13th conference on Advanced Materials Research (AMR 13)

APRIL 8-10

Ecole Européenne d'Ingénieurs en Génie des Matériaux

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13th AMR conference
8-10 April 2026
Ecole Européenne d'Ingénieurs en Génie des Matériaux

	Wednesday April 8		**Thursday April 9**		**Friday April 10**
Morning	***Scientific sessions***		***Industrial session***		***Scientific sessions***
	Lunch		**Lunch**		**Lunch**
Afternoon	***Scientific sessions***		***Social event***		***Lab tours***
			Gala diner		

Wednesday April 8

8:15-9:00 am	**Welcome**
9:00-9:10 am	**Opening**
	Session chairs: Antonio TRAVIESO and Flavio SOLDERA
9:10-9:30 am	***Alexia Chabot, Romain Giaux, Harena Rakotozafy, Mazarine Remy-Paquay, Stéphane Godet*** *Additive Manufacturing & microstructural design @ 4MAT, Université Libre de Bruxelles*
9:30-9:45 am	***Oliver Maurer*** *Reconditioning of metallic powder materials from industrial waste for Additive Manufacturing*
9:45-10:00 am	***Miguel Grande Molina, Jessica Calvo, José María Cabrera, Sara Fernandez, Felip Fenollosa, Pere Barriobero, Juan Francisco Almagro, Andrés Núñez, Esther Real, Itsaso Arrayago*** *3D Printing of stainless steel components by means of a novel Direct Energy Deposition Technology*
10:00-10-15 am	***T. Lourdet, C Jobard, R. Comte, N. Ramenatte, L. Portebois, S. Matthieu, M. Vilasi*** *Surface functionalization of additively manufactured Inconel 625 components with complex internal geometries*
10:15-10-30 am	***Walter Crupano, Jordi Llumà, David Sánchez-Molina, Ramón Jerez-Mesa, J. Antonio Travieso-Rodríguez*** *Viscoelastic Behavior of FFF-Manufactured PLA/PHB: Effects of Print Orientation and Processing Conditions*
10:30-10:40 am	***poster pitch 1***
10:40-11:20 am	**Coffee break and poster session 1**
	Session chairs: Alessandro MARTUCCI and Raul BERMEJO
11:20-11:35 am	***Raul Bermejo*** *Architectural design of 3D-printed alumina-based multimaterial components with enhanced damage tolerance*
11:35-11:50 am	***S. A. Razavi, M. Morales, T. Vilella, I. Serrano, M. A. Laguna-Bercero, L. Llanes, D. Rodríguez, M.-P. Ginebra, J. Llorca, G. Fargas*** *Additive manufacturing of cobalt–zirconia monolithic catalysts via direct ink writing for hydrogen production*
11:50-12:05 pm	***Maria Basso, Elena Colusso, Alessandro Martucci*** *Pulsed laser-annealed VO2 thin films for smart windows and gas sensors applications*
12:05-12:20 pm	***Alejandro Carlos Iglesias-Jaime, Alexandre Desforges, Alexandre Bouché, David Horwat*** *X-ray Photoelectron Spectroscopy Study on Copper Growth by ALD on High-Conductivity Al-Doped ZnO*
12:30-02:00 pm	**Lunch break**
	Session chairs: Karen LIENKAMP and Clemens HOLZER
02:00-02:15 pm	***Clemens Holzer*** *Research at the Institute of Polymer Processing, Leoben, Austria*
02:15-02:30 pm	***Yannic Wagner, Marc C. Thiel, Karen Lienkamp*** *Powder Aerosol Deposition and Polymers – Is There Hope for A Common Future?*
02:30-02:45 pm	**Carole Arnal-Herault, Thomas Arzoumanian, Maria Valentina Velasco Rueda, Bouchra Belaissaoui, Marc Ponçot, Nathalie Steunou, Subharanjan Biswas, Eliane Espuche and Anne Jonquieres** *New Composite Membranes with Very-High ZIF-8 Metal-Organic Framework Loading with high Performance for* CO_2 *Capture*
02:45-03:00 pm	**Chaimaa Raouf, Taha El Assimi, Firouze Oukhey, Monica Bertoldo, Redouane Beniazza, Mohammed Lahcini** *Sustainable Amphiphilic Poly(Ɛ-caprolactone)-g-Carboxymethyl Cellulose Bio-composites for coating water-soluble fertilizers for slow release*
03:00-03:30 pm	***François Montaigne*** *- Research activities at Institut Jean Lamour*
03:30-03:45 pm	***poster pitch 2***
03:45-04:15 pm	**Coffee break and poster session 2**
	Session chairs: Irene CALLIARI and Ilana TIMOKHINA
04:15-04:30 pm	***Andrea Gomez, Zainab Al-Maqdasi, Mikko Aleksi Salonen, Asmaa M. Ahmed, Juha P. Heiskanen, Roberts Joffe*** *Overview of recent results in development of partially biobased polyesters from furfural-derived monomers for environmentally friendly composites*
04:30-04:45 pm	**I.Calliari, F.Ahani, R.Elvira, T.Faraji, M.Oliboni, M.Pigato, J.E. Rey, A.Villalta, P.Barriobero-Vila** Low density steel for forged automotive components
04:45-05:00 pm	***Ilana Timokhina, Marta-Lena Antti*** *Correlative Microscopy: Concepts and Materials Science Applications*
05:00-05:15 pm	***Romain Giaux, Alexia Chabot, Eduardo Reverte, Alan Michel Vaissiere, Julien Zollinger, Stéphane Godet*** *High-throughput exploration of Fe-Si alloy printability using compositionally graded DED samples*

Thursday April 9

08:45-09:00	**Welcome and opening**
09:00-09:10	***Sébastien Borraccino - Direction des Partenariats de l'Université de Lorraine***
	Session chairs: Zoubir AYADI and Julien ZOLLINGER
09:10-9:35 am	***Julien Zollinger - Institut Jean Lamour*** *Solidification of metallic alloys: current status and challenges*
09:35-10:00 am	***Fabrice Wavelet - Ferry Capitain*** *How to reduce cracking sentivity of high alloyed steels straight from solidification ?*
09:00-10:25 am	***Cécile Bernardi - Institut de Soudure*** *Solidification microstructure using Wire-arc additive manufacturing processes: influence of processing parameters*
10:25-11:00	**Coffee break**
11:00-11:25 am	***Nazim Abdedou - ABS Centre Métallurgie*** *Steel Solidification at ABS: Process-Oriented Study of Solidification in Steel Casting*
11:25-11:50 am	***Pascal Lamesle - IRT M2P*** *High-Performance Circular Metals: When Residuals Decide the Limits of Recycling*
	Session chairs: Jessica CALVO and Julien ZOLLINGER
11:50-12:45 am	**Roundtable - Topical discussion**
01:00-02:30 pm	**Lunch break**

Friday April 10

08:40-09:10 am	**Marie-Odile Simonnot -** *Research activities at Laboratoire Réactions et Génie des Procédés*
	Session chairs: Antonio MATEO and Rachid HAKKOU
09:10-09:25 am	***Rachid Hakkou*, *Salma En-naji, Safaa Mabroum, Samira Moukannaa, Yassine Taha, and Mostafa Benzaazoua*** *Valorization of Phosphate Mine Wastes through Geopolymerization Technology for Construction and Depollution Applications*
09:25-09:40 am	***Youssef Ech'chalh*, *Raymond Michels, Catherine Lorgeoux, Valérie Burkle-Vitzthum*** *Thermochemical Conversion of Polypropylene: Influence of Waste Road Asphalt on Pyrolysis Products*
09:40-09:55 am	***Stefanie Arnold*, *Volker Presser*** *Hybrids for Electrochemical Technologies: From Batteries to Desalination and Closed-Loop Recycling*
09:55-10:10 am	***L. Jacob*, *T. Gries, G. Marcos, M. Hautier, T. Czerwiec*** *Looking for alternative surface treatments to coatings for bipolar stainless steel PEMFCs plates*
10:15-10:30 am	***poster pitch 3***
10:30-11:20 am	**Coffee break and poster session 3**
	Session chairs: David BUSQUETS-MATAIX and Thierry CZERWIEC
11:20-11:35 pm	***V. Amigó, A. Cárcel, M.D. Salvador, E. Giménez-Torres, A. Borrell, David Busquets*** *Research lines at the Materials Technology Institute at UPV*
11:35-11:50 am	***A. S. Ahmed*, *D. W. Mueller, J. F. Pierson, F. Muecklich*** *Surface Modification of Copper Alloys for Antibacterial Applications*
11:50-12:05 am	***Jan Grzelak*, *Ji-Young Yoon, Ines Bueso-Inchausti, Anna Cloeren, Maria-Pau Ginebra*** Engineered Hydroxyapatite Nanotopographies for Antimicrobial Bone Graft Substitutes
12:05-12:20 pm	**closing**
12:30-02:00 pm	**Lunch break**

Color code

Additive manufacturing

Surface engineering and thin films

Polymers and composites

Metallic materials and related methods

Materials science for energy and valorization of materials

Activities of research laboratories

Poster session 1 - Wednesday April 8 Morning

R. Demol, B. Laubie, J.L. Morel, G. Echevarria, M.N. Pons, M.O. Simonnot
Agromining: an innovative, low-impact approach to metal recovery

C. Jobard, T. Lourdet, R. Comte, R. Rouyer, L. Portebois
Detecting alpha case in titanium: beyond standard chemical etching

Jacob Hungerford, Tobias Prötsch, Serkan Nohut, Martin Schwentenwein, Raul Bermejo
Tailoring the pore morphology in 3D-printed porous alumina ceramics

Mahima Haque, Abdullah Jabr, Martin Müller, Raul Bermejo
Data-Driven Microstructure Optimization of Spark Plasma Sintered Ceramics

Yannic Wagner, Marc C. Thiel, Karen Lienkamp
Powder Aerosol Deposition - Influence of Polymer Substrate Characteristics and Deposition Parameters on Early TiO_2 Film Growth

Meryem Boutgoulla, Yousra Zghoundi, Filippo Balzano, Piergiorgio Tataranni, Cesare Sangiorgi, Yahya Agzenai Ben Salem and Rachid Hakkou
Enhancing the self-healing potential of cold-recycled asphalt pavements: a combined heating and recompaction approach

Khaoula Mouih, Safaa Mabroum, Fatima Ezzahra El Abbassi, Yassine Taha, Rachid Hakkou
Sustainable Construction Materials from Phosphate Mining By-Products: Performance and Environmental Evaluation of Compressed Earth Bricks

Hamza Beniddar, Inés Garcia Lodeiro, Fatima-Ezzahra El Abbassi, Rachid Hakkou
Phosphate waste rocks as an alternative source of durable fine aggregates for mortar production

Oumaima Fakraoui, Leonard Ionut Atanase, Slim Salhi, Isabelle Royaud, Mourad Arous and Zoubir Ayadi
Smart pH-Responsive Packaging Films Using Anthocyanin-Based Alginate/PVP/ZnO Nanocomposites

Poster session 2 - Wednesday April 8 Afternoon

Ahmed Anflousse, Abdelghani El Mouat, Jérémy Ternel, Michel Ferreira, Hervé Bricout, Hicham Ben Youcef, Sébastien Tilloys, Eric Monflier, Mohammed Lahcini

Crosslinked chitosan with hydrophobic jojabo oil as eco-friendly coating materials for water-soluble fertilizers

Fatima-ezzahrae Bammou, Mouad Balbal, Essaleh Mohamed, Rachid Bouferra, Younes Bahammou

Thermo-mechanical properties of Bentonite clay for Eco-Construction through Compaction and Sintering

T. Lourdet, C Jobard, R. Comte, A. Guéguen, L. Portebois

Development of a corrosion test bench and evaluation of the corrosion resistance of aluminium heat exchangers

Anna Seltmann, Volker Presser

Ionic Liquids as Electrolytes in Supercapacitors

K. Glücks, K. Ademmer, K. Lehnert, A. Rosenhahn, K. Lienkamp

Towards Sustainable Antifouling Materials for Marine Applications: Bromotyrosine-Functionalized Polymer Coatings

Emrick MURAT, Isabelle ROYAUD, Quentin GODINOT, Christophe ROOS, Maxime CHEVALIER

Élaboration et caractérisation physico-chimiques de nouveaux biocomposites
à base d'algues Sargasses et de déchets de l'agro -
industrie antillaise à visée d'exploitation en qualité d'isolants thermique
et phonique

Delphine Veys-Renaux, Ana Gasco-Owens, Charlotte Beaudouin, Emmauel Rocca

Designing white anodized layers on Al

KAIL Nathan, FOURMENTIN Claire, ROUXEL Tanguy, GODET Stéphane

Glasses of R_2O-B_2O_3-Al_2O_3 systems: study of structure-mechanical properties relationships at the metaluminous joint (R = Li, Na, K)

L. Martínez Orozco, N. León, R. M. Michell, O. Santana Pérez, M. Ll. Maspoch Ruldua

Impact of Reactive Extrusion on Crystalline Family Distribution
in Opaque Recycled PET Revealed by SSA–DSC

J. Gómez-Monterde, T. Abt, N. Candau, M. Sánchez-Soto, M. Ll. Maspoch

Integration of Computed Tomography and Numerical Simulation for the Mechanical and Fracture Behavior Analysis of Polypropylene

Yahya Ait fora, Seif Eddine Cherif, Fatima-ezzahra El Abbassi

Role of Motar in Enhancing the Seismic Performance of Traditional Masonry Structures

Poster session 3 - Friday April 10 Morning

Yousra Zghoundi, Meryem Boutgoulla, Piergiorgio Tataranni, Cesare Sangiorgi, Rachid Hakkou, Hicham Fenniri, Yahya Agzenai Ben Salem
Microwave-induced rejuvenator release for on-demand self-healing asphalt mixtures using biopolymeric capsules

Oumaima Fakraoui, Isabelle Royaud, Mourad Arous and Zoubir Ayadi
Mechanical–Dielectric Dynamic Correlations Using Fractional Modeling: Application to Polylactic Acid

Khouloud Bahloul, Zoubir Ayadi, Mourad Arous, Isabelle Royaud
Pollen-Reinforced PVA/Gelatin Biocomposites: A Sustainable Approach for Active Food-Packaging Films

J.-P. Tinnes, M. Bouita, , K. Ben Hafsia, I. Royaud, M. Ponçot
Mesophase, stress induced crystallization and strain tensor at the macromolecular scale : input of Raman spectroscopy

Laia Ortiz-Membrado, Antonio Mateo, Emilio Jiménez-Piqué
Large-Area Nanoindentation Mapping for Microstructure–Property Analysis in Heterogeneous Materials

Ramón JEREZ-MESA, Eric VELÁZQUEZ-CORRAL, Murat Sarp KOÇAK, Kevin BARRERA-LLANGA, J. Antonio TRAVIESO-RODRÍGUEZ
Decoding Ultrasonically Textured Surfaces: Multimodal Height Analysis and Functional Layer Identification via GMM

Cristian Franco, Lisbeth Cot, Luis Llanes, Miguel Morales, Gemma Fargas
3Y-TZP Gyroid Ceramic Monoliths produced by Digital Light Processing: Slurry Optimization and Post-Processing

Mariola Cano-Morenilla, Jordi Rodrigo, C. García-Mintegui, M. Vedani, Marta Pegueroles
Resorbable Polymeric and Zn-Based biomaterials for vascular applications

Tània Vilella, Daniel Rodríguez, Carlos Botero, Lars-Erik Rännar, Gemma Fargas
In-situ alloying of Titanium-Niobium alloys via Electron Beam Melting

A. Barjola, D. Busquets, A. Cárcel, E. Giménez Torres, O. Sahuquillo, A. Sonseca
Synthesis of MXene Nanomaterials with EMI Shielding Functionalities

Mohamed Aklalouch, Houda Anter, Asma Ihchemeyn, Manar Khachane, Aziza Khalil and Mohammed Mansori
From waste to worth: Efficient regeneration of NMC111 from spent motorcycle LIBs

Abstracts for oral sessions of April 8 and 10

Additive Manufacturing & microstructural design @ 4MAT, Université Libre de Bruxelles

Alexia Chabot[1,a], Romain Giaux[1,b], Harena Rakotozafy[1,c], Mazarine Remy-Paquay[1,d], Stéphane Godet[1,e]

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[d] mazarine.remy.paquay@ulb.be, [e] stephane.godet@ulb.be

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Keywords (6 maximum): AM, FGM, alloy development, LMD, microstructural design

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Abstract: The Additive Manufacturing (AM) research group within the 4MAT department of the *Université Libre de Bruxelles* focuses on developing a fundamental understanding of microstructural mechanisms in order to design novel metallic alloys with optimised macroscopic properties. The main research goal is to establish clear relationships between the processing conditions, microstructure evolution, and resulting material performances – notably mechanical and magnetic. Here, the macroscopic properties are optimized by controlling thermally and mechanically activated phenomena at the microscopic level. Such phenomena include phase transformations, precipitation, and texture development. This approach of “microstructural design” has enabled to develop optimised materials dedicated to a wide range of applications, including magnetic or aeronautics fields. The AM group’s research activities rely on state-of-the-art experimental tools gathered within the Nanoview platform. In this presentation, the platform’s resources will be introduced, covering the entire workflow: from numerical design and synthesis of novel alloy compositions to advanced, multi-scale microstructural characterisation. This workflow will be particularly illustrated on the design of new alloys using a high-throughput approach, combining numerical tools, multi-powder AM and casting processes, with innovative testing and microstructural characterisation methods. Eventually, these concepts will be illustrated through several representative case studies, focusing on aluminum, titanium, and Fe-Si magnetic alloys.

Reconditioning of metallic powder materials from industrial waste for Additive Manufacturing

Oliver Maurer

Re-Manufacturing Group at the Institute of Production Engineering, Saarland University, Campus A5.1, 66123 Saarbrücken, Germany

Keywords (6 maximum): Metal powder, Reconditioning, Recycling, Powder quality, Additive Manufacturing

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Abstract: The increasing demand for Additive Manufacturing (AM) has led to a significant generation of metallic powder waste, including oversized particles in virgin powder production or AM-specific residues such as smoulder and sieve residue. The efficient reconditioning of these waste streams is crucial for increasing both, economic and ecologic efficiency of AM, and conserving valuable resources. Our research focuses on the development of innovative methods for the processing and characterization of metallic powder waste, with the aim of utilizing them as feedstock for AM, specifically in Laser Powder-Bed Fusion. In addition to optimizing particle size and shape, investigations of the impact of chemical surface properties on the feasibility to recondition metallic powders is of special interest. This includes the effects of oxidation, which can lead to changes in color, as well as oxide removal. Furthermore, magnetic properties of nominally paramagnetic powders such as the austenitic stainless steel 316L can change to ferromagnetic behavior due to thermal history or mechanical loads. Additionally, examinations and tailoring of the flowability of the powders, which is a critical parameter for their successful application in AM, have to be part of reconditioning routines.

Investigating these research objectives, we aim to establish a comprehensive understanding of the complex relationships between powder properties and the possibilities of reconditioning, and to develop novel processing techniques or process chains that can efficiently convert waste powders into a high-quality feedstock for AM. By sharing our research objectives and first results, we look for collaborations with interested partners in the EEIGM or AMASE consortium.

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1. D. J. Turner, A. Seingell, Journal of Advanced Biology, 2025, 10, 1458936.
2. R. Thornton, I. Caplan, J. Stockton, r. Roberts, Journal of Physics, 2020, 125, 13879-13895.
3. …
4.
5. …

3D Printing of stainless steel components by means of a novel Direct Energy Deposition Technology.

Miguel Grande-Molina [a,b] , Jessica Calvo [a,b] , José María Cabrera [a,b] , Sara Fernandez [b] , Felip Fenollosa [a,b] , Pere Barriobero [a,b] , Juan Francisco Almagro [c] , Andrés Núñez [c] , Esther Real [a] , Itsaso Arrayago [a]

[a] Universitat Politècnica de Catalunya (UPC) – Barcelona Tech, Barcelona, Spain

[b] Fundació Centre CIM (CIM UPC), Barcelona, Spain

[c] Acerinox Europa SAU, Cádiz, Spain

Keywords (6 maximum): TIG, WAAM, Double-Wire, DED, Stainless-Steel, 3D printing

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Abstract:

The document should be 1 page maximum. Abstracts will be uploaded on the EEIGM Website. It will also be sought to upload the entire collection of abstracts on the HAL open platform except if you are against this possibility: https://hal.science/

Wire Arc Additive Manufacturing (WAAM) has emerged as a disruptive technology for large-scale metallic components due to its high deposition rates and material efficiency. This study presents the implementation and validation of a novel Direct Energy Deposition (DED) system based on a double-wire TIG-WAAM configuration, specifically designed for the fabrication of complex monomaterial and multimaterial structures. Using this setup, rectangular hollow sections of up to 2000 mm in length were manufactured employing a range of stainless steels, including austenitic (ER308L, ER309L), duplex (ACX 903), and ferritic (ACX 530) grades. Physical characterization confirmed high-quality builds characterized by the absence of porosity or inter-layer impurities. Microstructural analysis revealed a strong crystallographic texture oriented parallel to the build direction, a result of the rapid cooling cycles inherent to the process. While this led to anisotropic mechanical behavior, the ultimate tensile strength and hardness values remained consistent with as-welded material standards. Furthermore, the stability of these properties across the full length of the specimens demonstrates the high process homogeneity and thermal control of the double-wire TIG system. These results validate the potential of this technology for large-scale structural applications, although further optimization of process parameters is recommended to mitigate the effect of anisotropy in highly demanding industrial environments.

Surface functionalization of additively manufactured Inconel 625 components with complex internal geometries.

T. Lourdet[a], C Jobard[a], R. Comte[a], N. Ramenatte[b], L. Portebois[a], S. Matthieu[c], M. Vilasi[c]

[a] EEIGM, Halle des Matériaux – University of Lorraine

[b] Air Liquide – Reasearch & Development

[C] IJL – University of Lorraine

Keywords: surface functionnalization, additive manufacturing, complex geometries, slurry-based coating process

Abstract:

Additive manufacturing enables the production of components with complex geometries, including internal volumes that are difficult to access. However, many applications require surface state modification to ensure proper functionality, and the treatment of internal surfaces often represents a major challenge.

This presentation provides feedback on work carried out at the Institut Jean Lamour (IJL) and at the Ecole Européeenne d'Ingénieurs en Génie des Matériaux (EEIGM) from University of Lorraine concerning the surface functionalization of Inconel 625 components manufactured by laser powder bed fusion (LPBF), within the framework of the collaborative FAIR project (Additive Manufacturing for Reactor Intensification). Led by Air Liquide and involving several academic and industrial partners (Auer, Adisseo, Poly-Shape, IRCER, IJL, LRGP, CIRIMAT), the project aimed to develop compact intensified equipment for various industrial sectors (chemical, automotive, aeronautical, etc.).

The developments that ultimately led to an innovative process based on a suspension, enabling the single-step coating of several thousand internal channels with a protective alumina-forming β-NiAl coating, will be described.

Finally, the presentation will open perspectives on novel technological solutions developed at the University of Lorraine (IJL, EEIGM – Halle des Matériaux) related to corrosion protection, enhancement of conductivity, and surface smoothing of additively manufactured components.

Viscoelastic Behavior of FFF-Manufactured PLA/PHB: Effects of Print Orientation and Processing Conditions

Walter CRUPANO[a], Jordi LLUMÀ[a], David SÁNCHEZ-MOLINA[a], Ramón JEREZ-MESA[a], J. Antonio TRAVIESO-RODRÍGUEZ[a]

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Keywords: PLA/PHB blend; fused filament fabrication; viscoelasticity; stress relaxation; Prony series; additive manufacturing; print orientation; processing parameters

Abstract:

Additive manufacturing of polymeric components by fused filament fabrication (FFF) enables high geometrical flexibility and rapid customization, but it also introduces pronounced anisotropy and strong process dependence in the mechanical response, particularly under time-dependent loading. Accurate characterization of viscoelastic behavior is therefore essential for the reliable structural design of printed polymer components. This study investigates the viscoelastic properties of a commercial biodegradable PLA/PHB blend processed by FFF by means of long-term stress-relaxation experiments combined with a factorial design of experiments. The influence of filament processing state (unextruded filament, extruded filament, and printed parts), printing speed, and build orientation (intralayer and interlayer directions) is systematically quantified. Stress-relaxation curves were measured over time windows up to 1000 s and modeled using a quasi-linear viscoelastic formulation with a three-term Prony series. Excellent agreement between experiments and model predictions was obtained, with coefficients of determination exceeding 0.998 for all specimens. Analysis of variance demonstrates that both print orientation and printing speed significantly affect most viscoelastic parameters, while the processing state strongly modifies the relaxation moduli due to polymer chain alignment induced during extrusion and deposition. Specimens printed along intralayer directions exhibit higher stiffness and larger relaxation moduli than interlayer specimens, highlighting the critical role of interfacial bonding quality in layered manufacturing. Lower printing speeds are associated with longer characteristic relaxation times, indicating enhanced resistance to short-term stress decay and a more stable transient mechanical response.

Extruded filaments display higher initial relaxation moduli and maximum stresses than printed parts, whereas long-term relaxation behavior tends to converge, suggesting a partial homogenization of the viscous response at extended timescales. The extracted Prony parameters enable direct computation of time-dependent effective moduli and provide a physically interpretable link between microstructural features, processing conditions, and macroscopic viscoelastic performance. Overall, the proposed experimental–modeling framework supports predictive modeling, material selection, and process optimization for durable and sustainable additive-manufactured polymer components.

1. W. Crupano, B. Adrover-Monserrat, J. Llumà, D. Sánchez-Molina, R. Jerez-Mesa, J. A. Travieso-Rodriguez, *Progress in Additive Manufacturing*, 2026, **1**.
2. R. Tobajas, D. Elduque, E. Ibarz, C. Javierre, A. F. Canteli, L. Gracia, *Polymers 2018*, **10**(6).
3. A. M. Seifans, S. Ayyagari, M. Al-Haik, Aerosp Sci Technol, 2021, **111**, 106562.
4. R. Sattler, R. Zhang, G. Gupta, M. Du, P. M. Runge, H. Altenbach, M. Beiner Macromolecules, 2024, **57**(**7**), 3066.

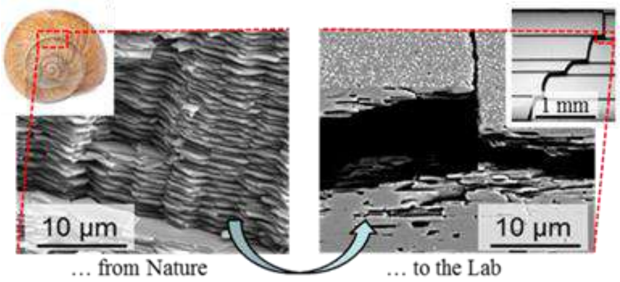
Architectural design of 3D-printed alumina-based multimaterial components with enhanced damage tolerance

Raul Bermejo

Chair of Structural and Functional Ceramics, Montanuniversität Leoben, Austria

Keywords: 3D-printing, alumina, texture, residual stress, multi-materials, damage tolerance.

Abstract: The combination of ceramics with other materials has enabled the fabrication of hybrid systems with exceptional structural and functional properties. However, a critical issue affecting the functionality and reliability of these systems is the initiation and uncontrolled propagation of cracks in the brittle ceramic parts. Bio-inspired design concepts in current ceramics engineering have proved successful in obtaining highly reliable ceramic materials with enhanced fracture resistance. For instance, tuning the location of "protective" layers within a ceramic multilayer architecture can significantly increase its fracture resistance, while retaining high strength. The use of tailored residual stresses in embedded layers can act as an effective barrier to the propagation of cracks from surface flaws, providing "damage tolerance". Moreover, by orienting (texturing) the grain structure, similar to the organized microstructure found in natural systems such as nacre, crack propagation can be tailored. The potential of additive manufacturing technology to design novel alumina-based ceramic systems is explored in this work. The first design concept (Fig. 1, left) is based on a multi-material design approach, where 3D-printed alumina layers are embedded between alumina-zirconia (ZTA)-layer regions. The tailored compressive residual stresses in the embedded alumina layer regions act as an effective barrier to crack propagation, providing a minimum strength for the architecture. The retained strength after thermal shock of the multi-material ceramic is significantly higher than that of the monoliths, owed to the crack arrest capability of the embedded layers. In the second design concept (Fig. 1, right), spatially tailored porosity in 3D-printing ceramic parts is introduced to enhance their damage tolerance. In such architectures, porous interlayers are printed by using a polymeric pore forming agent, which is removed after the debinding process. The specific strength of the architecture is governed by the outer-dense alumina regions, whereas the damage tolerance is enhanced through the weak porous interlayers. Based on the case studies, damage tolerant demonstrator components with tailored residual stresses (multi-ceramic blade) and porosities (ceramic nozzle) are designed, which may open new application fields in the future.



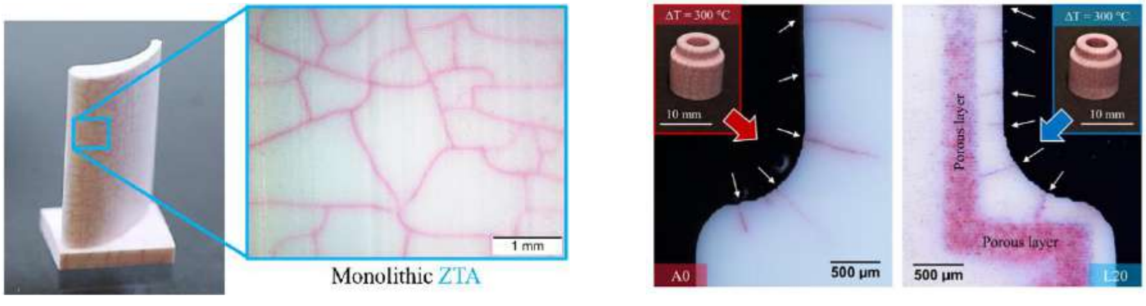


Fig.1: Examples of 3D printed multi-material architectures with tailored microstructures and architectures.

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Additive manufacturing of cobalt–zirconia monolithic catalysts via direct ink writing for hydrogen production

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Keywords; Direct Ink Writing; Zirconia; Monolithic catalyst; Co_3O_4; Ethanol steam reforming

Abstract: Additive manufacturing enables the fabrication of ceramic catalysts with tailored geometries while reducing manufacturing steps. This work proposes a single-step fabrication process of cobalt–yttria doped zirconia (Co–YDZ) monolithic catalysts based on Direct Ink Writing (DIW) of Co-enriched hydrogel-based ceramic inks and a single debinding/sintering thermal treatment at 600ºC. The effect of Co precursor amount (3.0–7.0 wt% Co) on ink rheology and catalytic performance in ethanol steam reforming for hydrogen production is investigated. Rectilinear monoliths were printed at 50% infill using inks with constant solid loading (70 wt%). The results show successful incorporation of Co into the monolith structure and strong Co enrichment at the surface after sintering. Catalytic tests between 200 and 600ºC reveal that Co-containing monoliths reach higher ethanol conversion and H_2 yield than the YDZ reference. The 5.0 wt% Co monolith provides the best balance between printability, structural integrity and catalytic performance, achieving complete ethanol conversion and the highest H_2 production between 300 and 600ºC.

INTRODUCTION

Heterogeneous catalytic reactions are typically carried out in fixed-bed reactors using powder or pellet catalysts, although these systems may present limitations such as low space rate, high pressure drop and hot spot formation in exothermic reactions. Honeycomb-like structured reactors can reduce pressure drop and diffusional resistance, but their conventional fabrication routes limit the achievable geometries and surface-to-volume ratio. Additionally, traditional monolithic catalyst preparation methods (e.g., impregnation and wash-coating) usually require multiple coating/drying cycles and multiple thermal treatments, which reduce reproducibility and increase processing time.

Additive manufacturing (AM) technologies, and particularly Direct Ink Writing (DIW), allow tailoring the geometry of structured catalysts (channel geometry, porosity and surface-to-volume ratio) to maximize mass and heat transfer. However, many AM approaches still rely on post-treatments for catalyst functionalization, again requiring multiple steps and thermal treatments [1, 2].

This work aims to fabricate Co–YDZ monoliths through DIW followed by a single-step thermal treatment to perform ethanol steam reforming as a proof-of-concept reaction for hydrogen production. Co is selected as a relatively cheap non-noble metal with proven catalytic activity for ethanol steam reforming, while yttria-doped zirconia (YDZ) is selected as a mechanically and chemically stable support [3].

METHODS

A Pluronic® F-127 hydrogel (32 wt% Pluronic, 68 wt% water) was prepared and cooled at 4ºC for 24 h. YDZ powder (3 mol% yttria partially stabilized zirconia) and cobalt (II) acetate tetrahydrate were mixed to obtain DIW inks with constant solid loading (70 wt%) and Co contents of 0, 3.0, 5.0, and 7.0 wt% (YDZ, 3Co-YDZ, 5Co-YDZ, and 7Co-YDZ). Rectilinear monoliths (22 mm diameter, 20 mm height) were printed at 50% infill using a ~580 µm nozzle at 4 $mm \cdot s^{-1}$, dried under controlled humidity, and treated in air in a single cycle up to 600ºC (200ºC 1 h, 450ºC 1 h, 600ºC 2 h; 1.5$ºC \cdot min^{-1}$ heating/cooling). Rheology, microstructure/chemistry (FE-SEM/EDS, Raman, XRD, XPS, HR-TEM/STEM-EDS), and catalytic performance in ethanol steam reforming (200–600ºC) were evaluated.

RESULTS

All formulated inks exhibited shear-thinning behavior, with viscosity decreasing as shear rate increased, which fulfills a key requirement for DIW processing. The Ostwald–de Waele fitting parameters showed a flow behavior index n below 0.4 for all compositions, confirming pseudoplastic behavior. Increasing Co precursor content led to an increase of the flow consistency parameter k, consistent with higher viscosity, while the yield stress decreased as Co content increased. Despite this reduction, storage modulus values in the linear viscoelastic region were above 105 Pa for all inks and the 3-interval thixotropy test indicated rapid recovery of solid-like behavior after high shear, supporting shape retention after extrusion. However, the 7 wt% Co formulation displayed the lowest yield stress, which may limit self-standing capability for larger prints.

Rectilinear monoliths were successfully printed at 50% infill and subsequently consolidated using a single-step thermal treatment in air that combined precursor decomposition, organic debinding and sintering. The thermal profile included plateaus at 200ºC, 450ºC and 600ºC, with the final dwell at 600ºC selected to prevent excessive Co_3O_4 particle growth. After sintering, cobalt-containing monoliths showed a pronounced color change compared to the YDZ reference, attributed to the conversion of cobalt(II) acetate tetrahydrate into cobalt oxide. Surface FE-SEM observations revealed semi-spherical macro-particles (approximately 50–200 µm) whose concentration and size increased with Co content. EDS mapping confirmed that these macro-particles were Co-rich and also showed Co presence distributed across filament surfaces, particularly for 5Co-YDZ and 7Co-YDZ. Cross-sectional observations evidenced macro-porosity inside the filaments, which became more relevant as Co content increased, Figure 1. Shrinkage values were nearly identical for YDZ, 3Co-YDZ and 5Co-YDZ, while 7Co-YDZ exhibited lower shrinkage, which was attributed to the formation of closed macro-pores generated by gas release during decomposition of the high amount of Co precursor required to reach 7 wt% Co.

Raman spectroscopy identified the macro-particles as spinel Co_3O_4. XRD patterns confirmed zirconia as the main phase and showed increasing intensity of Co_3O_4-related peaks with increasing Co content, demonstrating successful incorporation of cobalt oxide phases in the monoliths. XPS analysis further revealed that the Co concentration at the monolith surface was higher than the theoretical ink composition (4.6 wt% Co for 3Co-YDZ, 6.5 wt% for 5Co-YDZ and 9.3 wt% for 7Co-YDZ), indicating segregation and enrichment of Co species at the surface during sintering. At the nanoscale, STEM-EDS and HR-TEM analyses showed larger YDZ particles in close contact with smaller cobalt oxide nanoparticles. Co_3O_4 nanoparticles supported on YDZ particles (100–200 nm) were typically 5–15 nm, which is suitable for catalytic applications.

In ethanol steam reforming between 200 and 600ºC, Co–YDZ monoliths clearly outperformed the YDZ reference, reaching higher ethanol conversion at lower temperatures. Both 5Co-YDZ and 7Co-YDZ achieved high conversion already at 250ºC (around 81% and 96%, respectively), while complete conversion of YDZ required 350ºC. In terms of H_2 yield, 5Co-YDZ and 7Co-YDZ exhibited the highest values at 250–300ºC; above 300ºC, the H_2 yield of 5Co-YDZ remained very close to 3Co-YDZ and higher than 7Co-YDZ. Overall, 5Co-YDZ provided the best balance between printability, structural integrity and catalytic performance, achieving complete ethanol conversion and the highest H_2 production between 300 and 600ºC, Figure 2.

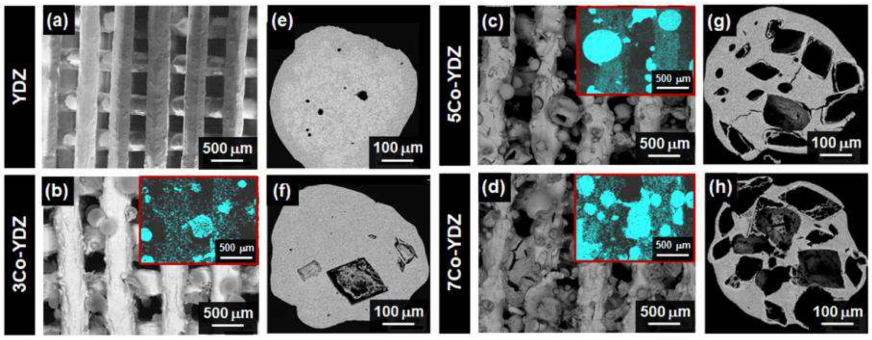

Fig. 1 – Surface/cross-section SEM and Co EDS maps (microstructure and Co distribution).

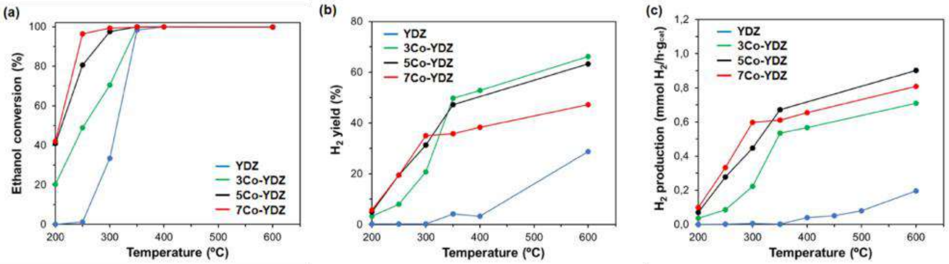

Fig.2.– Ethanol conversion, H_2 yield and H_2 production vs temperature (catalytic performance).

CONCLUSIONS

Co–YDZ monolithic catalysts were successfully fabricated using a novel single-step approach combining Direct Ink Writing of Co-enriched ceramic inks and a single debinding/sintering thermal treatment at 600ºC. Rectilinear monoliths (50% infill) were printed using inks with constant solid loading (70 wt%) and Co contents of 3.0–7.0 wt%. Characterization confirmed effective incorporation of Co, formation of Co_3O_4, and strong Co enrichment at the monolith surface. Catalytic tests in ethanol steam reforming showed improved performance compared to YDZ, with the 5.0 wt% Co monolith providing the best compromise between printability and catalytic activity, reaching complete ethanol conversion and the highest H_2 production between 300 and 600ºC.

Acknowledgments

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Pulsed laser-annealed VO_2 thin films for smart windows and gas sensors applications

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Keywords: sol-gel ; laser anealing, VO2 ; gas sensors ; smart windows.

Abstract: The temperature-dependence of the optical/electrical properties of vanadium dioxide (VO_2) thin films can be exploited for several applications, including thermal regulation of buildings and gas sensing. Upon a specific transition temperature (T_c ~ 68°C), which can be opportunely decreased, VO_2 undergoes an insulator-metal transition (MIT) with a concurrent structural and electronic variation. Current challenges of solution-based VO_2 thin films concern the high temperatures required for the crystallization step and the difficulty of controlling the final stoichiometry. As conventional thermal annealing requires temperatures above 400°C and controlled atmospheres, an alternative is offered by nanosecond pulsed laser annealing (ns-PLA). The latter allows for local temperature increases in the film within the ns pulses, without involving the substrate.VO_2 thin films were synthetized from a non-carcinogenic precursor and irradiated with a KrF excimer laser at room temperature. PLA was performed in air, without the need for an inert atmosphere. The optimal working window was defined by tuning the atmosphere during deposition, drying temperature, film aging, and crystallization time. The parameters were evaluated by GIXRD, SEM, TEM, UV-Vis-NIR and Raman spectroscopy, volt-amperometric measurements, XPS, RBS. Simulations were used to estimate the temperature distribution within the films during laser pulses. Different types of VO_2 films were prepared by varying the laser parameters and their efficiency was tested for two applications, i.e., smart windows and H_2 gas sensors. Crystallization was found to start from the first few pulses and full conversion to crystalline VO_2 (M1) was reached within a few seconds. Similar thermochromic efficiencies, in terms of modulation of the NIR transmittance, could be reached through multiple combinations of sol-gel parameters. Exploiting the environmental moisture, the drying temperature was reduced to 100°C. The morphology and crystallinity degree were found to affect the gas sensing activity, evaluated through resistivity variations upon H_2 exposure. The versatility of sol-gel reactions was combined with ultrafast PLA for the simultaneous crystallization and nanostructuring of VO_2 thin films. The applicability of the latter as energy-saving smart windows and H_2 gas sensors was proven. This widens the possibilities to exploit the MIT of VO_2 in multiple fields.

X-ray Photoelectron Spectroscopy Study on Copper Growth by ALD on High-Conductivity Al-Doped ZnO

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Keywords (6 maximum): Atomic Layer Deposition, Metal-oxide interface chemistry, X-ray photoelectron spectroscopy.

Abstract:

Understanding how metals interact with oxide surfaces at the atomic scale is essential for optimizing conductivity, stability, and device performance [1]. X-ray photoelectron spectroscopy (XPS) provides key insight into these interactions by revealing chemical states and surface transformations [2], while atomic layer deposition (ALD) offers the precision needed to study them through its self-limiting, layer-by-layer growth [3]. When copper is deposited by ALD, its controlled nucleation and oxidation pathways on conductive oxides such as ZnO become particularly relevant to transparent electronics and photoelectronic technologies [4]. In this work, we investigate copper deposited by ALD onto high-conductivity Al-doped ZnO substrates, correlating evolving surface chemistry with emergent transport behavior. Using XPS alongside Auger electron spectroscopy (AES), conductivity and morphology analysis, we trace the evolution from initial ALD adsorption events to continuous overlayer formation. Across broad deposition ranges and ambient conditions, we identify recurring oxygen-related signatures and copper aggregation or oxidation tendencies. From these trends, we propose a general growth model describing the transition from early adsorption to continuous overlayer formation on high-conductivity substrates.

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Research at the Institute of Polymer Processing, Leoben, Austria

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Keywords (6 maximum): polymer processing, recycling, additive manufacturing, applied research

Abstract:

Montanuniversitaet Leoben (MUL) is a specialised technical university focusing on circular engineering – from raw material via the production processes to the final product and back to the material via recycling. The Institute of Polymer Processing is part of the Department of Polymer Engineering and Science at MUL. Selected research work of the Institute of Polymer Processing in the fields of polymers and the production processes will be presented. The research is a balanced mix of basic and applied research with the aim of effectively transferring research results into practice and teaching. The focus is on these topics: extrusion, injection moulding, recycling, additive manufacturing, process simulation and material data. Together with the other institutes of the department– Chemistry of Polymeric Materials, Designing Plastic and Composite Materials, Processing of Composites, and Materials Science andTesting of Polymers – the whole process chain is covered.

Powder Aerosol Deposition and Polymers – Is There Hope for A Common Future?

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Keywords: polymer ceramic composites, powder aerosol deposition, powder processing, thin films, vacuum cold spray.

Abstract:

Powder aerosol deposition (PAD) has been developed in order to gain access to thin functional ceramics layers without sintering, which is important for many applications.[1] Compared to the body of work dedicated to the deposition of pure ceramics, research focusing on polymers-ceramics co-deposition is scarce, as reviewed recently.[2] This includes materials that can be applied as biocompatible coatings for implant materials, magnetic shielding films, hydrophobic and transparent glass coatings, and porous thermal insulation coatings. Also, important work investigating the co-deposition mechanism has been published.[3] Notably, the presence of polymers either as a substrate or a particle during co-deposition causes a shock-absorption effect, leading to plastic deformation of the polymer component and a substantial deviation from the room temperature impact and consolidation mechanism.[3] In recent work, we investigated two aspects in more detail: First, the deposition of only polymer particles onto polymer substrates using PAD, which is barely investigated so far;[4] second, the effect of the polymer powder processing on the formation and morphology of the resulting co-deposited polymer ceramic thin film.[5]

The deposition of pure polymer layers by PAD so far has not been investigated in great detail. Since the dynamics and mechanism of all-polymer PAD should differ substantially from the known PAD mechanisms, we started our investigations of this process by studying the high-velocity impact of spherical polystyrene (PS) particles onto polymer substrates both *in silico* using molecular dynamics simulations, and in reality by PAD. In the molecular dynamics simulations, the particle diameter and impact velocity were systematically varied. In the real-life experiments, a regular PAD set-up was used, but the substrate motion relative to the nozzle was set so high that single particle impacts could be observed. The simulated particle shapes showed good agreement with the experimental ones, deforming into shapes resembling cylindrical domes. Scaling behavior extracted from the simulations provided estimates of the otherwise not directly measurable experimental impact velocities and showed that the particle deformation mechanism during impact was driven by a temperature increase causing viscoplastic flow.[4]

We also prepared isotropic and multilayer-like polystyrene/titanium dioxide composite thin films by PAD. Such composites combine functionality, mechanical robustness and flexibility. However, their fabrication is challenging, because the high temperatures typically needed for ceramics processing are incompatible with most polymers. PAD offers a solvent- and sinter-free avenue towards PCCs. So far, it has not been investigated how the preparation route of the composite powders dictates the film formation process during PAD, or the microstructure of the obtained films. To address this, we investigated the system PS-titanium dioxide (TiO_2). PS particles synthesized via emulsion polymerization were combined with TiO_2 particles to yield either inhomogeneous or homogeneous powders. When deposited on polycarbonate and steel, they produced fundamentally different microstructures, with organized, multilayer-like films emerging from the inhomogeneous powders, and isotropic films from the homogeneous ones. These structural differences correlated with variations in crystallite size revealed by X-ray diffraction, which - combined with quantification of the actual polymer content of the films determined by thermogravimetric analysis - provided new insight into the role of internal shock absorption of the polymeric component absorption during impact.

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New Composite Membranes with Very-High ZIF-8 Metal-Organic Framework Loading with high Performance for CO_2 Capture

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Keywords: composite membranes; mixed matrix membranes; segmented copolymer; CO_2 separation.

Abstract (< 300 words):

Reducing greenhouse gas emissions is a key objective of the 2030 International Agenda for Sustainable Development, particularly in the fight against climate change. This study focuses on the development of highly efficient composite membranes, called mixed matrix membranes (MMMs,) for CO_2 capture. A CO_2-philic poly(urea-imide) segmented copolymer containing a high proportion of poly(ethylene oxide) (70 wt%) [1] was used as a rubbery, non-crystalline polymer matrix and combined with the metal–organic framework ZIF-8.

To achieve high filler loadings and good compatibility between the polymer and ZIF-8, a multiple priming technique was employed, allowing ZIF-8 contents of up to 70 vol%. The structure and morphology of the resulting membranes were investigated using FTIR, DSC, and SEM analyses to assess polymer–filler interactions. Gas permeation properties for CO_2 and N_2 were measured under post-combustion capture conditions (2 bar and 35 °C) using time-lag experiments.

The membranes exhibited excellent performance at high ZIF-8 loadings. At 70 vol% of ZIF-8, CO_2 permeability reached 471 Barrer, representing an 8.5-fold increase compared to the pristine polymer, while maintaining a high CO_2/N_2 selectivity of 30.2. Several theoretical models (Maxwell, Bruggeman, Lewis–Nielsen, and Pal) were applied to predict gas transport behavior. All models accurately described membrane performance at low ZIF-8 contents (≤ 20 vol%), but only the Maxwell model remained effective up to 60 vol%. None of the models could predict performance at 70 vol% due to particle aggregation and interfacial defects.

Overall, the best-performing membrane ranks among the most efficient MMMs reported for CO_2 capture using rubbery polymers and ZIF-8 [2]. The introduction of a third component to form ternary MMMs is proposed as a promising strategy to further enhance performance and overcome permeability–selectivity trade-offs in a future work.

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Sustainable Amphiphilic Poly(Ɛ-caprolactone)-g-Carboxymethyl Cellulose Bio-composites for coating water-soluble fertilizers for slow release

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Keywords : Slow-release fertilizer, amphiphilic polymer, poly(ε-caprolactone), carboxymethyl cellulose, green chemistry, controlled release.

Abstract:

To overcome the main drawbacks related to conventional fertilizers, industrials and researchers have proposed a new generation of water-soluble fertilizer to work around the unbalanced and fast nutrients release from conventional granular fertilizers[1]. In fact, the coating materials are generally used to encapsulate fertilizers to delay the nutrients release rate. However, the non-biodegradation of the coating's material remains the major limitation of the proposed solution[2]. In that regard, an amphiphilic and biodegradable co-polymer has been prepared following the grafting from approach[3], where the poly(ε-caprolactone) (PCL) chains were grafted from carboxymethyl cellulose CMC (PCL-g-CMC). It is worth mentioning that the synthesis pathway adopted has been carried out in free solvent condition and without the use of organometallic catalysts. A high molecular weight of PCL chains grafted was estimated using size exclusion chromatography (26000 g.mol-1). Furthermore, the amphiphilic biocomposite prepared was fully characterized using 1H NMR, FTIR, X-ray diffraction, DSC, TGA, and DLS to investigate its structural, thermal and morphological properties. In the second part, spherical diamonium phosphate (DAP) granules were coated by the prepared copolymer (PCL-g-CMC) using a laboratory rotary drum to obtain a uniform coating thickness (~45µm) as confirmed by cross section images observed by SEM. Afterwar, the release rate of phosphorus (P_2O_5) in water and sandy soil was investigated to approve the nutrient slow-release property. Finally, the efficiency of the prepared fertilizers (DAP-coated) has been tested in wheat (Triticum Durum) compared to conventional fertilizer (DAP-uncoated).

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Overview of recent results in development of partially biobased polyesters from furfural-derived monomers for environmentally friendly composites

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Keywords : biobased, furan, UPR, thermosets, thermo-mechanical properties, moisture

Abstract: Today most composites are still produced with fossil-based resources, with high environmental impact. Biobased polyester resins can be seen as a sustainable alternative to reduce consumption of fossil-based polyester. As these resins are intended to be used in green composites, they are likely to be combined with natural (e.g. cellulosic) fibers, having enormous potential to improve sustainability of composite materials. This study is focused on a fully biobased 5,5'-sulfanediyldi (furan-2-carboxylic acid) (SFA) replacement monomer for fossil-based phthalic acid, and fumaric acid (FA) in combination with different proportions of diols and curing agents at different concentrations [1]. For each formulation, mechanical, thermal, electrical properties as well as moisture influence on stiffness have been studied to determine their performance compared to current commercial polyester resins. In the study, Vickers' hardness was used as an indicator of stiffness and revealed that moisture absorption (below 2 w/w%) leads to a reduction in hardness [2,3,4]. Formulations containing glycols show greater susceptibility than those with propanediol. These formulations were narrowed down to 4 candidates, which their properties (e.g. UPR_2 tensile modulus E_t = 3.70 ± 0.07 GPa, maximum tensile stress σ_m= 68±8.9 MPa, and elongation at break ε_b= 2.72 ± 0.99 %) were strongly comparable to conventional fossil-based polyesters (Reference material with tensile modulus E_t = 3.93 ± 0.18 GPa, maximum tensile stress σ_m= 40.9 ± 11.1 MPa and elongation at break ε_b= 1.26 ± 0.34 %). Reinforcing their potential for high performance, structural composite applications.

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Low density steel for forged automotive components.

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Reducing vehicle weight is a key strategy to lower fuel / electricity consumption / CO2 emissions and achieve automotive emission reduction goals Development of high-strength, low-density steels is essential to achieving industry emission limit objectives. Fe-Mn-Al-C low-density steels represent a promising class of materials that provide an alternative to both traditional steels and other lightweight alloys, but steels have a huge range of compositions, and properties, mostly linked to different microstructures. Low density Fe–Mn–Al–C steels are promising candidates for structural forged parts owing to their capability to contribute in reducing the vehicle weight mainly achieved through alloying with aluminium (Al) (~1.3% density reduction per 1 wt.% Al).In the present project a focus on high-strength low-density steels (<7 g/cm3; 800-1200 MPa) is done, aimed at the most promising microstructures and forging properties Thermodynamical Calculations were used to study the effect of Alloying on the Solidus Temperature, Austenite Fraction, and carbides . The selected compositions (C=0.6-1.1%), Mn=25-30%, Al=7-9%) were casted , hot forged and hot rolled and the characterized by SEM, XRD, Compression Tests, Thermal Hardness test, corrosion tests, Preliminary results on hot working parameters and recommended chemical composition were obtained , taking account the balance between strength and ductility, precise control of microstructure evolution and prevention of detrimental intergranular precipitates. Duplex ferrite-austenite microstructure show very low hot ductility, Intergranular kappa carbides promote cracks while fully austenitic microstructure presents the best hot ductility . Cost-Performance Balance suggests that the economic feasibility of Fe-Mn-Al-C steels requires balancing higher costs against performance benefits and environmental compliance advantages.

Laboratory Research → **Industrial Application**

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Correlative Microscopy: Concepts and Materials Science Applications

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Keywords: correlative microscopy, atom probe tomography, segregation to grain boundary, clusters.

Abstract: Advanced steels designed for sustainable and energy-efficient applications exhibit complex microstructures governed by chemical and structural heterogeneities spanning multiple length scales. Capturing these features requires the combined use of complementary characterisation techniques. This extended abstract describes the principles and application of correlative microscopy, integrating scanning electron microscopy (SEM), transmission electron microscopy (TEM), and atom probe tomography (APT), to establish robust microstructure–property relationships in advanced high strength steels. The approach enables direct correlation between crystallographic features and local chemistry, providing critical insights into phase transformations, solute redistribution, and strengthening mechanisms relevant to industrial alloy development.

INTRODUCTION

The development of modern advanced high-strength steels (AHSS) relies on precise control of microstructure through alloy design and processing [1]. Critical phenomena such as solute segregation, atomic clustering, nanoscale precipitation, and phase transformations occur over length scales ranging from micrometres down to individual atoms. Conventional characterisation techniques typically provide only partial information, which can lead to incomplete or ambiguous interpretations of microstructural evolution. Correlative microscopy has therefore emerged as a powerful strategy to overcome these limitations by integrating complementary analytical techniques to deliver a comprehensive and self-consistent description of microstructure across multiple length scales.

Atom probe tomography (APT) enables three-dimensional, atom-by-atom mapping of chemical distributions within metallic materials [2]. Electron backscatter diffraction (EBSD) in the scanning electron microscope (SEM) provides crystallographic information and phase identification at the microscale, while high-resolution transmission electron microscopy (TEM) offers direct imaging of nanoscale structural features and defects [3]. The combined application of these techniques allows direct correlation between local chemistry, crystallography, and microstructure, thereby revealing how nanoscale features govern strength and toughness. Such understanding enables researchers and steelmakers to design steels with improved performance, reduced weight, and lower energy demand during production, supporting the transition toward more sustainable and fossil-free steel manufacturing.

METHODS

SEM-based electron backscatter diffraction (EBSD) was performed using an FEI Quanta 3D field-emission gun scanning electron microscope (FEG-SEM) operated at an accelerating voltage of 20 kV and a probe current of 8 nA. EBSD maps were acquired with a step size of 0.25 μm. Transmission electron microscopy (TEM) investigations were conducted using a JEOL JEM-2100 conventional LaB_6 TEM operated at 200 kV (Fig. 1).

APT specimens were prepared from selected regions of interest using a focused ion beam (FIB) in situ lift-out technique in the same FEI Quanta 3D FEG-SEM (Fig. 1) [3]. Atom probe tomography analyses were carried out using a CAMECA LEAP 4000HR operating in voltage-pulsed mode with a pulse fraction of 20%, a pulse repetition rate of 200 kHz, and a specimen temperature of 60 K. Three-dimensional reconstructions and data analysis were performed using IVAS software version 3.6.12.

RESULTS

One representative example of correlative microscopy is the investigation of non-equilibrium niobium (Nb) segregation at prior austenite grain boundaries (PAGBs) induced by welding thermal cycles. This phenomenon was experimentally studied using combined SEM–EBSD and APT in a steel containing 0.06 at.% Nb (Fig. 2) [3].

PAGBs were first identified based on EBSD orientation maps, after which site-specific APT specimens were prepared along the selected boundaries (Fig. 2).

The Nb concentration at the PAGBs was measured to be approximately 0.115 at.% following a simulated welding thermal cycle with a holding time of 10 s during cooling from 1320 to 950 °C. Using the experimentally determined APT data and a non-equilibrium segregation model, the diffusion coefficient of Nb associated with the desegregation process was calculated at 950 °C and determined to be 9.23×10^{-11} $m^2 s^{-1}$ (Fig. 3). Based on these results, the kinetics of non-equilibrium Nb segregation at PAGBs during welding were established, and the competing segregation and desegregation processes were discussed in detail [3].

Another example of correlative microscopy is the analysis of precipitation hardening in ferritic steels through the combined use of aberration-corrected high-resolution transmission electron microscopy (HR-TEM) and APT. This approach provides direct atomic-scale evidence for the precipitation pathway, revealing the evolution from embryo clusters to fully developed nanoscale precipitates (Fig. 4). Carbide precipitation initiates with disc-shaped embryo clusters consisting of only a few atomic layers, which are fully coherent with the ferritic matrix. With increasing diameter, these embryo clusters evolve into solute-rich clusters analogous to Guinier–Preston zones observed in aluminium alloys [4]. Upon further growth, particularly in thickness, the clusters undergo a structural transformation into a new phase with a NaCl-type crystal structure, thereby contributing significantly to precipitation strengthening.

DISCUSSION

The results presented demonstrate the strength of correlative microscopy in resolving complex, multiscale phenomena governing the microstructural evolution of advanced steels. By combining SEM–EBSD and APT, non-equilibrium Nb segregation at PAGBs induced by welding thermal cycles could be directly quantified with both spatial and chemical resolution. EBSD enabled reliable identification and reconstruction of PAGBs, while site-specific APT provided atom-by-atom compositional information along the selected boundaries. This approach overcomes the limitations of conventional characterization techniques, which are unable to simultaneously capture crystallographic context and nanoscale chemical segregation.

The measured Nb enrichment at PAGBs following the simulated welding thermal cycle confirms that rapid thermal excursions can promote non-equilibrium segregation even for substitutional alloying elements with relatively low diffusivity. The calculated diffusion coefficient of Nb at 950 °C, derived from APT data and a non-equilibrium segregation model, is in good agreement with literature values, lending confidence to both the experimental methodology and the kinetic interpretation. These results highlight the competing nature of segregation and desegregation processes during welding, which are highly sensitive to thermal history and cooling rates. Such insights are critical for understanding grain boundary stability, phase transformation behavior, and susceptibility to grain boundary-related degradation mechanisms in welded steels.

The second example further illustrates the capability of correlative microscopy to elucidate strengthening mechanisms at the atomic scale. The combined use of aberration-corrected high-resolution TEM and APT reveals a continuous precipitation pathway in ferritic steels, from solute-rich embryo clusters to fully developed nanoscale precipitates. The observation of disc-shaped, coherent embryo clusters supports the concept that precipitation initiates through chemically ordered clusters analogous to Guinier–Preston zones. The subsequent structural transformation into a NaCl-type phase with increasing size and thickness provides direct evidence for the gradual transition from clustering to precipitation, rather than a sharp nucleation event. This atomic-scale understanding is essential for linking processing conditions to precipitation kinetics and mechanical strengthening.

Together, these examples demonstrate how correlative microscopy enables the direct correlation of crystallography, morphology, and chemistry across multiple length scales. Such integration is particularly valuable for advanced steels, where mechanical performance is governed by subtle nanoscale features that are strongly influenced by processing history.

CONCLUSIONS

Correlative microscopy provides a powerful and robust framework for investigating microstructural phenomena in advanced steels across length scales ranging from the micrometre to the atomic level. The combined application of SEM–EBSD, TEM, and atom probe tomography enables comprehensive characterization of both structural and chemical heterogeneities that cannot be resolved by individual techniques alone.

In the case of Nb-containing steels subjected to welding thermal cycles, correlative SEM–EBSD and APT analysis allowed direct quantification of non-equilibrium Nb segregation at prior austenite grain boundaries and enabled the determination of diffusion kinetics governing segregation and desegregation processes. In ferritic steels, the integration of aberration-corrected TEM and APT provided atomic-scale evidence of the precipitation pathway from coherent embryo clusters to nanoscale precipitates, clarifying the mechanisms responsible for precipitation strengthening.

These results underline the critical role of correlative microscopy in advancing the fundamental understanding of microstructure–property relationships and in supporting the development of optimized alloy compositions and processing routes. As steel production moves toward more sustainable and energy-efficient technologies, such integrated characterization approaches will be essential for accelerating alloy and process design while ensuring reliable industrial implementation.

FIGURES/SCHEMATICS/TABLEs.

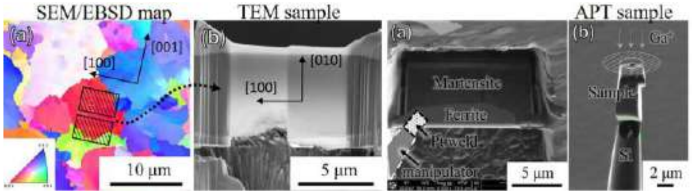

Fig. 1. Site-specific sample preparation for correlative microscopy

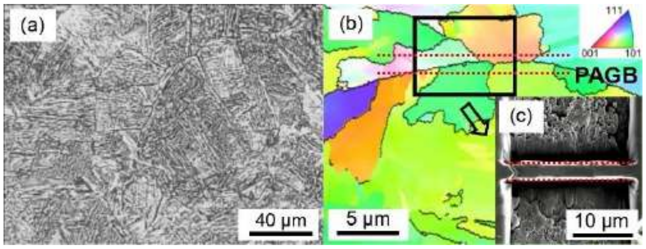

Fig. 2. APT sample preparation from PAGB found by EBSD.

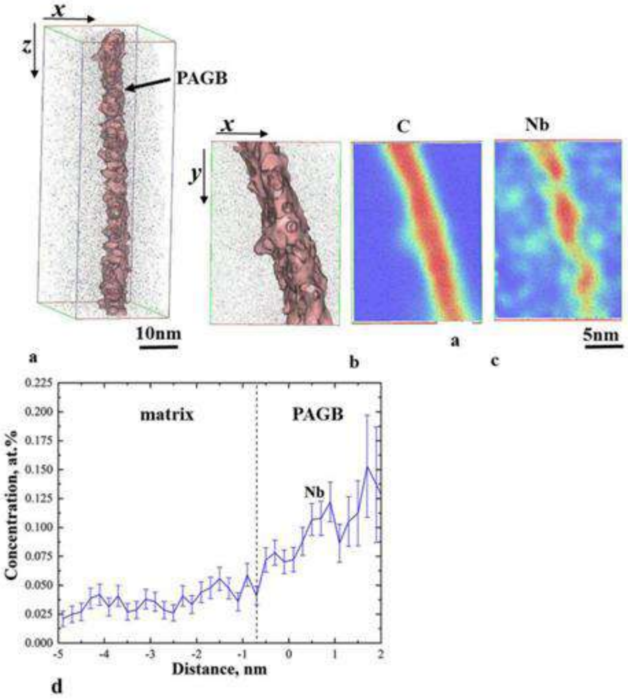

Fig. 3. (a) iso-surface (2.9 C and 0.12 Nb at.%) in two orientations, (b) 2-D C and Nb, (d) Nb proximity histogram accross the PAGB in (b).

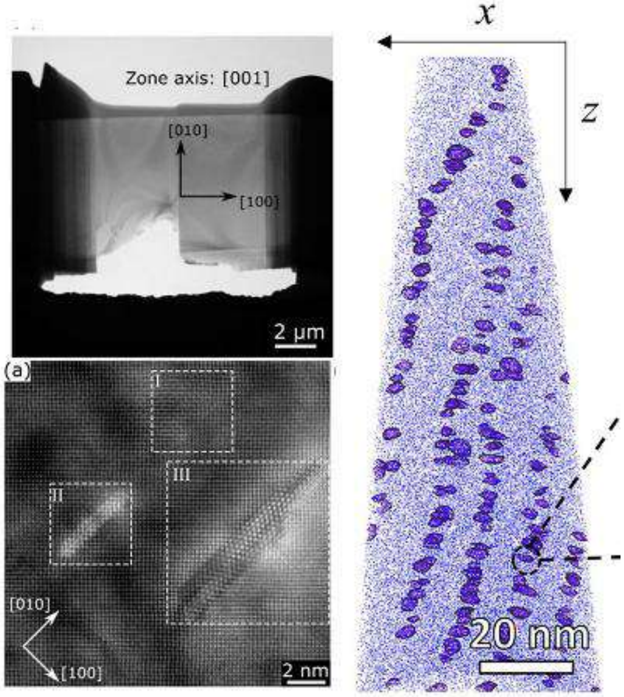

Fig. 4. (a) TEM sample and bright field image showing formation of GP zone and clusters, (b) atom probe map from similar region.

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High-throughput exploration of Fe-Si alloy printability using compositionally graded DED samples

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Keywords (6 maximum): Fe-Si alloys, Direct Energy Deposition, Cracking behaviour, High-throughput

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Abstract:

Iron Silicon (Fe-Si) alloys are considered key functional materials for electrical and magnetic applications due to their high electrical resistivity, low core losses, and favourable soft-magnetic properties. Increasing the silicon content improves magnetic performance but also leads to a marked loss of ductility, making alloys with Si content above 4wt% difficult to process using conventional and additive manufacturing methods. In this study, compositionally graded Fe-Si samples were produced by Direct Energy Deposition (DED) to investigate the printability and cracking behaviour of high Si iron alloys.

Multiple graded samples were printed with silicon content ranging from 6.5wt% to 3wt% in 0.5wt% increments, using powder mixing of Fe-6.5wt%Si and pure Fe powders. This method enables rapid exploration of composition and property relationships within a single build, making it particularly attractive for high-throughput materials research. Optical microscopy revealed crack formation in the Fe-6.5wt%Si region, while the regions with 6wt% silicon content and below showed crack free behaviour. These observations highlight a sharp printability threshold linked to the intrinsic brittleness and cracking susceptibility of high-Si iron alloys under the conditions imposed by DED.

The printing campaign was conducted at the Institut Jean Lamour (IJL) in Nancy and represents an initial step in a newly established and growing collaboration between IJL and the Université Libre de Bruxelles (ULB). Beyond providing insight into Fe-Si alloy printability, this work demonstrates the potential of compositionally graded DED as a powerful tool for accelerated alloy development. Future work will focus on microstructural analysis, crack-mitigation strategies, and comparative studies with LPBF printed Fe-Si alloys, further strengthening this emerging partnership.

Valorization of Phosphate Mine Wastes through Geopolymerization Technology for Construction and Depollution Applications

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Keywords: *Phosphate mine wastes; Geopolymerization; Alkaline activation; Acid activation; Sustainable construction materials, Depollution.*

Abstract:

The phosphate mining industry produces large quantities of waste, which causes environmental and management issues. In Morocco, phosphate mine wastes such as clays, and marls are generally deposited on the surface within mine sites, leading to large land occupation and costly management approaches [1]. Although these mineral by-products are inert and chemically stable, their valorization remains a major concern. Geopolymers, generally considered as alternative cements to Portland cement, offer a sustainable route for waste valorization. The viability of geopolymerization technology as an environmentally friendly management and valorization method for Moroccan phosphate mine wastes was investigated. Different by-products from phosphate interlayers and beneficiation plants were used as raw materials. Depending on their nature, various treatments and activation methods were applied. Calcination was used to increase the reactivity of the materials before using them as precursors in geopolymerization, which were then activated through alkaline activation, alkaline fusion, or acid activation using phosphoric acid. Fly ash and metakaolin were used in some formulations to adjust the chemical composition. The results showed that the reactivity of phosphate mine wastes strongly depends on the nature of the precursor and the activation method. Red clay-based mortars in a fly ash matrix reached compressive strengths up to 39 MPa [2]. Calcined phosphate sludge achieved compressive strengths up to 62 MPa when combined with 50 wt% fly ash, and alkaline fusion allowed its incorporation up to 70 wt% in the formulations [3,4]. Calcined yellow clays and marls led to geopolymer binders with compressive strengths of 25 MPa and 38 MPa, respectively [1,5].Mineralogical and microstructural analyses revealed that alkaline activation led to the formation of new crystalline phases and amorphous binding gels, predominantly C-A-S-H, N-A-S-H, and N-C-A-S-H. In contrast, acid activation using phosphoric acid produced crystalline phosphate phases such as aluminum phosphate ($AlPO_4$), monetite ($CaHPO_4$), brushite ($CaHPO_4 \cdot 2H_2O$), and newberyite ($MgHPO_4 \cdot 3H_2O$), coexisting with an amorphous Si–Al–P gel [6,7]. The acid-activated geopolymers, prepared by adding 25 wt% calcined yellow clay to metakaolin, were successfully applied for methylene blue adsorption, exhibiting a removal efficiency of 98%, which was rapidly achieved due to the increase in specific surface area from 38.4 m^2/g to 115.9 m^2/g. Overall, the results confirm that geopolymerization, through both alkaline and acid activation, is a promising approach for the sustainable management and valorization of phosphate mine wastes, contributing simultaneously to construction applications and the depollution of wastewater.

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Thermochemical Conversion of Polypropylene: Influence of Waste Road Asphalt on Pyrolysis Products

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Keywords : Pyrolysis, Polypropylene, waste road asphalt, synergy, bitumen, reflux

Abstract:

Over the past five decades, global plastic production has increased by nearly a factor of twenty, driven by the steady expansion of human activities and industrial demand [1]. By 2015, packaging applications accounted for approximately 40% of total plastic production, largely due to the functional and economic advantages of plastic materials in this sector [1]. However, nearly 95% of plastic packaging items are discarded after a single use, leading to major challenges in waste management and generating an estimated annual economic loss of $80–120 billion [2].

Pyrolysis represents an attractive approach for the recovery and valorization of plastic wastes[3] in the context of circular economy strategies[4]. In this work, the thermal conversion of polypropylene (PP), waste road asphalt, and their blends was investigated in order to identify possible synergistic interactions and to maximize liquid product formation. The experiments were conducted in a semi-batch reflux reactor, operating under an inert nitrogen atmosphere[5]. The feedstocks were heated from room temperature to 500°C at a rate of 10°C min^{-1}. The resulting products were analyzed using GC–MS/FID, µ-GC, XRD, SEM and ^{1}H-NMR.

Introducing asphalt into the feedstock significantly influenced the distribution of products, although the chemical profile of the volatile fraction remained largely unchanged. A pronounced synergistic effect was observed during the co-pyrolysis of PP and asphalt, resulting in an oil yield of 76.4 ± 1.2 wt.% at a PP-to-asphalt mass ratio of 2 : 1 corresponding to an increase of approximately 6.3 ± 0.7 wt% compared to pure PP. This enhancement is attributed to the role of bitumen, which mitigates the catalytic cracking effects of minerals. The liquid products obtained from the mixtures exhibited chemical properties similar to those derived from PP pyrolysis, with only slight variations in viscosity, indicating that asphalt addition does not adversely affect oil quality.

Overall, the results demonstrate that the co-pyrolysis of polypropylene and asphalt waste is a promising route for the concurrent upgrading of polymeric and bituminous residues into valuable hydrocarbon streams, supporting sustainable chemical recycling pathways.

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Hybrids for Electrochemical Technologies: From Batteries to Desalination and Closed-Loop Recycling

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Keywords (6 maximum): Energy storage, MXene, batteries, desalination

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Abstract: MXenes, a rapidly expanding family of two-dimensional transition metal carbides and nitrides, have emerged as a highly versatile materials platform for electrochemical energy storage and beyond. Owing to their high electrical conductivity, tunable surface chemistry, and layered structure, MXenes can serve multiple roles in electrochemical devices, ranging from active electrode materials to conductive scaffolds and stabilizing matrices.[1]

Here, we present recent advances in MXene-based hybrid materials for lithium-ion batteries, sodium-ion batteries, electrochemical desalination, and sustainable recycling concepts. First, the effect of interlayer engineering is demonstrated by silica-pillaring of $Ti_3C_2T_z$ MXenes, increasing the interlayer spacing to ~1.75 nm. This structural modification significantly enhances ion transport, resulting in improved capacities, rate capability (>150 mAh/g at 1 A/g) and cycling stability (71% capacity retention after 500 cycles). However, the charge storage remains largely governed by ion intercalation, limiting the achievable capacity.[2]

To overcome this limitation, MXenes were hybridized with alloying and conversion-type materials. Antimony/MXene hybrids exhibit high reversible capacities up to 450 mAh/g at 0.1 A/g and excellent rate performance (365 mAh/g at 4 A/g) in sodium-ion batteries, with the best performance achieved for homogeneously distributed Sb rather than maximum Sb loading or particle size minimization.[3] Extending this concept to more abundant materials, SnO_2/MXene composites with an optimized 1:1 ratio deliver stable capacities of 525 mAh/g over 700 cycles and high-rate performance of 340 mAh/g at 8 A/g, enabled by the synergistic buffering and charge-transport properties of MXenes.[4]

Beyond energy storage, asymmetric MXene/activated-carbon cells are shown to enable efficient electrochemical desalination without membranes, achieving charge efficiencies up to 85% in seawater-level salinity and desalination capacities up to 12 mg/g.[5] Finally, a closed-loop recycling strategy for binder-free MXene electrodes is presented, achieving >90% capacity recovery and enabling upcycling into TiO_2/C composites for catalytic applications.[6]

Together, these results highlight MXenes as a multifunctional materials platform enabling high-performance, resource-efficient, and sustainable electrochemical technologies.

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Looking for alternative surface treatments to coatings for bipolar stainless steel PEMFCs plates

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Bipolar Plate, PEMFC, thermochemical treatment,

Abstract:

Proton exchange membrane fuel cells (PEMFCs) are a key technology in the hydrogen sector. They convert hydrogen and oxygen into electricity through electrochemical reactions, operating silently and without polluting emissions. To be competitive, PEMFCs must meet stringent requirements such as durability, high performance, and low cost. A crucial component of PEMFCs (approximately 30 % of the total cost) is the bipolar plate (BPP), which handles current transfer, gas distribution and separation, and water management. Various materials can be used, but stainless-steel plates offer a good compromise between mechanical strength and manufacturing cost. Their electrical conductivity needs however improvement. To address this, protective coatings are typically applied to the BPPs. The use of these coatings faces cost and durability challenges. We hereby explore the possibilities of thermochemical treatments to replace coatings on stainless steel BPPs.

Using post-discharge plasma oxidation processes provides a uniform surface coverage, even on complex geometries, and offers fine chemical selectivity through adjustment of process parameters. In this context, we will present in this communication the results obtained on the selective oxidation of iron contained in stainless steel. The characterizations we conducted are based on chemical and structural analyses (GDOES, SEM, TEM, XPS) and interfacial contact resistance (ICR) measurements to evaluate the influence of thermochemical parameters on surface composition and morphology. Our observations show that post-discharge plasma treatment promotes the migration of iron atoms to the surface, resulting in the formation of a continuous iron oxide layer a few nanometers thick covered with discontinuous iron oxide agglomerates.

Surface Modification of Copper Alloys for Antibacterial Applications

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Keywords: direct laser interference patterning, ultrashort pulsed laser, copper-alloys, surface modification, antibacterial properties

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Abstract:

The widespread use of antibiotics has led to a rapid increase in multidrug-resistant (MDR) microorganisms, contributing to a growing number of severe and often fatal hospital-acquired infections[1]. One approach to limit their spread is the use of surfaces that actively kill bacteria on contact[2]. Combining such antimicrobial materials with advanced surface engineering can enhance their effectiveness[3]. Copper shows strong antibacterial activity, but its mechanical properties restrict practical applications. Targeted alloying offers a way to broaden its use, as long as the antibacterial effect is preserved.

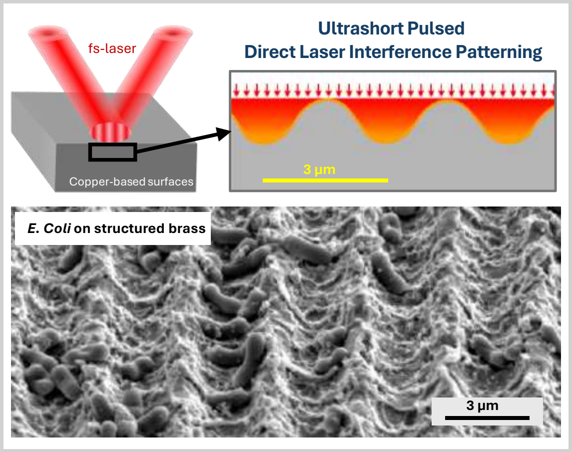

Figure 1: SEM image of brass structured by femtosecond-pulsed direct laser interference patterning (fs-DLIP) showing periodic microscale features ~3 µm, comparable to a single E. coli cell.

In this study, copper-based alloy systems were investigated for their suitability in laser-assisted surface functionalization. Ultrashort-pulsed direct laser interference patterning (USP-DLIP)[4] was used to produce periodic surface structures with dimensions comparable to bacterial cells on various copper alloys, aiming to enhance contact-dependent bacterial killing. The influence of alloy composition on laser-material interaction, surface chemistry, and wetting behavior was examined, followed by an assessment of the antibacterial performance of the modified surfaces[5]. Correlating USP-DLIP-induced topographical and chemical modifications with variations in antibacterial activity provides insight into the role of alloying elements and supports the identification of copper alloy systems suitable for optimizing antimicrobial surfaces.

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Engineered Hydroxyapatite Nanotopographies for Antimicrobial Bone Graft Substitutes

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Keywords (6 maximum): calcium phosphate; hydroxyapatite; titanium oxide; antimicrobial surface; high-aspect-ratio nanotopography; bone graft

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Abstract: Bacterial infections associated with implantable biomaterials remain a critical challenge in orthopedic and maxillofacial applications. Bioinspired nanostructured surfaces offer a promising, antibiotic-free strategy to reduce bacterial colonization through physical mechanisms. Here, we investigated the controlled formation of mechanobactericidal nanotopographies on calcium-deficient hydroxyapatite (CDHA) produced by hydrolysis of α-tricalcium phosphate. Reaction temperature, pH, duration, and amount of nucleation seed were systematically varied to tailor surface nanostructure. Three representative morphologies: interconnected nanosheets, densely packed needles, and nest-like nanowire assemblies, were selected for biological evaluation. To decouple topographical from material effects, analogous nanotopographies were accurately replicated on TiO_2 surfaces and their antibacterial performance against *Pseudomonas aeruginosa* and *Staphylococcus aureus* was directly compared. All nanostructured surfaces exhibited significant bactericidal activity against *P. aeruginosa*, with no statistically significant differences between CDHA and TiO_2, confirming the dominant role of nanotopography. For *S. aureus*, reduced metabolic activity was observed without detectable cell wall damage, particularly for needle-like structures, indicating an indirect antibacterial mechanism. These results demonstrate that diverse antibacterial nanotopographies spanning a broad range of dimensions can be engineered on calcium phosphate materials through controlled processing, highlighting their potential for antibiotic-free infection-resistant biomaterials.

Abstracts for the industrial session of April 9

Solidification of metallic alloys: current status and challenges

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Keywords : Solidification, Alloys, Nucleation, Growth, Microstructures

Abstract:

Solidification is a phase transformation involving a wide range of spatial and temporal scales, both for describing its fundamental mechanisms and for its consequences during product manufacturing. These multiple spatial and temporal scales require a multi-physics approach. In this presentation, we will provide an overview of current research in the field of solidification, ranging from phase nucleation (nanometer and picosecond scales) to macrosegregation in ingots (meter and tens-hour scales). A particular focus will be made on growth, microsegregation et mass transport is liquid and solide liquide misxtures leading to chemical heterogeneities at the scale of products.

We will conclude by discussing the experimental and numerical challenges that must be addressed to answer the scientific questions that remain unanswered today, in relation to industrial issues.

How to reduce cracking sentivity of high alloyed steels straight from solidification ?

Fabrice Wavelet[a]

[a] Ferry Capitain, France

Keywords (6 maximum): refractory steels, carbide network, cracking sensitivity, solidifcation

Abstract:

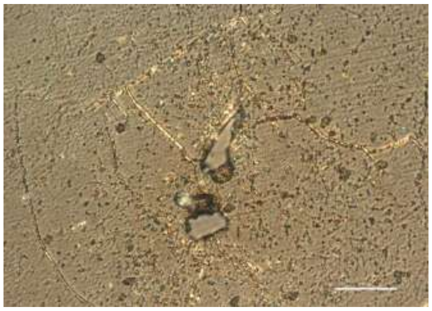
Industries such as Aerospace, Metal working and Oil and Gas are using high alloyed steels in very different and demanding service conditions. Reliability and durability are key words for these applications, ie Super Plastic Forming, Steckel Mills and Oil field explorations.

The steel grades used shall undergo high temperatures and stresses, or highly demanding service and safety conditions.

Typically, these steels have an austenitic matrix associated with a large number of carbides, either precipitating in the grain interfaces or inside the grains.

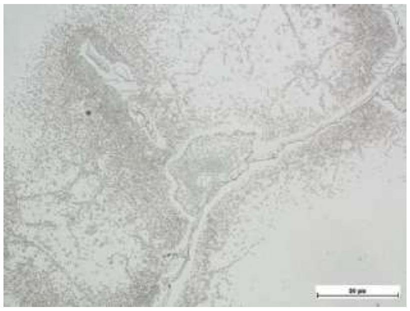
Principal mode of failure is cracking. Recent analysis demonstrated that cracks are mostly initiated in the grain boundaries where a continuous carbide network can be found. As such, these carbides, formed at high temperatures during the solidification process, are both useful for the global resistance and detrimental to the cracking sensitivity, tearing it down to very low values against fracture mechanics ,and making welding repairs of cast defects very difficult. Finding a correct compromise between these beneficial and detrimental effects of carbides goes through a modified distribution of carbides in the grain boundaries.

Goal of a presentation of this topic in the Advanced Material Research Conference will be to introduce the current state of understanding from an industrial perspective and the consequence on different products and applications of such carbide distribution. Available data can be shared with academics should further investigations be considered of interest.



Steel 15% Mn, 1.8% Mo, 0.6% V

Cracks in 15% Mn Steel



Steel 20% Cr, 37% Ni, 0.4% Nb

Full circumferencial crack, coiler drum

Thermal fatigue crack, SPF matrix

Arc-DED Fabrication: Impact on Microstructure and Material Properties

Etienne Krafft,[a] Cécile Bernardi[a]

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Keywords (6 maximum): Arc Directed Energy Deposition (DED-arc), Thermal Cycles, Deposition Strategy, Microstructure Evolution, Mechanical Properties, Additive Manufacturing

Abstract:

Arc-based Directed Energy Deposition (DED-arc) is an additive manufacturing process that involves the layer-by-layer deposition of material using an electric arc as a heat source. Similar to conventional welding processes, the thermal cycles experienced by the material during fabrication play a critical role in determining the resulting microstructure and mechanical properties. These thermal cycles are primarily governed by welding energy input, as well as preheating and post-heating conditions. However, in the context of additive manufacturing, an additional parameter must be considered: the deposition strategy, including bead size, geometry, and spatial distribution.

The deposition strategy significantly influences heat accumulation and dissipation within the material. Unlike traditional welding, where a single pass or limited number of passes are applied, DED-arc involves multiple successive layers, leading to complex and repeated thermal cycling. Variations in bead overlap, interpass temperature, and deposition sequence can alter cooling rates and thermal gradients. As a result, different regions within the fabricated component may experience heterogeneous thermal histories, which directly affect microstructural evolution.

Microstructural observations reveal that these thermal conditions can lead to the formation of diverse grain morphologies. For instance, high heat input and slower cooling rates tend to promote grain growth and coarser microstructures, whereas optimized deposition strategies can refine the grain structure and enhance material homogeneity. The presence of reheating cycles may also induce phase transformations or tempering effects, further modifying the microstructure across deposited layers.

Mechanical properties are closely linked to these microstructural features. Tensile strength, hardness, and ductility can vary significantly depending on the thermal history imposed by the deposition strategy. Experimental results typically show that finer and more homogeneous microstructures lead to improved mechanical performance, while excessive heat accumulation may degrade properties due to grain coarsening or the formation of defects.

In this study, illustrative micrographs are presented to highlight the relationship between deposition strategy and resulting microstructures. These are complemented by mechanical testing data, which demonstrate how variations in process parameters influence material performance. The combined analysis underscores the importance of carefully designing deposition strategies in DED-arc processes to control thermal cycles, tailor microstructures, and optimize mechanical properties.

Overall, understanding the interplay between process parameters, thermal history, and material response is essential for advancing the reliability and performance of arc-based additive manufacturing technologies.

Steel Solidification at ABS: Process-Oriented Study of Solidification in Steel Casting

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Keywords : Steel solidification, Continuous casting, Ingot casting, Macrosegregation, Numerical simulation, Solidification strucutres

Abstract:

Solidification control remains a fundamental challenge for Acciaierie Bertoli Safau (ABS), an Italian long-product steelmaker. While production takes place at the Udine plant, covering continuous casting of various profiles (round, square, octagonal) and ingot casting, the research and development efforts are centralized at ABS Centre Métallurgique (ACM) in Metz. This presentation details how ACM integrates advanced metallurgical characterization with numerical simulation to better comprehend the solidification process.

The first axis of the R&D activities at ACM covers experimental characterization. This involves chemical etching to reveal solidification structures, precise measurements of primary and secondary dendrite arm spacings, and chemical macro-segregation mapping. These experimental data points serve as the foundation for the calibration of numerical models. By integrating temperature measurements from the casting lines, ACM aims to develop high-fidelity digital twins designed to predict and prevent internal defects and scrap.

A second central axis concerns the numerical simulation of solidification processes, both for continuous casting and ingot casting. Simulations are performed using Thercast (Transvalor), a software dedicated to casting and solidification processes, offering robust capabilities for thermal, mechanical, and metallurgical coupled computations. This model is used to investigate solidification kinetics, chemical segregation [1], as well as various other phenomena such as asymmetric cooling conditions and their effects on bloom bending. For ingot casting, studies address inclusion flotation and the effect of hot-top design and exothermic powder thickness on solidification.

In parallel, ACM is a partner in the development of OpenSolid, a new 3D finite volume model for segregation simulation in continuous casting and ingot casting. This initiative brings together several major industrial groups and academic laboratories, including IJL, with the ambition of simulating complex solidification phenomena ranging from detailed modeling of chemical segregation to columnar-to-equiaxed transition (CET) prediction.

Future developments aim to introduce the complexity arising from electromagnetic stirring (EMS) and submerged entry nozzle (SEN) in the liquid steel flow for continuous casting, as well as bulging and soft-reduction effects on segregation. For ingot casting, attention is also given to the role of canal geometry on filling flow and to accounting for inhomogeneous thermal conditions introduced by neighboring ingots.

The ultimate goal of this dual approach, combining experimental measurements with numerical simulations, is to minimize casting defects and optimize yields through a high-fidelity digital representation of the solidification process.

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High-Performance Circular Metals: When Residuals Decide the Limits of Recycling

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Keywords (6 maximum): Recycling, circularity, residuals, properties, sustainability, sovereignty

Abstract:

Recycling of titanium, steels and foundry aluminium addresses both strategic issues (securing supply, industrial sovereignty) and environmental ones (reducing energy use and CO_2 emissions). For titanium, the recovery of well-controlled production scraps makes it possible to produce high-quality materials, whereas end-of-life parts, which are more contaminated and more oxidised, require sorting, deoxidation or dilution, because increased residual levels (O, H, Fe, etc.) degrade ductility, toughness and sometimes aeronautical or medical qualification.

For steels, the development of the electric-arc route increases the share of recycled scrap, but copper and tin, which are almost impossible to remove in secondary metallurgy, become limiting: above very low contents, they cause hot shortness and loss of ductility, which requires intensive scrap sorting and controlled dilution to maintain high-grade steels.

In foundry aluminium alloys, the increase of Fe in recycled streams leads to the formation of Fe-rich intermetallic phases that are brittle, their effect being worsened by silicon; fatigue resistance and ductility are reduced, and composition (Mn, adjustment of Si, possibly Cu) and solidification conditions must be tuned to remain within an acceptable performance window. In this context, one key research line focuses on second-melt foundry aluminium alloys. The objectives are to characterise the effective properties (mechanical behaviour, corrosion resistance, etc.) of selected recycled Al cast alloys in relation to forming routes and appropriate heat treatments, and to assess the variability of these properties as a function of compositional fluctuations induced by residuals, in order to define acceptable tolerance thresholds.

For all these recycling scenarios, it is also necessary to confirm the technical and long-term feasibility of producing such alloys from end-use residues and to quantify the associated environmental impacts across the entire production cycle, in terms of greenhouse gas emissions and recycled content.

Taken together, these sectors illustrate the same trade-off: more circularity and a lower environmental footprint, at the cost of increasingly precise control of residual elements to preserve in-service properties.

PAM-CHR - Plasma Arc Melter, Cold Hearth Refiner

Titanium scraps

Abstracts for poster session 1

April 8 morning

Agromining: an innovative, low-impact approach to metal recovery

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Keywords: phytoextraction; hydrometllurgy; nickel; Life Cycle Assessment

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Abstract:

Agromining is a chain of processes aiming at extracting metals from soils using hyperaccumulator plants, and treating plant biomass to produce compounds based on the target metal [1]. This method addresses the growing demand for metals without competing with conventional mining industries.

The approach has been particularly developed for nickel (Ni), a critical metal in numerous sectors, with nearly 500 hyperaccumulator plant species naturally occurring on ultramafic soils (rich in Ni). Similar processes have also been developed for zinc, cadmium, aluminum, and rare earth elements.

The sector operates on two main pillars: i) agronomic optimization: development of technical pathways to maximize Ni yields (several hundred kg/ha) [2]; ii) hydrometallurgical processing: metal recovery through plant combustion (producing Ni-rich ash), followed by separation processes (leaching, precipitation, etc.) to obtain Ni salts [3].

The development of agromining is systematically accompanied by techno-economic analyses and environmental impact assessments. At the pilot demonstrator stage, a Life Cycle Assessment (LCA) is conducted. A recent study, focusing on two Ni-hyperaccumulator plants and various amendments, demonstrated that agromining not only recovers metals but also reduces heavy metal concentrations in soils [4]. However, these environmental benefits depend on rigorous process optimization.

Research on agromining has been transferred to industry [5] through the creation of ECONICK, a company stemming from work by LSE and LRGP, as well as the joint venture BOTANICKEL with APERAM.

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Detecting alpha case in titanium: beyond standard chemical etching

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[b] Solcera

Keywords: titanium, alpha-case, quality control, surface contamination.

Abstract:

This work focuses on analytical methods that can be used to detect oxygen contamination on surface of titanium and its alloys (atomic diffusion inside the crystal structure), phenomenon also called "alpha case" [1]. It can be highly problematic in high performance applications, as the contaminated area will be fragile and can initiate cracks and failures of components.

The standard NF EN 2003-009 – method A (observation after chemical etchning) [2] is commonly used for detection of alpha case on cross section samples. However this technique has some drawbacks, especially the complexity of cross-section analysis for low-thickness contaminations (< 10 µm), which can make discrimination between contaminated and clean areas uncertain. There's also hygiene and safety concerns, as the chemical etchning implies the use of fluorhydric acid (HF).

Based on literature datas, alternative methods have been considered in the present work to improve the reliability of the conclusion. Thus, several approaches that have been set up by La Halle des Matériaux from EEIGM, that successfully discriminated contaminated from clean samples and evaluated the depth of diffusion of oxygen (even for low thickness contaminations) :

- Microscopic cross section observation without chemical attack in specific lighting modes (destructive method);
- Microhardness measured at low loading on extreme surface of cross-section samples (destructive method);
- XRD characterization (non-destructive method);
- SEM-EDS surface examination (non-destructive method).

In conclusion, these approaches complement the EN 2003-009 standard and may provide more relevant information, while enabling non-destructive control of titanium surfaces.

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Tailoring the pore morphology in 3D-printed porous alumina ceramics

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Keywords: alumina, additive manufacturing, vat photopolymerization, porosity, microstructure, rapid sintering

Abstract:

Alumina-based ceramics are known for their high strength, hardness and stiffness as well as being a lightweight material thanks to its low density. For applications in filters, membranes, catalysts or biostructures, porous alumina ceramics are from high interest. In recent studies, the Lithography-based Ceramic Manufacturing (LCM) process, was used for the fabrication of porous alumina ceramics with spatially resolved porosity by the use of poly(methyl methacrylate (PMMA) as pore-forming agent. It was proven that complex-shaped porous alumina ceramics, processed using LCM and followed by conventional debinding and sintering, result in comparable mechanical properties as conventionally processed parts, while enabling the fabrication of ceramic parts with complex shapes and high precision.

The mechanical and structural properties of porous ceramics are affected by the pore morphology, which depends not only on the size distribution and concentration of pore forming agents, but also on the sintering protocol. In this study, the formation of the pore morphology during the sintering is investigated for additive manufactured alumina with a PMMA content of 20%, using two sintering routes: (i) conventional sintering with low heating rates, which leads to an interconnected pore network, and (ii) non-conventional sintering with high heating rates, which leads to isolated spherical pores. For the non-conventional sintering technique with rapid heating, a “pressureless” Spark Plasma Sintering setup is used, enabling the sintering of additive manufactured porous parts while tailoring both microstructure and pore morphology.

Using optimized sintering protocols for tailoring the pore morphology of porous alumina, it is possible to specifically tailor the properties of the ceramic for the use in structural (i.e., lightweight, high specific strength) or functional applications (i.e., specific surface area, gas permeability).

Data-Driven Microstructure Optimization of Spark Plasma Sintered Ceramics

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Keywords: Spark plasma sintering, machine learning, simulation, microstructure, densification

Abstract:

Spark plasma sintering (SPS) is a field-assisted sintering technique that enables rapid densification of powders through Joule heating, achieved by the simultaneous application of an electric current and uniaxial pressure. The key advantages of SPS include reduced sintering time and temperature, which minimize grain growth and promote a fine-grained microstructure. Due to these advantages, SPS has found applications in several key sectors, including aerospace, automotive, electronics, and biomedical industries.

Despite its benefits, several studies have reported non-uniform microstructures in ceramic samples processed by SPS. The simultaneous application of pressure and temperature can lead to pronounced thermal and stress gradients within the sample.

In this work, decoupling thermal and mechanical loading during SPS is proposed as a strategy to improve microstructural homogeneity and tailor densification mechanisms in ceramic systems. This is achieved using a specialized die configuration in which the electrode connections are decoupled from the high-pressure punches, enabling independent pressure application at defined stages of the sintering process. However, this tooling concept has received limited research to date.

The primary objective of this study is to understand the sintering mechanisms under the modified SPS setup and to optimize processing conditions for improved microstructural control and enhanced densification. To establish the process–property–microstructure relationship, a combination of simulations, experiments, and data-driven methods is employed. Since direct measurement of the sample temperature during SPS is not feasible, simulations are used to predict temperature gradients within the specimen. These models are validated through experiments conducted at varying processing parameters. The resulting microstructures are analyzed for grain size distribution and porosity using AI-driven image analysis, which enables automated, objective, and reproducible evaluation of large datasets while extracting comprehensive microstructural information. Machine learning techniques are subsequently applied to optimize sintering parameters and achieve performances better than that of conventional SPS configurations.

Powder Aerosol Deposition - Influence of Polymer Substrate Characteristics and Deposition Parameters on Early TiO_2 Film Growth

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Keywords: polymer ceramic composites, powder aerosol deposition, thin films, vacuum kinetic spray, vacuum cold spray

Abstract:

Powder Aerosol Deposition (PAD) is a process used to deposit thin, mostly ceramic, films from powder particles without the need of sintering. Its room temperature conditions and only short temperature spikes on particle impact make PAD a promising method for creating polymer ceramic composites (PCC's), with the polymer as either film component or substrate. While PCC's show potential in various fields like electronics[1], sensors[2], or protective coatings[3], PAD fabrication of them has so far been limited. The few studies done on coating polymer substrates with PAD confirm that the method can produce successful coatings, but there still is no comprehensive understanding of how deposition takes place on them.

This study[4] aims to identify at what intensity various process parameters important for PAD influence the film properties when using polymer substrates. Namely, experiments varying the gas flow rate, the number of scans, scanning speed and the nozzle to substrate distance were done. In addition, the effect of substrate hardness and surface roughness were investigated. The films were characterized by their thickness using laser scanning microscopy and roughness measurements were done with atomic force microscopy. Cross-sections of the films and their interface with the substrate were researched using frequent ion beam and scanning electron microscopy.

The study[4] shows the differences in deposition behavior of polymer substrates compared to steel substrates, especially when varying the gas flow rate, as well as in the resulting film surface. A difference in initial film formation speed was observed for polycarbonate substrates compared to polyamide 6 and high-density polyethylene, giving evidence to the process being influenced by differences in substrate toughness and not just their hardness.

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Enhancing the self-healing potential of cold-recycled asphalt pavements: a combined heating and recompaction approach

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Keywords: *Cold recycled asphalt, thermomechanical treatment, healing capacity, Sustainable pavements*

Abstract:

Cold recycled asphalt pavements have gained growing attention for their ability to restore mechanical performance through externally induced healing methods, notably induction and microwave heating. Nevertheless, heating alone often fails to fully restore the initial strength of damaged mixtures. In response to this limitation, this study investigates a thermomechanical treatment (microwave heating followed by recompaction) applied to cold recycled asphalt mixtures incorporating reclaimed asphalt pavement and varying proportions of black steel mill slag. Healing performance was quantified through repeated indirect tensile strength test, and Healing Rate (%HR) was used to assess the influence of heating duration and slag content on the recovery potential. The results demonstrated that the highest healing rates enabled complete restoration of the initial ITS, confirming the effectiveness of the proposed thermomechanical treatment in reestablishing mixture integrity. Chemical and mineralogical characterization by XRD and XRF revealed iron-oxide-rich phases in the steel slags with favorable dielectric behavior, enhancing microwave absorption and heating efficiency relative to the reference mixture. Although slag addition increased air voids due to its angularity and porosity, all mixtures met the required ITS thresholds, indicating adequate structural integrity. Overall, the findings support microwave-assisted healing combined with recompaction as a promising, sustainable, and cost-effective strategy to extend pavement service life, reduce maintenance needs, and promote environmentally responsible rehabilitation practices.

Sustainable Construction Materials from Phosphate Mining By-Products: Performance and Environmental Evaluation of Compressed Earth Bricks

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Keywords: *Compressed earth bricks; phosphate mining waste rocks; life cycle assessment; global warming potential;*

Abstract:

Phosphate mining represents a significant industrial activity in Morocco, that holds over 70% of the world's phosphate rock reserves. The extraction of sedimentary ore generates large quantities of by-products and residues. This work investigates the feasibility of recycling Moroccan phosphate mining waste rocks for the production of compressed bricks. Compressed earth brick constitute a cost-effective alternative with low embodied energy and favorable thermal and mechanical properties. The study focused on the semi-pilot scale evaluation of the technical performance of these blocks , ther with an assessment of their environmental impacts. The geotechnical, chemical, and mineralogical characteristics of the raw materials were studied. Local soil of Benguérir was used to substitute the fine fraction of the brick's matrix. Various formulations were prepared on semi-pilot scale using cement and lime as chemical stabilizers. The findings show that the use of 8% cement and 10% local soil provides the most suitable balance in terms of mechanical and durability perspectives. The use of cement and local soil improves the compressive strength, reaching 5.2 and 2,06 MPa in dry and wet states, respectively. Furthermore, the obtained water absorption coefficients and abrasion resistance comply with relevant stnadrds requirements. The life cycle assessment analysis demonstrate that the blocks can be considered environmentally friendly materials, exhibiting lower environmental impacts across all categories, mainly because of using available locally sourced materials and the elimination of high-energy firing process. Nevertheless, cement is still to be the dominant contributor to environmental burdens, especially in the Global Warming Potential category, driven by the clinker production and limestone calcination. Compressed blocks demonstrate a GWP of 120.29 kg CO_2 eq/ 1t of blocks. The study's findings underscore the importance of using locally available materials to limit the environmental footprint related to transport and mechanical preparation, supporting adequate structural integrity.

Phosphate waste rocks as an alternative source of durable fine aggregates for mortar production

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Self-compacting mortar, Phosphate mining waste, Recycled dolomite aggregate, durability performance, Acid resistance, Alkali-carbonate reaction

Abstract:

The depletion of natural river sand and the increasing demand for environmentally sustainable construction materials underscore the need to valorize mining by-products as alternative aggregates while ensuring comparable durability performance (Bederina et al., 2013; Beniddar et al., 2024; Huang et al., 2005). Morocco holds approximately 70% of global phosphate rock reserves, with extraction operations generating substantial quantities of waste rock, predominantly carbonate-rich interlayers including dolomite, that are typically stockpiled without further use (Hakkou et al., 2016; Oubaha et al., 2024). This study examines the durability performance of self-compacting mortars (SC-M) incorporating phosphate-derived dolomite sand as partial and total replacement for conventional siliceous river sand. A multi-scale experimental investigation was conducted to evaluate transport properties, hygrothermal characteristics, chemical resistance, and alkali-aggregate reactivity.

Dolomite incorporation enhanced key durability indicators. Although water-accessible porosity increased and density decreased marginally at higher replacement levels, capillary absorption was substantially reduced, indicating diminished capillary network connectivity despite elevated total porosity. Ultrasonic pulse velocity values remained within ranges characteristic of high-quality cementitious materials, confirming structural integrity across all mixtures. Dolomite-containing mortars exhibited reduced thermal conductivity and decreased drying shrinkage compared to the reference, showing improved insulation potential and dimensional stability.

Chemical durability was assessed under accelerated acid exposure. Dolomite mortars showed superior resistance to both HCl and H_2SO_4 environments, with performance trends dependent on acid type. In HCl, partial replacement provided optimal performance, whereas complete replacement yielded superior results in H_2SO_4, reflecting distinct microstructural versus chemical degradation mechanisms governing behavior in each environment. Compressive strength retention following sulfuric acid exposure was markedly enhanced relative to control specimens.

Alkali–aggregate expansion testing in NaOH solution revealed innocuous behavior with negligible expansion. Complementary microstructural characterization (TGA/DSC, FTIR, SEM–EDS) following selective TMAH dissolution of different dolomite size fractions showed minimal dedolomitization and only trace brucite/calcite formation, confirming low alkali–carbonate reaction susceptibility. These findings demonstrate that phosphate-derived dolomite sand constitutes a viable sustainable alternative to natural river sand in self-compacting mortars, offering enhanced durability under aggressive exposure conditions while valorizing mining waste streams.

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Smart pH-Responsive Packaging Films Using Anthocyanin-Based Alginate/PVP/ZnO Nanocomposites

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Keywords : anthocyanin, intelligent packaging, ZnO nanoparticles, pH-responsive indicator, sodium alginate

Abstract:

Ensuring food safety remains a major global challenge due to the health risks associated with food spoilage and contamination [1]. The prevention and early detection of foodborne infections are therefore critical priorities in modern food safety systems [2]. In this context, pH-responsive intelligent packaging has emerged as a promising approach for rapid and user-friendly monitoring of food freshness. Among natural colorimetric indicators, anthocyanins have attracted considerable interest owing to their vivid pH-dependent color changes and valuable functional properties, including antioxidant, anti-inflammatory, and anti-carcinogenic activities [3].

In this study, bioactive and pH-sensitive color-changing packaging films were developed using a simple casting technique. Anthocyanins extracted from red cabbage were incorporated into a biopolymer matrix composed of sodium alginate and polyvinylpyrrolidone (PVP), with zinc oxide nanoparticles added as a reinforcing agent. The resulting alginate/PVP/ZnO nanocomposite films exhibited excellent antioxidant activity, providing multifunctional performance beyond pH responsiveness. Importantly, the films displayed clear and distinct color transitions in response to pH variations, demonstrating their effectiveness as visual indicators for real-time food freshness monitoring. These findings highlight the strong potential of anthocyanin-based intelligent packaging films for enhancing food safety and consumer awareness.

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Abstracts for poster session 2

April 8 afternoon

Crosslinked chitosan with hydrophobic jojoba oil as eco-friendly coating materials for water-soluble fertilizers

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Keywords (6 maximum): *Hydroformylation, jojoba oil, chitosan, crosslinking, coatings, biodegradable materials*

Abstract:

The rapid growth of the global population, which is expected to reach nearly 10 billion by 2050, will require a substantial increase in worldwide food production (estimated at approximately 50%) to meet future nutritional demands and ensure food security [1]. To address these challenges, conventional fertilizers play a crucial role by supplying essential nutrients, particularly nitrogen (N) and phosphorus (P). However, the rapid release of these nutrients results in low use efficiency, as a significant fraction is lost through runoff, leaching, fixation, or volatilization, leading to environmental problems. To overcome these limitations, traditional fertilizer coating technologies have mainly relied on synthetic polymers, including thermosetting resins (e.g., epoxy, urethane, and silicone resins) and thermoplastic polymers (e.g., polyethylene, poly(acrylic acid-co-acrylamide), and poly(vinyl acetate)) [2,3]. In this study, we report the synthesis and application of biodegradable polymer coatings based on chitosan crosslinked with hydroformylated jojoba oil (CS:J-CHO) for controlled-release fertilizers. Hydroformylated jojoba oil (J-CHO) was synthesized by single-step hydroformylation of the double bonds present in jojoba oil, thereby introducing reactive aldehyde groups capable of forming imine bonds. These aldehydes were then cross-linked with the amine groups of chitosan (CS) to produce a biodegradable polymer network (CS:J-CHO) through the formation of a Schiff base. The resulting films exhibited adjustable swelling, improved thermal stability, increased hydrophobicity, and an amorphous structure, as confirmed by FTIR, TGA, XRD, and contact angle measurements. The application of CS:J-CHO as an internal coating, combined with an external layer of paraffin wax, on diammonium phosphate (DAP) granules significantly slowed phosphorus release from 3 to 96 hours. Furthermore, the biodegradability of the coating ensures environmental sustainability and offers a promising platform for controlled-release fertilizers with reduced environmental impact.

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Thermo-mechanical properties of Bentonite clay for Eco-Construction through Compaction and Sintering

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Keywords : Bentonite, Compaction intensity, Sintering temperature, Eco-construction materials, Thermal properties, Mechanical properties

Abstract:

Bentonite is widely used in construction and geotechnical applications; however, its swelling behavior and sensitivity to water limit its direct use without treatment. This study evaluates the physical behavior of bentonite clay under different compaction intensities (14–210 MPa), water contents (0–20%), and thermal states (as-prepared, dried at 105 °C after 28 days, and sintered between 700 °C and 1000 °C). Results show that increasing compaction intensity significantly increases apparent density, thermal conductivity, and compressive strength due to pore volume reduction, while electrical conductivity and permittivity decrease with higher densification and reduced water mobility. Sintering leads to strong densification, with apparent density reaching about 2.28 g/cm³ at 1000 °C, and significantly improves mechanical performance while reducing moisture sensitivity. Thermal conductivity increases with sintering temperature, whereas electrical conductivity decreases due to the loss of conductive phases and reduced porosity. Water content strongly affects electrical properties in the as-prepared state, with electrical conductivity and dielectric permittivity increasing with moisture due to enhanced ionic transport, while thermal conductivity increases up to about 15% water content and then stabilizes. Among the investigated conditions, sintering at 800 °C provides the best compromise between densification, mechanical performance, Thermal stability, and energy efficiency, making it the most suitable temperature for construction applications. These results highlight the strong influence of compaction, moisture, and thermal treatment on bentonite performance and demonstrate their key role in optimizing bentonite-based materials for durable and sustainable construction.

Development of a corrosion test bench and evaluation of the corrosion resistance of aluminium heat exchangers.

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Keywords: test bench, acidic buffer solution, localized corrosion, heat exchanger, aluminium alloy.

Abstract:

As part of a project carried out for Air Liquide, the EEIGM "Halle des Matériaux" developed a laboratory-scale corrosion test bench to evaluate the environmental resistance of brazed aluminum alloy heat exchangers. This device reproduces corrosive conditions representative of a specific application in order to evaluate the behavior of three heat exchangers in parallel, in an acidic bufferd environment (pH ≈ 3.8) at 40°C.

The corrosion study is based on variations in the physicochemical properties of the electrolyte and on the analysis of microstructural degradations undergone by the equipment, in both cases over time. Measurements of the physicochemical properties of the electrolytes (non-destructive) provided overall kinetic data on corrosion processes and enabled a comparison between the three tested exchangers. Post-mortem analyses (destructive) revealed three main modes of localized corrosion: galvanic [1], pitting [2], and intergranular [3]. These phenomena are directly related to specific microstructures, which themselves depend on the design and manufacturing process of the exchangers.

Finally, the development of the corrosion test bench allowed to reproduce the operating conditions defined by Air Liquide for more than 50 days, and generated reliable data on the behavior of the three heat exchangers. This work opens prospects for further testing, particularly under other environmental conditions (pH, temperature, etc.) or on different exchanger designs and base materials.

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Ionic Liquids as Electrolytes in Supercapacitors

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Keywords: Energy storage, ionic liquids, supercapacitor

Abstract Ionic liquids (ILs) are low-temperature molten salts (melting points below 100 °C) and consist solely of ions, meaning they can be used without an added solvent. Their negligible vapor pressure and high thermal stability make them attractive electrolytes, and many ILs also offer a comparatively wide electrochemical stability window - although viscosity, conductivity, and toxicity can vary strongly with the specific cation-anion pair.[1] Owing to this broad stability window (and often adequate ionic conductivity), ILs have been widely explored for high-energy electrical double-layer capacitors (EDLCs, also known as supercapacitors). In EDLCs, charge is stored through reversible ion electrosorption at the electrode-electrolyte interface rather than via Faradaic reactions, which typically enables high power density and high cycling stability. However, their energy density generally remains well below that of batteries. A key advantage of IL-based electrolytes is the large design space: by selecting and tailoring the ions, IL properties can be tuned toward wider operating voltages and improved ion transport, which is critical for narrowing the energy-density gap while maintaining long-term stability.[2]

Building on this idea, we first tested whether introducing a second, smaller inorganic cation into a neat IL could improve pore filling and thus capacitance in nanoporous carbons. Using activated carbon black (BP2000) electrodes, a symmetric EDLC with [EMIM][TFSI] delivered about 122 F/g at a rate of 0.2 A/g, while the 9:1 mixture $[K]_{0.1}[EMIM]_{0.9}[TFSI]$ showed a very similar 118 F/g.[3] In other words, the “binary-cation” concept did not immediately and directly translate into a clear device-level gain. This is consistent with the broader mechanistic picture from theory and experiment: mixed-cation ILs can enhance charging in certain pores and polarities, but in a full two-electrode cell, the overall performance is often limited by the “weaker” electrode, in particular when pore accessibility for the counter-ion becomes the bottleneck.[3]

To overcome this intrinsic asymmetry, we next moved from binary to quaternary electrolytes by adding lithium salts to $[EMIM][BF_4]$, thereby introducing both a small cation (Li^+) and a smaller counter-anion that can better access micropores on the oppositely charged electrode. In symmetric activated-carbon EDLCs (0-3 V), neat $[EMIM][BF_4]$ reached 103 F/g at 0.5 A/g, while capacitance increased to 135 F/g with 0.5 mol/dm³ $LiNO_3$ (and to 122-123 F/g with $LiPF_6$ or $LiClO_4$).[4] The $LiNO_3$ mixture also delivered 50.2 Wh/kg at 50 W/kg and still 45 Wh/kg at 1500 W/kg. It retained more than 88% of its initial capacitance after 5000 cycles at 2 A/g.[4] Importantly, the magnitude of the improvement depends on the electrode pore structure, with more microporous activated carbons benefiting more strongly than carbon black.[4]

Overall, these results underline that IL “tunability” only pays off if the electrolyte design is matched to the electrode pore-size distribution and both electrodes can be utilized efficiently - otherwise, the cell is capped by the limiting side.

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Towards Sustainable Antifouling Materials for Marine Applications: Bromotyrosine-Functionalized Polymer Coatings

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Keywords: Marine antifouling, Copolymers, Coatings, Polyacrylamides, Bromotyrosine

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Abstract:

Biofouling in marine environments poses a significant challenge, particularly for surfaces that are difficult to access, such as underwater camera lenses or fish tagging chips. Conventional antifouling approaches often rely on the release of toxic biocides or require frequent cleaning, which is either not sustainable or costly. Therefore, there is a growing need to find alternative approaches to minimize ecological impact while maintaining effectiveness.[1]

To address this, we developed novel copolymer coatings based on *N,N*-dimethylacrylamide, a bromotyrosine-derived antifouling unit[2,3] and benzophenone as a photo-crosslinker.[4,5] Molecular characterization of the copolymers was performed using nuclear magnetic resonance spectroscopy, infrared spectroscopy and size exclusion chromatography. Thin, smooth coatings were prepared by spin coating and comprehensively characterized. Film thickness was analyzed using ellipsometry, and wettability was determined through contact angle measurements. The surface morphology was assessed by atomic force microscopy, and the surface charge was evaluated using an electrokinetic analyzer for zeta potential determination.

The functional performance of the coatings was investigated through protein adhesion studies using surface plasmon resonance spectroscopy, and incubation experiments with the diatom *Navicula perminuta*. Cytocompatibility tests with human keratinocytes (HaCaT cells) indicate low toxicity, suggesting that these coatings may provide a safe alternative, however further experiments with marine-specific organisms are required.

Overall, the results suggest that these polymer coatings can provide promising antifouling properties without toxicity, offering new solutions for marine antifouling applications.

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Élaboration et caractérisation physico-chimiques de nouveaux bio-composites à base d'algues Sargasses et de déchets de l'agro-industrie antillaise à visée d'exploitation en qualité d'isolants thermique et phonique.

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Keywords (6 maximum): Sargassum, Sugarcane bagasse, Bio-composite, Natural fiber

Abstract:

The massive beaching of sargassum at the Caribbean coast constitutes a major problem due to the release of toxic and corrosive hydrogen sulfide (H_2S) and ammonia (NH_3) and heavy metals such as arsenic, thereby having a massive impact on tourism and electronic component longevity. The insular context of this region forces the application of the circular economy, thus the valorization of waste and by-products generated by numerous industries, such as sugarcane bagasse, a by-product of the sugar industry. Many studies have labeled this by-product as a promising role for renewable construction material[3,4]. Energy consumption is another important subject now and for the future. Developing more effective and high-end thermal insulators is critical for the conservation of heat in building industry, thus reducing CO_2 emissions. This study aims to mitigate the impact of sargassum by incorporating the extract from the brown algae as a binder for delignified bagasse fiber due to the presence of polysaccharides such as alginate or fucoidan showing binding properties[5]. Three biocomposites have been manufactured to suit many applications, from acoustic insulation to rigid interior wall impregnated by thermoplastic . The new processed composite has been physico-chemically characterized. The material showed a thermal diffusivity between 9.10^{-8} m^2.s^{-1} and 12.10^{-8} m^2.s^{-1}. Its behavior under flame has been characterized. The material thermal conductivity (λ = 0,12 W.m^{-1}.K^{-1}) is comparable to the one of cork, and the stiffness of impregnated biocomposites (3–4 GPa) is similar to thermoplastics (PLA, ABS, PET). An arsenic monitoring protocol needs to be established to keep inorganic arsenic concentration under harmful levels by extracting it after neutralization[6-8].

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Designing white anodized layers on Al

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Keywords (6 maximum): Anodizing, H2 release, electrochelmistry, SEM

Abstract: The achievement of white glossy anodized surface remains challenging in aluminum surface finishing industry. It relies on light scattering by objects of about 150-200 µm. This may be induced by the incorporation of pigments in the pores of the anodic layer, provided that they have a suitable diameter, which requires specific conditions, especially a very high anodizing voltage. Alternatively, the morphology of the anodic layer may be modulated and an efficient route has been recently developed [1-2] by applying a pulse bipolar anodizing process in H_2SO_4. Actually, the implementation of a periodic cathodic step induces the generation of "voids" through the anodic layer thanks to H2 release. The created defects work as as light scattering areas and both their repartition and their influence directly the optical properties of the coating.

Thanks to the combination of electrochemical measurements to investigate the electrochemical behavior over the anodic and cathodic steps, cross-section microscopic characterization and optical measurements, a deep understanding and an optimization of the procedure described above has been reached on a model substrate, namely pure Al (99.999%) with mirror-like surface finishing and under controlled conditions.

Process aspects were investigated on pure Al. The bath stirring and the substrate orientation seem to have no influence on H_2 generation and release, on the formation of voids and consequently, the white aspect of the coating is maintained. However, the initial surface roughness is crucial for further optical properties. While mirror-like mechanical finishing and chemical etching result in the same targeted optical properties after the pulse bipolar anodizing procedure, a rough initial surface induces a modification in the morphology of the created defects and a downgrading of the visual aspect.

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Glasses of R_2O-B_2O_3-Al_2O_3 systems: study of structure-mechanical properties relationships at the metaluminous joint (R = Li, Na, K).

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Glassy materials have long been considered fragile, exhibiting purely elastic behavior before breaking when subjected to stress[1]. However, the advent of fine-scale mechanical characterization techniques, such as instrumented micro-indentation, has revealed that glass can exhibit permanent deformations when subjected to sharp contact loading, induced by the appearance of two mechanisms: densification and shear flow[2]. Although developed on a local scale, these mechanisms govern the mechanical properties of glass on a macroscopic scale. Increasing crack initiation resistance therefore requires a highly adaptable matrix, both at short and medium range.

Alkali aluminoborate glasses are of particular interest due to their high flexibility, especially within the first few coordination spheres of the network. In particular, studies of these systems close to the metaluminous joint ($R=Al_2O_3/R_2O=1$) have already shown increased properties when subjected to localized stress[3]. This study therefore focuses on the relationships between the concentration of each formative element at R=1 and the mechanical properties obtained. Some of the obtained glasses displayed the highest crack resistance value ever reported for an oxide glass (CR $\approx$ 50 N). The crack initiation resistance results are then correlated with the different structural variations obtained by RAMAN micro-spectroscopy.

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Impact of Reactive Extrusion on Crystalline Family Distribution in Opaque Recycled PET Revealed by SSA–DSC

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Abstract

This work explores the use of successive self-nucleation and annealing (SSA) by differential scanning calorimetry (DSC) to characterize the crystalline heterogeneity of opaque recycled PET (rPET-O) and its reactively extruded (REX) counterparts, and to analyze how REX –induced changes in molecular architecture affect the distribution of crystalline families. Calendered sheets of rPET-O and REx-rPET-O containing 1 and 1.5 wt% of a styrenic–acrylic multiepoxide (SAmfE) chain extender were analyzed by conventional DSC to assess their global thermal behavior. Rheological tests on the reactively extruded materials suggest a change in molecular architecture, consistent with chain extension and long-chain branching, and with the presence of additional structural irregularities introduced by SAmfE. Self-nucleation (SN) experiments were performed on rPET-O to identify the SN domains and optimal self-seeding temperature, which was applied to all materials to design SSA protocols. The final SSA heating scans reveal five distinct melting components for all samples and show that the contribution of the low-temperature part of the endotherm (Tm ≤ 240 °C) increases from about 30% in rPET-O to roughly 40% in the material with 1.5 wt% SAmfE, while the main melting maximum remains nearly unchanged at ~247 °C. A simple Gibbs–Thomson estimate, using literature values for PET ($Tm_0 \approx 523$ K and $\Delta H_{f0} \approx 140$ J/g), indicates that these components correspond to effective lamellar thicknesses spanning r from ~4–5 nm for the low-temperature peaks to ~7–8 nm for the high-temperature ones, with REX mainly enriching the thinner-lamella fraction rather than changing the average thickness. Overall, the results demonstrate that SSA–DSC, supported by rheological evidence of long-chain branching, is a sensitive and relatively simple tool to probe how REX modifies the lamellar size distribution in opaque recycled PET, providing quantitative microstructural indicators that can support the optimization of advanced recycling routes for PET-O-based materials.

Key words

rPET-O, DSC, SSA, Reactive extrusion, Molecular architecture, Crystalline families.

Acknowledgments

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The authors thank the María de Maeztu Units of Excellence Programme CEX2023-001300-M funded by MICIU/AEI/10.13039/501100011033. We also acknowledge the accreditation TECNIO as a technology transfer agent by ACCIÓ, given to the CCP. L. Martínez Orozco acknowledges funding support from the FI-SDUR fellowship from the Generalitat de Catalunya (expedient: 2022 FISDU 00319) during the initial phase of the doctoral program, and the one-year doctoral extension granted by the Universitat Politècnica de Catalunya (UPC).

Integration of Computed Tomography and Numerical Simulation for the Mechanical and Fracture Behavior Analysis of Polypropylene

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Abstract

This study presents an integrated methodology for modeling and simulating the mechanical and fracture behavior of microcellular glass fiber-reinforced polypropylene composites. The proposed approach combines advanced three-dimensional characterization using computed tomography (CT) with structural analysis through the finite element method (FEM), enabling a direct correlation between the material's microstructure and its mechanical and fracture response.

High resolution CT scans of solid and micro-foamed specimens were processed to reconstruct accurate three-dimensional representations of the internal structure. A segmentation procedure was applied to differentiate the polymer matrix, glass fibers, and cellular regions, enabling a detailed volumetric description of the material. The resulting data guided the generation of high-resolution conformal meshes that preserved the key morphological features influencing structural behavior. Boundary conditions, matrix–fiber interfaces, and material properties were defined in the numerical models to replicate the mechanical and fracture tests performed on the same materials. The simulated results were compared with experimental data for validation. This analysis highlights the effect of foaming as well as fiber orientation and spatial distribution on local stress concentrations, stiffness reduction and the material's resistance to crack initiation and propagation.

Keywords: Glass fiber-reinforced polypropylene, microcellular polymers, computed tomography (CT), numerical methods, structural simulation.

Acknowledgments

This work was supported by the Catalan Government through the quality accreditation and funding of the research group eb-POLICOM: Polímers i Composits Ecològics i Biodegradables (Grant 2021 SGR 01042), by the project EcoPolyRub PID2023-151338NB-I00 funded by the Ministerio de Ciencia, Innovación y Universidades (MICIU), the Agencia Estatal de Investigación (AEI) and FEDER funds.

The authors thank the María de Maeztu Units of Excellence Programme CEX2023-001300-M funded by MICIU/AEI/10.13039/501100011033. We also acknowledge the accreditation TECNIO as a technology transfer agent by ACCIÓ, given to the CCP.

role of Motar in Enhancing the Seismic Performance of Traditional Masonry Structures

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Keywords: Seismic performance, Energy dissipation, Failure mechanisms, Sustainable construction, Earthquake-resistant, structures.

Abstract:

Traditional masonry structures in seismic regions commonly use earth and lime-based mortars due to their availability, low cost, and environmental sustainability. However, these mortars generally exhibit low tensile strength, limited ductility, and poor energy dissipation capacity, increasing the vulnerability of buildings to seismic loading. Recent research has highlighted the critical role of mortar properties in governing the seismic response of masonry systems.

Experimental and analytical studies, particularly under dynamic loading, show that mortar characteristics significantly influence displacement capacity, damage evolution, failure mechanisms, and energy dissipation. Consequently, material-based strategies have gained increasing attention as a sustainable alternative to invasive structural strengthening techniques.

Among these approaches, fiber reinforcement has proven effective in improving tensile strength, ductility, and post-cracking behavior.[1] demonstrated that polypropylene fibers enhance shear resistance and energy dissipation while limiting crack propagation. Additionally, the optimization of binder composition has been shown to play a key role. [2] reported that mortars based on natural hydraulic lime exhibit improved ductility and mechanical compatibility with traditional masonry. From a sustainability perspective, Resketi and [3] showed that eco-friendly mortars incorporating industrial by-products such as fly ash and metakaolin improve long-term mechanical performance while reducing environmental impact. Furthermore, [4] highlighted that Textile Reinforced Mortar (TRM) systems based on lime–pozzolan matrices significantly enhance ductility and energy dissipation capacity of masonry walls under seismic loading.

Overall, the literature confirms that optimizing mortar formulation represents an effective and sustainable strategy to reduce the seismic vulnerability of traditional masonry structures, particularly in earthquake-prone regions relying on local and traditional materials.

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Abstracts for poster session 3

April 10 morning

Microwave-induced rejuvenator release for on-demand self-healing asphalt mixtures using biopolymeric capsules

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Keywords (6 maximum): Asphalt durability, healing effeciency, Encapsulation technology, Microwave irradiation.

Abstract:

Encapsulated rejuvenators offer a promising route to enhance the self-healing capability of asphalt pavements; however, conventional systems rely mainly on traffic-induced rupture, which limits control over the healing process [1]. This study investigates a microwave-activated healing strategy based on biopolymeric Alginate capsules encapsulating a rejuvenator and modified by microwave absorbent materials to enable external activation.

Alginate capsules were synthesized through ionic cross-linking and evaluated for their thermal stability, mechanical resistance, encapsulation efficiency and microwave responsiveness. Results showed that the capsules remain stable under asphalt mixing conditions, tolerating temperatures up to 150 °C and representative compaction loads of 10N[2], [3]. Microwave irradiation induced localized heating and initiated controlled rejuvenator release in less than 30seconds microwave exposition, confirmed by infrared thermography and mass loss measurements.

When incorporated into asphalt mixtures at low contents up to 1%, the capsules did not compromise the mechanical performances. In contrast, healing efficiency was significantly enhanced under microwave activation. From short irradiation times onward, capsule-modified mixtures consistently exhibited higher healing efficiency compared to control mixture, achieving full recovery of mechanical properties after only 2minutes microwave irradiation exposure.

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Mechanical–Dielectric Dynamic Correlations Using Fractional Modeling: Application to Polylactic Acid

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Keywords : fractional model, mechanical properties, dielectric properties, relaxation phenomena, theory and modeling, polylactic acid

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Abstract:

To fully exploit polymer materials and optimize their performance, understanding the correlation between mechanical and dielectric dynamic behaviors is essential [1–2]. Such correlations provide deeper insight into material behavior and enable prediction of property evolution under varying conditions.

This study examines the relationship between the mechanical and dielectric responses of polylactic acid (PLA), a widely used polymer characterized by distinct relaxation processes [3]. Experimental data were analyzed using the Mechanical Fractional Model (MFM) and the Dielectric Fractional Model (DFM) [4–5], revealing increased molecular mobility with rising temperature, particularly near the glass transition. To go beyond separate analyses, a fractional calculus–based correlation model was developed to link the complex mechanical modulus and relative permittivity of PLA. This approach enables prediction of the real permittivity $\varepsilon'(T)$ from the real modulus $E'(T)$, demonstrating the potential to predict dielectric behavior from mechanical data and vice versa.

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Pollen-Reinforced PVA/Gelatin Biocomposites: A Sustainable Approach for Active Food-Packaging Films

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Keywords: biocomposites, biodegradability, pollen, food packaging, gelatin, PVA.

Abstract:

Pollen was incorporated into a polyvinyl alcohol–gelatin (PVA–gelatin) copolymer to develop innovative active food-packaging films using the solvent-casting method [1]. PVA/gelatin/pollen biocomposites with different pollen contents (1%, 3%, and 5% by weight) were prepared to investigate their influence on the structural, vibrational, and dielectric properties of the polymer system. Scanning electron microscopy (SEM) revealed a homogeneous film morphology and a uniform dispersion of pollen within the PVA–gelatin matrix [2], indicating good interfacial compatibility between the components. X-ray diffraction (XRD) analysis was employed to assess the effect of pollen on the crystalline structure of the polymer, while Fourier-transform infrared spectroscopy (FTIR) confirmed the presence of physicochemical interactions, particularly hydrogen bonding, between pollen and the polymer matrix [3].

Furthermore, pollen incorporation altered the dielectric behavior of the films by influencing relaxation processes and dipolar mobility, highlighting strong interactions between the functional groups of pollen and those of the polymer network. From a functional standpoint, the addition of pollen improved key film properties, notably mechanical stiffness and viscoelastic behavior, while simultaneously reducing moisture content and water solubility—features that are highly desirable for food-packaging applications [4]. Overall, these findings demonstrate that PVA/gelatin/pollen biocomposites constitute a promising, sustainable, and eco-friendly platform for the development of active packaging materials aimed at food preservation [5].

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Mesophase, stress induced crystallization and strain tensor at the macromolecular scale : input of Raman spectroscopy

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Keywords (6 maximum): polymers, X-ray diffraction, Raman spectroscopy, crystallization, strain measurement

Abstract:

Understanding the behavior of polymers under various loads is a challenging task due to the multitude of physical micro-mechanisms that develop during deformation stages [1]. To simplify the study, it is helpful to differentiate the various phases present in the polymer, even though they contribute cooperatively. The mesophase is particularly complex to analyse due to its thermodynamic instability, often resulting from the destruction of the crystalline phase or an arrangement of the amorphous phase [2]. Combining various characterization techniques, such as Raman spectroscopy and X-ray diffraction, has proven effective in detecting the different mechanisms responsible for the stretching of polymers, studying them, and quantifying their effects. Coupling these analysis method with the VidéoTractionTM and a digital image 3D stereocorelation (DIC) systems or Differential Scanning Calorimetry (DSC) provides valuable information at the level of the representative elementary volume (REV). This approach has led to the proposal of Raman criteria for crystallinity, macromolecular orientation, volume damage and multiscale true strain fields of thermoplastics as illustrated on figures 1 and 2 [1,3,4,5].

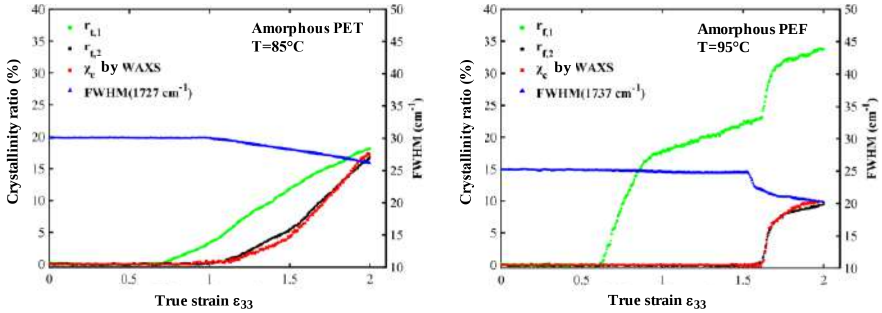

Figure 1: *Crystallinity ratio evolution, determined by Raman spectroscopy ($r_{t,1}$, $r_{t,2}$, $r_{f,1}$ and $r_{f,2}$ criteria) and by WAXS (χ_C), during a tensile test at 5.10^{-4} s^{-1}, for amorphous PET and PEF, respectively at 85°C and 95°C.*

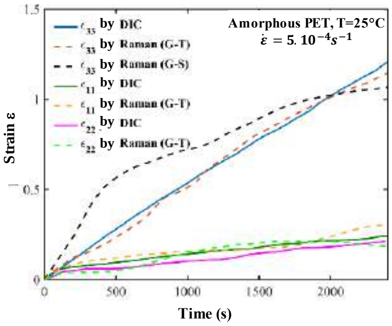

Figure 2 : *True strain fields obtained by DIC and Raman spectroscopy, for amorphous PET in uniaxial tension below T_g.*

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Large-Area Nanoindentation Mapping for Microstructure–Property Analysis in Heterogeneous Materials

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*Speaker

Keywords (6 maximum):

Nanoindentation mapping, micromechanical characterization, statistical analysis, multiphase materials, unsupervised learning

Abstract:

High-speed nanoindentation mapping enables the acquisition of large-area, high-density micromechanical datasets, shifting nanoindentation from isolated point measurements to a spatially resolved characterization approach. By sampling thousands of indents over representative areas in less time, this technique provides direct access to spatial variability in hardness and elastic modulus response, offering a powerful link between local mechanical behaviour, microstructural length scales, and macroscopic material performance.

Large nanoindentation maps allow the quantification of mechanical heterogeneity across microstructural constituents and regions, revealing property contrasts, gradients, and characteristic zones that are not captured by conventional testing strategies. Beyond average values, the statistical representativeness of these datasets enables analysis of full property distributions, including scatter and asymmetry.

To fully exploit this information, advanced statistical tools and new analysis approaches are employed to interpret overlapping mechanical responses in a statistically robust and physically meaningful way. Unsupervised learning is used to extract population-level parameters such as mean values, scatter, and relative fractions, together with their spatial organization across the mapped area. This enables direct visual correlation between mechanical populations and microstructural features observed by electron microscopy, while naturally accounting for asymmetric distributions.

This approach has been aplied to several industrially relevant case studies, including cemented carbides, superduplex stainless steels, advanced high-strength steels, and steel-based multi-material assemblies produced by additive manufacturing, illustrating the current reach of nanoindentation mapping for microstructure–property correlation. A structured characterization framework has been outlined, combining mapping design guidelines with reproducible data processing and statistical analysis into a coherent workflow intended for application across different material systems. Current limitations are resolution constraints imposed by indentation interactions, sensitivity to test conditions and surface preparation, and ambiguities near interfaces where mechanically overlapping responses challenge classification of properties.

Decoding Ultrasonically Textured Surfaces: Multimodal Height Analysis and Functional Layer Identification via GMM

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Keywords: texturing, surface topology, surface metrology, vibration-assisted machining, multimodal analysis

Abstract:

The metrological characterization of mechanically textured surfaces remains a significant challenge, particularly when dealing with complex topographies generated by advanced manufacturing processes. In this contribution, we present a methodological approach to deal with surfaces textured by Radial Ultrasonic Vibration-Assisted Machining (RUVAM), a process capable of producing highly repeatable crater-like patterns whose functional behavior depends critically on their detailed morphology[1]. Traditional surface parameters defined in ISO 25178-2:2021 often fail to capture the true complexity of these textures, especially given their strongly non-Gaussian and frequently multimodal height distributions[2]. Our work introduces a metrological approach that uses Gaussian Mixture Models (GMM) to segment the surface into functional layers, providing a more accurate representation of the distribution of peaks, cores, and valleys.

To explore the limitations of classical descriptors, a series of RUVAM textures was generated by systematically varying process parameters such as insert nose radius, feed rate, and depth of cut. These conditions produce distinct sets of crater geometries, which were captured through surface measurements and filtered to produce scale-limited height datasets. We observed that conventional amplitude parameters, typically effective for simpler, near-Gaussian surfaces, are not representative enough for characterizing these textures. Similarly, functional parameters based on the bearing area curve (BAC) provided an inadequate partition of material into functional zones, failing to reflect the actual physical structure of the cratered surfaces. Instead, feature parameters demonstrated a much stronger capacity to describe the complexity of RUVAM textures, successfully capturing key aspects of crater morphology and distribution. This highlights the need for metrological tools that are sensitive to local geometrical features rather than relying solely on global statistical descriptors.

Given the inherent multimodality present in the height distributions of these surfaces, GMM provides a natural statistical model for texture segmentation. Our analysis confirms that RUVAM surfaces can often be stratified according to tetramodal or pentamodal distributions, reflecting the layered structure characteristic of crater-like patterns[3]. By fitting Gaussian components to the height data, GMM yields probabilistic boundaries that more faithfully separate peaks, cores, and valleys than the deterministic thresholds imposed by ISO functional parameters. This data-driven truncation method aligns with the actual morphology of the texture, offering a more physically meaningful interpretation of the surface's functional zones. The resulting segmentation is thus not only mathematically justified but also consistent with the manufacturing process that generated the texture. [upcommons.upc.edu]

The implications of this work extend beyond the specific case of RUVAM texturing. Many engineered surfaces, particularly those produced by additive manufacturing, laser structuring, or other processes that generate patterned or multimodal height distributions, share similar analytical challenges. The GMM-based approach presented here provides a transferable framework for metrologically interpreting such surfaces, enabling a more accurate linkage between topography and functional behavior[4]. By moving toward a probabilistic and feature-oriented description of surface texture, this research contributes to the development of more robust, physics-informed metrology capable of supporting the next generation of textured surface engineering.

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3Y-TZP Gyroid Ceramic Monoliths produced by Digital Light Processing: Slurry Optimization and Post-Processing

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$3Y - TZP$ DLP; ceramic slurry; gyroid; post-processing

Abstract: This work details the development and optimization of yttria-stabilized zirconia ($3Y - TZP$) ceramic monoliths using Digital Light Processing (DLP). The primary goal was to manufacture complex gyroid structures with a target porosity of 50% for potential catalytic applications, balancing mechanical strength and active surface area. The methodology focused on optimizing the ceramic suspension (slurry) through rheological and sedimentation studies, establishing an optimal formulation of 30 wt% zirconia and DisperBYK dispersant. Printing was conducted using a bottom-up DLP configuration, where critical parameters such as exposure times and build plate roughness were adjusted to achieve high geometric precision. Post-processing stages were guided by Thermogravimetric Analysis (TGA/DTG) to of slow heating ramps to ensure progressive resin removal, ensuring structural integrity. Characterization via SEM and EDS confirmed the achievement of stable, dense structures with fine equiaxial grains, validating the feasibility of the proposed fabrication route.

INTRODUCTION

Additive manufacturing (AM) of ceramics allows for the creation of intricate geometries that are difficult to achieve through traditional subtractive methods, opening new possibilities for functional components where mass transfer, surface area and structural efficiency are relevant. Among ceramic materials, yttria-stabilized zirconia ($3Y - TZP$) is widely valued for its high mechanical strength and toughness, as well as its thermal and chemical stability. For these reasons, zirconia-based structures are of interest for applications such as structured reactors and catalytic supports [1].

DLP is a vat photopolymerization technique capable of producing parts with high resolution and smooth surfaces. However, DLP processing of ceramics requires the formulation of stable and printable photocurable suspensions (slurries), where the ceramic loading, dispersant content and rheology must be carefully controlled. Insufficient stability can lead to sedimentation during printing, resulting in gradients, delamination, or defects that may be amplified during debinding and sintering [2], [3].

In this work, gyroid architectures were selected as a representative monolithic geometry due to their potential to combine mechanical stability and high surface area. A target porosity of 50% was used as a design criterion. This work focuses on optimizing slurry formulation, printing parameters and thermal post-processing conditions to obtain stable zirconia gyroid monoliths by DLP [2], [4].

METHODS

Material Selection and Slurry Formulation

The ceramic phase consisted of TOSOH $\mathrm{TZ} - 3\mathrm{YSB} - \mathrm{E}$ zirconia powder (3 mol% yttria) with an average particle size is $30 \pm 1\ \mu m$. The photocurable matrix was based on ELEGOO Water-Washable resin. To achieve a stable suspension, DisperBYK was employed as a dispersant to provide steric stabilization and reduce viscosity [5], [6].

Optimization involved testing zirconia loadings from 15% to 75%. In the absence of a dispersant, sedimentation occurred within 10–60 minutes, which is insufficient for printing. The addition of

DisperBYK significantly extended stability beyond 220 minutes for the 30 wt% loading, ensuring homogeneity during the DLP process.

DLP Printing and Parameter Optimization

Printing was executed on an DLP printer ELEGOO Saturn 4 Ultra in a bottom-up configuration. In this setup, the piece grows inverted, reducing the required volume of slurry but introducing mechanical stresses during the separation of the cured layer from the vat. Critical parameters were optimized to prevent delamination and "overgrowth": Bottom Exposure, Normal Exposure, and Layer Height [7].

Post-processing: TGA-Guided Thermal Treatment

The "green" parts required debinding to remove the organic binder followed by sintering. TGA/DTG analysis of the formulation identified key degradation events at ~350°C (additives), ~415°C (resin matrix), and ~515°C (residual carbon oxidation). These findings were essential for designing slow, controlled heating ramps (0.2–0.5 °C/min) to prevent cracks and structural failure. Final sintering was performed at 1450°C to achieve densification. Sintered samples were characterized by SEM and EDS [8], [9].

RESULTS

Slurry Stability and Rheology

Rheological analysis showed that while pure resins are nearly Newtonian ($n \approx 1$), the ceramic slurries exhibit pseudoplastic behavior. Formulations with 30 wt% zirconia and dispersant remained within the printable viscosity range (≤ 3 Pa·s), providing the best balance between stability and flowability.

Geometric Fidelity of Gyroid Monoliths

Gyroid structures were designed using SolidWorks to achieve 50% porosity. Initial prints with a 25 wt% loading exhibited delamination and phase separation due to sedimentation and exposure time issues. Subsequently, optimization of the exposure time yielded successful monoliths with high geometric fidelity and no visible surface defects, Figure 1.

Figure 1. $3Y-TZP$ Gyroid Ceramic Monoliths produced by Digital Light Processing

Characterization of the samples

SEM: Sintered samples exhibited a dense microstructure consisting of fine, equiaxed grains with localized residual intergranular porosity, Figure 2.

EDS: Elemental analysis confirmed the presence of Zirconium (67.43 wt%) and Yttrium (7.88 wt%), aligning with the expected stoichiometric composition of 3Y-TZP, Figure 2.

Micro-CT: Non-destructive internal porosity analysis and structural integrity assessment were performed via micro-computed tomography.

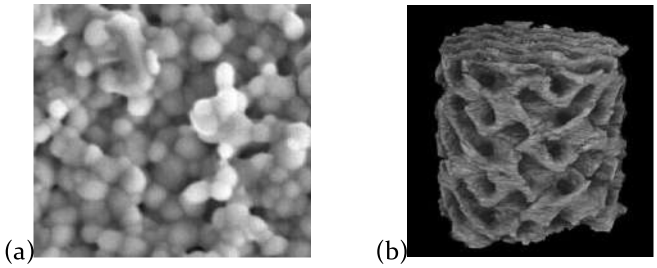

Figure 2. (a) SEM micrograph of microstructure. (b) Micro-CT analysis

CONCLUSIONS

This work successfully developed and optimized a fabrication process for $3Y - TZP$ ceramic monoliths using DLP, establishing that an optimal formulation of 30 wt% zirconia with % DisperBYK provides the stability and rheological behavior necessary for producing complex, reproducible gyroid geometries. Precise adjustments to printing parameters, specifically bottom and normal exposure times, were critical to achieving high geometric fidelity and preventing common defects such as delamination or overgrowth. Furthermore, the implementation of controlled, slow-heating thermal cycles guided by TGA and DTG analysis proved to be the essential solution for ensuring structural integrity during resin removal and achieving full densification. Final characterization through SEM and EDS confirmed a dense, homogeneous microstructure with fine equiaxial grains and a consistent chemical composition, validating the feasibility of these structures for potential use as catalytic supports. Future work should explore ceramic loadings above 30 wt% to improve final density, exploring alternative dispersants for higher solid concentrations, and evaluating the textural properties and specific catalytic performance.

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Resorbable Polymeric and Zn-Based biomaterials for vascular applications

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Keywords (6 maximum): Bioresorbable materials, Poly(lactic-co-caprolactone), Zinc alloys, 3D-printing, Surface modifications, Endothelialization

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Abstract: Bioresorbable materials offer temporary vascular support while avoiding long-term complications of permanent stents. This study evaluates poly(L-lactide-co-ε-caprolactone) (PLCL) and Zn–2Ag alloys as candidates for bioresorbable stents. PLCL stents fabricated by solvent-cast direct-write (SCDW) 3D printing showed Tg values near body temperature, suitable mechanical performance, and favorable endothelial response. Zn–2Ag alloys processed by equal-channel angular pressing (ECAP) exhibited grain refinement, uniform degradation, and antibacterial activity. Both materials demonstrate strong potential for next-generation bioresorbable vascular devices.

INTRODUCTION: In vascular applications, non-permanent implants are desirable to restore vessel patency while avoiding long-term complications of permanent stents, such as chronic inflammation and late thrombosis [1–3]. Bioresorbable devices provide temporary support and degrade after healing. Bioresorbable polymers offer good processability and biocompatibility [4,5], and 3D printing enables customized structures. Alternatively, bioresorbable metals provide superior mechanical strength; zinc (Zn) is particularly attractive due to its suitable degradation rate compared to iron and magnesium [6,7], although early early surface degradation may hinder endothelialization. This work evaluates poly(L-lactide-co-ε-caprolactone) (PLCL) and Zn-based alloys as candidates for vascular devices. PLCL stents were fabricated by solvent-cast direct-write (SCDW) 3D printing, while Zn alloys were structurally modified by equal-channel angular pressing (ECAP) and assessed for mechanical performance, degradation behavior and biological response.

METHODS: PLCL stents with 85:15 (P85) lactic to caprolactone ratio and a blend (B70) consisting of a mixture of P85 and 70:30 (P70) lactic-caprolactone molar ratio were dissolved in chloroform at 30.25% w/v and fabricated using the solvent-cast direct-writing (SC-DW), [5], with a custom-made Power DIW 3D printer (CIM-UPC, Spain) and thermally treated at 65°C. Strut thickness and morphology (OM), thermal (DSC, DMTA), and mechanical properties (compression and radial test) were characterized. Biological characterization was conducted through dynamic endothelialization (HCAECs) and thrombogenicity assays. Cold-rolled Zn-2Ag (2 wt.% Ag) underwent one (E1) and two (E2) ECAP cycles with a 90° internal angle via route BC at room temperature (RT). Microstructure (EBSD), mechanical properties, corrosion behavior (PDP and immersion in Hank's solution), cell adhesion (HUVECs), and antibacterial response (S. aureus) were evaluated.

RESULTS AND DISCUSSION: PLCL bioresorbable stents were successfully 3D-printed with controlled geometry and a strut thickness of ~230 μm. Thermal analysis showed Tg values close to body temperature, particularly for B70 (Tg=32°C; χc=20.3%.) compared to P85 (Tg=42°C; χc=26%). DMTA showed Tg values around 37°C for both formulations, with shape recovery at body temperature of 21% for P85 and 79% for B70. In terms of mechanical performance, P85 showed higher compression resistance at 50% deformation (P85: 0.035 N/mm; B70: 0.023 N/mm) and elastic recovery (P85: 98.01%; B70: 95.3%). All stents demonstrated high cell viability, endothelial adhesion, and reduced thrombogenicity for thinner designs.

Zn–2Ag alloys presented grain refinement and a homogeneous microstructure after two ECAP cycles, consisting of a Zn matrix with an AgZn3 secondary phase. Although UTS and YS decreased after ECAP (from 125 ±1 MPa and 100 ± 7 MPa to 81 ± 3 MPa and 52 ± 4.2 MPa, respectively), isotropic behavior and superplasticity were observed. ECAP-processed samples showed uniform degradation in Hanks' solution, with a corrosion rate of ~90 mm/year. Ag-containing alloys reduced bacterial adhesion, with E2 samples exhibiting enhanced antibacterial performance attributed to their homogeneous degradation behavior.

CONCLUSIONS: 3D-printed PLCL stents with Tg close to body temperature showed favorable mechanical and biological performance. Zn–2Ag alloys exhibited homogeneous degradation and antibacterial activity after ECAP processing. Both materials demonstrate strong potential as candidates for bioresorbable vascular devices.

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In-situ alloying of Titanium-Niobium alloys via Electron Beam Melting

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Keywords: Titanium, Niobium, 3D printing, Electron Beam Melting

Abstract: Biocompatible Titanium-based alloys have long held a significant position in the biomedical field due to their mechanical strength and biocompatibility. In particular, Nickel-Titanium (NiTi) alloys have gained attention for their unique shape memory properties. However, concerns regarding Ni-hypersensitivity have motivated the exploration of Ni-free Ti-based alloys alternatives. Titanium-Niobium (TiNb) alloys have emerged as promising candidates, offering not also the shape memory properties in some cases, but also reducing the Young's modulus of the material, thereby mitigating the stress-shielding effect and promoting osseointegration. This study explores the fabrication of Ti-xNb alloys (x=0, 15, 20, 25, 30 wt.%) using Electron Beam Melting (EBM) with an Arcam ST12 system. Elemental Ti and Nb powders (particle sizes <150 and <74 µm, respectively) were utilized to evaluate the feasibility of in-situ alloying. To define the process window, a gradient of parameters was established by varying beam speeds (1000–1400 mm/s) and currents (4–4.8 mA) to analyse the impact of energy density on consolidation. Comprehensive characterization was performed using XRD, DSC, and SEM-EDS, alongside microhardness (HV) and nanoindentation to assess mechanical shifts. SEM observations revealed a multi-phase microstructure in samples containing Nb, where the presence of some unmelted Nb particles indicated the influence of energy density on atomic diffusion. Furthermore, a clear gradient in hardness and mechanical response was observed as a function of niobium content. These results confirm that EBM allows for the precise tuning of TiNb alloy properties, offering a viable pathway for customized, low-modulus orthopaedic implants.

Synthesis of MXene Nanomaterials with EMI Shielding Functionalities

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Keywords: MXene sybthesis, electromagnetic characterization, EMI shielding.

Abstract:

MXenes are a recent class of 2d transition metal carbides and nitrides with singular properties that make them suitable for multiple applications in catalysis, energy storage electrodes, sensors, nano reinforced polymer films, tunable optoelectronic properties, etc. In addition, their high electric conductivity offers efficient EMI shielding capabilities (1).

Their name Mxene comes from their 2D laminar structure, similar to graphene. Mxenes have a general formula M n+1AXn , where n = 1 to 4. They are obtained from their parent MAX phases, where M refers to an early transition-metal element (Ti, Nb..), A is a group 13–16 element, and X is carbon and/or nitrogen.

Ti3C2T was the first obtained Mxene (2) and to date, it is the most widely studied. It was initially obtained by etching the max phase Ti3AlC2 with concentrated hydrofluoric acid. This synthesis process creates a mixture of =O, –OH, and –F terminations. Physical and chemical properties of MXenes vary with the composition of these surface terminations. When the MAX A-site atoms are etched, the exposed transition metal atoms are immediately coordinated by anions from the etchant, giving the surface terminations Tx of Mn+1CnTx MXenes. Control of the type and position of these moieties is a relevant research topic (3,4,5) and opens opportunities to optimize the mxene properties.

In this work we present preliminary results that explore alternative synthesis routes to the traditional etching of the MAX phases in harmful HF acid, obtaining several types of Mxenes: Ti3C2Tx, Ti3CNTx, V2CTx and with different Tx functional terminations. The scope was to synthesize MXene-based nanomaterials with enhanced EM shielding and absorption properties and to obtain hybrid composites by integrating MXene into polymeric matrices. EMI shielding values ranged from 30 to 60 dB, similar to those measured by others (6), confirming the effective EMI shielding efficiency properties.

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From waste to worth: Efficient regeneration of NMC111 from spent motorcycle LIBs

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Keywords (6 maximum): Lithium-ion batteries ; Cathode regeneration ; NMC111 ; Hydrometallurgical-combustion route ; Sucrose-assisted synthesis.

Abstract:

The global transition toward sustainable mobility, particularly the rapid electrification of the two-wheeler sector, has catalyzed an unprecedented demand for lithium-ion batteries (LIBs). With electric vehicles projected to command 30% of the global transport market by 2030 [1], the synergy between battery storage and renewable energy is essential for grid reliability and energy efficiency. However, this expansion brings critical end-of-life management challenges. The extraction of battery-grade metals like lithium, cobalt, and nickel remains ecologically taxing and economically volatile, with raw materials accounting for nearly 50% of production costs. Consequently, there is an urgent need for scalable recycling technologies that minimize environmental footprints while recovering high-value materials [2, 3].

Current recycling methodologies generally rely on pyrometallurgical, hydrometallurgical, or direct recycling techniques. While hydrometallurgical routes are favored for their low energy consumption and high metal purity, they often struggle with the trade-offs between "green" organic acids and the superior leaching efficiency of inorganic acids [4]. For instance, while organic acids are environmentally friendly, hydrochloric and sulfuric acids offer near-total leaching efficiency, though they carry risks of toxic gas generation or adverse sulfur contamination in reprocessed cathodes [5]. To optimize these processes, nitric acid has emerged as a compelling leaching agent, as it has been shown to yield recycled products with higher battery capacities compared to sulfuric-leached counterparts.

In this study, we propose a streamlined upcycling route that bypasses the complexities of conventional co-precipitation or sol-gel methods, which are often time-consuming and parameter-sensitive [6]. By integrating nitric acid leaching with a sucrose-assisted auto-combustion method, we developed a rapid, low-energy process that eliminates the need for a separate metal recovery step [7]. Sucrose serves as an inexpensive, effective fuel agent that promotes the formation of nanosized particles, offering a significant industrial advantage over more complex fuel mixtures [8].

The resulting $LiNi_{0.3}Mn_{0.3}Co_{0.3}O_2$ (NMC111) particles exhibited a pure layered phase with a well-defined, quasi-spherical morphology and an average particle size of 342 nm. The regenerated cathode material demonstrated robust electrochemical performance, delivering an initial charge capacity of 170 mAh/g and a discharge capacity of 137 mAh/g at a C/5 rate. Furthermore, the material maintained a cycling stability of 93% after 50 cycles. These findings validate the hydrometallurgical–combustion route as a highly effective and simplified strategy for transforming spent motorcycle batteries into functional, high-performance cathode materials for the next generation of energy storage.

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