase. The progress of this process eventually leaves the lengths of surface and sub-surface layers of MgAl₂O₄ immersed in liquid aluminium, i.e., cut-off from the supply of Mg from the substrate. This development facilitates the nucleation and growth of Al₂O₃ crystallites at the expense of the spinel phase, which eventually vanishes. In the next stage, ready presence of numerous adsorption sites at the faces of growing Al₂O₃ crystallites affects a change in the mechanism of dissolution of the MgO substrate, i.e., from the one involving nucleation of MgAl₂O₄ at the substrate into immediate transfer of oxygen into the expanding Al₂O₃ crystallites with Mg transferred into the Al drop and evaporating into the vacuum. The growth of the Al₂O₃, expected to be the most stable phase in the Al – Mg – O system under conditions of this study, should have stabilized the situation at the reaction front, but the SEM/TEM observations negated it.

The final stage of the wetting process of the Al/MgO couple is triggered by strong enrichment of liquid Al in Mg at the reaction front. This situation arises due to overextended distance to the drop/vacuum acting as a sink for magnesium. Exceeding critical concentration of Mg in Al stops the growth of Al₂O₃ dendrite-like crystallites and starts nucleation of MgAl₂O₄ at their sides. The growth of the spinel in this case is originally very fast due to on-site accumulation of all three necessary components. However, it eventually slows down relying on efficiency of sucking in liquid aluminium into the reaction front area as well as/and efficiency of dissolution of MgO. It shows that the reactive wetting in this system involves a unique situation, i.e., the reaction product phase which is nucleated first is soon after dissolved, only to be re-nucleated and grow later. The above result proves that the local condition, i.e., the amount of the Mg dissolved in liquid Al at the reaction front during reactive wetting of the MgO with Al is the subject of the strong changes caused by extending the diffusion path up to the drop and vacuum. The lack of any Mg at the MgO and Al interface in the thin foils analysed using TEM/EDS techniques is explained by time span between the stop of a dissolution of the MgO and temperature decrease down to that proper for solidification of aluminium, which could be used either for building-in into spinel or expelling into drop/vacuum area. Interestingly, it also shows that at high Mg concentration, Al₂O₃ is much preferable to MgO as it concerns nucleation of MgAl₂O₄ allowing for fast expansion of the reaction product zone. Nucleation of a layer of the spinel phase at the MgO, as at the beginning of the Al and MgO interaction, would practically stop the progress of the reaction front. It is the explanation of the late stages of reactive wetting of MgO with liquid Al, what represents a new look at this mechanism. The results obtained on single crystal MgO substrates are compared with available literature data on interaction between liquid Al with polycrystalline MgO ceramics.

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SURFACES AND INTERFACES (S1)



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STATISTICAL MODELING OF THE INFLUENCE OF OXYGEN ON THE SURFACE TENSION OF BINARY ALLOY

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A Langmuir-based oxygen adsorption model for binary alloys is developed from a statistical approach by correlating the fractions of oxygen allocation to a probability distribution of oxygen adsorbed by each metallic element to predict and interpret the oxygen-induced surface tension shift. The parametric model integrates a modified Langmuir equation with Bayesian inference on experimental data, updating the posterior distribution by providing physical information. The model was successfully applied to liquid Fe-Ni to predict its temperature-dependent surface tension under the influence of oxygen.

SURFACES AND INTERFACES (S1)



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A COHERENT SET OF MODEL EQUATIONS FOR VARIOUS SURFACE AND INTERFACE ENERGIES IN SYSTEMS WITH LIQUID AND SOLID METALS AND ALLOYS

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In this paper first a generally valid model is derived from the two fundamental equations of Gibbs for temperature and composition dependences of all types of interfacial energies. This general model is applied here to develop a coherent set of particular model equations for surface tension of liquid metals and alloys, for surface energy of solid metals and alloys, for high-angle grain boundary energy in metals and alloys, for solid/liquid interfacial energy in metals and alloys, for liquid/liquid interfacial energy in alloys and solid/solid interfacial energy in metals and alloys. The latter case is sub-divided into models on coherent, incoherent and semi-coherent interfaces with the same phases and with different phases on the two sides of the interface. Model parameters are given here as an example for the 111 plane of fcc metals and alloys. For other crystal planes or other crystal structures the model parameters should be adjusted, while the model equations remain the same. The method is demonstrated on various surface and interfacial energies of pure Au, on solid/liquid interfacial energy in the Al-Cu system, on different types of solid/solid interfacial energies in the Au-Ni system, on solid/solid, solid/liquid and liquid/liquid interfacial energies in the Al-Pb system and on the coherent, incoherent and semi-coherent interfacial energies between ordered and disordered fcc phases in the Ni-rich part of the Ni-Al system. The ability of this method is demonstrated to predict surface and interface transition along free surfaces and grain boundaries and also negative interfacial energies in nano-systems.

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SURFACES AND INTERFACES (S1)



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PHASE EVOLUTION AT THE LIQUID SOLDER Sn – Zn – Ag – Al/Cu INTERFACE

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Key Words: wetting, interphase, lead-free solders

Wetting phenomena concerning Sn-Zn eutectic alloys with the addition of Ag and Al were studied in contact with Cu substrate at 250, 275, 300 °C in the presence of ALU33 flux. The chosen wetting times were 5, 20, 60, 180, and 600 s. Selected, solidified solder-pad couples were cross-sectioned and subjected to SEM-EDS and TEM analyses with focus on the evolution of the interfacial microstructure. The intermetallic phases within the Cu–Zn system were identified. The kinetics of the formation and growth of the intermetallic layer were determined. The values of interfacial tension for the Sn-Zn solders with a presence of ALU 33 flux, and contact angles for those alloys with Cu substrates, were calculated based on a force versus time diagram, obtained using the wetting balance method.

Acknowledgement

This work was financed by the National Science Centre of Poland (Project 2018/29/B/ST8/02558).

MICRO- AND NANOJOINING (N2)



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ATOMISTIC MODELING OF IMMISCIBLE NANO-MULTILAYERS FOR NANOJOINING APPLICATIONS BY LIQUID-ASSISTED PROCESSES

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Understanding liquid-assisted processes is of crucial importance in the advanced manufacturing of metal-based materials, covering welding, joining, brazing, coating, and 3D printing applications. In this talk, we will discuss the recent advances made by the Modeling & Simulations team at Empa in understanding such processes, focusing on nanojoining applications of immiscible nano-multilayers. We will cover the topics of enhanced solid-state interfacial diffusion, interface premelting, as well as melting point depression of copper in nano-confinement with AlN [1]. We will show that all these different phenomena can be characterized by different orders of magnitude of in-plane diffusion coefficient, giving simple criteria for distinguishing such processes at the nanoscale. In addition, melt nucleation at the metal-ceramic interfaces can be closely followed by characterizing atomic displacements of Cu atoms, showing that the local chemistry at the interface plays an important role in such a stochastic process. Specifically, we demonstrate that only the Al-terminated interface promotes melt nucleation, while both Al- and N-terminated interfaces exhibit enhanced solid-state interfacial diffusion and reduced liquid-state interfacial diffusion compared to the bulk copper.

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MICRO- AND NANOJOINING (N2)



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THE MICROSTRUCTURE AND TENSILE PROPERTIES OF CAST Ti64 ALLOY CONTAINING TRACE TiC NANOPARTICLES

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In this work, a high-strength Ti64 cast alloy containing trace TiC nanoparticles was fabricated by adding nano-TiC/Al master alloys to the molten Ti64 alloys. The trace addition of the TiC nanoparticles (0.1 wt%) simultaneously reduced the size of the β grains, the α laths, and the α colony size of the lamellar structure during casting. The yield strength of TiC/Ti64 was increased by ~170 MPa, and the elongation of TiC/Ti64 was similar to the Ti64 alloy. TiC particles could provide nucleation sites for the metastable phases (α' , α'' , metastable β) and suppress the coarsening of the α laths during the solution treatment. The size of the weave-basketed acicular martensite phase and the aspect ratio of α phases of TiC/Ti64 alloy is smaller than those of Ti64 after heat treatment. The ultimate strength of TiC/Ti64 alloy is about 1200 MPa, and the elongation is ~6% after heat treatment.

MICRO- AND NANOJOINING (N2)



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STUDY OF INTERFACIAL INTERACTIONS BETWEEN INDIUM BUMPS AND GOLD WETTING LAYERS FOR PHOTONIC PACKAGING APPLICATIONS

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Photonic components such as micro-displays or infrared detectors are developed by 3D integration, where devices are stacked on top of each other or packaged. The electrical, optical and thermal connections are created between stacks by interconnects. One interconnection consists mainly of a solder material (e.g. indium bumps) and Under-Bump Metallization (UBM) that contains several metallic layers which serve different purposes. The contact in this configuration is ensured by wetting of the top gold layer of UBM by liquid indium and the formation of Au-In intermetallics (IMCs) at interfaces.

Increasing the resolution and thus the quality of detectors images requires an increase in the number of pixels by surface and consequently a decrease in the size of interconnections as well as the distance between them, which is called "interconnection pitch". However, this miniaturisation highlighted some phenomena that were previously negligible for larger dimensions. In particular, the increase of IMCs proportion in interconnection with pitch reduction which leads to a change in their mechanical behavior which may cause problems during manufacturing of components and/or their use. Therefore, thickness reduction of IMCs seems mandatory to reach pitches below 7.5 μm . The first step to control the intermetallic growth is to understand the phenomena that occur at the interface indium bumps/standard UBM. In this work, we first characterized the interfaces In bumps/Au pads at different pitches (30 μm , 15 μm and 7.5 μm) before and after hybridization. Thicknesses and compositions of IMCs are determined using scanning electron microscopy (SEM) coupled with energy-dispersive spectroscopy (EDS). Afterwards, growth kinetics of IMCs are determined in the temperature range 20-200°C using solid/solid and solid/liquid diffusion couples. Finally, wetting and spreading kinetics of liquid indium on solid gold are studied by using the dispensed drop method. The very early stages of wetting are followed by using a rapid camera (up to 1500 frames/s) which allows to distinguish different stages of wetting and determine the relationship between the contact angle of liquid In over unreacted solid Au and intermetallics that are formed at the liquid In/solid Au interface during the reactive wetting process.

MICRO- AND NANOJOINING (N2)



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THE EFFECT OF INTERFACE STRESS ON THE GRAIN BOUNDARY GROOVING IN NANOMATERIALS: APPLICATION TO THE THERMAL DEGRADATION OF Cu/W NANO-MULTILAYERS

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Nano-multilayers (NMLs) are functional nano-architectures, which physical properties can be tailored by smart microstructural and interfacial design. Upon thermal treatment, the layered structure of NMLs of immiscible metals degrades, forming the nanocomposite (NC) microstructure. The driving force of the degradation is of capillary nature, i.e. the system tends to decrease the energies of interfaces by wetting.

In the present work, the degradation upon thermal annealing (400 – 800 °C; duration of 100 min) of sputtered Cu/W NMLs with different nanolayer thicknesses (3, 5, 10 nm) is rationalized. It was revealed that the transition to NC starts only when the major part of initial compressive residual stresses ($-0.5 \div -3.0$ GPa for Cu; $-3.0 \div -7.0$ GPa for W) is released. The high internal stress values can be related to the large magnitude of interface stress f , which is defined as the work necessary to strain Cu/W interfaces (strong Cu{111}<-101>||W{110}<-111> texture presents in all NMLs): the calculated value is 11.25 ± 0.56 J/m². The interface stress increases the work required to create the unit of strained Cu/W interface. It was found that the magnitude of interface stress f decreases linearly to zero when temperature is raised to the onset of NML degradation (700 – 800 °C). Thus, the grooving of W/W grain boundaries is favored when the interface stress is close to zero. Kinetically, the Cu/W NML degradation process is rate-limited by the mobility of W along phase and grain boundaries.

For more details see: A.V. Druzhinin, C. Cancellieri, L.P.H. Jeurgens, B.B. Straumal: The effect of interface stress on the grain boundary grooving in nanomaterials: Application to the thermal degradation of Cu/W nano-multilayers, Scr. Mater. **199** (2021) 113866.

WETTABILITY (W1)



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WETTING AND INTERFACIAL REACTIVITY IN FeNiCrBSiMoC-MeB₂ SYSTEMS

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The wetting behavior of molten alloys in contact with ceramics is of key importance in the synthesis of metal-ceramic composites. This study aims to investigate the wetting and interfacial reactions of TiB₂, ZrB₂, CrB₂ with FeNiCrBSiMoC self-fluxing alloy in order to select the reinforcing additives for FeNiCrBSiMoC-based composite materials. The wetting behaviour of the alloy on the ceramic substrates has been studied by the sessile drop technique in a vacuum environment at 1100 °C. Structural characterization of the solidified alloy-ceramics couples was carried out using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy and electron backscatter diffraction.

The structure of original FeNiCrBSiMoC alloy consists of the precipitates of nickel borides, chromium and chromium-molybdenum carbides and carboborides, uniformly distributed in the Ni(Fe)-based matrix. Under conditions of this study, the FeNiCrBSiMoC alloy shows non-wetting behavior ($\theta = 100^\circ$) with the ZrB₂ substrate. Moreover, under the cooling the drop is separated from the ZrB₂ substrate. The FeNiCrBSiMoC alloy demonstrates good wetting with TiB₂ ($\theta = 38^\circ$) and CrB₂ ($\theta = 0^\circ$). The formation of new hard phases, such as chromium-molybdenum and chromium-iron carboborides, have been found to occur at the interface in the TiB₂-FeNiCrBSiMoC system. The CrB₂-FeNiCrBSiMoC system is characterized by intensive interfacial interaction with the formation of additional chromium boride and carboboride phases. From the analysis of experimental results it was concluded that the TiB₂-FeNiCrBSiMoC system is proved to be a promising candidate for the development of wear-resistant composite materials and coatings.



SOLID-STATE DEWETTING OF THIN METAL FILMS ON ROUGH SUBSTRATES

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We studied the initial stages of solid-state dewetting of thin nanocrystalline gold (Au) films deposited on an oxidized surface of a biomedical titanium-aluminum-vanadium (Ti-6Al-4V) alloy. The interrupted annealing technique was employed to study the evolution of holes in the film and film flattening. While a high number of dewetting holes was formed in the film at the early stages of dewetting at the temperature of 300 °C, the nanoporous but continuous film morphology remained stable even after prolonged anneals at the higher temperature of 500 °C. We developed kinetic models describing solid-state dewetting of thin metal films on rough surfaces with topographical features typical of oxidized Ti-6Al-4V. These models considered metal self-diffusion along the grain boundaries as a controlling factor of the film flattening. The stabilization of nanoporous film structure was attributed to the slowdown of the dewetting holes' expansion at the sharp edges of the substrate. Our work demonstrates that solid-state dewetting of thin metal films deposited on the rough surface of oxidized metal can be utilized for producing continuous nanoporous metal coatings.

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WETTABILITY (W1)



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12 -16 September, 2022, Krakow, Poland

IN SITU OBSERVATION OF DYNAMIC WETTING OF LIQUID CU ON AN ULTRA-LOW CARBON STEEL PLATE

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To reduce CO₂ emissions, utilization of steel scrap has been sharply increasing in recent days. Steel scrap, however, contains harmful tramp elements such as Cu and Sn, which cannot be removed in the refining process due to thermodynamic characteristics. As the contents of these tramp elements gradually increase, hot shortness, caused by the penetration of liquid Cu, becomes a severe problem in the hot rolling processes. Although hot shortness is a well-known phenomenon, it has not been investigated in detail from the viewpoint of high-temperature capillarity. This study investigated the dynamic reactive wetting of liquid Cu on the ultra-low carbon steel plate using a confocal laser scanning microscope [1-3]. The wetting angle changes were investigated, and corresponding spreading kinetics were analyzed. It was found that the viscous wetting occurred at 1085 °C, while the secondary wetting occurred at 1135 °C. The cross-sectional image analysis found that the wetting transition happened between 1085 and 1135 °C. Even when the grain boundary wetting did not occur, applying tensile stress to yield elastic strain behavior of the sample accelerated the grain boundary wetting, lowering the wetting transition temperature. The present results would be helpful in predicting the threshold condition for hot shortness in the rolling process. Applying this technique, a new rolling condition, and an alloying element to prevent the hot shortness can be searched.

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SURFACES AND INTERFACES (S2)



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SURFACE TENSION, INTERFACIAL SEGREGATION, AND GRAPHITE SHAPE IN CAST IRONS

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Using literature data on iron melts, an expression of the surface tension of cast iron melts as a function of temperature and composition was obtained. Its predictions were satisfactorily compared with reported experimental results. Then, an analysis of experimental information of sessile drop experiments with cast iron melts onto graphite substrates showed a strong adhesion between these two phases when sulfur is present and its dramatic decrease when the sulfur activity is reduced by the addition of spheroidizers such as Mg and Ce. Finally, analysis and discussion of results on the segregation of impurities and trace elements at the graphite matrix interface in cast irons led to the proposal of a scheme for their effect on graphite shape.

SURFACES AND INTERFACES (S2)



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SURFACE TENSION OF LIQUID Ti, V AND THEIR BINARY ALLOYS MEASURED BY ELECTROMAGNETIC LEVITATION

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Ti-based alloys are prime candidates for construction materials used for applications in extreme conditions and environments, due to the combination of lightweight with high-strength, high-temperature stability, large corrosion resistance as well as their biocompatibility. Alongside this rising industrial demand comes an increasing need for precise knowledge of the thermophysical properties of Ti-based alloys, as input for process optimization, phase calculation and atomic modelling.

The Ti-Al-V system is currently the most relevant alloy system when it comes to industrial application. While, now, there are some data regarding the thermophysical properties of the Ti-Al [1] side of the system, reliable and systematic data for the Ti-V system are still sparse. One of the major reasons for this is the difficult processability of liquid Ti-V at elevated temperatures, due to the high chemical reactivity of the system. Electromagnetic levitation offers a container-less measurement method for density as well as surface tension without the risk of contamination of the sample by container walls.

Density is hereby measured in a shadowgraph technique where an expanded laser is directed onto the levitating sample. The molar volume is subsequently obtained from integration over the profile edge curve of the sample shadow captured by a camera on the other side of the sample. Surface tension is measured by means of the oscillating drop technique. Here a high-speed camera records the surface oscillations of the molten droplet and the frequency spectra are evaluated according to the sum formula of Cummings and Blackburn.

The obtained density and surface tension data are analyzed by means of different thermodynamic models. In case of the density, it is found that Ti-V obeys the ideal solution model, so no excess volume needs to be considered [2]. The surface tension data can be evaluated using Butler's thermodynamic model. Again, near-perfect agreement is found only with the ideal solution model.

Even while utilizing the levitation method, contact between the melt and the surrounding atmosphere cannot be completely prevented. Oxygen, plays a crucial role for metallic melts in most applications, since already small oxygen contents can greatly influence the thermophysical properties of liquid alloys [3]. Therefore, in a second step, the influence of oxygen on the before studied thermophysical properties of the Ti-V system will be reviewed.

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SURFACES AND INTERFACES (S2)



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DIFFUSION-INDUCED RECRYSTALLIZATION DURING THE EARLY STAGES OF SOLID-STATE DEWETTING OF Ni-Pt BILAYERS

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Nanoparticles of Ni-Pt alloys attract great attention because they can replace more expensive Pt in several catalysis-related applications. The shape, size, distribution, and crystallographic orientation of such nanoparticles can affect the functional properties of the Ni-Pt nanoparticles.

We studied the solid-state dewetting and intermixing processes in Ni-Pt bilayers deposited on a sapphire substrate. We report the phenomenon of diffusion-induced recrystallization (DIR) that occurs during short anneals (shorter than 1 min) at the temperature of 950 °C [1]. These annealings resulted in heterogeneous intermixing, with the isolated intermixed regions containing randomly oriented new grains of varying concentrations being spread in the original bilayer with little intermixing. Further annealing resulted in preferential growth of {111}- and {100}-oriented grains with random in-plane orientations at the expense of the randomly textured matrix formed during DIR. A clear advantage of the {111}-oriented grains at the later stages of grain growth was observed.

Longer annealing at the temperature of 1000°C resulted in full solid-state dewetting of the bilayer and the formation of homogeneous faceted single-crystalline Ni-Pt nanoparticles. The majority of nanoparticles exhibited {111} orientation, while a minority of smaller nanoparticles had a {100} orientation. An additional ordering annealing at 550 °C reversed the distribution of the particle orientations, with the majority of small nanoparticles exhibiting the (001) orientation and L10 long-range order.

These findings demonstrate that the DIR process plays an important role in the diffusion intermixing of Ni-Pt bilayers and the final geometry of metal nanoparticles formed during solid-state dewetting.

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SURFACES AND INTERFACES (S2)



HTC 2022 10th International Conference on HIGH TEMPERATURE CAPILLARITY
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HIGH-TEMPERATURE INTERACTIONS BETWEEN BASIC REFRACTORY AND COPPER SLAGS USING HOT STAGE MICROSCOPY AND OTHER EXPERIMENTAL METHODS

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Copper is one of the most important metals among all non-ferrous metals, both in Poland and worldwide. In 2020, according to the International Copper Study Group, Poland was 13. the biggest producer of Cu worldwide in terms of copper mine production, 7. producer in smelted copper production, and 9. producer in refined copper production. Refractory materials are indispensable for the production of copper. They work as thermomechanical linings in all the heating devices for copper refining. According to statistics from the European Ceramic Industry Association, refractory materials account for 18 % in terms of the total value of the ceramic market in Europe, while 11 % of it corresponds to refractories in the non-ferrous industry.

In this work, chemical interactions between MgO-based refractory and copper slags of different chemical and phase compositions will be presented. The slag samples applied in the work derived from one stage technology of blister-copper (Cu>98.5%) production - Outkumpu technology. They are the product of Cu-Pb-Fe alloy cleaning in a copper converter.

The MgO-based refractory was tested in terms of chemical resistance against copper slags using numerous experimental methods, including hot stage microscopy, contact corrosion test, and powder corrosion test up to 1450°C. The high-temperature interactions which occurred in the test system were explored by the X-Ray Diffractometry, Scanning-Electron Microscopy coupled with an Energy Dispersive X-ray Spectrometry for examination of chemical composition in selected microareas. The results obtained were verified in terms of thermodynamic equilibria. Moreover, the SEM images were explored to obtain quantitative information on the after-corrosion materials. For this purpose, dedicated image recognition and object detection algorithms were developed and applied to assess the amount of glassy slag that penetrated the materials due to corrosion.

This work shows that all the copper slags exhibit low-melting character; PbO and CuO_x are most aggressive towards MgO-based refractory, with Cu diffusing to magnesia grains mostly. Moreover, new products are generated due to corrosion, with magnesia component, thus, MgO is most prone to interaction with copper slag.

Acknowledgment

The research was supported by the funds of the National Centre for Research and Development, Grant no. LIDER/14/0086/L-12/20/NCBR/2021 (PI: I. Jastrzębska).



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GRAIN BOUNDARY WETTING PHENOMENA IN THE HIGH ENTROPY ALLOYS

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In this talk the phenomenon of grain boundary (GB) wetting by melt and second solid phase is analyzed for multicomponent alloys without principal components (also called high-entropy alloys or HEAs) containing titanium. GB wetting can be complete or partial. In the former case, the second phase forms the continuous layers between solid grains and completely separates them. In the latter case of partial GB wetting, the second phase forms the chain of droplets in GBs, with certain non-zero contact angles. The GB wetting by the melt can be observed in HEAs produced by all solidification-based technologies like crystallization from the melt (arc or induction melting in vacuum or argon), plasma spark sintering, electric current assisted sintering, laser or plasma cladding deposition of coatings, additive manufacturing by the laser-powder bed fusion or laser-metal deposition, self-propagating high-temperature synthesis (SHS), and by brazing within the brazing joints. The phenomenon of GB wetting by the second solid phase can also be observed in HEAs produced by all solidification-based technologies. Usually, long annealing is needed to reach the thermodynamic equilibrium. However, the particle chains or continuous layers of a second solid phase can form in GBs also without the mediation of a liquid phase, for example by solid-phase sintering or coatings deposition. The GB wetting by the melt leads to the appearance of novel GB tie lines T_{wmin} and T_{wmax} in the multicomponent HEA phase diagrams. In case of GB wetting by the second solid phase, the new GB tie-lines should be considered in the two- or multiphase areas in the multicomponent phase diagrams for HEAs. The so-called grain-boundary engineering of HEAs permits the use of GB wetting to improve the HEAs' properties or, alternatively, its exclusion if the GB layers of a second phase are detrimental.

Acknowledgement

The support from the University of the Basque Country (GIU19/019 project) is acknowledged. The Institute of Solid State Physics, University of Latvia, as a center of excellence, has received funding from the European Union's Horizon 2020 Framework Programme H2020 - WIDESPREAD - 01 - 2016 - 2017 - Teaming Phase2 under grant agreement no. 739508, project CAMART2.

WETTABILITY (W2)



HTC 2022 10th International Conference on HIGH TEMPERATURE CAPILLARITY
12 -16 September, 2022, Krakow, Poland

ADVANCING AND RECEDING CONTACT ANGLES OF LIQUID METALS ON A TILTING PLATE

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The tilting plate method consists in gradually and slowly increasing the slope of the support of a sessile drop until the drop starts sliding down. At this specific moment, the contact angles measured on the downhill and uphill sides correspond to the maximum advancing and receding angles respectively.

This technique is commonly and widely adopted for room temperature liquids such as aqueous and organic solutions and it has also been used for metals which are liquid at room temperature such as Hg and Ga. However, technical issues arise when dealing with liquid metals at high temperatures.

In this work, the feasibility of the tilting plate method to measure the maximum advancing and receding contact angles in liquid metals/ceramic systems is shown and their relationship with hysteresis is discussed. Namely, liquid pure metals such as Sn, Ag, Au in contact with monocrystalline and polycrystalline Al_2O_3 (sapphire) and SiC were considered. These systems were chosen according to two main characteristics. First, the metal-ceramic couples are non-reactive; this means that neither interfacial reactivity nor dissolution of the solid into the liquid exists, which can modify the chemistry of the liquid or affect the integrity of the solid and the condition of a flat and rigid surface. Secondly, these pure metals are widely used as base metals in many high-temperature applications involving liquid metals in relative movement with a solid substrate such as brazing or casting processes, etc.



EFFECT OF METAL DEPOSITED NANOPARTICLES AND THE NANOPARTICLE DOPED FLUX ON SOLDER JOINTS BETWEEN LEAD-FREE SOLDERS AND METAL SUBSTRATES

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The Sn-based alloys are widely used as lead-free solders (LFS) in the electronics industry, and a number of studies intended for enhancement of their characteristics were carried out. Doping Co, Cu and Ni nanoparticles into flux significantly lowers the total IMC thickness of solder-substrate joints. The in-situ targeted alloying method is an effective way to control the morphology and growth of IMC layer by reactively dissolving metallic nanoparticles at solder/substrate interface. The metal and ceramic nanoaddmixtures, as well as carbon nanotubes reinforce the basic alloy and suppress the IMC growth, increase the microhardness and the wetting angle and decrease the spreading rate. Therefore, investigations of different properties of the Sn-based alloys with nano admixtures in the liquid and solid states are quite important.

The morphology and the growth behavior of the IMC which grows between Sn-based solder and Cu substrate under the influence of flux doped with the Cu, Ni and Co nanoparticles, were investigated. The influence of metal nanoparticles as a "bridge materials" between the reinforcement and solder matrix was studied. These metal nanoparticles are regarded as ideal "bridge materials" since they are apt to react with Sn-based solder alloys to form IMCs during a soldering process. For this reason, CNTs were coated with gold, copper and nickel nanoparticles. The effect of different pure CNTs and CNTs coated with metals with determined parameters on structure and structure-sensitive properties of the SAC305 and SAC387 alloys was evaluated.

The results contain necessary information on physical, mechanical and technological properties of the possible solder materials and the corresponding joints and they will serve as a background for evaluation of other physical and mechanical properties of prospective high-temperature solders.



WETTING BEHAVIOUR AND INTERFACIAL REACTIVITY OF TIN SOLDER WITH NANOCRYSTALLINE Ni AND Ni-Mo COATINGS

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The wetting behaviour of liquid tin with nanocrystalline Ni and Ni-Mo coatings, obtained by electrodeposition technique, was investigated using the classical sessile drop method to verify these layers as a barrier between Cu substrate and Sn solder, possible to be applied in the electronic interconnections. Next to the commonly used electroless processes for ENIG coatings deposition, the electroplating technique is a convenient way of coating production without problematic phosphorus inclusions and the brittle Ni₃P phase crystallization. Moreover, the introduction of the refractory metal into the nickel lattice can effectively stabilize the microstructure of alloys during the heat treatment (up to 500 °C for Ni-Mo), hindering diffusion processes and slowing down the rate of the reaction at the substrate and coating interface (1). However, investigations of the interfacial reactivity and phase evolution in the lead-free solder/electrodeposited Ni-based layers interconnection are limited.

The Ni-Mo coatings, containing 14 at.% of Mo, were obtained by the electrodeposition technique on the copper substrate, from aqueous, citrate-ammonia electrolyte solutions, under galvanostatic conditions (5 A/dm²). Additionally, Ni coatings deposited from the citrate-ammonia and commercial Watts baths were used as the reference samples. Wetting experiments were conducted by the sessile drop method, in a tubular furnace system, equipped with externally heated alumina flanged tubes, operating under vacuum conditions (10⁻⁴ Pa) (2). Coatings, covered with a 2 nm Au layer (as in ENIG coatings) with solder drop were held at the temperature of 110 °C for 3 minutes and then moved with the magnetic manipulator to the hot zone (260 °C) for 60 seconds.

The type of used electrolyte bath (selection of components, its concentration, type, size, the molecular weight of anions etc.) determines the microstructure of formed layers. Therefore, the nickel coatings deposited from the Watts solution had a columnar microstructure (consisting of grains/clusters of grains of about 500 nm) and pyramidal surface morphology (3). For coatings obtained from citrate baths, a nanocrystalline, equiaxed microstructure and smoother, globular-like surface morphology were observed. These differences determine the wettability of the coatings' surface by the molten tin. Hence, the contact angle for Ni coatings deposited from the Watts bath, with slightly higher surface roughness and coarse structure, reached higher values (32-35 °C), than that observed for the more compact Ni coating deposited from citrate-ammonia solution (23-25 °C).

In the Ni-Mo alloy/liquid tin couple, a good wetting was noted, but a pinning effect of the solder spread on the globular surface of the substrate was observed, resulting in the scatter of contact angle values in the range of 36-56 °C. However, the reactions between the alloy and the molten solder are of a different nature. Regardless of Ni coatings surface morphology and microstructure, a dissolution of Ni in liquid solder leads to the nucleation and growth of a continuous Ni₃Sn₄ intermetallic layer. The Ni-Mo alloy sublayer stabilizes the system and hinders thermally induced processes occurring at the interface. Diffusion of Ni atoms towards the solder causes a Mo content increase on the top part of the Ni-Mo coating (about 10 at.% Ni, 35 at.% Mo, and 55 at.% Sn), which may impede further atoms movement. Hence, the formation of the Ni₃Sn₄ phase is limited to the local growth of small, whiskers structures in the solder volume containing approximately 10 at.% Ni, 10 at.% Mo, and 80 at.% Sn.

Acknowledgment: This work was financed by the Ministry of Science and Higher Education of Poland as the statutory financial grant of the Polish Academy of Sciences and supported by the ERASMUS+ (staff mobility for training).

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A MODIFIED CAPILLARY MODEL FOR THE PENETRATION OF THE NON-WETTING POROUS MEDIUM SYSTEM

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In the present study, a modified capillary model to describe the penetration behaviour of molten metal into the porous medium was suggested. The suggested model is especially valid for the non-wetting system, i.e., the contact angle between the porous medium and penetrating liquid is greater than 90° and when the pressure for the penetration is relatively lower.

In material processing, it is often required to suppress or promote the penetration of molten metal. For instance, in the case of sand mould casting, it is required to suppress the penetration of molten metal into the sand mould to minimize the surface defects of casting components. On the other hand, it is required to promote the penetration of molten metal to produce the Metal Matrix Composite (MMC). It is essential to understand the penetration behaviour quantitatively to make a guideline to suppress or promote the penetration of molten metal into a porous oxide medium.

The capillary model is widely used conventionally for both room temperature and higher temperature systems to analyze the penetration behaviour. For the porous medium, which consists of solid particles such as sand mould, the equivalent pore radius (effective capillary radius) was conventionally used for the capillary model analysis. However, in the conventional studies, the equivalent radius of the capillary was often treated as constant. In the present study, the equivalent radius was reconsidered, and the change in the equivalent radius against the penetration depth was discussed for a more appropriate evaluation of the maximum penetration depth. It was explained that the equivalent pore radius decreases with increasing the penetration depth, and then the penetration is finally stopped due to the decrease of the equivalent pore radius. In other words, the critical pressure, the pressure required for the penetration, becomes higher due to the decrease of the equivalent pore radius at the deeper location. Based on the modified capillary model, a characterization method of the porous media for the change in the equivalent pore radius at the relatively lower pressure was suggested. The analysis method was applied for the different types of metals (cast iron, steel and mercury), and reasonable results for the estimation of the penetration depth were obtained.

The analytic melt penetration models usually do not include the heat exchange between the melt and sand grains when the penetration front advances in the mould. In the present study, based on the suggested modified capillary model, a finite element model of metal penetration in a sand mould was created with the aim to study the mechanisms that affect the metal penetration depth and speed. The simulation results reveal the two possible cases. In the first case, the melt penetration stops due to the penetration front reaching the solidus temperature of the penetrating metal. It happens when a relatively quick melt penetration in straight pores takes place. In the second investigated case, the melt penetration stops due to the pressure balance between the capillary pressure in the pore and the external pressure exerted by the melt. The penetration front is still molten state at a relatively high temperature. It happens at a relatively slow melt penetration in tapered pores where the capillary pressure gradually increases, thereby making the melt advance slower.

PROCESSING (P2)



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BENEFITS AND LIMITATIONS OF THE USE OF Pb, Sn AND Bi ALLOYING ELEMENTS IN THE HOT DIP GALVANIZING BATH

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Lead, bismuth and tin are currently the main group of alloying additives for zinc galvanizing baths. These metals improve the fluidity of liquid zinc, which has a positive effect on the consumption of zinc and improves the appearance of the coating. However, their presence in the bath, despite the beneficial effects, is controversial, due to the increased risk of LME. Recent studies also show that these additives may contribute to lowering the corrosion resistance of coatings. The article analyzes the existing knowledge on the alloying additions of lead, tin and bismuth to the bath, and investigates the possibility of producing coatings in an alternative bath without these additives. Therefore, this work presents the benefits and limitations of the use of these additives in the example of tests of coatings on different steels obtained by hot dip galvanizing at the temperature range of 440-460°C. The microstructure was disclosed and the chemical composition in the micro-areas of the coatings on low-silicon steel and on steel from the Sandelin range was determined.



REACTIVE INFILTRATION OF MoO₃ TO PRODUCE MESOPOUROUS Mo₂C

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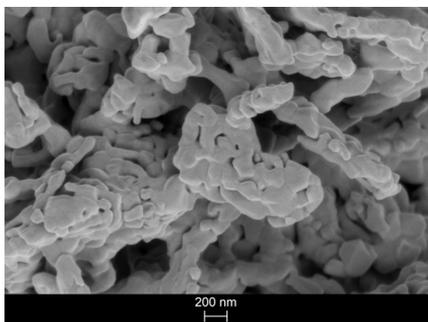
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Reactive infiltration is a well-known process that has been widely used in the manufacture of SiC, the so-called RBSC (Reaction Bonded Silicon Carbide), where liquid silicon is infiltrated into a carbon preform, giving rise to SiC [1]. Alloys have also been used in order to prepare materials with better mechanical properties, such as SiC/MSi₂ [2]. The use of oxides in a liquid state is not a common practice, only in the case that we use vitreous materials in a "liquid" state. Mo₂C is a material that is of great interest in catalysis since its catalytic activity is similar to that of noble metals, specifically it has been thought of as a substitute in some reactions where platinum is used since its surface has a similar electronic structure.

One of the advantages it presents with respect to traditional catalysts is that it does not need support (zeolites, carbon, gamma alumina,...) but rather that all the material is catalytic. The main problem is that Mo₂C has a very low surface area, about 5 m²/g, and much more is needed. In the literature there are different processes, which are typical in catalysis, such as the impregnation of a Mo precursor (ammonium molybdate, i.e.) on a carbon material, subsequently heated in an inert atmosphere between 600-900 °C, obtaining the molybdenum carbide with low crystallinity, but with an acceptable surface area [3].

In this research, a new synthetic route is proposed in which MoO₃ is used in liquid state as an infiltrating agent on a bed of activated carbon, the process has been optimized (T, t, carbon/MoO₃ ratio), and it is compared with the reaction in gaseous state. The results show that the reactive infiltration is much more effective than the reaction in the gaseous state, and that it is possible to obtain Mo₂C with a surface area of 200 m²/g, and what is more important, mesoporous.

The synthesized material has been used in various reactions proving its great catalytic power [4].



High-resolution SEM image of Mo₂C obtained by reactive infiltration

Authors acknowledge financial support from "Ministerio de Ciencia e Innovación" (PID2020-116998RB-I00).

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PROCESSING (P2)



HTC 2022 10th International Conference on HIGH TEMPERATURE CAPILLARITY
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INDUCTION VACUUM MELTING AND MICROSEGREGATION OF γ - γ' CO-BASED SUPERALLOYS

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Alloys based on the Co-Al-W system can be presented as a new class of heat-resistant materials that have become the subject of intense scientific research. The new γ - γ' Co-based superalloys were first time presented in 2006, when occurrence of γ' -Co₃(Al,W) as analogous phase to γ' -Ni₃(Al,Ti) was observed [1]. Since discovery of γ' in Co-based alloys, numerous authors have been investigating the effects of different alloying elements on the γ' stability, morphology, solvus temperature, volume fraction, lattice misfit, and high-temperature strength of the alloys. This current study shows technological aspects concerning fabrication of new γ - γ' Co-based superalloys via Vacuum Induction Melting (VIM) and gravity casting. Moreover, the characterization of microsegregation of different Co-Al-W-based alloys is included.

Firstly, the effects of the feedstock materials morphology and the method of introducing different components during smelting are analyzed. Furthermore, the impact of using solid crucibles and compacted crucibles is evaluated, and the loss of refractory mass in the smelting process is discussed. The different Co-based superalloys are analyzed in view of microsegregation during solidification. Moreover, the effect of cooling rate on microstructure and microsegregation of Co-Al-W alloys is studied.

The study revealed that γ - γ' Co-based alloys may be effectively produced by VIM technique and gravity casting. The Co-Al-W-based superalloys exhibit a proper microstructure, with low porosity and microsegregation. A decreased cooling rate increased the microsegregation of the alloys, especially in the case of Al-distribution.

Acknowledgment

This work is financed from the budgetary funds for science for the years 2018–2022, as a research project within the Diamond Grant programme (0069/DIA/2018/47).

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PRELIMINARY STUDY OF SELECTIVE LASER MELTING PARAMETERS FOR PROCESSING CO-BASED ALLOY WITH HIGH GLASS FORMING ABILITY

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Amorphous metals have been an interest of researchers since the 1960s of the XX century. For the last 5 years, interest has focused on developing amorphous metal by the Selective Laser Melting (SLM) technology. For 5 years, many alloys with high glass forming ability have been studied, mostly based on Fe and Zr. The technology of Selective Laser Melting was taken under studies for amorphous metals because its insurance a very high speed of cooling rate of a melted spot and allows to produce theoretically unlimited size of elements.

In this work, preliminary experiments of a Co-based alloy with high GFA were carried out by using SLM. Studies involve two Co-based alloys with high GFA printed from powders with SLM technology. Because Co-based alloys were never studied concerning SLM printing, the parameters for the first stage of the experiment were taken from similar research of Fe-based alloys. The matrix of different laser powers P (55, 75 and 95 W) and scanning speed v (from 600 to 1800 mm/s with a step of 200 mm/s) were tested. Constant parameters included the thickness of the layer (25 μm) and the hatch spacing (77 μm). The second stage of parameters involved the optical evaluation of the samples. The best parameter sets were exploited for the next printing with a minor step of P , v , and limiting the energy density per unit volume (ED). Namely, alloy CoBFeSiNb was printed with a laser power 45-75 W (step 5 W), scanning speed 500-800 (step 100 mm/s) and energy between 42-68 J/mm³. Alloy CoBFeTaSiCu with laser power 60-95 W (step 5 W), scanning speed 700-1200 mm/s (step 100 mm/s), ED between 41-50 J/mm³. After the second stage, the samples were examined with optical and scanning electron microscopy (SEM), X-ray diffractor (XRD) and differential scanning calorimetry (DSC). Those tests allowed to conduct the best sets of parameters for further studies by criterium of density (porosity) of samples and presents of crystal phase. The results show that the best parameters for CoBFeSiNb are 65W, 500 mm/s (67.5 J/mm³) and for CoBFeTaSiCu 70 W, 800 mm/s (48.7 J/mm³). The quality (number of cracks) of the second alloy is worse than in the first alloy. However, cracks are present in sample of both alloys. This phenomenon is known in the literature for other printed alloys. XRD analysis for CoBFeSiNb shows the crystallization peak around 44° (2 theta angle). The analysis of CoBFeTaSiCu shows several strong peaks, which means that crystallization occurred in more volume than in CoBFeSiNb.

PROCESSING (P3)



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JOINING OF SILICON-BASE CERAMICS AND COMPOSITES TO HIGH-TEMPERATURE ALLOYS VIA LIQUID-PHASE BONDING

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Vacuum-brazed silicon nitride and silicon carbide ceramics and their composites with high-temperature alloys (Inconel-625 and titanium) with and without metallic interlayers (W, Mo, Ni, and Ta) were produced and characterized for microstructure (OM, SEM, TEM), composition (EDS) and mechanical behavior (microhardness, shear strength). The effect of braze composition, and type and arrangement of the interlayers on the microstructure of each interface, composition, and mechanical behavior was investigated. The Knoop microhardness scans mimicked the interlayer arrangement and mechanical tests revealed improved shear strength in joints made using the interlayers compared to directly brazed joints without interlayers. Observations on elemental distribution, interphase formation, and mechanical behavior of the joints will be discussed in the context of prior research studies and theoretical understanding of thermal stress management in dissimilar material joints with the aid of interlayers.



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MICROSTRUCTURAL AND MECHANICAL CHARACTERIZATION OF THE INTERFACIAL PHENOMENA OCCURRING IN BRAZING Ti-6Al-4V ALLOY USING TiZrCuPd AMORPHOUS RIBBONS

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In the contemporary technology of liquid-assisted joining of dissimilar materials by brazing, interfacial phenomena strongly affect structure-properties relationships of brazed joints. In the case of titanium alloys, their thermophysical properties determine important parameters of brazing process such as temperature, time and number of cycles. The limitations in design of the technology of joining titanium alloys result from changes in the structure and properties which occur above the temperature of phase transformation $\alpha+\beta\rightarrow\beta$. The promising group of materials, which can be used as brazing materials to join Ti alloys are metallic glasses. The amorphous alloys, in comparison with crystalline metallic materials, reveal unique physical, chemical and mechanical properties due to their amorphous structure, which does not contain defects of crystalline structure. The advantage of amorphous ribbons consists in their higher chemical homogeneity even after crystallization as well as their narrow temperature range of phase transformations and melting.

The results obtained in this study allowed describing the relations between the structure of the joint and its mechanical behavior under various testing conditions (temperature, load), depending on the chemical composition, the crystallization mechanism and mechanical properties of the alloy used as a filler material. Moreover, it was shown that the amorphous Ti-Cu-Zr ribbons with the addition of palladium can be treated as a perfect candidate for filler metal to join Ti-6Al-4V alloys.

The important aspect of brazing process is wetting phenomenon of base metal by filler alloy. Wetting tests were carried out by the sessile drop method. The investigation demonstrated that with increasing temperature the contact angle is decreasing, being beneficial for wettability while the palladium addition impedes the formation of brittle and undesirable intermetallic phases during the brazing process. Furthermore, the use of amorphous foil as a bonding material without the need for flux, allowed to obtain a homogeneous, non-porous joint of high quality and strength.

The microstructure of joints was characterized by scanning electron microscopy, transmission electron microscopy and X-ray SYNCHROTRON diffraction techniques. The last method allowed for visualization of the distribution of the intermetallic phases in the volume of the material or at the interface between dissimilar materials, as well as, revealing the subtle chemical composition changes at the interfaces. To characterize the mechanical properties of the induction and resistance brazed joints were tested for shear strength using a specially designed holder for such samples.

Acknowledgement

The work is financed within the Grant no UMO-2013/11/B/ST8/04286 by the National Science Centre Poland and realized as statutory project of IMMS PAS in Krakow in years 2019-2022 in the Accredited Laboratories of IMIM PAS.

PROCESSING (P3)



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12 -16 September, 2022, Krakow, Poland

SAMPLE PREPARATION FOR EXPERIMENTAL STUDIES OF THE MECHANICAL RELAXATION OF PURE IRON IN THE PRESENCE OF HYDROGEN

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Hydrogen embrittlement (HE) is the most devastating, unpredictable and least understood, mechanism of failure in engineering components. Therefore, information on factors affecting HE of different materials is of a great practical importance. Particularly, it is needed for the selection or design of proper materials for applications as parts of metallurgical appliances to be used in high-temperature hydrogen-assisted processing of metallic components by liquid-assisted techniques. The presence of hydrogen leads to severe degradation in mechanical properties and consequently a loss of structural integrity in a vast range of metals and alloys. All metals are susceptible to HE to some extent. Even though the deleterious effect of H on the mechanical properties of metals and alloys has been studied extensively for almost one hundred years the mechanisms responsible for the embrittling process are not understood. There are considerable disagreements in the scientific literature concerning the underlying processes. The mechanisms with the most experimental and theoretical basis are Hydrogen-enhanced decohesion (HEDE) and Hydrogen enhanced localized plasticity (HELP). HEDE is thought to be caused by attenuation of interatomic cohesion forces at H-concentration sites. HELP suggests that hydrogen embrittlement is the result of increased dislocation mobility due to the presence of hydrogen. Within HELP, the fracture process is a highly localized process of plastic fracture rather than brittleness. Unlike HEDE, HELP is supported by experimental observations showing increased mobility of dislocations and the formation of localized shear bands near the top of cracks in samples with increased hydrogen content. However, the physical processes behind increased dislocation mobility have not been well studied. A deeper understanding of the HELP mechanism requires more detailed study than can be provided by phenomenology. The problem arises from the fact that the mobility of dislocations cannot be measured directly. One of the methods by which the ongoing processes can be assessed is by studying the mechanical relaxation of the metal. Even in simple, pure metal systems the practical study of the phenomenon is complicated because of the wide range of variables to be considered. During this study it was determined that the commercially available samples were varying in a wide range of properties. Therefore, significant efforts have been made to standardize them in order to increase the accuracy of the study. This includes the implementing of various processes such as annealing under argon and electropolishing with specially designed equipment.



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MICROSTRUCTURAL EVOLUTION STUDY DURING TEMPERING OF A HIGH-CARBON NANOBAINITIC CAST STEEL AFTER CONTROLLED THERMOMECHANICAL TREATMENT

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The carbon-enriched retained austenite in the steel with a nanobainitic microstructure during heating processes, e.g., in the welding process, can transform to martensite or decompose to ferrite and cementite mixture [1]. Hence, the thermal stability of the high-carbon nanobainitic steel microstructure consisting of thin plates of bainitic ferrite and retained austenite in the shape of thin films and blocks was studied by tempering cycles of 2h at temperatures ranging from 150°C to 600°C. The high-resolution dilatometry, X-ray measurements, microstructural observations using scanning and transmission electron microscopy, and hardness measurements were employed to determine the mechanism of the nanostructured microstructure decomposition during heating. After different tempering conditions, the steel microstructure changes from nanobainitic to microstructures consisting of ferrite and cementite in the case of the treatment at 600°C. X-ray results indicated that the carbides precipitation could occur along with increasing tempering temperature because the decreasing of the volume fraction of retained austenite and simultaneously reduction of the carbon content in its were noticed. Additionally, the X-ray results confirmed that the cementite is presented after tempering at 600°C. Therefore the hardness of nanobainitic steel decreases from 544 ± 9 HV5 at 150°C to 464 ± 7 HV5 at 600°C and can also be associated with losses of bainitic ferrite tetragonality, dislocation density reduction in its, and coarsening of bainitic laths.

Acknowledgment

This research was supported by The National Research and Development Centre, Poland (Project No POIR.01.01.01-00-0418/19-00).

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SELECTION OF THE CHEMICAL COMPOSITION OF INCONEL 740 CASTINGS WITH THE USE OF THE CLASSIFICATION AND NEURAL-FUZZY ALGORITHM

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The aim of the work was to compare two inference algorithms, the classification algorithm and the neural-fuzzy algorithm, for solving the problem of selection of the chemical composition of the molten alloy during metal casting process, as a factor affecting structure and properties of solidified castings. The research was performed on the example of Ni-based superalloy Inconel 740. In the first part of the work, the literature on Inconel 740 castings, classification algorithms and neural-fuzzy systems was analyzed. On this basis, a set of data was prepared, which is a key element of the analysis. Next, one classification and neural-fuzzy algorithm, each suitable for solving the problem, were selected. The second part involved the data analysis process with the use of Statistica and MATLAB computing packages. The obtained results were discussed and the testing process and its results presented in the context of all compared algorithms. An algorithm better suited to solve this problem was indicated. The results in the case of the CART tree algorithm and the mixed algorithm using the Mamdani fuzzy inference system differ slightly. The situation is different in the case of the neural - fuzzy algorithm using the Sugeno fuzzy inference system. In the relevant case, two samples numbered 1 and 3 significantly differ from the other results. The CART tree method in the analyzed case tends to overestimate the results, and the values for samples no. 3 and 5 significantly differ from the values predicted by the other algorithms. In the case of the fuzzy inference system, Mamdani and Sugeno coincide except for the sample number 1, where their values significantly differ from each other. Comparing the algorithms, taking into account each output variable, it can be concluded that in most cases, the CART tree predicts values safely and does not generate values significantly different from other algorithms. The greatest number of outliers can be seen in the case of the neural-fuzzy algorithm using the Sugeno fuzzy inference system. One of the reasons that could influence this behavior is the insufficient training database or the insufficient number of input fields to the models. The execution of descriptive statistics and then the CART tree model was a quick process, and the analysis of the obtained data took the most time. In the case of the neural-fuzzy algorithm, most of the time was spent on selecting the input parameters; so, MATLAB could count the models without memory problems. The used Sugeno inference system and the ANFIS algorithm were very helpful during the analyzes, and the process of generating the membership function and conditions was fast and did not involve much interference. The mixed algorithm was the most time consuming task because it required collecting the conditions generated in Statistica, creating scopes for each membership function, and typing the conditions into a part of the program called the Rule Viewer. Creating this algorithm required a lot of concentration and knowledge. It is worth adding that performing analyses using MATLAB is relatively more difficult than in Statistica, and is intended for users who have programming experience. In the case under consideration, it can be concluded that all the algorithms correctly predicted the values for the output variables of the models, while the algorithm using the CART tree and the mixed algorithm have similar results in each of the models.

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CIRCUMVENTING SOLIDIFICATION CRACKING SUSCEPTIBILITY IN AL-CU ALLOYS PREPARED BY LASER POWDER BED FUSION

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Laser powder bed fusion (LPBF) of Al-Cu alloys shows high susceptibility to cracking due to a wide solidification temperature range. In this work, 2024 alloys were manufactured by LPBF at different laser processing parameters. The effect of processing parameters on the densification behavior and mechanical properties of the LPBF-processed 2024 alloys was investigated. The results show that the porosity increases significantly with increasing laser power, while the number of cracks and lack-of-fusion defects increase distinctly with increasing scan speed. The solidification cracking susceptibility of the LPBF-processed 2024 alloys prepared at different processing parameters was analyzed based on a finite element model, which was accurately predicted by theoretical calculations. Dense and crack-free 2024 samples with a high densification of over 98.1% were manufactured at a low laser power of 200 W combined with a low laser scan speed of 100 mm/s. The LPBF-processed 2024 alloys show a high hardness of 110 ± 4 HV0.2, an ultimate tensile strength of 300 ± 15 MPa, and an elongation of $\sim 3\%$. This work can serve as reference for obtaining crack-free and high-performance Al-Cu alloys by LPBF.

PROCESSING (P4)



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DEVELOPMENT OF NEW ACTIVE BRAZING ALLOYS USING HIGH-THROUGHPUT THERMODYNAMIC CALCULATIONS

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Active brazing metals are used in many different industries for the brazing of metals to ceramics. An active element, which in most cases is titanium, is wetting the ceramic by the formation of an intermediate layer with good wettability for the brazing alloy. Most active brazing is based on copper, gold or Ag-Cu eutectic alloys. Precious metals containing alloy have an issue with the price while copper-based alloys show limited corrosion resistance. Both groups of alloys suffer from poor high-temperature properties and are limited by their low melting temperature.

The development of new alloys requires much experimental effort, especially if multicomponent alloys are considered. This paper describes the development of new active brazing alloys for metal-ceramic joining that are based on binary eutectic Ni-Ta or Ni-Nb alloys. However, different metal bases (steel, Ni-Base, Co-base alloys) require an adaption of the simple eutectic compositions in order to avoid the formation of brittle intermetallic phases such as Laves or μ -phase.

Therefore, a simulation-based high throughput approach was chosen to find suitable alloy compositions. Thousands of single equilibrium calculations were run by a TC-Phyton script that delivered solidus and liquidus temperature as well as the primary and secondary recrystallizing phases. The alloys were based on two ternary eutectic monovariant lines in the Ni-Nb-Ta system that are $L \Rightarrow (Ni) + Ni_3Ta$ and $L \Rightarrow Ni_3Ta + \mu$ -phase. The addition of Cr, Co, Fe, Mn and Mo was studied using ThermoCalc V2022a and the TCNI11 thermodynamic database. The calculations were evaluated according to specific selection criteria such as an optimization of the liquidus temperature and melting range. Brittle intermetallic phases, such as μ -Phase, Laves-Phase or sigma phase must be avoided to guarantee suitable mechanical properties of the joint. The evaluation was performed using Jupyter Notebooks and multivariate data analysis with the software SIMCA® by Sartorius.

Selected alloys were finally prepared by vacuum arc melting from high purity elements. The melting range was determined by DTA and DSC measurement and correlated to the simulation results. The resulting microstructure after DTA was investigated by SEM/EDX on metallographic samples. The focus was on the determination of the primary phase, its phase fraction and the composition of the phases forming the eutectic. The segregation of the alloying additions and the formation of additional phases was determined. The experimental data were correlated to the thermodynamic calculations.

In a next step, promising alloys were tested for wettability. The wetting angle on alumina samples was determined as a function of temperature and wetting time. The wetting tests will be the basis for metal-ceramic brazing trials in future.



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EFFECT OF SUB-RAPID SOLIDIFICATION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF Al-Mg-Si ALLOY FABRICATED BY TWIN-ROLL CASTING

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Traditional Al-Mg-Si strip manufacturing involves direct-chill (DC) casting and thermo-mechanical processing, which needs a lengthy follow-up procedure, making it costly and time-consuming [1,2]. The twin-roll casting (TRC), an economical and efficient technology, has attracted widespread attention [3-5]. In this work, Al-1.5Mg-3Si strips were fabricated by permanent mold casting (PMC) and vertical-type twin-roll casting (TRC), respectively. The effect of different cooling rates and solidification processes on the microstructure and mechanical properties of the alloy was studied. The sample prepared by TRC showed a heterogeneous structure composed of the columnar dendrites around the near-surface region and the near-equiaxed grains around the central region, and the TRC sample had finer grain size and eutectic structure due to the route of sub-rapid solidification (SRS) compared with the PMC sample. However, central macro-segregation occurred in the TRC strips due to the higher cooling rates and the roll force. Therefore, we eliminated the central segregation by changing the process parameters and so on.

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PROCESSING (P4)



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12 -16 September, 2022, Krakow, Poland

NOVEL EFFECTIVE METHODS TO PRODUCE AlN/Al AND AlN/Cu JOINTS

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The reliability of wide-bandgap (WBG) semiconductors used as power electronics is closely related to the high thermal conductivity of AlN-metalized substrates. Thus, the bonding of AlN ceramics with metals is a key issue for producing reliable AlN-metalized substrates. This work focuses on two AlN/metal interfaces, AlN/Al, using CuO interlayer, and AlN/Cu, using porous-copper layer and Ag foil.

(a) The method for joining Al to AlN comprises two steps: The surface of AlN is coated with a CuO thick film and sintered at 1100 °C. Then, the Al foil is put on the pretreated AlN surface and bonded to AlN through pre-heating at 400 °C and final heating at 660 °C in N₂-5%H₂ reduction atmosphere. The experimental results, obtained by optical and scanning electron microscope (SEM) observations as well as by X-ray diffraction analysis at the cross-sections of the joints and the fracture surfaces, suggest that the reaction mechanism starts with the reaction of CuO with AlN to form CuAlO₂ when the AlN, coated with CuO, is heat-treated at high temperature. A Cu layer is produced by the reduction of CuO and Cu diffuses in the Al foil, forming strong AlN/Al joints. Elongated crystals of Al₂Cu are developed in the reaction zone at the interface between AlN and Al. The peeling-off strength of the Al foil from the surface of the AlN substrate was 15.4 MPa for the AlN/Al couples produced after 30 min of heat treatment at 660 °C. The fracture strength of the produced AlN/Al joints depends on the phases formed at the interface and their microstructure.

(b) The method for producing AlN/Cu joints takes place through a film-metallization production process, which involves a porous network of Cu layer and Ag foil. The microstructure and the phases formed at the interface of the AlN/Cu joints produced at various brazing temperatures and times were analyzed by scanning electron microscopy and X-ray diffraction analysis. Strong joints with a shear strength of 48.5 MPa were produced after brazing at 850 °C for 10 min. The typical microstructure at the interfacial reaction zone was Cu / Ag(s)+Cu(s) / CuAlO₂+Al₂O₃+Cu(s) / AlN. The experimental results manifest the crucial role of the Ag-Cu eutectic liquid phase, formed by the reaction between the Cu layer and the Ag foil, and of the porous network of the Cu layer deposited on the surface of the AlN ceramic substrate, since both of them effectively favor the reduction of the residual thermal stresses in the joint, resulting in strong ceramic/metal joints.



CONSTRUCTION OF NACRE-MIMETIC Al/Al₂O₃ CERAMIC-METAL COMPOSITES WITH A “BRICK-MUD” STRUCTURE VIA BIDIRECTIONAL FREEZE CASTING AND PRESSURE INFILTRATION

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The construction of a hierarchically ordered “brick-and-mortar” structure with a high content of the “brick” phase is a key challenge in the preparation of nacre-mimetic composites. Herein, we put forward a new method to construct the fine structure of nacre, exploiting the natural phenomenon that secondary ice crystal nucleation induces the formation of transverse cracks in ceramic lamellae during the directional freeze casting. These cracks were subsequently filled with molten metal to construct “mud” in the composites. After hot pressing, a “brick–mortar” structural Al/Al₂O₃ composite with ceramic content up to 70–75 vol.% was successfully prepared. The hot-pressed composites exhibited the best bending strength and fracture toughness when the ceramic scaffolds were sintered at 1300 °C with 1.0% TiO₂ as sintering aid. The excellent fracture toughness of “brick–mortar” structural composites stemmed from the high intrinsic toughness of the continuously distributed metal matrix and crack deflection. This work provides new research data for the preparation of lightweight, high-strength and tough bionic materials for practical applications.

PROCESSING (P5)



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ACHIEVING ULTRA-HIGH STRENGTH IN Al-Zn-Mg-Cu Alloy BY TRACE IN SITU NANOPARTICLES IMPROVED MULTISTAGE MICROSTRUCTURES

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The addition of alloying element is a common method to strengthen aluminum alloy. However, too high alloying element content is bound to aggravate the element segregation in the prepared material, which not only increases the difficulty in the casting and subsequent machining deformation but also is harmful to mechanical properties. Here, the alloying element content of Al-Zn-Mg-Cu was improved based on 7075 alloy, and a new economical in-situ reaction method in Al melt was developed to introduce nanoparticles into alloy. Cast Al-Zn-Mg-Cu alloy with various Zn/Mg contents reinforced with 0.5 wt.% nanoparticles was prepared. It was found that cast Al-6.60Zn-3.15Mg-1.60Cu manipulated by 0.5 wt.% TiC-TiB₂ nanoparticles, exhibited excellent strength-plasticity synergy. The yield strength, elongation and the product of tensile strength and elongation were increased by 31.6%, 108.9% and 186.0% compared with cast 7075 alloy. Based on this research result, the Al-6.60Zn-3.15Mg-1.60Cu alloy-based composite was conducted to hot extrusion and friction stir welding (FSW) subsequently, in order to realize the fabrication of large-size Al-Zn-Mg-Cu alloy with outstanding mechanical performances. It was suggested that the manipulation of nanoparticles concurrently accelerated precipitate nucleation, promoted dislocation multiplication, increased the proportion of high-angle grain boundary and low-angle grain boundary, and increased the density of LAGB and subgrain boundary. Also, nanoparticles can effectively inhibit the abnormal grain growth during post-weld heat treatment, and then the joint was strengthened. The Al-6.60Zn-3.15Mg-1.60Cu alloy-based composite reached the yield strength of 827 MPa and maintained large plastic strain of 10.2%. The FSW-ed Al-Zn-Mg-Cu joint achieved ultra-high yield strength of 847 MPa and proper plastic strain.

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W_01

MICROSTRUCTURAL PHENOMENA AT INTERFACES OF LIQUID Sn-Zn BASED SOLDERS WITH Ni SUBSTRATE

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Wetting phenomena of Sn-Zn eutectic alloys with the addition of Ag, Al, and Li, in contact with Ni pads, were studied at 250 °C, in the presence of ALU33 flux. The chosen wetting times were 5, 20, 60, 180, and 600 s. Selected, solidified solder-pad couples were cross-sectioned and subjected to an SEM-EDS study of the interfacial microstructure. The intermetallic phases of the Ni-Zn system that formed at the solder/substrate interface were identified and gamma IMCs. The kinetics of the formation and growth of the intermetallic layer were determined. The values of interfacial tension for the Sn-Zn solders with a presence of ALU 33 flux, and contact angles for those alloys with Ni substrates, were calculated based on a force versus time diagram, obtained using the wetting balance method.

Acknowledgement

This work was financed by the National Science Centre of Poland within the project 2018/29/B/ST8/02558.

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12 -16 September, 2022, Krakow, Poland

WETTING AND INTERFACIAL INTERACTIONS OF TiAl-X (X = Fe, Ni) ALLOYS ON ALUMINA

W_02

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In this work we show how the addition of a third element, namely Fe or Ni, influences interfacial behaviour of TiAl alloys on polycrystalline alumina. Wetting was studied by the sessile drop method at 1500 °C and the interfacial microstructures were investigated so that the relationship between the contact angle and the interfacial reaction with the help of the relevant phase diagrams can be determined. The equiatomic TiAl intermetallic exhibited a contact angle of 60°; on the other hand, increasing concentrations of Fe and Ni were found to improve the wetting behaviour of ternary TiAl-X alloys with contact angles decreasing to 29° and 32° for third-element weight concentrations of 7.2.

Neither reactive-formed interlayers nor newly formed phases were observed for any binary or ternary composition. Moreover, the metal-ceramic interface after contact between TiAl and alumina did not indicate macroscopic dissolution during liquid/solid contact. Conversely, for the ternary alloys a slight dissolution of the ceramic substrate was observed.



GRAIN BOUNDARY WETTING PHENOMENA IN THE EZ33A MAGNESIUM-BASED ALLOY

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Low-temperature phase transitions in the EZ33A magnesium-based alloy have been investigated. Based on the structure assessment of the alloy after annealing at 150°C (1826 hours) and at 200°C (2371 hours) a grain boundary (GB) wetting transition by a second solid phase was documented. Within a 50°C temperature range, substantial differences in the $\alpha(\text{Mg})$ grain boundary fraction wetted by the $(\text{Mg,Zn})_{12}\text{RE}$ intermetallic were observed. In contrast to what was reported in the literature, two different types of precipitates were found within $\alpha(\text{Mg})$ grains. With increasing annealing temperatures, both types of precipitates dissolve.

The wetting phase transition of grain boundary and their triple junctions (GB TJ) by the second solid phase in the EZ33A alloy was also studied. The condition for complete wetting for the GB TJ ($\sigma_{\text{GB}} > \sqrt{3} \sigma_{\text{SS}}$) is weaker than for GBs ($\sigma_{\text{GB}} > 2 \sigma_{\text{SS}}$). Therefore, if the transition from partial to complete wetting occurs with increasing temperature, then all GB TJs should become completely wetted at a temperature T_{wTJ} , which is lower than the temperature T_{wGB} , at which all GBs become completely wetted. For the first time, it was experimentally found that GB TJs are completely wetted at $T_{\text{wTJ}} = 380 \pm 10^\circ\text{C}$, which is approximately 70°C lower than $T_{\text{wGB}} = 450 \pm 10^\circ\text{C}$. The wetting phase at the grain boundaries is the intermetallic compound $(\text{Mg,Zn})_{12}\text{RE}$. A similar phenomenon was previously observed for GB TJ wetting with a liquid phase [Acta Mater. **56**, 925 (2008)].



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INVESTIGATION OF WETTING PROCESSES AND INTERPHASE INTERACTION IN THE SYSTEM OF Sn - NiO POWDER

W_04

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Wettability of powders with liquid metal plays a significant role in many technologies, such as coatings, dispersions, powder processing and other practical uses. Such significance has driven to development of ways to evaluate the wettability of powders with liquids. Typically, the wettability of solid surfaces is estimated via measuring the wetting angle formed by the liquid under study on a certain solid surface.

One of the most common methods of measuring the wetting angle is the sessile drop method, when a drop of liquid is placed on the surface and the wetting angle is measured directly at the contact boundary of the three phases (liquid, solid and vapour). For powder samples, this method requires compacting the powder into the disk using high pressure and then measuring the wetting angle by the fluid under investigation. Another way to use the sessile drop method is to deposit a layer of investigated powder on the substrate and then measure the wetting angle.

These two methods were used to study the wetting of nickel and nickel oxide by liquid tin in this work. The wetting process was investigated in the temperature range of 450-1250 K. It was established that below the temperature of 1034 K the tin does not wet the pressed nickel oxide, and at the temperature of 1034 K there is an intense chemical interaction of nickel oxide and tin. The uncompressed powder of nickel oxide is not wetted with tin up to a temperature of 1000 K. With further heating, the wetting angle sharply decreases.

The result of the interaction of liquid tin with nickel oxide was investigated by X-ray diffraction and scanning electron microscopy methods.

In the case of interaction of liquid tin with a compacted substrate of nickel oxide in the temperature range from the melting point to 750 K, the process of dewetting the oxide surface is observed. In the process of interaction of tin with a block of nickel oxide particles at a temperature of 1025 K, the process of spontaneous infiltration of tin into this block was observed.



EFFECT OF hBN COATING ON WETTABILITY AND REACTIVITY BETWEEN LIQUID MAGNESIUM AND COPPER SUBSTRATE

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In liquid-assisted processes of metallic components, liquid alloys have a direct contact with refractory metallurgical appliances. Therefore, information on high-temperature interaction between molten Mg and its alloys with different solid materials is of practical importance. Particularly, special attention should be paid to selecting suitable materials for the crystallizer in directional solidification of different alloys because such materials should not only be non-wetting and non-reactive in contact with molten alloys but also they should have high thermal conductivity.

This research is focused on understanding factors affecting high-temperature interaction between liquid pure Mg and solid Cu that was selected as a material for the crystallizer in directional solidification of Mg alloys because of high thermal conductivity of Cu. The sessile drop method, combined with non-contact heating and capillary purification of the Mg drop from a native oxide film, was applied for real time observation and recording of wetting behavior of liquid Mg on Cu substrates. The sessile drop tests were performed in Ar (99.999 wt.%) or Ar+5%H₂ protective atmosphere at the test temperature of 700 °C. Two types of Cu substrates were used: (1) freshly polished and (2) additionally coated with hBN layer by spaying from the hBN slurry. After wettability tests, the detailed structural characterization of the solidified Mg/Cu and Mg/hBN/Cu couples was performed by light microscopy (LM), scanning electron microscopy (SEM) and energy dispersive X-ray spectrometry (EDS).

Under conditions of this study, liquid pure Mg showed very good wetting of uncoated Cu substrate. For this substrate, LM/SEM/EDS analysis evidenced reactive wetting mechanism caused by the dissolution of the Cu substrate in the Mg drop and reactively formed continuous layer of intermetallic compound at the Mg/Cu interface. On the contrary, the hBN-coated Cu substrate showed non-wetting and non-reactive behavior in contact with liquid pure Mg.

Acknowledgement

This research was financially supported by the National Science Centre of Poland within OPUS 16 funding scheme, Project, no. 2018/31/B/ST8/01172 in the years 2019–2022.



HIGH-TEMPERATURE INTERACTION OF LIQUID MAGNESIUM WITH STEEL SUBSTRATES

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Information on the interaction of liquid magnesium and its alloys with different refractory materials is of practical importance in many liquid-assisted processings of Mg-based components.

In this study, high-temperature sessile-drop wettability tests were conducted with pure Mg on two types of steel substrates. The tests were performed at a temperature of 700°C in a protective atmosphere (Ar + 5%H₂) using non-contact heating combined with capillary purification of Mg drop from a native oxide film directly at the test temperature in an experimental chamber by squeezing the drop from a graphite capillary placed above steel substrates.

The high-temperature behavior of Mg/substrate couples was recorded with a high-resolution, high-speed CCD camera and collected images were used for measurements of the contact angle values formed between the liquid Mg and selected substrates. The solidified Mg/substrate couples were subjected to detailed microstructural observations by light microscopy (LM), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDS).

LM, SEM and EDS analysis used for structure and chemistry characterization of interfaces in Mg/substrate couples revealed the effects of morphology, quantity, and distribution of the graphite phase in steel used in this study on the wetting behavior of liquid Mg and its bonding with selected substrates.

The obtained results were analyzed from the point of view of the suitability of steel for metallurgical appliances in liquid-assisted processing of Mg-based components.

Acknowledgment

This research was financially supported by the National Science Centre of Poland within OPUS 16 funding scheme, Project no. 2018/31/B/ST8/01172 in the years 2019–2023.

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12 -16 September, 2022, Krakow, Poland

W_07

PULSED CURRENT-DRIVEN WETTING OF 3YSZ BY LIQUID COPPER AND ULTRAFAST JOINING WITH NICKEL

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A prerequisite for metal-ceramic bonding by brazing is good wettability between the brazing filler and the ceramic. However, ceramics such as ZrO_2 are usually not wetted by liquid metals due to the large difference in physicochemical properties between them. In this work, we present a method based on electrochemically-driven wetting and bonding at the metal/zirconia interface. A current-coupled sessile-drop device was set up to study the wetting behavior of pure Cu droplets on 3 mol% Y_2O_3 -doped ZrO_2 (3YSZ) substrates at 1373 K under the action of pulsed currents. The wettability was significantly improved within a few seconds to tens of seconds after the application of the pulsed current. Moreover, the robust joining of 3YSZ to Ni was achieved in an ultrashort time (≤ 1 min) using pure Cu as the brazing filler. The changes in the interfacial microstructure and shear strength of Ni/Cu/3YSZ joints under different processing parameters (duty cycle, energization time, frequency) were examined, and their relationships were revealed. This work provides a novel, effective and scalable strategy for the regulation of interfacial wetting and bonding between oxygen ion-based electrolyte ceramics and metals.



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WETTING AND PECULIARITIES OF CONTACT INTERACTION IN THE NiTi-Cr(Ti, Zr)B₂ SYSTEMS

W_08

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Today the problem of protection of shipbuilding and hydropower equipment parts as well as of oil and gas producing floating platforms from the action of aggressive media, in particular sea water, is urgent.

Special attention is paid to the development of high-efficiency devices for utilization of the wave energy. In addition to the assurance of the produced signal stability, such devices must withstand the destroying action of aggressive media as well as the cavitation and hydroabrasive wear, providing failure-free operation of the system as a whole, in other words, it must possess high wear and corrosion resistance in water media.

One of the actual and promising trends is the development of novel composite powder materials on the basis of intermetallics NiAl and NiTi for deposition of such protective coatings, which, on the one hand, exhibit high resistance to electrochemical corrosion in sea water and, on the other hand, withstand the cavitation wear.

Wetting and peculiarities of contact interaction in the NiTi-CrB₂, NiTi-TiB₂ and NiTi-ZrB₂ systems have been studied. It was established that process of the intermetallic spreading over the surface of a diboride sample is the most intense for chromium diboride: herein minimal contact angles are formed. Examination of the microstructure and chemical composition of interaction zones revealed that in the NiTi-CrB₂ and NiTi-ZrB₂ systems a new boride phase, TiB₂, is formed owing to the chemical interaction between the starting components. The most promising refractory addition in creating NiTi-based composite materials is considered to be chromium diboride.



PECULIARITIES OF CONTACT INTERACTION AND STRUCTURE FORMATION DURING WETTING OF ZIRCONIUM AND CHROMIUM DIBORIDES WITH NiAl AND NiTi INTERMETALLICS

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Composite materials based on NiAl intermetallide are intended for high-temperature applications including friction units. Composites based on NiTi intermetallide using solve the problem of protection of shipbuilding and hydropower equipment parts from the action of aggressive media. For developing new composite materials, special attention is paid to studying the physical and chemical compatibility of the initial components. In this work wettability in NiAl (NiTi)-ZrB₂, NiAl (NiTi)-CrB₂ systems was investigated. Wetting kinetics were studied and wetting contact angles were determined.

It was established that when zirconium diboride is wetted with NiAl intermetallic the contact angle $\theta = 20^\circ$ is formed. The NiAl-CrB₂ system is characterized by complete spreading of the metal drop with the formation of zero contact angles.

When zirconium diboride is wetted with NiTi alloy, the contact angle of wetting is $\theta = 33^\circ$, and chromium diboride is $\theta = 5^\circ$. Spreading of NiAl and NiTi intermetallics for all studied systems took place in a vacuum for 12 min at $T = 1670^\circ\text{C}$ and $T = 1350^\circ\text{C}$, respectively.

The microstructure, phase analysis, chemical composition of contact zones after wetting were investigated using Scanning Electron Microscopy. With the help of a Transmission Electron Microscope, the features of phase and structure formation of interaction zones will be studied.

It was established that the NiAl-CrB₂ system is characterized by the formation of a new Ni_{0.5}Cr_{1.5}B₃ boride phase. In the NiTi-CrB₂ and NiTi-ZrB₂ systems, as a result of chemical interaction between the initial components, titanium boride grains are formed, which can serve as additional strengthening elements of the composite. It should be noted that a new Ni₃Ti intermetallic phase is formed from the original NiTi alloy (as a result of its depletion by titanium during the formation of TiB₂ grains).

The formation of new additional boride phases will have a positive impact on the development of wear-resistant materials and coatings based on them.



IN SITU SYNTHESIS OF MeC TYPE CERAMIC PHASES IN THE ASPECT OF HIGH-TEMPERATURE WETTABILITY EXAMINATIONS

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Metal-matrix composites (MMCs) based on Fe alloys reinforced with ceramic particles are characterized by good mechanical properties and high wear resistance [1]. The manufacturing of local composite zones and layers reinforced with TiC particles is being extensively discussed in many scientific studies. The process of producing metal-ceramic composites reinforced by TiC particles can be performed by ex situ and in situ techniques [2]. Titanium carbide is characterized by: high hardness, high melting point, thermodynamic and chemical stability, as well as high wettability in Fe-based alloy at high temperatures and different atmospheres [3]. The technological processes of producing metal matrix composites (MMCs) are conditioned by obtaining the relevant bonding at the interface between a metal matrix and a ceramic reinforcing phase [4]. Therefore, it is necessary to control the interaction of metals in the liquid state with ceramics in order to ensure good quality of products, and thus the reliability of their functioning under operating conditions [5]. The production of MMCs reinforced with titanium carbide using infiltration methods is a widely developed topic of research at the turn of the last dozen or so years. Among many methods used, one can find those based on the phenomenon of reactive infiltration.

This paper is focused on the in situ synthesis and characterization of TiC reinforced cast iron formed by reactive infiltration of the substrate composed of Ti and graphite powders and liquid gray cast iron. High-temperature interaction between molten gray cast iron and Ti+C_{gr} substrates was investigated by a sessile drop method combined with non-contact heating of the couple of materials to the test temperature of 1330°C in an inert gas atmosphere (argon). During high-temperature testing, the images of the couple were recorded by high-speed high-resolution CDD camera and used for estimation of the values of contact angles vs time.

The analysis of wettability kinetics of different Ti+C_{gr} substrates by molten cast iron and detailed structural characterization of solidified couples with the help of light microscopy, electron microscopy (SEM, TEM), X-ray diffraction and energy dispersive X-ray spectroscopy allowed to explain the mechanism of high-temperature interaction in the selected couples. The obtained results made it possible to illustrate the course of the titanium carbide synthesis reaction taking into account the intermediate chemical reactions taking place in the Fe-Ti-C ternary system. It was evidenced that good wetting and fast infiltration of all examined substrates with selected alloy have a reactive nature due to reactively formed wettable TiC phase.

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POSTER SESSION – WETTABILITY



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W_11

WETTABILITY AND INTERFACIAL MICROSTRUCTURE OF NEW GENERATION LEAD-FREE SOLDER ALLOYS ON COPPER SUBSTRATE

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Over the past 15 years, the electronics industry has experienced a key change in the manufacturing process. In 2006, the RoHS EU directive was implemented in most European countries. These regulations had a significant impact on the soldering process of electronic components. The use of Sn37Pb solder has been restricted due to the lead content. Therefore intensive research began to find a substitute for SnPb alloy and the Sn-Ag-Cu (SAC) composition was most often proposed as a lead-free alloy in production processes. Despite the popularity of this solution, many parameters related to the joints' quality and reliability have not been achieved compared to the Sn37Pb solder. Defects such as microcracks, voids, thick intermetallic layers or microstructure inhomogeneity are still unresolved.

The search for new solders, devoid of the negative features observed in the interconnections made using SAC alloys, is very advanced. The result of these works is the appearance of new compositions of alloys on the commercial market. However, their application in production practice, carries a significant risk due to the lack of reliable tests. Therefore, a series of experiments were designed to verify the quality of the interconnections of new alloys with copper in relation to the reference SAC305 alloy.

The wetting and spreading behaviour of molten solder alloy on Cu substrates were investigated by the improved sessile drop method coupled with the capillary purification procedure applied. It was accompanied by non-contact heating of the solder/Cu couple and mechanical cleaning of a molten solder from its native oxide film directly in a vacuum chamber. For this, the solder was placed in a graphite capillary located above the Cu substrate. After reaching the test temperature, the liquid solder alloy was squeezed through a hole in the capillary and the drop was deposited on the substrate. The sessile drop tests were performed in a vacuum. The reaction zone of the solidified solder/Cu couples was characterized in terms of morphology and microstructure together with the chemical composition analysis using the scanning and transmission electron microscopy. In the next step the samples were annealed in the solid state at different periods of time to study the microstructure evolution of solder/copper interface.

Acknowledgement

Research was carried out as part of the "Implementation Doctorate" program of the Ministry of Education and Science in Poland, project No DWD/3/29/2019 realized at Krakow School of Interdisciplinary PhD Studies and Fideltronik SA Company. The SEM and TEM research was conducted in the Accredited Testing Laboratories at the IMMS PAS.



WETTING OF NANOCOATINGS ON SILICON OXIDE BY METAL SOLDERS

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W_12

One of the soldering options is the metallization of nonmetal surfaces by adhesive-active metal nanocoatings. To develop such a soldering technology, it is necessary to study the wetting and adhesion of nanocoatings to the surface of silica glass. In our work, metallic nanocoatings on SiO₂ were studied in order to develop a technological process for soldering silica glass with aluminum.

The effect of metal nanocoatings on the wetting of SiO₂ with lead-based solder melts (Pb–5In and Pb–2.5Ag, wt%) by a sessile drop method using the capillary purification technique was studied in vacuum (1×10⁻³ Pa) at a temperature of 500 °C. Single coatings (Mo, Cr, V, Nb, Ti) and double coatings with a second Cu layer of constant thickness were deposited by electron beam evaporation of metals in vacuum. The metals of the first coating layer were chosen so that there was practically no mutual solubility of the solder and the coating, as well as with different chemical affinities for oxygen, which affected the adhesion activity of the coating metals to SiO₂.

The contact angle depends on the thickness δ of (Ti, Nb, Cr, V, Mo) nanocoatings (Fig. 1) showing the difference in the slope of the curves from the contact angle (θ) at $\delta = 0$ to an angle equal to the wetting angle of the compact metal of “threshold” coating. The character of the dependence is a linear decrease with increasing thickness coating from the wetting angle of silicon oxide to the wetting angle of the compact metal by the solder melt. The “threshold” coating thickness for different metals depends on the chemical affinity of the metal to oxygen. The higher the chemical affinity, the greater the “threshold” thickness coating. The value of $\Delta_r G_{298}^\circ$ for coating metal oxides (TiO₂→NbO→V₂O₃→Cr₂O₃→MoO₂) decreases in the series, respectively, 444.3→379.4→379.0→353.0→266.6 (kJ/mol). In the same series, the “threshold” film thickness decreases 70→65→62→60→50 (nm) upon wetting of (Ti, Nb, V, Cr, Mo) films on SiO₂ by lead–indium alloys. In this case, the contact angles differ slightly and are equal to 20–30°.

The structure of Cu, Ni, Mo, Cr, Nb, Ti coatings after annealing at temperatures of 600, 900, 1200 °C was studied. The initial freshly-sprayed metal films have preserved integrity predominantly. After annealing, dispersion takes place and the integrity film decreases with increasing temperature. The dependence of the strength on the shear of a solid Pb–2.5Ag, Pb–15In, and Pb–40Sn alloys on the thickness of Cr, Nb, Ti nanocoatings on SiO₂ (Fig. 2) was examined. The shear strength of solder joints is sufficient (up to 20 MPa) to use this method for brazing quartz with an aluminum. The wetting by liquid metal of the metal coating deposited on the surface oxide is determined : by the thickness coating, by coating structure, by the affinity of the metal to oxygen (adhesive attraction the coating – substrate), which determines the shape of the “islands” and the ease of dispersion; by dissolution of the coating in the melt; by wetting of oxide by the coating metal.

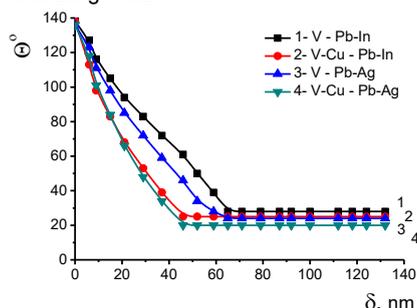


Fig. 1. Wetting of V (1, 3) and V–Cu (2, 4) coatings deposited on SiO₂ by Pb–15In (1, 2) and Pb–2.5Ag (3, 4) melts

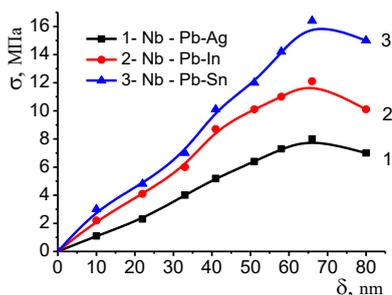


Fig. 2. Dependence of the shear strength of solid Pb–2.5 Ag (1), Pb–15 In (2), and Pb–40Sn (3) alloys on the thickness of Nb nanocoating on SiO₂

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W_13

WETTING BEHAVIOUR AND REACTIVITY OF LIQUID COPPER ON 304 AND 316L STAINLESS STEELS SURFACES

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Joining stainless steel to copper is widely used in the automotive industry, where the presence of a continuous interfacial interaction zone between the connected elements is necessary to obtain durable and high-quality joints. Additionally, the 304 and 316L steels are the most commonly used in many applications where corrosion resistance is critical. The combination of the high-melting point, thermal expansion coefficient, heat and electrical conductivity of copper with the high strength of steel provides exceptional functional properties of the produced steel/Cu joints. Simultaneously, the large differences in the physical and chemical properties of copper and steel cause the formation of the good quality metallurgical bond (with the participation of the liquid phase) between these dissimilar materials is very difficult with no steel surface modification. Moreover, a wide metastable miscibility gap is present in the Cu-Fe phase diagram in the range of high temperatures, what additionally makes the bonding process more challenging.

For this reason, the improved sessile drop method coupled with the capillary purification procedure accompanied with non-contact heating of the Cu/steel couple was applied for the study of copper wettability on selected stainless steel substrates. The oxide film on molten copper was mechanically removed directly in a vacuum chamber. After reaching the test temperature, the liquid Cu placed in the graphite capillary above the steel was squeezed through a hole in the capillary and the drop was deposited on the substrate. Next, the description of the microstructure evolution and chemical characterization of the Cu/steel interface in the micro- and nanoscale using scanning and transmission electron microscopy was performed directly after wetting tests and after annealing in the solid state at different periods of time.

The obtained results deliver new knowledge on copper wetting and spreading behaviours on steel substrates as well as on the conditions needed to obtain thermodynamically stable diffusion couples thus allowing to predict the functional reliability of components produced by liquid-assisted joining copper with stainless steel.



INVESTIGATION OF THE EFFECT OF HEAT EXPOSURE ON THE WETTING OF LASER BEAM TREATED ALUMINIUM SHEETS

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W_14

Thanks to hybrid material combinations, there is a big boom in the development of adhesive technology. Bonding is also becoming increasingly important in industrial applications. The expectation is to achieve a sufficiently high strength bond on all surfaces. The materials to be bonded can be metals, polymers, ceramics, fibre-reinforced materials, which can be used in many areas of industry such as automotive, architecture, etc. Depending on their application, these materials can even be given coatings, which may be required for aesthetic and protective reasons [1].

Today, these materials are expected to be bonded together in a way that does not significantly increase the weight of the structure, as well as aesthetically, we must aim to achieve the most beautiful appearance. The technology that best meets these requirements is adhesive technology. But to bond such a variety of surfaces, we need well-designed bonds. With adhesives, we can bond two surfaces together in an insoluble way. The adhesive can only form a proper bond on the surface if it is in a liquid state when applied. It can then spread over the surface and create adhesion between materials over a large area [2].

Before bonding, it is important to ensure that the surfaces are properly cleaned and determine how strong adhesion is created between the surface and the adhesive. Several ways of determining these adhesion forces have been developed, many researchers are still working on the subject today. The adhesion bond is created by intermolecular forces, these forces come from second-order chemical bonds, which can be van der Waals forces and hydrogen bonds. There are several theories of these bonding forces, which form the basis of adhesion bonding. Or these forces to develop, the adhesive and the surface to be bonded must be no more than five angstroms apart. This can be achieved if the adhesive wets the surface well [3].

There are several theories for determining surface wetting. These theories are based on Young's equations. He says that wettability depends on the extent to which liquids are spread over solid surfaces. This can be characterized by the contact angle (Θ) along the two phases. The degree of wetting is determined by the interactions between the solid and the liquid molecules at the interfaces. It follows from this principle that the wettability can be deliberately influenced by designing and modifying layers with the appropriate structure and orientation [4].

In our research, we have succeeded in developing laser beam surface treatments that increase the adhesion on the surface of the aluminium sheet. Laser surface treatment has created a special structure on the surface and activated it. We have improved the bonding technology for aluminium sheet due to the increased surface adhesion. In the experiments, we observed that the modified adhesion of laser-treated surfaces is affected by the thermal effects on the material. To investigate this phenomenon, the laser-treated aluminium sheets were annealed at different temperatures and the adhesion was then investigated by static droplet wetting measurements.

Acknowledgment

This research is supported by 2020-1.1.2-PIACI-KFI-2021-00260 project.

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W_15

GRAIN BOUNDARY WETTING IN THE W–Ni ALLOYS

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The aim of the investigation is the grain boundary wetting phase transitions in the binary W–Ni alloys. Influence of the alloys' composition and annealing temperature on the structure of grain boundaries has been studied. The study was carried out by means of high-resolution electron microscopy (HREM) and analytical electron microscopy.

Different kinds of wetted GBs were revealed by HREM, namely:

- (i) "dry" GBs (partial wetting),
- (ii) GBs with thick (complete wetting) and
- (iii) thin layers of bulk Ni-rich phase (pseudo-incomplete wetting).

The continuation of tie-line of the GB wetting phase transition into one-phase (W) area is constructed in the bulk W–Ni phase diagram.



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W_16

HIGH-TEMPERATURE INTERACTION OF LIQUID MAGNESIUM WITH A PURE SILVER SUBSTRATE

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Magnesium and silver can be considered potentially biodegradable materials for bone imitation implants with variable and directed porosity. They have mechanical properties comparable to human bone, excellent bioactivity, non-toxicity and antibacterial properties [1]. However, to successfully use such materials for an implant in the human organism, its surface must allow absorption of proteins and formation of a layer in a physiological environment, which will support cell/osteoblast growth [1,2]. In addition, full knowledge of how and in what to produce such a material is also needed. Therefore, the wettability of magnesium and silver plays a key role in identifying the possibility of using such materials in medicine.

The main objective of this study was to determine the interaction of liquid magnesium in contact with pure silver (99.99 wt.%) substrate. High-temperature wettability studies were performed in a protective atmosphere (Ar-5% H_2) at a temperature of about 700 °C using the sessile drop method. To eliminate the effect of native oxide layer on Mg drop on contact angle measurements, the non-contact heating of tested couple of materials to the investigated temperature was combined with in situ purification of Mg-Ag drop directly in a high-temperature chamber using a capillary purification procedure [3]. For this, an Mg sample was placed in a graphite capillary above an Ag substrate and after reaching the desired temperature, the Mg drop was mechanically squeezed from the capillary and deposited on the substrate [3,4]. The images of the drop/substrate couple were recorded with two high-resolution high-speed digital cameras from two directions of observation. From these images, the contact angle values (θ) formed between the liquid magnesium and the silver substrate was measured.

It was noted that for the Mg/Ag couple with CP procedure, immediately after contact with the Ag substrate, liquid Mg drop free of native oxide film showed complete wetting and spreading over the substrate, i.e., the contact angle reached a value of $\theta < 5^\circ$ in a very short time. Once the wettability tests were performed, the samples were subjected to detailed microstructural observations by SEM with EDS analysis.

Acknowledgment

This research was financially supported by the National Science Centre of Poland within OPUS 21 funding scheme, Project no. 2021/41/B/ST5/02787 in the years 2022–2025.

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NEW SOLDER, BASED ON Sn-Zn EUTECTIC WITH ADDITION OF Ag, Al AND Li

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New Sn-Zn-based solder with the addition of Ag, Al, and Li (SZAAL) was designed. The influence of alloying additions on mechanical and electrical properties, microstructure, and melting point was investigated. Tensile tests showed enhanced mechanical properties of the SZAAL alloy compared to Sn-Zn eutectic and Sn-Zn-Ag alloys. Investigation revealed also a change in electrical behavior with the addition of aluminum to the alloy.

Acknowledgement

This work was financed by the National Science Centre of Poland, grant 2018/29/B/ST8/02558.

Keywords: led-free solder, DSC, surface tension, electrical resistivity.

POSTER SESSION – SURFACES AND INTERFACES



HTC 2022 10th International Conference on HIGH TEMPERATURE CAPILLARITY
12 -16 September, 2022, Krakow, Poland

EXTENSION OF THE GIBBS-DUHEM EQUATION TO PARTIAL MOLAR SURFACE THERMODYNAMIC PROPERTIES OF SOLUTIONS

S_01

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In this paper, the Gibbs-Duhem equation is extended to the partial molar surface thermodynamic properties of solutions. According to the surface Gibbs-Duhem equations the sum of the mole fractions of the components in the surface region of a bulk solution multiplied by different partial molar surface quantities should equal zero if summation is taken by all components of the solution. There are four different partial molar surface quantities identified in this paper for which the surface Gibbs-Duhem equation is proven to be valid: i). the reduced surface chemical potential, ii). the surface chemical potential, iii). the partial molar surface area, iv). the partial molar excess surface Gibbs energy = the product of partial molar surface area and the partial surface tension. The first one is known since Guggenheim (1940), but the other three are presented here for the first time. It is also demonstrated here how to apply the surface Gibbs-Duhem equations: i). it is proven that the model equation applied by us recently for the reduced chemical potential [Adv Coll Interf Sci 283 (2020) 102212] obeys one of the surface Gibbs-Duhem equations, ii). On the contrary, it is proven that the model equation suggested by us recently for the partial molar surface area [same citation as above] contradicts one of the surface Gibbs-Duhem equations; therefore, a new (and simpler) model equation for the partial molar surface areas of the components is suggested here that obeys the surface Gibbs-Duhem equation. It is also shown that the Butler equation obeys one of the surface Gibbs-Duhem equations. It is also concluded that surface composition in equilibrium should be such that it ensures the minimum of surface tension.

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INVESTIGATION OF INTERFACIAL TENSION BETWEEN SILICON STEEL AND ALUMINA

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Carbon neutrality by 2050 is the most important and urgent global mission. Therefore, increasing the efficiency of motors and transformers is very important. Silicon steel (electrical steel) is a core material in these applications, and controlling grain orientation is critical to achieving high energy efficiency. Silicon steel generally has a higher silicon content than general carbon steel and shows excellent electrical and magnetic properties. By adding silicon, the eddy current loss and magnetic anisotropy can be suppressed, yielding the improved magnetism of electrical steel. Electrical steel sheets are produced through preliminary annealing, cold rolling, annealing, and insulation coating. The surface defects of cold-rolled steel products are generally caused by the incorporation of mold flux, debris from the clogged nozzle, and re-oxidation inclusions. In order to reduce these defects, improvements in steel cleanliness, prevention of reoxidation, and optimization of mold flux flow have been implemented. The control of bubbles and inclusions in the continuous casting process is essential to improve the surface quality. Therefore, it is very important to understand the behavior of bubbles and inclusions in front of the solid-liquid interface during solidification. Recently, Jeong in our group reported the interfacial tension between low alloyed steel and alumina inclusions and explained the behavior of inclusions in front of the solid-liquid interface [1]. In this study, the surface tension of silicon steel and the interfacial tension between silicon steel and alumina are reported. The surface tension is investigated with an advanced sessile drop method, the constrained drop method [2]. In addition, the contact angle is investigated with the sessile drop method. Then, the interfacial tension is evaluated from Young's equation. The experimental results will be summarized and reported at the conference.

This work was supported by POSCO.

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HTC 2022

10th International Conference on HIGH TEMPERATURE CAPILLARITY
12 -16 September, 2022, Krakow, Poland

A DIP DIVE INTO THE THERMAL AND PHYSICO-CHEMICAL PROPERTIES OF COPPER SLAGS

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S_03

Slag is a crucial component in the technology of copper production. The role of the slag is to absorb impurities from the refined bath and thermally protect the copper against oxidation. The most important features of slag are composition, and viscosity which depends on slag chemistry and process conditions (temperature, p_{O_2}). In general, the slag can be described as an ionic structure occurring in the liquid state with cations (e.g. Si, Fe, Ca, Mg, Mn, Na) and non-metallic anions (e.g. O, S, F). The small cations of a high charge attract oxygen forming tetrahedral complex like SiO_4 . These acid structural groups, which share the common oxygen, are called network formers, and they increase the viscosity of slag. On the other hand, basic oxides (e.g. CaO, MgO, FeO) called modifiers, reveal O^{2-} to the liquid slag, breaking the network and lowering the viscosity. The viscosity of slag is one of the most important features during copper refining. For instance, the high viscosity of fayalite slags increases the amount of entrapped copper and matte, whereas, calcium ferrite slags possess lower viscosity, lower Cu solubility and higher basicity, removing impurities more effectively. On the other hand, the high viscosity of slag causes stronger wettability of refractory lining.

As copper slags are composed mostly of oxide compounds, they can interact with refractory lining easily, thus, provoking its corrosion and causing accelerated consumption. The greatest corrosion by slag is observed in converters where slag is much enriched in copper oxide, which attacks magnesia grains in refractory. Components of slag very easily penetrate the refractory via its open pores when the slag is of low viscosity. Additionally, reactions between components of slag and the refractory can cause the liquid phase appearance. This can accelerate, or even terminate, the use of refractory lining. Thus, the prediction of high-temperature phase composition and the properties of slag are significant to be well explored.

This work presents the crucial thermal and physico-chemical properties of copper slags, using numerous experimental methods as well as theoretical simulations based on equilibria conditions. Theoretical simulations of alternations in phase composition of slags vs. temperature were calculated with the aid of FactSage software. X-Ray Fluorescence Spectroscopy was used to determine the chemical composition of slags. X-Ray Diffractometry was applied to identify the phase composition of the slags. Melting behavior of the slags was evaluated using Hot Stage Microscopy non-isothermally up to 1450°C with $p_{O_2}=0.21$. The microstructure of slags was observed with Scanning-Electron Microscopy coupled with Energy Dispersive Spectrometer to measure chemical composition in microareas. Image analysis of SEM photographs was applied for the qualitative assessment of glassy and crystal components of the slag.

Acknowledgment

The research was supported by the funds of the National Centre for Research and Development, Grant no. LIDER/14/0086/L-12/20/NCBR/2021 (PI: I. Jastrzębska).



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S_04

STRUCTURAL CHARACTERIZATION OF INTERFACES FORMED BETWEEN MOLTEN Al-Si ALLOY AND TiB₂ SUBSTRATES

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The Al-Si/TiB₂ couples produced by the sessile drop method in [1] were subjected to detailed characterization of structure and chemistry of interfaces formed between commercial TiB₂ ceramic and near eutectic AlSi12 alloy (12 wt% Si) during isothermal heating in vacuum at different temperatures (900-1300°C).

Comparative observations of cross-sectioned couples were performed by light microscopy (LM), scanning electron microscopy (SEM) and transmission electron microscopy (TEM) combined with energy dispersive X-ray spectroscopy (EDS) analysis. Besides Al, eutectic Si and TiB₂ phases, the selected structural characterization techniques showed the presence of small crystals of alumina (Al₂O₃) occasionally formed at the drop/substrate interface. Mainly, these crystals were nucleated at the surface of TiB₂ near its grain boundaries, and they grew inside the AlSi12 drop. Additionally, the Al₂O₃ crystals were decorated with fine Si precipitates, nucleated at the Al₂O₃ surface during solidification of Al-Si alloy upon cooling after sessile drop tests.

LM/SEM/TEM observations suggest that under the conditions of this study, the introduction of 12 wt% Si into Al does not influence the interaction in the Al/TiB₂ couple. Similar to the results obtained with the same commercial TiB₂ ceramic and pure Al [2], it is accompanied by redox reaction between liquid Al and partially oxidized titanium diboride, i.e. $4Al + 3TiO_2 = 2Al_2O_3 + 3Ti$.

Acknowledgement

The research was supported by the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences (Task Z-13).

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HTC 2022

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REACTIVITY BETWEEN SAC305 ALLOY AND ELECTROLESS Ni-P AND Ni-P-Re COATINGS ELECTROLESS PLATED ON COPPER

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Electroless Ni-P coatings are widely used in the electronic industry, known as Electroless Nickel Immersion Gold (ENIG) coatings, where top thin layer of gold is applied to improve the wetting properties, while the nickel underneath protects the copper prior to diffusion. Lead-free soldering technology forced the use of solder materials characterized by higher soldering temperatures than previously Sn-Pb ones. This in turn caused on one hand, demand for higher thermal stability of the nickel coatings, and on the other hand, uncontrolled growth of the intermetallic phases at the coating/solder interface. Thin and continuous layer of the intermetallics is desired to be formed at the interface zone, however, massive detachment of the intermetallic phase and its movement toward the solder brings the failure of such interconnection. Current study is dedicated to the modification of the Ni-P coatings by addition of the rhenium in order to improve the thermal stability of such coating, and finally, it may possibly serve as the avoidance tactics for spalling of the intermetallics. The structure of nickel coatings strongly depends on the phosphorus content and may be amorphous (high phosphorous content), amorphous with Ni nanocrystallites (medium content of phosphorous) or crystalline (low content of phosphorous). This translates into their thermal stability (crystallization temperature) but also to the type of Ni_xP_y phases that are created at elevated temperatures. The possible phase transformations can be correlated with the structure of the coating, chemical composition, deposition parameters and the rhenium content. Authors continued the previous studies [1-3] regarding Ni-P and Ni-P-Re coatings, this time taking up the research problem involving phenomena occurring at the interface between Ni-P-Re coating and classical SAC305 alloy. It is expected that addition of rhenium causes retarding or eliminating massive detachment of the intermetallic phases from the interface toward the solder after their interaction. In presented study Ni-P and Ni-P-Re coatings with composition similar to commercially ones, containing 6–8 wt.% of phosphorus were deposited. The coating surface morphology, chemical composition, its wetting behaviour and reactivity with liquid SAC305 alloy using the sessile drop method were studied. The substrate/coating and coating/solder interfaces were examined by scanning and transmission electron microscopy, with particular interest in determination of the interface zone quality and verification of the formed intermetallic phases.

Acknowledgment

This work was financed by the Ministry of Science and Higher Education of Poland as the statutory financial grant of the Polish Academy of Sciences and supported by the ERASMUS+ (staff mobility for training).

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S_05



HTC 2022

10th International Conference on HIGH TEMPERATURE CAPILLARITY
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S_06

COPPER/STEEL INTERACTION IN EXPLOSIVE WELDING PROCESS – IN-DEPTH ANALYSIS OF REMELTING AREAS THROUGH THE WETTABILITY TESTS

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Explosive welding (EXW) is a high-energy joining technique efficiently used for metals and alloys challenging to be combined, thanks to collision of the plates at high speeds. The collision takes place due to the detonation of an explosive charge, which accelerates the top flyer plate towards a base plate. During EXW process, the metallic plates or alloy sheets are subjected to extremely high pressure and locally temperature, therefore they are significantly deformed. Additionally, melting of the top surface layers often takes place and depending on the process parameters and type of joined materials, a variety of interface zones is observed in terms of the microstructure and phase compositions.

Presented studies are dedicated to the Cu/P265GH steel interface zone, where copper acting as a flyer plate collided with the steel with the 2500 m/s detonation velocity, and further the weld was subjected to stress relief annealing. As the interface zone was locally decorated with the melted regions the additional wetting experiment was performed to analyse the liquid-assisted phenomena. The modified sessile drop wetting test comprising of squeezing the liquid copper via the capillary on the steel substrate was performed. Highly deformed steel surface has been applied in this test, obtained from the previously EXW Cu/P265GH couple via the cutting off copper followed by its grinding.

The Cu/steel interface zones, formed both in explosive welding as well as after the wetting test, were subjected to complex microstructure characterization as well as the chemical and phase composition analysis using scanning and transmission electron microscopy techniques.

Acknowledgments

This research was financially supported by the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences within the statutory work "The interface zone of the welds obtained with the use of explosion energy" Z-5/2021 and Z-5/2022. The SEM and TEM research was conducted in the Accredited Testing Laboratories at the IMMS PAS.

POSTER SESSION – SURFACES AND INTERFACES



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INTERFACIAL PHENOMENA BETWEEN GRAPHENE ON Cu SUBSTRATE COATED BY Ni, Cu, W LAYER, AND LIQUID Ga-Sn-Zn ALLOY

S_07

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To optimize the parameters of wetting and interfacial properties, Cu substrate was coated by graphene. Nonetheless, preliminary experiments showed that the liquid metal mechanically destroys the graphene layer [1]. Therefore, a Ni, Cu, or W layer was sputtered to protect graphene. The presence of graphene was verified by micro-Raman spectrometry. The thickness of the Cu, Ni, or W layer was ~25 nm, compared to previous work [2], where the thickness of the Ni-W deposited layer varied from 8 to 10 μm , and the electrochemical deposition method was used. To observe the changes at the interface, the experiments were performed with Ga-Sn-Zn eutectic alloy and the wetting process was monitored using the sessile drop method at various temperatures of 100, 150, and 250 °C with holding times 1, 240, and 2400 h. The topology of the obtained samples was evaluated by atomic force microscopy. The microstructure of cross-sections was observed in a scanning electron microscope equipped with energy dispersive X-ray spectroscopy device. To identify the phases developed at the interface, X-ray diffraction analysis was conducted. The results showed that such a very thin Ni, Cu, or W layer is not sufficient to protect the Cu substrate from liquid metals, such as the eutectic Ga-Sn-Zn, which can be used in cooling systems [3]. The results from XRD and microstructure observations showed that CuGa_2 phase forms at interface and dissolution of Cu substrate occurs with increasing temperature and time. The CuGa_2 phase grows very slowly at the interface at annealing temperatures below 150 °C in graphene/Ni and graphene/W coatings. Accordingly, these coatings can be used to protect copper substrate in cooling electronic devices.

Acknowledgments

This work was financed by IMMS PAS (Z1: Environment-friendly technologies and materials).

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POSTER SESSION – PROCESSING



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P_01

GRAIN BOUNDARY OXIDE LAYERS IN THE NdFeB-BASED PERMANENT MAGNETS

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The microstructure of grain boundaries (GBs) in the commercial NdFeB-based alloy for permanent magnets has been studied. It is generally accepted that the unique hard magnetic properties of such alloys are controlled by the thin layers of Nd-rich phase in Nd₂Fe₁₄B/Nd₂Fe₁₄B GBs. These GB layers ensure the magnetic isolation of Nd₂Fe₁₄B grains from each other. It is generally supposed that such GB layers contain metallic Nd or Nd-rich intermetallic compounds. However, the commercial NdFeB-based permanent magnets frequently contain a tangible amount of neodymium oxide Nd₂O₃ at the triple junctions between Nd₂Fe₁₄B grains. The goal of this work was to check whether the Nd₂Fe₁₄B/Nd₂Fe₁₄B GBs could also contain the thin layers of Nd₂O₃ oxide phase. Indeed, the screening with EELS-based elemental analysis permitted to observe that some of these Nd-rich layers in Nd₂Fe₁₄B/Nd₂Fe₁₄B GBs contain not only neodymium, but also oxygen. More detailed analysis of such GBs with high-resolution transmission electron microscopy (HR TEM) showed these GB layers are crystalline and have the lattice of neodymium oxide Nd₂O₃. In turn, the Lorentz micro-magnetic contrast in TEM permitted to observe that the Nd-oxide GB layers prevent the migration of domain walls from one Nd₂Fe₁₄B grain to another during remagnetization. This finding proves that the GB oxide layers, similar to those of metallic Nd or Nd-rich intermetallic compounds, can ensure the magnetic isolation between Nd₂Fe₁₄B grains needed for high coercivity. Therefore, the GB oxide layers can be used for further development of NdFeB-based permanent magnets.



DETERMINATION OF HIGH-TEMPERATURE CORROSION MECHANISM OF NOVEL Cr-FREE REFRACTORY MATERIAL FOR THE COPPER INDUSTRY BY EXPERIMENTAL COUPLED WITH SIMULATION APPROACH

P_02

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Refractory materials are indispensable for the production of metals, like steel or non-ferrous metals including copper, lead, zinc, etc. According to statistics from the European Ceramic Industry Association, refractory materials account for 18 % in terms of the total value of the ceramic market in Europe, while 11 % of it corresponds to refractories in the non-ferrous industry. Refractories work as thermomechanical linings in all heating devices, being subjected to harsh conditions of thermal, chemical and mechanical stresses - acting simultaneously - during operation. In the copper industry, numerous devices are lined with refractory materials, including fluidized-bed furnaces, electric-arc furnaces, converters, or anode furnaces.

Currently, magnesia-chrome refractories are used in Cu devices, as they exhibit excellent properties (high thermal shock resistance, corrosion resistance and erosion resistance). However, the application of this type of refractories directly links with the possible formation of hexavalent chromium compounds, which belong to 1. Group carcinogens as classified by the International Agency for Research on Cancer. The formation of Cr(VI) compounds is favored at increased temperature, over 600°C, in the access of air, and, especially, in the presence of basic (e.g. CaO) and alkali oxides (e.g. Na₂O, K₂O), when chromates and di-chromates are generated. For instance, in the CaO-Cr₂O₃ system, four compounds are formed, where three of them contain Cr(VI) while one of them contains only Cr(III). Additionally, increased oxygen partial pressure favors the oxidation of Cr(III) into Cr(VI). So, conditions of most copper industrial devices carry a risk of Cr(VI)-compounds formation. Therefore, the development of new alternative Cr-free refractories is necessary.

In this work, the new, intelligent Cr-free refractory material is proposed. The work presents a corrosion model of novel refractory which was developed applying a combined experimental and simulation approach. In the experimental route, different methods of corrosion testing were applied to reflect better the actual working conditions, and obtain diverse information on the chemical answer of material to interaction with copper slag. The high-temperature corrosion was tested up to 1450°C, using a pellet test, contact test, and hot stage microscopy test. The results were interpreted using XRD and SEM/EDS methods. In the theoretical route, the high-temperature reactions occurring in a temperature range of 700-1400°C between refractory material and slag were simulated using FACTSage software. Coupling the results of the experiment and simulation allowed to determine the corrosion mechanism, which revealed that passive corrosion predominates, with the formation of new compounds which protect the materials against deeper infiltration of aggressive slag components.

Acknowledgment

The research was supported by the funds of The National Centre for Research and Development, Grant no. LIDER/14/0086/L-12/20/NCBR/2021 (PI: I. Jastrzębska), and partly supported by the statutory funds of the Faculty of Material Science and Ceramics AGH University of Science and Technology, Poland Agreement no. 16.16.160.557.

POSTER SESSION – PROCESSING



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12 -16 September, 2022, Krakow, Poland

P_03

MECHANICAL MIXING OF METALS DURING HIGH-PRESSURE TORSION

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Recently the attention of researchers has been attracted to the phenomenon of turbulent material flow during high-pressure torsion (HPT) leading to mixing of materials in the solid state. In the production of metal compositions, the intensity of mixing processes plays a fundamental role. For its quantitative assessment, a stirring efficacy index Q was introduced [1]. The index quantifies the degree of homogeneity of the resulting distribution of the components in the sample volume. The report presents a method for determining this index from images of the microstructure of the processed multi-layered samples. The problems arising in the calculation of Q are analyzed and approaches to overcoming them are proposed. It is shown that the stirring efficacy index is a potent tool for studying solid-state synthesis by the HPT method.

Acknowledgment

The authors acknowledge funding support from The Volkswagen Foundation through the Cooperation Projects Az.: 97 751.

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METAL-CERAMIC PROSTHETIC RESTORATIONS PRODUCED BY SELECTIVE LASER MELTING

P_04

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The aim of the present work is to evaluate the SLM (selective laser melting) technique for manufacturing CoCrMo alloys, which can be coated by dental ceramic porcelains to produce dental metal-ceramic prosthetic restorations [1]. The experiments were carried out according to ISO 9693 [2]. The experiments were performed in two stages: (a) full characterization of the alloy substrate produced by SLM in order to be qualified for stage (b), and (b) production of the alloy/ceramic-porcelain final product and chemical as well as mechanical characterization of the ceramic/metal interface.

(a) In the first stage of experiments, the effect of repeated heat-treatment (HT) cycles, as precisely applied in common dental practice to produce metal-ceramic restorations, on microstructure (studied through XRD and SEM/EDS analyses), mechanical properties, and corrosion resistance (evaluated by potentiodynamic polarization measurements in artificial saliva-simulating solution at 37°C) of SLM CoCrMo dental alloys (i.e., fabricated with the advanced digital technique of SLM, with the aid of CAD technology), was thoroughly investigated [3]. Compared to the as-prepared alloy, the heat-treated alloy presents a markedly refined microstructure with a notable carbide/Laves phase dissolution and extinction (to a large extent) of the dendritic/cellular morphology. A small reduction in the values of the mechanical properties was recorded, i.e. Young's modulus was reduced from 240 to 210 GPa, Vickers microhardness from 280 to 250 GPa, and yield strength from 841 to 752 MPa. However, the HT improved the resistance of the samples toward general corrosion, which is mainly ascribed to the stress relief and refined/uniform microstructure due to the HT. The alterations caused to the investigated samples due to HT were relatively small, suggesting that the Co-Cr dental alloys produced by SLM are qualified for safe use in preparing metallic substrates aimed to produce metal-ceramic dental restorations.

(b) In the second stage of experiments, specimens of the aforementioned (as built) CoCrMo dental alloy were bonded (by the use of a bonding agent) with dental porcelain, which was positioned in layers (i.e., opaque, dentin, enamel layers) [4]. The bond strength was measured through 3-point bending strength tests. Elemental (by SEM/EDS) and crystallographic analyses (by XRD) were conducted on the fracture surfaces. The metal-ceramic bond strength was 51.87 ± 7.50 MPa. Cohesive fracture occurred (on the porcelain side). Specimens produced by the SLM technique cover the lowest acceptable metal-ceramic bonding strength of 25 MPa specified in the ISO 9693, manifesting, therefore, a satisfactory bonding strength for clinical use.

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MANUFACTURE OF BRASS COMPONENT USING ADDITIVE TECHNOLOGY

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Additive manufacturing is currently one of the fastest growing areas due to the great advantages it represents, especially for rapid prototyping or to manufacture complicated parts that are impossible using other techniques. In the case of metals, the Selective Laser Melting technique (SLM) is the most widespread. Currently, medium-sized pieces are manufactured using this technology. The grain size is mainly controlled by the size of the original powder. The other two factors of great importance are the power of the laser and the scanning speed. Since very high scanning speeds or very low powers would generate a porous material due to lack of sintering, which leads to poor mechanical and transport properties. Therefore, the process must be optimized. Some pure metals such as Ti, or tool steel are fully optimized, but Cu and its alloys present various problems when using this technology, so it is important to investigate this process for these alloys. One of the most interesting proposals today is the use of sacrificial alloys for the manufacture of three-dimensional Metal-Organic Frameworks (MOFs) where the metal of the cluster MOF matches that of the alloy [1-3].

In the present investigation, a series of brass monoliths have been optimized and developed for their partial transformation into MOFs, brass has the advantage over other alloys since Cu and Zn are the basis of two of the most important MOFs, due to their stability and catalytic and adsorption properties (HKUST-1, ZIF-8). Table 1 shows the optimization process followed for the manufacture of brass pieces. Once the process has been optimized, monoliths with different geometries have been made, and they have been exhaustively characterized (mechanical properties and transport properties). Subsequently, they have been partially transformed into the desired ZIF.

Table 1. Manufacture parameters used in SLM

	5000 mm/s	4000 mm/s	3000 mm/s	2000 mm/s	1000 mm/s	500 mm/s	250 mm/s
100W	A1	B1	C1	D1	E1	F1	G1
200W	A2	B2	C2	D2	E2	F2	G2
300W	A3	B3	C3	D3	E3	F3	G3
400W	A4	B4	C4	D4	E4	F4	G4

Authors acknowledge financial support from "Ministerio de Ciencia e Innovación" (PID2020-116998RB-I00).

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PRODUCTION OF PURE IRON STRIP SPECIMENS BY ELECTRODEPOSITION FROM A CHLORIDE BATH FOR USE IN TENSILE TESTS

P_06

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Electrochemical deposition is a promising method for the laboratory-scale preparation of high-purity iron specimens which has the advantages of being relatively low cost and offering great versatility in controlling the dimensions of the resulting deposit. In the present work we describe our effort in creating a complete process for the production of high-purity iron strip specimens for tensile tests. Pure iron strip is electrodeposited from a chloride bath in a specially designed by us electrochemical cell, compliant with the specifics of the process. An emphasis is put on producing uniform deposits, free of void defects and cracks. Hydrogen bubbles, generated onto the cathode by parasitic reactions, are found to be the main reason for the incorporation of void defects into the deposits while the diffusion of hydrogen leads to hydrogen embrittlement. A rotating cathode with automatic reverse in the direction of rotation is employed. The purpose of rotation is to equalize the current density over the cathode surface, while the automatic reverse ensures the rapid removal of hydrogen bubbles. The surface tension of the electrolyte is studied, and a suitable surfactant additive is found, that remains stable during the electrodeposition process and doesn't contaminate the resulting iron specimen. Uniform and free of void defects pure iron deposit is achieved with a combination of the described hydrodynamic conditions in the cell and the use of a surfactant additive in the electrolyte. Due to the nature of the process, the electrodeposited iron coating's microstructure consists of needle-like highly structured and anisometric grains, which renders them unusable for tensile tests. The microstructure of the electrodeposited pure iron strip is modified with the use of an annealing procedure. A special apparatus is developed for the annealing of the iron strips under argon, which facilitates the rapid desorption of hydrogen from the crystal grains and their recrystallization. The combination of a carefully controlled electrodeposition and recrystallization anneal is used to produce high-purity iron strips, with microstructure resembling that of hot rolled specimen, which are suitable for tensile tests. Grain size of the pure iron specimens can be varied by changing the annealing regime.

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PHENOMENA OBSERVED AT A1050/S355J2N INTERFACE FORMED DUE TO EXPLOSIVE WELDING SUPPORTED BY AN IN-DEPTH ANALYSIS OF LIQUID ALUMINIUM WETTABILITY TEST ON S355J2N CARBON STEEL

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Aluminium is characterized by good resistance to atmospheric corrosion and high thermal and heat conductivity combined with other materials based on steel, enable to get composite material of good mechanical properties, especially strength and improved corrosion resistance. Therefore, such Al/steel combination can be perspective one and may be applied as, for example, an anode for active electrochemical corrosion protection.

This study is divided into two experimental parts. The first is associated with the application of the explosive welding process to successfully join the Al and steel plates, while the second part is related to the wetting experiment with the same pair of materials.

The explosive welding is currently one of the most promising techniques that allows the production of double or multi-layer systems as a result of collision of joined metallic sheets generated by controlled detonation of an explosive. This fascinating technique allows for the combination of materials with either the same, similar, or strongly different properties thanks to the jetting phenomenon of removing oxides and impurities from the surface, application of high pressure and local melting of the joined materials. Within the present study the top flayer plate of a 5 mm thickness sheet of A1050 aluminium alloy was shot into the base material – a 5 mm thick S355J2N carbon steel plate with the detonation velocity of 2200-2300 m/s.

The second part of the study (wetting test) involved the modified sessile drop method, consisted of squeezing the aluminium droplet through an alumina capillary at the test temperature and its deposition on the deformed after explosive welding S355J2N carbon steel substrate. Prior to this test the A1050/S355J2N clad obtained by explosive welding was cut to the required dimensions, grinded to expose the highly deformed interface surface of the steel side, which was then metallographically prepared using papers of progressively decreasing gradation.

Both experiments were followed by a series of electron microscopy techniques to expose the microstructure features, phase composition at various magnification scales and thus enable the analysis of the occurring the interface phenomena.

Acknowledgement

This research was financially supported by the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences within the statutory work "The interface zone of the welds obtained with the use of explosion energy" Z-5/2022. The electron microscopy research was conducted in the Accredited Testing Laboratories at the IMMS PAS.



FORMATION OF AN INTERFACIAL BOUNDARY WHEN BRAZING DISSIMILAR MATERIALS KOVAR - Ti (Mo)

P_08

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Brazed assemblies made of dissimilar materials are widely used to create structures in various industries. When obtaining such joints, many problems arise related to the difference in the physicochemical properties of the materials to be joined, wetting the base metal with brazing filler metal, diffusion along the base metal grain boundaries, and the formation of intermetallic compounds at the brazing filler metal-base metal interface. In this regard, when creating brazed assemblies from dissimilar materials, the microstructural studies of the brazing filler metal - base metal interface play an important role. During vacuum high-temperature brazing of dissimilar materials, diffusion processes occur between the solid base metal and liquid brazing filler metal, which lead to the formation of reaction layers (intermetallic compounds) at the interface and, consequently, the deterioration of mechanical properties. Such processes are especially active when brazing an adhesive-active element - titanium with other metals. It is possible to limit the diffusion of titanium into the seam and exclude the formation of intermetallic interlayers by using barrier coatings, which various methods apply to titanium alloys. Using optimal temperature-time parameters of the brazing process and barrier coatings can reduce or eliminate the interaction and diffusion between the materials to be joined and improve the performance of the obtained brazed joints.

This paper presents the results of studies of chemical inhomogeneity and X-ray microanalysis of brazed joints of dissimilar materials Kovar-titanium (molybdenum) obtained using vacuum brazing. The influence of the thermal regime of brazing on the formation of the reaction layer at the interface between the base metal (Mo) and brazing filler metal is shown.



PHASE COMPOSITION, MICROSTRUCTURE, AND SURFACE HARDNESS OF SURFACE PLASMA GAS NITRIDED Ti-10V-2Fe-3Al WITH INDIRECT PLASMATRON

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The purpose of this study is to investigate and analyze the influence of plasma gas nitriding on the phase transformations, microstructure, and surface hardness of titanium alloy Ti-10V-2Fe-3Al. The limits of the used power in this process are a minimum of 12 kW and a maximum of 35 kW. Treatment times vary from 5 to 30 minutes. It is found that the phase composition of the surface layer after plasma gas nitriding consists of α -Ti, (N, O), TiN, and TiO₂. Titanium oxides are detected only on the outer surface of the nitrided layer. The hardness of the plasma gas nitrided layers obtained of Ti-10V-2Fe-3Al is up to 650 HK0.05.

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A STUDY ON THE STICKING PHENOMENON DURING IRON ORE REDUCTION BY HYDROUS GAS

P_10

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As a technological alternative that can dramatically reduce CO₂ compared to the blast furnace production method, it is necessary to increase the use of alternative iron sources in the steelmaking process, so it is necessary to develop energy-saving DRI manufacturing technology. Therefore, it is required to derive energy-saving fine particle iron ore as the raw materials and multi-stage fluidized reduction process conditions using hydrous gas from ironmaking by-product gas. On the other hand, the sticking phenomenon that occurs in the fluidizing reduction process causes a bottleneck and lowers the reduction efficiency due to the agglomeration of the reduced ore particles. Therefore, it is necessary to evaluate the morphology and consider the sticking phenomenon under the reduction of the iron ore in the fluidizing reduction process.

In this study, the kinetic behavior and the generation of whisker were observed by analyzing the difference in the morphology of iron ore after reduction according to the effect of temperature and composition of reducing gases such as H₂, CO, and CH₄. As the H₂ ratio increases, the reduction rate increases, and the length of the whiskers become shorter and thicker. Inside the iron particle, the point-shaped metallic iron crystal nuclei were connected making adhered morphology, this part seems to be a carbon deposition as the C content was high. Finally, the mechanism of the sticking phenomenon during iron ore reduction using hydrous gas was investigated and the conditions for preventing sticking were presented.

This work was supported by Korea Institute for Advancement of Technology(KIAT) grant funded by the Korea Government(MOTIE) (P0002019, Human Resource Development Program for Industrial Innovation) and also supported by the Korean Institute of Energy Technology Evaluation and Planning (KETEP) and the Ministry of Trade, Industry & Energy (MOTIE) of the Republic of Korea (No. 20172010106310).

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POSTER SESSION – PROCESSING



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P_11

THE INFLUENCE OF TiC AND TiB₂ PARTICLES ON THE MICROSEGREGATION AND MICROSTRUCTURE EVOLUTION OF CAST IN718 ALLOY

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Inconel 718 is currently the most widely used superalloy with an excellent high-temperature performance below 650°C. Typically, the 718 alloy is one of the few alloys with high Nb elements, and thus the mechanical properties of the alloy might be seriously deteriorated by severe microsegregation. Homogenization at high temperature could eliminate microsegregation but it requires a very long time. Previously, we found that the trace addition of TiC nanoparticles effectively refined the as-cast grains and reduced microsegregation in a Ni-Fe-based cast superalloy. The yield strength, ultimate tensile strength, and elongation were simultaneously improved. However, the mechanism is unclear. In the present work, we compared the effect of TiC and TiB₂ particles on the microsegregation and microstructure evolution during heat treatments to illustrate the influence of the particle type and size.



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STUDY ON THE PHASE COMPOSITION AND MICROSTRUCTURE OF Ti-8Al-1Mo-1V AFTER SURFACE PLASMA GAS NITRIDING WITH INDIRECT PLASMATRON

P_12

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The application of titanium alloys is limited due to their low surface hardness and wear resistance, especially for parts operating under friction and contact loads. One of the most widely used technologies for the thermochemical treatment of titanium alloys is gas nitriding. A new method in this direction is surface plasma gas nitriding using indirect arc plasmatrons operating in a chamber with a controlled nitrogen atmosphere. In the present work, the changes in the phase transformations, microstructure, and surface hardness of titanium alloy Ti-8Al-1Mo-1V after plasma gas nitriding at the power of 18 kW, 25 kW, and 35 kW, for a time between 5 and 30 minutes are studied. The plasma gas nitriding with indirect plasmatron of the titanium alloy produced continuous surface layers. Analysis of the surface showed the presence of TiN and TiO₂. The thickness of the plasma gas nitrided layers ranges between 25 μm and 350 μm, depending on the technological parameters.

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P_13

HIGH-TEMPERATURE SURFACE TENSION PHENOMENA IN VISCOUS GLASSES TO PRODUCE CERAMIC FOAMS

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This work aims at presenting the utilization of high-temperature surface tension phenomena to produce ceramic foams from viscous glasses, through heating at their softening point. The raw material mainly consists of regular glasses, collected from glass cullet, thoroughly pulverized into fine glass powder with a size of 1 - 20 μm . There are studies which report on the addition of carbon powder to the glass powder, whereby CO and CO₂ gasses, acting as foaming agents, are spontaneously produced at high temperatures, 500 - 600 °C, close to the pyroplastic state of glass, according to the following reaction:



Except for pure carbon, the use of fly ash from fuel oil has also been proposed. Nevertheless, this production method is not environmentally friendly because it involves production of H₂S that fills the pores of the produced foams.

In the present study, SiC was utilized as a foaming agent, added in a small amount of 1 wt.%. At the softening point of the glass cullet, SiC is oxidized, releasing CO₂, which facilitates the foaming of the viscous glass. Surface tension phenomena crucially determine the formation, size and shape of the pores as well as the struts among the pores. Viscous sintering is the dominant sintering mechanism of the soda-lime-silica glass in the range of 600 – 800 °C. Accordingly, densification starts as soon as the glass reaches the softening temperature at which the atoms have enough mobility to diffuse towards any vacancy-concentration gradient.

In this work, the ceramic foams were produced by adding to the above glass-SiC a mixture of reinforcing oxides, such as AD glass (i.e. a glass which is crystallized to anorthite-diopside glass-ceramic) as well as red mud [1, 2]. The results showed that densification satisfactorily occurred within the range of 750 - 800 °C, while crystallization takes place at >800 °C. The results showed that the additives affect the microstructure of the produced foams (i.e., the pore size and pore number, and the microstructure and microporosity of the struts), and consequently can improve the mechanical and thermal properties of the resultant ceramic foams.

Acknowledgment

This work was co-financed by the European Union and Greek national funds through the Operational Program "Competitiveness, Entrepreneurship and Innovation", under the call RESEARCH—CREATE—INNOVATE (acronym: SEMI-WEB; project code: T2EDK-02073).

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REACTIONS BETWEEN Al_2O_3 - SiO_2 REFRACTORY CERAMICS DOPED WITH ALKALINE-EARTHS AND MOLTEN Al

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This study reports on the interfacial interactions between molten aluminum and aluminosilicate refractory ceramics doped with alkaline earth oxides of nanosize, aiming to act as potential pore-blocking and non-wetting agents [1-3]. Ceramic crucibles were produced in the laboratory by slip casting as well as regular plastic forming techniques. Powders of α - Al_2O_3 , SiO_2 as natural diatomite, and BaO, or CaO, or MgO, through BaCO_3 , CaCO_3 , or $4\text{MgCO}_3 \cdot \text{Mg}(\text{OH})_2 \cdot 5\text{H}_2\text{O}$, respectively, were used to prepare the ceramic crucibles. The ceramics were sintered at 1400 °C for 1 h in air. The influence of nanosized powders on preparing the crucibles and their properties was also investigated. More specifically, nano BaSO_4 , with a size of ~100 nm, prepared by chemical precipitation, was also added to the alumina and silica mixtures. Cylindrical specimens with a diameter of 13 or 30 mm were formed by uniaxial pressing at ~200 MPa. The characterization of the produced ceramics showed that the amount and the nature of the alkaline earth oxide added to aluminosilicates affect ceramic microstructure and refractoriness.

The produced crucibles were filled with metal aluminum which was melted. The results showed that the ceramics' composition and microstructure (i.e. crystallinity, porosity), as well as the temperature and holding time, influence the interfacial reactions. A reaction zone formed in the ceramic consists of a porous ceramic structure infiltrated with Al. The formation of precipitates of intermetallic particles, attached at the interface or dispersed in the bulk metal, are important features, whose role was thoroughly investigated and will be comprehensively discussed. Temperature and holding time affect attachment/detachment of the precipitates. Therefore, the precipitates influence the reaction kinetics at the metal/ceramic interface and control infiltration process, ceramic erosion, and melt purity. Consequently, the investigated additives define phase formation and microstructure of the sintered ceramics and subsequently control the metal/ceramic interfacial reactions, which are always intensive.

Acknowledgment

This work was carried out in the framework of the project "Advanced Aluminosilicate Refractories and Magnesia Refractories of High Efficiency using Nanotechnology" that is co-financed by the European Regional Development Fund in the context of the special action "Industrial Materials" of the Operational Programme "Competitiveness, Entrepreneurship & Innovation (EPAnEK)", ΕΣΠΑ 2014-2020, (acronym: NanoRefrMat; project code: T6YBP-00386).

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REACTIONS BETWEEN MOLTEN ARTIFICIAL SLAGS OF OXIDE GLASSES AND REFRACTORY CERAMICS OF MgO-C DOPED WITH NANO- Al_2O_3 AND TiO_2

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This study reports on the interfacial interactions between molten oxide glasses made of oxide mixtures that represent the compositions of typical industrial slags and novel refractories based on MgO-C and doped with nano-alumina and nano-titania. The MgO-C refractories were prepared by adding up to 7 wt.% aluminum and/or titanium oxides nanoparticles (NPs) [1, 2]. Sol-gel method was applied to synthesize the Al_2O_3 NPs, while a microwave-assisted hydrothermal method via sol-gel process was used for the synthesis of TiO_2 NPs [3]. The NPs were added at $\text{Al}_2\text{O}_3:\text{TiO}_2$ ratio of 0/1, 0.5/0.5, 0.75/0.25, 0.25/0.75, and 1/0, in replacing equal amount of MgO fine powder. Pure MgO-C conventional refractories were also prepared for comparison purposes. Ceramic specimens in the form of crucibles and pellets were produced by conventional manufacturing techniques and sintering was carried out at 1400 °C [4]. The characterization of the produced ceramics showed that the nano-additives in the magnesia-carbon matrix improve the sintering process, resulting in refractory ceramics with higher density, lower porosity as well as higher cold crushing strength in comparison to that of conventional compositions.

The artificial slags had a composition of (in wt.%) 56CaO-11SiO₂-33Al₂O₃. Typical wetting experiments were performed at the temperature range of 1500 - 1650 °C for 15 - 120 min [5]. This presentation will present and discuss the results regarding the influence of the chemical composition of the ceramics on the interfacial reaction and the refractory behaviour of the produced ceramics.

Acknowledgment

This work was carried out in the framework of the project "Advanced Aluminosilicate Refractories and Magnesia Refractories of High Efficiency using Nanotechnology" that is co-financed by the European Regional Development Fund in the context of the special action "Industrial Materials" of the Operational Programme "Competitiveness, Entrepreneurship & Innovation (EPAnEK)", ΕΣΠΑ 2014-2020 (acronym: NanoRefrMat; project code: T6YBP-00386).

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10th International Conference on HIGH TEMPERATURE CAPILLARITY
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REFRACTORY BEHAVIOR OF NOVEL MgO-C CERAMICS, DOPED WITH NANO- MgO, TiO₂ AND ZrO₂, TOWARDS ARTIFICIAL OXIDE SLAGS

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The aim of this study was to assess the impact of nanoparticles' addition (MgO nanoparticles or combinations of TiO₂ and ZrO₂ nanoparticles) on the physical, chemical, including refractory properties, and mechanical properties of conventional MgO-C refractories with a graphite content of 9%. The MgO nano-powder additions studied were 1%, 3% and 5%, whereas the combinatorial additions of TiO₂ and ZrO₂ nanoparticles studied were 1%/1%, 2%/2%, 3%/3%. Nanopowders were produced in the lab by the sol-gel method and added to the mixture after dispersion of the resin (Novolac). The mixtures of raw materials were uniaxially pressed (250 MPa) and cylindrical specimens ($r = 1.3$ cm, $h = 0.5 - 0.8$ cm) were obtained. After polymerization of the resin, three sintering temperatures (1400, 1500, and 1600 °C) were investigated for the same time span (4 hours) in an electric furnace. Bulk density, apparent porosity, and water absorption were measured by Archimedes method, while mechanical properties were evaluated by CCS tests. The microstructures of the as-developed specimens were examined by XRD and SEM analyses. The specimens with MgO nanoparticles showed maximum density after firing at 1500 °C. The physical properties of TiO₂/ZrO₂-containing specimens were similar for all the sintering temperatures and slightly better in comparison with the nano-MgO-containing specimens. Eventually, combination syntheses gave the highest CCS values for the 2% TiO₂ / 2% ZrO₂ content.

Then, to investigate the refractory behavior of the produced materials, the interfacial interactions between molten oxide glasses made of oxide mixtures that represent the compositions of typical industrial slags and the qualified specimens were studied. More specifically, the artificial slag had a composition of (in wt.%) 56CaO-11SiO₂-33Al₂O₃. Typical wetting experiments were performed at the temperature range of 1500 - 1650 °C for 15 - 120 min [1]. This presentation will present and discuss the results regarding the influence of the chemical composition of the produced ceramics on the interfacial reaction zone and the refractory behavior of the produced ceramics.

Acknowledgment

This work was carried out in the framework of the project "Advanced Aluminosilicate Refractories and Magnesia Refractories of High Efficiency using Nanotechnology" that is co-financed by the European Regional Development Fund in the context of the special action "Industrial Materials" of the Operational Programme "Competitiveness, Entrepreneurship & Innovation (EPAnEK)", ΕΣΠΑ 2014-2020 (acronym: NanoRefraMat; project code: T6YBP-00386).

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THE CELLULAR PRECIPITATION OF A SUPERSATURATED SOLID SOLUTION IN THE Co-32 wt.%W ALLOY AT DIFFERENT ANNEALING TEMPERATURES

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Cellular precipitation of supersaturated solid solutions is a precipitation during which not two but only one new phase is released from the metal base against the background of a depleted solid solution. Cellular precipitation has its own peculiarity – it begins at high-angle grain boundaries, and if it does not occur in full volume (when the precipitation of supersaturated solid solution occurs at the same time by other mechanisms) it can lead to embrittlement of the material. After detecting the temperature limits of the cellular precipitation existence, it is possible to achieve the grinding of the grain structure of the material only through the heat treatment. However, what is interesting is the existence of two or more stages of cellular precipitation, where two identical solid solutions in the material with different parameters of the crystal lattice co-exist. Co-W alloys in the temperature range of 600-900 °C age reveal exclusively the cellular mechanism. By analysing the change of the material's volume during aging, it is possible to construct the dilatometric isotherms, which characterize only the general picture of this change. Since the precipitation can be caused not only by primary, but also by secondary cellular reactions, it is necessary to separate these phenomena. On the other hand, when studying Pb-Sn system alloys, the technique of numerical differentiation was applied after the measurements of change in electrical resistance during aging, which made it possible to separate the stages of precipitation in time. According to the kinetics of the process of formation of new pseudo boundaries, it occurs at the junctions of overgrown primary cells, where the nuclei of secondary cells can appear, when there is sufficient supersaturation of the solid solution at a given temperature [1]. By applying the method of numerical differentiation to dilatometric isotherms, it was possible to obtain the co-existence of two solid solutions due to the cellular reaction in the examined in this work cobalt-tungsten system at different annealing temperatures. As the studied alloy is a ferromagnetic, therefore it also reveals two Curie points, which makes it an interesting technological material, as determination of the time parameters of each stage creates the basis for designing the materials with desired properties.

Acknowledgements

The research was done as a part of a doctoral thesis conducted at «Kinetic and thermodynamic parameters of diffusion processes at grain boundaries of metal solid solution», state registration number 0107U009634. Dr. Yulia Vronska's research stay at the Institute of Metallurgy and Materials Science of the Polish Academy of Sciences is funded through a support program launched within the scientific cooperation between the Polish Academy of Sciences and the National Academy of Sciences of Ukraine.

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