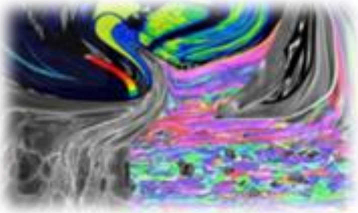

# Advanced materials through low temperature processing!

June, 3rd 2025

This meeting is organized in collaboration with the **ANR project "Pre-cold'it"**

ICMCB, Institut de Chimie de la Matière Condensée de Bordeaux
87 avenue du Dr. Albert Schweitzer - Pessac

# Program

**Sponsored by :**

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This event was organized in the frame of the SF2M-SO activities, represented by the **members of the SF2M-SO Scientific Council**: Moukrane Dehmas (President), Christophe Drouet (Vice-president), Denis Delagnes (Treasurer), Angeline Poulon, Nicolas Pradeilles, Fabrice Salles and Joël Alexis.

## Special thanks

We warmly thank the **ICMCB laboratory** for hosting this SF2M-SO/ANR *pré-Cold'it* event in Bordeaux, as well as all the attendees for the rich discussions that will arise from this Conference/workshop dedicated to low(er) temperature processing approaches for advanced materials.

We are also very grateful to our **Invited Speaker, Prof. Samuel Bernard** from IRCER Limoges, for straightforwardly accepting our invitation!

We particularly wish to thank **our sponsors** for their precious support in the organization of this event:

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Finally, great thanks go to additional **key members of the Organizing Committee**: Catherine Elissalde, Dylan Chatelain, Katarina Eliot, Thomas Hérisson de Beauvoir, Virginie Cosseron-Da Costa, and Caroline Job.

**Welcome of participants**

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|---|---|
| **9h15 – 9h30** | Introduction to the day by the Vice-President of SF2M Sud-Ouest and coordinator of the ANR pre-Cold'it project |
| **9h30 – 9h45** | U.C. Chung: Presentation of the Sintering Platform |
| **9h45 – 10h45** | **Invited Lecture – Dr Samuel BERNARD** (DR CNRS, IRCER Limoges)<br>"Polymer-derived ceramics (PDC) chemistry as a powerful tool for the low-temperature processing of functional inorganic materials" |

**Coffee Break**

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| **11h15 – 12h30** | **Session 1: Low(er) Temperature Material and Processing** – *Chairperson C. Estournès*<br>▪ J. Locs, K. Rubenis, L. Dauge, D. Loca - **Cold Sintering of Amorphous Calcium Phosphate Ceramics**<br>▪ D. Chatelain, Alain Denoirjean, Fabrice Rossignol, Nicolas Tessier-Doyen, Vincent Guipont – **Local sintering in the consolidation of hydroxyapatite coatings obtained by cold gas spraying**<br>▪ N. Bencharef, H. Debeda, C. Castro Chavarria, S. Buffière, P. Legros, D. Bernard, M. Josse, M. Maglione, U-C. Chung, C. Elissalde - **Optimizing low temperature Spark Plasma Sintering of KNN ceramics: microstructure and properties relationships** |

**Lunch & Poster session**

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|---|---|
| **14h00 – 15h30** | **Session 2: Cold sintering approach** – *Chairperson C. Elissalde*<br>▪ M. Biesuz, L. Karacasulu - **Cold sintering of volcanic ash from Mount Etna**<br>▪ T. Hérisson de Beauvoir, C. Estournes - **Characterization of microstructural evolution during Cold Sintering Process by impedance spectroscopy**<br>▪ Y. Denis, G. Philippot, E. Roitero, G. Goglio, M. Suchomel, U-C. Chung, T. Hérisson de Beauvoir, H. Reveron, J. Chevalier, C. Estournes, C. Elissalde - **Cold Sintering of Zirconia and Yttria Stabilized-Zirconia from reactive hydroxides precursors**<br>▪ N. Albar, C. Manière, T. Hérisson de Beauvoir, E. Sanchez, G. Chevallier, A. Weibel, C. Estournès - **Study of the kinetics of sintering by cold sintering process of zinc oxide** |

**Coffee Break**

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| **16h00 – 17h30** | **Session 3: Alternative Low Temperature Processing Routes** – *Chairperson S. Bernard*<br>▪ H. Tortajada, J. Topart, B. Belleville, F. Brouillet, D. Grossin, S. Panier - **Why the community of materials scientists need to support Open-Feedstock Binder Jetting project IP50 for low temperature processing: from scratch to proof of concept with calcium phosphate cement**<br>▪ T. Nogueira, O. Toulemonde - **Molten salt approach for perovskite synthesis**<br>▪ R. Troncy, L. Guibbert, F. Ansart, S. Duluard - **Low temperature synthesis of ZrO2 and YSZ via propoxide sol-gel routes: how acetylacetone chelating agent and nitrates influence the microstructure and composition of the intermediate compounds**<br>▪ J. Garcia, M. Busquet - **Development of a sol-gel based coating for aluminum fire protection** |
| **17h30 – 17h45** | Best presentation award ceremony |

**Closure of the day**

Poster session:

- Bertille Belleville, David Grossin, Fabien Brouillet, Christèle Combes, **Bone substitutes elaborated by low-temperature reactive additive manufacturing**
- Flavie Gaudin, Christophe Drouet, Christèle Combes, Philippe Barthelemy, Tina KAUSS, Clémentine Aubry, **Development of a new biomaterial to combat antibiotic resistance: focus on oligonucleotide/apatite interactions**
- Levent Karacasulu, Mattia Biesuz, Cekdar Vakifahmetoglu, **Reactive hydrothermal liquid-phase densification (rHLPD) of perovskite-based ceramics**
- Sabrine Medjouel, Yohann Thimont, Jeremy Soulie, Antoine Barnabé, Prescillia Lagarrigue, **Freeze-Casting of 3D Copper Foams for Advanced Porous Thermoelectric Materials**
- Hugues Tortajada, Jules Topart, Stephane Panier, **Open-Source Desktop Binder-Jetting Printer for Researchers**

# Invited lecture

## PDC chemistry as a powerful tool for the low-temperature processing of functional inorganic materials

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The PDC (Polymer-Derived Ceramics) method is an attractive synthetic approach in the design of advanced catalytic formulations that are based on non-noble transition metals with high degree of structural and compositional homogeneity. Nowadays, PDC chemistry gathers plenty of fascinating strategies to prepare materials from solution state precursors. Low temperature chemistry, reproducibility, and high surface to volume ratios of obtained products are features that add merit to this route. The development of different and fascinating procedures was fostered by the availability of preceramic polymers and transition metal complexes with the great advantage of tailoring the physicochemical properties of the materials through the manipulation of the synthesis conditions. Thus, the synthesis of preceramic polymers offers a number of features particularly advantageous to trigger the formation of metal NPs and trap them in a high SSA ceramic-based matrix upon heat-treatment at significantly lower temperatures than conventional processes. In addition, the shaping richness of preceramic polymers allows for 3D printing offering some unique properties for heterogeneous catalysis. The aim of this talk is to introduce an overview of the PDC synthesis of tailored and multifunctional inorganic materials including metal nanoparticles and ceramics) and their application in the main domain of electrocatalysis. One of the main achievements of this talk is to stress the versatility of PDC preparation by highlighting its advantage over other preparation methods through some specific examples of the synthesis of catalysts and 3D ceramic architectures.

# Oral Communications

# Cold Sintering of Amorphous Calcium Phosphate Ceramics

Janis Locs[1,2], Kristaps Rubenis[1,2], Linda Dauge[1,2], Dagnija Loca[1,2]

*[1] Institute of Biomaterials and Bioengineering, Faculty of Natural Sciences and Technology, Riga Technical University, Riga, Latvia.*
*[2] Baltic Biomaterials Centre of Excellence, Headquarters at Riga Technical University, Riga, Latvia.*

Amorphous calcium phosphate (ACP) is a promising material for biomedical applications due to its high biocompatibility, bioresorbability, and similarity to bone mineral. However, its utilization in bulk ceramic form has been limited by its inherent metastability and tendency to crystallize at elevated temperatures. This study presents a breakthrough in the densification of ACP through "cold sintering" under uniaxial compaction at room temperature (15–35 °C) and pressures up to 1500 MPa. The developed technique enables the formation of dense ACP ceramics with relative densities exceeding 98% without phase transformation, preserving the amorphous structure. These high-density ACP ceramics also demonstrate remarkable mechanical properties, such as Vickers hardness above 1.90 GPa and compressive strengths reaching 379 MPa. Furthermore, a novel drug delivery platform is proposed, where drugs such as gentamicin sulfate and strontium ranelate are co-sintered with ACP powder (avoiding thermal degradation of the drug), creating homogeneous ceramic matrices capable of sustained release. The integrated approach enables a single-step fabrication of multifunctional biomaterials suitable for multiple applications, offering tunable mechanical and drug-release properties by adjusting compaction parameters.

# Local sintering in the consolidation of hydroxyapatite coatings obtained by cold gas spraying

Dylan Chatelain[1], Alain Denoirjean[2], Fabrice Rossignol[2], Nicolas Tessier-Doyen[2], Vincent Guipont[3]

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3 *Centre des Matériaux UMR 7633, 91003 Evry, France*

Cold gas spraying (CGS) has emerged as a promising technique for the deposition of thermally sensitive materials, offering significant advantages over conventional thermal spray methods by operating at relatively low temperatures, thus avoiding phase decomposition and oxidation. While CGS has been successfully applied to ductile metals, its extension to brittle ceramic materials, such as hydroxyapatite (HA), remains a technical challenge due to the inherently low plasticity of ceramics, which impedes effective particle consolidation upon impact.

Hydroxyapatite, a calcium phosphate-based bioceramic, is widely recognized for its excellent bioactivity, biocompatibility, and chemical similarity to the mineral component of bone. These properties make HA coatings highly desirable for orthopedic and dental implants. However, maintaining its phase purity and crystallinity during deposition is critical for ensuring long-term implant performance. Conventional thermal spraying techniques often compromise these attributes due to high processing temperatures. In contrast, CGS allows for the deposition of HA at temperatures below its decomposition threshold, preserving its structural integrity. Nevertheless, the low impact ductility of HA powders results most of the time in poor bonding and low coating cohesion.

This presentation investigates the microstructural evolution and consolidation mechanisms of HA coatings deposited via CGS, with a focus on the role of local sintering phenomena. Coating morphology especially is characterized using SEM, SEM FIB, and image analysis. The findings suggest that even limited local sintering at contact zones between particles contributes significantly to mechanical integrity. These results provide a pathway toward understanding CGS mechanisms for the creation of coatings in the case of ceramic deposits.

# Optimizing low temperature Spark Plasma Sintering of KNN ceramics: microstructure and properties relationships

Nadia Bencharef[1,2], Hélène Debeda[1], Christopher Castro Chavarria[1,2], Sonia Buffière[2], Philippe Legros[3], Dominique Bernard[2], Michaël Josse[2], Mario Maglione[2], U-Chan Chung[2], Catherine Elissalde[2]

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[2] *CNRS, Université de Bordeaux, ICMCB, UMR 5026, 33600 Pessac, France.*
[3] *CNRS, Université de Bordeaux, PLACAMAT, UAR 3626, 33600 Pessac, France.*

Piezoelectric ceramic materials are highly developed and commonly used in advanced technologies. One particularly appealing application involves the conversion of ambient vibration energy into electrical energy, which can power autonomous "place and forget" small devices. MEMS (Micro-Electromechanical Systems) energy harvesting devices are particularly promising. Here, we focus on simple devices composed of a micro-beam (a "micro-cantilever") consisting of an active piezoelectric layer between two electrodes deposited on a flexible substrate. PZT-based ceramics are commonly used for their excellent piezoelectric properties. However, the toxicity of lead has stimulated research into lead-free material alternatives.

In this context, lead-free piezoelectric ceramics based on potassium sodium niobate (K0.5,Na0.5)NbO3 (KNN) are considered as promising candidates. Their performances are highly dependent on their composition, requiring preservation of the desired stoichiometry during sintering. The volatility of alkali metals at high temperatures, as well as issues related to sustainable development and energy consumption, have recently driven research towards non-conventional sintering processes (Flash, Cold Sintering Process (CSP), Spark Plasma Sintering (SPS), Spark Plasma Texturing (SPT), Ultrafast High-temperature Sintering (UHS)….) [1-4]. Despite significant advances, optimization of the KNN sintering process remains an active area of research, aiming to simultaneously optimize the density, microstructure, and piezoelectric properties of this lead-free material.

In terms of processes, SPS has proven to be effective for densification at low temperatures (<1000°C) and moderate pressures. However, post-annealing is required to eliminate oxygen vacancies that can form in a low oxygen partial pressure environment (graphite mold). In our preliminary study, an exploration of SPS experimental parameters was carried out using KNN nanoparticles. A specific temperature window was targeted due to the final objective of our study which is the SPS sintering of multilayers with a flexible StSt stainless steel substrate (StSt/electrode/KNN/electrode). Remarkable results were thus obtained by SPS at 700°C, without any additives or post-thermal treatment. KNN ceramics have a density of 97% and the average grain size is 400 nm. However, considering the piezoelectric properties, it is necessary to obtain controlled microstructures with a grain size >1 μm. Promising approaches were explored, playing on the powder reactivity and sintering kinetics while keeping the target temperature of 700°C. The impact of powder humidity and sintering parameters, including atmosphere (argon and high vacuum) and heating rate, on densification and microstructure will be analyzed. Structural, microstructural, dielectric and piezoelectric characterizations carried out to qualify the ceramics and study the influence of the different parameters will be presented (fig.1a-1.b). These results open up a strong potential for the development of MEMS devices for energy harvesting [5].

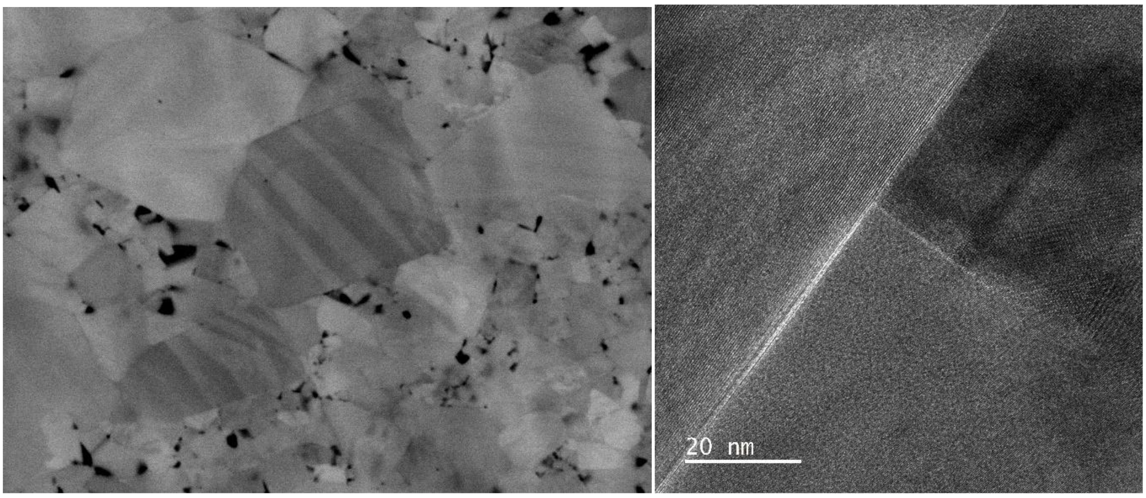

*Figure 1 : a. SEM and b. TEM images of the microstructure of a KNN ceramic by SPS at 700°C.*

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*[4] A. Kabir and al., Open Ceramics 17 (2024) 100551.*
*[5] H. Debéda and al., Chap 10 pp.219-255, Elsevier (2019).*

# Cold sintering of volcanic ash from Mount Etna

Mattia Biesuz[1], Levent Karacasulu[1]

[1] *University of Trento, Department of Industrial Engineering, Trento, Italy*

Volcanic ash disposal represents a significant economic, societal, health, and environmental challenge. This issue is more serious in the case of volcanoes with explosive and frequent eruptions, which eject large amounts of ash over the neighboring areas. Among the others, Mount Etna (Sicily, Italy) represents a clear example of the problems related to the disposal of such wastes.

Herein, we show that the cold sintering process, CSP, provides a unique tool to valorize the volcanic ash. A key advantage of CSP resides in the fact that the Etna ash can be processed without the addition of any additive (clay and/or binders), which limits the use of volcanic ash in the traditional ceramic industry to about 10-20wt% of the raw materials mass.

Different CSP conditions have been investigated, combining various pressures, liquid solutions, and temperatures. It is shown that strong porous ceramics can be obtained with flexural strength well-above that of traditional fired bricks (up to ≈ 70 MPa) and possessing relatively low thermal conductivity and diffusivity. On these bases, CSP represents an intriguing tool toward the re-use of a waste material to get compounds with properties competing with the traditionally fired artifacts.

# Characterization of microstructural evolution during Cold Sintering Process by impedance spectroscopy

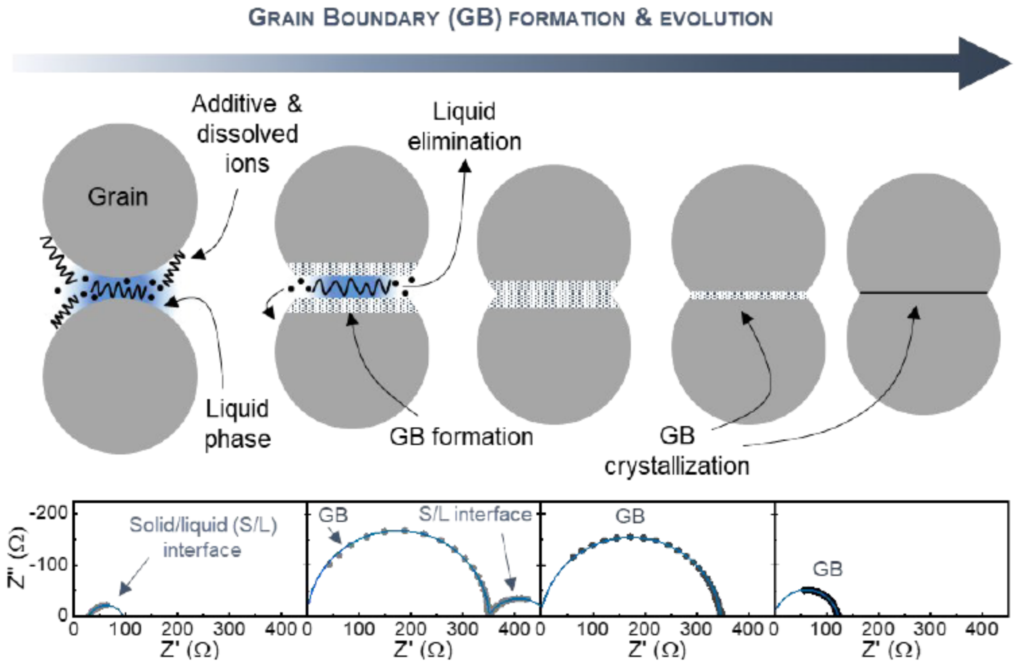
Thomas Hérisson de Beauvoir[1], Claude Estournes[1]

[1] *CIRIMAT, Université Toulouse 3 Paul Sabatier, CNRS-INP-UT3, Université de Toulouse, Toulouse 31062, France*

The development of new functional materials and devices has a close relationship with the development of processing technologies. Among them, sintering represents a key to prepare ceramics and composites, both in the bulk or multilayered forms. Recent discoveries of extremely low temperature sintering mechanisms, have led to a huge development of the Cold Sintering Process (CSP). Due to liquid phase mediated dissolution-precipitation, ceramics were sintered at temperatures < 300 °C such as ferroelectrics ($BaTiO_3$), piezoelectrics (PZT, KNN) or ionic conductors ($CeO_2$, YSZ), but also ceramic-metal and ceramic-polymers composites [1,2].

New concerns about the composition/nature/behavior of grain boundaries (GB) emerged, different than what is usually observed in high temperature solid-state diffusion sintering techniques. This leads to widely affected properties of sintered materials, and is of huge importance in the case of electroceramics, sensitive to GB properties. To better control this aspect, a first step is to highlight and track these GBs evolutions. An *in operando* impedance analysis, during the sintering process of ceramics, was developed and used to measure the evolution of electronic/ionic conductivity of GB and liquid phase present in the process [3] (see the figure below, on the relationship between change in impedance during dwell time and schematic evolution of solid-liquid mixture during CSP]).

Applying this to various ceramic materials allowed to shed light on unique GB evolution phenomena, but also highlighted the role and behavior of liquid phases used during sintering. The use of ionic liquids, often used for materials facing incongruent dissolution, was also explored and will be presented. The discussion will focus on the major trends, lessons learned from the use of *in operando* impedance analyses during CSP, offering opportunities to widely tune the properties of electroceramics through sintering at very low temperatures.



*References :*

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*[3] J. Eur. Ceram. Soc. 42 (2022) 5747-5755.*

# Cold Sintering of Zirconia and Yttria Stabilized-Zirconia from reactive hydroxides precursors

Y. Denis[1], G. Philippot[1], E. Roitero[3], G. Goglio[1], M. Suchomel[1], U-C. Chung[1], T. Hérisson de Beauvoir[2], H. Reveron[3], J. Chevalier[3], C. Estournes[2], C. Elissalde[1]*

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Recently, several non-conventional sintering methods were developed (Spark Plasma Sintering, Cold Sintering process (CSP), Hydrothermal Sintering) and have contributed to significant advances in the implementation of functional ceramics performed at low temperature with controlled properties and microstructures. In this context, our objective was to apply Cold Sintering Process to Yttria-Stabilized-Zirconia (YSZ) in order to produce dense ceramics at T ≤ 500°C. The issues raised using Yttria-Stabilized-Zirconia are the low solubility of zirconia and the incongruent dissolution between yttrium and zirconium, two limiting characteristics regarding the main mechanism of pressure solution creep involved in CSP. In this study, avenues are explored to reduce the impact of these issues in the CSP treatment of yttria-stabilized-zirconia compositions. Our approach is based on the use of hydroxide precursors of zirconium ($Zr(OH)_4$) and yttrium (YOOH) to achieve reactive cold sintering of dense YSZ ceramics at low temperatures. Starting from $Zr(OH)_4$, a monoclinic zirconia ceramic with a relative density of 83% was achieved in the presence of nitric acid at 500°C and 650 MPa. The material exhibits a Vickers Hardness of 6.5 GPa despite its low density and monoclinic structure. Additionally, yttria-stabilized-zirconia ceramic was obtained by reactive CSP, achieving a tetragonal phase percentage up to 72% and a relative density of 76%, with nitric acid at 400 °C and 450 MPa. The impact of the experimental parameters on both densification and structural characteristics is discussed. Analysis of the results reveals a complicated trade-off between densification and tetragonal phase stabilization for low-temperature CSP sintering. However, the potential of exploiting precursor reactivity to compensate for limited solubility, combined with concepts concerning the tailoring of specific microstructures to prioritize mechanical properties despite their structural aspects.

# Study of the kinetics of sintering by cold sintering process of zinc oxide

Nicolas Albar[1], Charles Manière[2], Thomas Hérisson de Beauvoir[1], Emeric Sanchez[1],
Geoffroy Chevallier[1], Alicia Weibel[1], Claude Estournès[1]

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Traditional ceramics are well known to the general public, whereas technical ceramics, which are much less familiar, have become essential to advanced materials industries. The production of these ceramics generally involves powder metallurgy with high-temperature sintering (i.e., >700°C) and cycle times ranging from several hours to several days. These conditions limit the range of usable materials to those with good thermodynamic and chemical stability [1].

In recent decades, significant advances in the properties of technical ceramics have been made possible by the emergence of, on the one hand, new methods for synthesizing nanometric powders (e.g., soft chemistry, sol-gel...) and, on the other hand, new shaping processes (e.g., Hot Pressing (HP), Hot Isostatic Pressing (HIP), Spark Plasma Sintering (SPS), Flash Sintering (FS)). These developments have notably enabled a substantial increase in densification kinetics and a lowering of sintering temperatures, allowing for better control of ceramic microstructures (grain size, porosity, etc.).

More recently, the emergence of low-temperature sintering techniques (e.g., Cold Sintering Process (CSP), Hydrothermal Sintering (HS), Cold SPS (C-SPS), or ultra-fast SPS (Flash SPS)) has made it possible, by activating mechanisms different from those involved in conventional sintering, to stabilize and densify metastable materials and/or to control microstructures (composition and thickness of grain boundaries). This has paved the way for the development of new materials and/or materials with enhanced properties [2–6].

While their effectiveness has been demonstrated, their development is still limited by a lack of fundamental understanding of the mechanisms involved. The use of kinetic models developed to describe sintering under load allows us to define parameters associated with these mechanisms and thus control densification and microstructure during CSP. Adapting these models to CSP sintering conditions (presence of a transient liquid phase) makes it possible to predict the sintering behavior of the studied material [7,8].

Ultimately, it could be possible to develop materials suitable for a wide range of applications, such as energy, transportation, or structural materials.

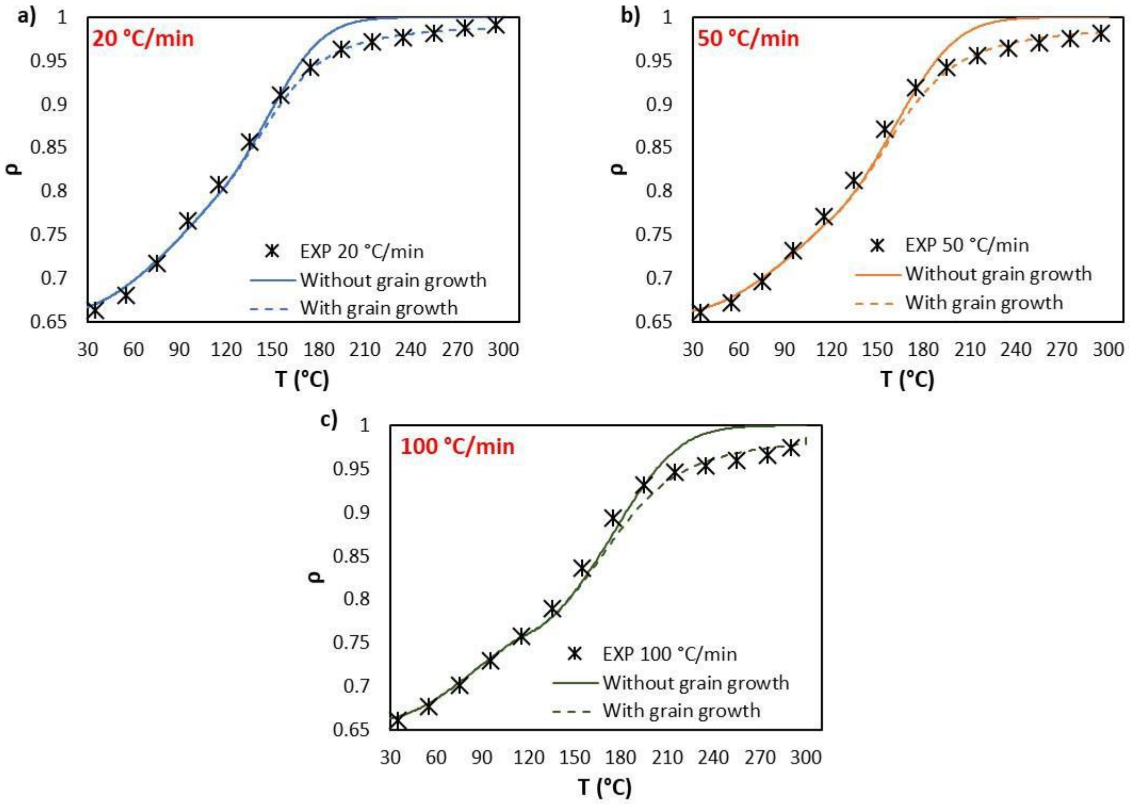

*Figure 1: Simulated $\rho$ = f(T) curves with and without grain growth and experimental ones at a) 20 °C/min, b) 50 °C/min and c) 100 °C/min.*

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# Why the community of materials scientists need to support Open-Feedstock Binder Jetting project IP50 for low temperature processing: from scratch to proof of concept with calcium phosphate cement

Hugues Tortajada[1], Jules Topart[1], Bertille Belleville[2], Fabien Brouillet[2], David Grossin[2], Stéphane Panier[1]

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[2] *CIRIMAT (UMR 5805 CNRS, Toulouse-INP, Université de Toulouse), Toulouse*

Keywords: additive manufacturing; binder jetting; calcium phosphate; cement; hydroxyapatite

Additive Manufacturing (AM) technologies are revolutionizing material science, notably through their ability to create complex geometries with precise control over composition. Binder Jetting (BJ) is a particularly promising AM technique [1] : it operates without supports, uses a wide range of powders, and offers scalability for industrial production. However, its accessibility remains severely limited due to the proprietary nature and high cost of existing BJ systems. In response to this challenge, the LTI Laboratory has initiated the IP50 project — an open- feedstock binder jetting 3D printer, designed from scratch to democratize access to BJ technology.

The IP50 project is unique in its dual innovation: alongside the hardware, LTI developed Powd3r, a dedicated 3D slicer tailored for binder jetting applications [2]. Powd3r generates specific toolpaths and control programs, overcoming a major software gap for BJ open-source projects. The entire machine ecosystem, encompassing both slicing and embedded control, has been made freely available, following the open-access philosophy promoted by initiatives such as Oasis 3DP [3]. By focusing on low-cost, modular, and accessible solutions, the IP50 project directly addresses the needs of academic researchers, small laboratories, and independent developers (with a small build volume of 50 × 50 × 50 mm). Although LTI provided a compiled (closed-source) slicing software with the machine, the system natively interprets standard G-code syntax and can therefore be operated using alternative software solutions. While LTI distributes the compiled slicing software, it remains proprietary and is not open-sourced.

Beyond technological democratization, the IP50 opens new scientific opportunities, especially in the domain of low-temperature processing of bioceramics. 3D-printed bioceramics, particularly calcium phosphate and carbonate-based materials, hold enormous potential for bone substitution applications [4]. These materials require manufacturing at room temperature due to their metastable nature. Traditional BJ processes involve high-temperature post-processing steps (debinding and sintering), which are incompatible with the preservation of the bioactive properties of such cements [4].

The IP50, combined with tailored cement formulations, addresses this critical limitation. In our proof-of-concept study, we adapted inorganic bone cements for binder jetting, synthesizing reactive calcium phosphate powders and formulating multi-component systems capable of setting at room temperature. Key challenges included powder characterization, adjusting the dissolution-recrystallization rate via additives, and selecting aqueous-based binders that trigger the cement setting reaction without compromising print fidelity.

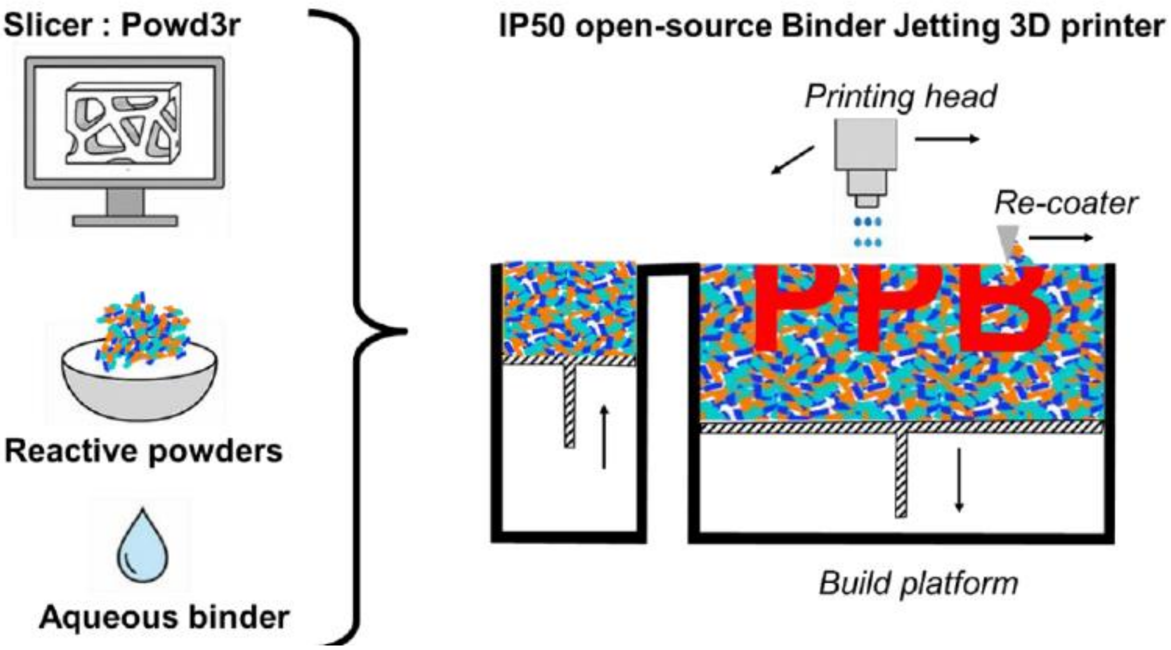

*Figure 1: The IP50 Project and Calcium Phosphate Cement Proof of Concept*

This approach offers a fully reactive AM process where consolidation occurs during printing itself, without any post-printing thermal treatment. It represents a major shift for the fabrication of personalized bone scaffolds, maintaining crucial physico-chemical properties for osseointegration while leveraging the advantages of AM. Moreover, by integrating low-temperature reactive formulations with open-source hardware and software, the IP50 establishes a new research platform for material scientists to explore room-temperature AM strategies.

In conclusion, the IP50 project embodies both a technological and scientific leap: it democratizes access to an otherwise restricted manufacturing technique and enables novel research into low-temperature additive manufacturing of sensitive materials like bioceramics. Community support is essential to further develop, validate, and expand this platform. By fostering a collaborative open-feedstock environment, material scientists can collectively push the boundaries of binder jetting technology making it more inclusive, versatile, and suited to emerging challenges in biomaterials and beyond.

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# Molten salt approach for perovskite synthesis

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Since almost 80 years, a lot of research on functional ferroelectric materials have been done. Until the 2000s, the properties improvement was the privileged factor with in particular the use of lead zirconate titanate (PZT) base materials which is an excellent insulator with significant polarization. Nevertheless, since then a change of paradigm took place considering environmental issues [1] and allowing the emergence of new interest phases called « lead-free ceramics ». Among materials/phases of particular interest, certain compounds such as Potassium Sodium Niobate (KNN), Barium Titanate (BT) and/or Sodium Bismuth Titanate (NBT) -based materials are promising to replace PZT [1].

In this work, a focus is made on two phases: Strontium Sodium Bismuth Titanate (SNBT) and high entropy Bismuth Sodium Barium Strontium Calcium Titanate (BNBSCT) for their dielectric permittivity higher than NBT [2-4] and regarding the recent study underlying an intriguing interplay between local polarization, perovskite A-site ionic size and octahedral tilt [5]. In this presentation, a cost-effective salt synthesis route « molten salt » was used allowing to significantly decreased both synthesis and sintering temperature.

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*[3] Liu and al. « Phase Evolution in (1-x)(Na0.5Bi0.5)TiO3-xSrTiO3 Solid Solutions: A Study Focusing on Dielectric and Ferroelectric Characteristics »*
*[4] Pu and al. « Dielectric Properties and Electrocaloric Effect of High-Entropy (Na0.2Bi0.2Ba0.2Sr0.2Ca0.2)TiO3 Ceramic »*
*[5] Sharma and al.« Interplay of Local Polarization, Ionic Size, and Octahedral Tilt in SrTiO3 - Na0.5Bi0.5TiO3 »*

# Low temperature synthesis of $ZrO_2$ and YSZ via propoxide sol-gel routes: how acetylacetone chelating agent and nitrates influence the microstructure and composition of the intermediate compounds

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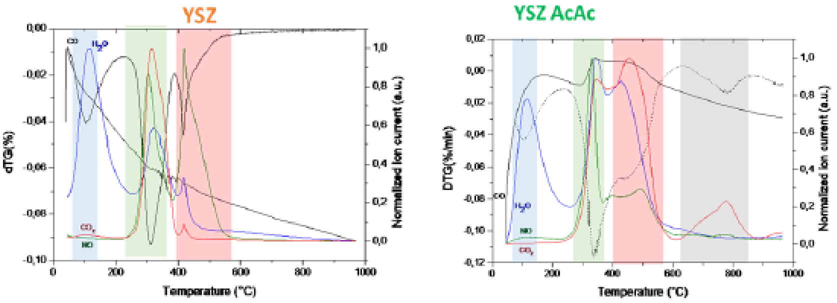
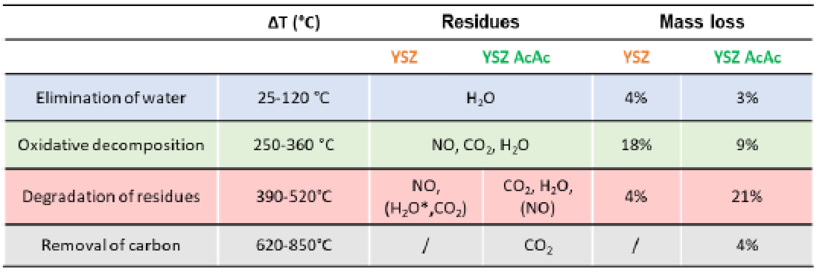
Low temperature syntheses via sol-gel route are known to allow both fine stoichiometry and crystallinity degree control for powder preparation. Alkoxide routes are of particular interest to tune finely the reactivity of the precursors and in fine the purity and microstructure of the intermediate compounds. However the presence of organics is a challenge that has to be overcome to obtain pure powder at the lowest temperature as possible. Such low temperature syntheses are needed to lower energy consumption in the production processes of powder but also for specific applications, for example to maintain high specific surfaces for catalysts, to get high intermediate reactivity for sintering or to avoid quenching of photoluminescent ceramic.

The different mechanisms taking place during the synthesis and heat treatments of zirconia based gels were studied depending on the presence of acetylacetone and/or yttrium nitrates. A characterization study was performed based on FTIR and NMR analyses to identify acetylacetone clusters nature before and after hydrolysis step. The thermal events were observed by thermal analyses coupled with mass spectrometry (Figure 1). The crystallinity ratio, crystallite sizes and nature of intermediate species were studied via XRD analyses at different temperatures (25-1000°C range), and the powder morphology via microscopies.

The reaction between propoxide organic groups and nitrates is known in the literature [1]. A specific result of this study is the elucidation of the acetylacetone impact, with the inhibition of this reaction as well as the presence of reaction intermediates with very high degradation temperature (over 700°C) in presence of acetylacetone.





| | ΔT (°C) | Residues | | Mass loss | |
|---|---|---|---|---|---|
| | | YSZ | YSZ AcAc | YSZ | YSZ AcAc |
| Elimination of water | 25-120 °C | $H_2O$ | | 4% | 3% |
| Oxidative decomposition | 250-360 °C | NO, $CO_2$, $H_2O$ | | 18% | 9% |
| Degradation of residues | 390-520°C | NO, ($H_2O$*,$CO_2$) | $CO_2$, $H_2O$, (NO) | 4% | 21% |
| Removal of carbon | 620-850°C | / | $CO_2$ | / | 4% |

*Figure 1: Analysis of residues via thermal analysis coupled with mass spectrometry of YSZ xerogels prepared without (YSZ) and with acetylacetone (YSZ Acac).*

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# Development of a sol-gel based coating for aluminum fire protection

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The fire resistance of equipment and structures, and in particular of some of the functional and/or structural parts that make them up, is a major issue for all industrial sectors, and more particularly for the transport. Indeed, the fire safety is a major concern for the certification and implementation of equipment in many industrial fields.

In the air transport sector, this function is regulated by the standard ISO 2685, which defines the fire resistance of parts when exposed to a flame. This flame is defined by a specific temperature (1100 °C) and heat flux (116 kW/m²). Resistance of such a flame for at least 15 minutes is required to obtain the "fireproof" classification.

To guarantee such fire resistance, it has been proposed in the prior art to use fire-resistant metallic materials such as titanium. Unfortunately, this results in a weight increase, which leads to excessive energy consumption especially in the field of transport, for example excessive fuel consumption in the aeronautical sector. This strategy therefore runs counter to current environmental and ecological objectives.

Alternative solutions have been proposed in the prior art, one of which involves substituting titanium with a lighter material, such as an aluminium alloy, and applying a protective coating to compensate for its poor fire resistance. Examples of such protective coatings include organic intumescent paints, which have good fire resistance. However, due to their chemical nature these paints release potentially toxic smokes when exposed to a flame. Furthermore, to be effective, they must be applied to the substrates to be protected in relatively thick layers, typically thicker than 600 µm, which also leads to a significant increase in the mass of these substrates. These paints display also a poor mechanical properties and lifespan when exposed to the in service environmental conditions of an aircraft.

For all these reasons, we have proposed to develop a new surface protection based on the sol-gel process. The advantages of this technology are:

- Good thermal and chemical properties (inorganic materials) even when applied with low thickness < 200 µm
- REACh compliances
- Easy-to-process with good compatibilities with well-known paint processes
- Chemical compatibility with several kinds of substrates when applied as a coating: metallic, ceramic and organic materials
- Low release of volatile organic compounds when exposed to high temperatures

The strategy for fire protection is based on a ceramisation of the sol-gel layer occurring during flame exposure. A protective ceramic layer will be formed during fire exposition with excellent thermal properties (low thermal conductivity and thermal stability under ceramic melting point temperature) which acts as a fireproof barrier and insulates the substrate from flames.

Thus, by selecting the more suitable sol-gel matrix with specific fillers and adjusting the chemical composition of the sol-gel formulation, we succeeded to improve significantly the fire resistance of a classical aluminium-based alloy Al 2024 T3 (widely used in aeronautics). This performance was demonstrated thanks to a home-made burnthrough test and compared to the raw material. Some results are shown in the figure below.

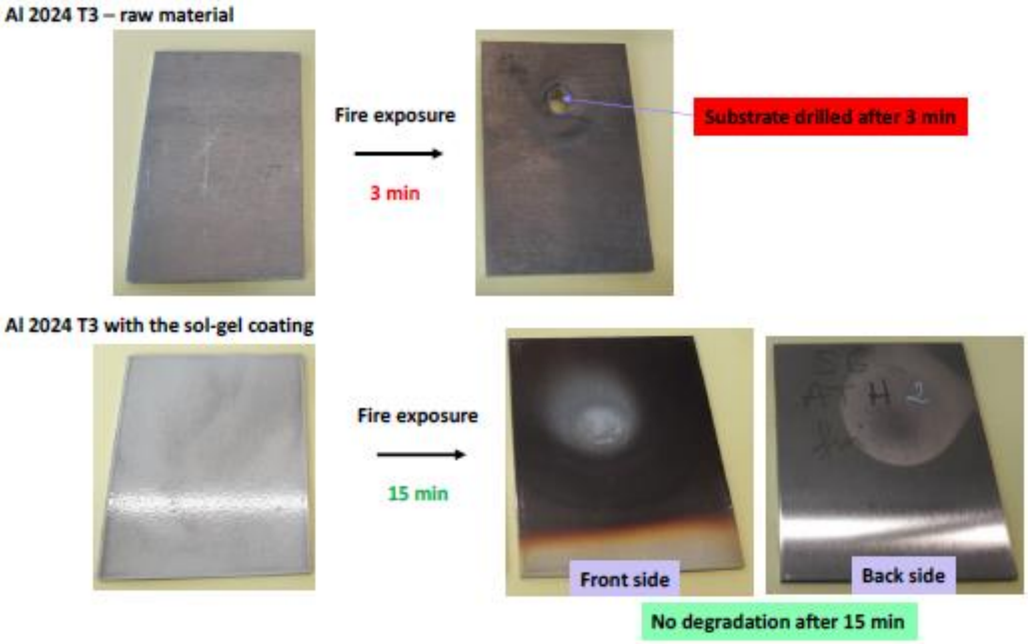

*Figure 1: Fire resistance assessment (burnthrough test)*

# Posters

# Bone substitutes elaborated by low-temperature reactive additive manufacturing

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3D-printed bioceramics are promising for bone substitution, due to their versatile composition and shape/porosity combined with the growing use of additive manufacturing (AM). AM offers several advantages as the ability to print complex and tailored scaffolds while maintaining essential physico-chemical properties, crucial for proper osseointegration.

We explore developing efficient and customizable bone substitutes by AM, using the reactivity of calcium phosphate and calcium carbonate metastable phases. Those materials need to be manufactured at low-temperature and possibly, in controlled humidity environment to preserve their metastability and biological properties. In our case, a key feature of reactive AM is the low-temperature consolidation, where the reactive shaping occurs during the printing process itself and thus, does not require post-printing thermal treatments. Such cement-based compositions can be tuned for AM thanks to their biocompatibility, the reactivity of the powders used and the setting mechanism occurring when adding water to the reactive powders. The aim is then to develop reactive binder jetting (BJ) and robocasting (or direct-ink-writing DIW), adapted to enable setting reaction to occur at low temperature while printing. This involves synthesizing beforehand reactive powders from mineral precursor salts suitable for both powder bed (for BJ) and the formulation of multi-component bone cements (as the solid phase for DIW). The process leverages the reactivity between the powders and the binder or liquid phase. The main challenge lies in controlling the evolution of the systems to develop printable modular compositions, besides setting appropriate printability parameters.

The first phase focuses on synthesizing and characterizing the three reactive powders studied: vaterite CaCO3 and brushite (CaHPO4·2H2O) and some seeds/particles of biomimetic apatite Ca10-x (PO4)6-x (HPO4, CO3)x (OH)2-x (avec $0 \leq x \leq 2$) [1], facilitating carbonated apatite nucleation. It includes optimizing the synthesis scaling-up to control crystal growth and ensuring the reproducibility and purity of powder batches. Powder pre-treatments are investigated [2], including co-grinding of the three-phase mixture in order to create reactive powders agglomerates, along with their impact on paste functional properties (extrudability for DIW and homogenization of the bed powder for BJ). The setting time can also be tuned with additives to accelerate or delay the setting reaction and adjust the dissolution-recrystallization rate. The binder / liquid phase formulation and biocompatible polymeric additives is intended to be studied, as well as the optimization of the printing conditions and printing parameters.

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# Development of a new biomaterial to combat antibiotic resistance: focus on oligonucleotide/apatite interactions

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Antibiotic resistance is a major challenge in medicine, especially for post bone reconstruction surgery infections. A solution to combat this resistance is to combine therapeutic oligonucleotides to antibiotics and calcium phosphate-based bone substitutes – specifically biomimetic apatites.

While this work presents a systematic study of oligonucleotide-apatite interactions, its aim is to enable the integration of such molecules into materials, particularly those produced at low temperatures, such as bone cements. This study investigates the adsorption of a model lipid oligonucleotide on apatite.

In order to carry out this study, it was first necessary to optimize the experimental parameters of the tests. A typical adsorption test is divided into two phases: the contact of the compounds of interest under agitation for a defined time, then the separation by centrifugation of the solid and liquid phases and their respective study. For each phase of the test, the fixed parameters have been optimized: speed of agitation, placement on the stirring plate, quantities used, centrifugation parameters.

Once the test parameters were optimized, kinetic studies were carried out and adsorption isotherms were plotted, giving information on the different adsorption energy sites. These preliminary studies demonstrate an efficient adsorption of the model oligonucleotide on the apatite.

Chemically modified oligonucleotides will further be studied in order to have a global understanding of the mechanisms of interaction oligonucleotides/apatite, and the release mechanisms investigated.

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# Reactive hydrothermal liquid-phase densification (rHLPD) of perovskite-based ceramics

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In recent years, various sintering approaches have been proposed to enable the densification of ceramic components at temperatures below 400°C, collectively referred to as cold sintering techniques. Among these, the reactive hydrothermal liquid-phase densification (rHLPD) process represents a distinct low-temperature densification route that enables simultaneous synthesis and densification through infiltration, hydrothermal reaction, and subsequent reactive crystallization.

In this study, perovskite (ABO3) ceramics via rHLPD were investigated with respect to their structure and electrical properties. Unlike other cold sintering methods, phase-pure TiO2 green bodies were first prepared by uniaxial pressing. These green compacts were then subjected to a hydrothermal reaction in an aqueous solution containing A-site cations. The produced samples were structurally characterized, and their electrical properties were investigated. Despite the low processing temperatures (≤ 240°C), monolithic perovskite-based samples were successfully formed, demonstrating promising properties without the need for post-annealing.

# Freeze-Casting of 3D Copper Foams for Advanced Porous Thermoelectric Materials

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The advantages associated with thermoelectric materials in the form of thin films and dense bulk structures are, respectively: (i) low thermal conductivity and enhanced radiative heat dissipation, and (ii) improved electrical transport, mechanical cohesion, and current percolation. Combining these characteristics would be highly beneficial for efficient heat-to-electricity conversion. In this context, the use of macroporous foams offers an attractive compromise, as their large surface area can promote intermediate thermal and electrical behavior, potentially leading to novel thermoelectric properties.

To be good candidates, thermoelectric materials must exhibit specific thermal properties as low thermal conductivities while maintaining high electrical conductivity and Seebeck coefficients. Copper iodide (CuI), which has so far been mainly obtained as thin films to ensure transparency for p-type Transparent Conducting [1], suffers from limited thermoelectric performance due to the thin films configuration. Developing CuI in bulk porous architectures—such as three-dimensional foams with oriented porosities—thus appears as a promising route to enhance its thermoelectric conversion efficiency. However, such materials remain challenging to fabricate and, to date, have not been thoroughly investigated.

In the aim to obtain such porous CuI architectures for thermoelectric applications, a two-step approach is required: 1) elaboration of copper foam, 2) iodination of the resulting metallic foam. However, the $Cu_0$ conversion in CuI is accompanied by a substantial volume expansion (up to four times) as copper is transformed into copper iodide. In dense copper bulks, this expansion often lead to incomplete transformation. To overcome these limitations and ensure good thermal management, it is essential to start from a copper scaffold with a well-controlled porous architecture, capable of accommodating the volumetric changes while maintaining overall structural integrity and porosity in the final CuI material.

Freeze-casting emerges as a particularly relevant cold-processing technique for this purpose. This process relies on the controlled freezing of a suspension, followed by sublimation to generate porosity. It enables precise control over the 3D porous structure (including pore size, orientation, and anisotropy) through careful adjustment of formulation and processing parameters. During freezing, solvent crystals grow directionally and act as sacrificial templates, while the suspended particles are progressively concentrated and organized within the polymer-rich phase as shown in the **figure** below. Subsequent freeze-drying removes the frozen solvent, yielding a green body with a well-defined, anisotropic porous architecture. By integrating debinding and partial sintering steps, robust copper foams with interconnected and aligned porosity are obtained. The directional pore channels not only improve iodine diffusion and reactivity throughout the foam, but also contribute to enhanced thermal dissipation—an asset for thermoelectric applications.

This study presents the fabrication of macroporous copper scaffolds by freeze-casting, using HydroxyEthyl Cellulose (HEC) as a binder. The initial efforts focused on optimizing HEC/Cu aqueous suspensions by varying both HEC concentration and HEC/Cu ratios to control the rheological behavior and thus, porosity features of the green bodies. Structural characterization of the freeze-cast samples prior to debinding and sintering confirmed the formation of anisotropic, interconnected porous architectures, as revealed by SEM imaging and porosity analysis. Subsequent thermal treatments were studied and applied to remove the binder and sinter the copper walls, preserving the oriented porous structure. XRD, TGA, and SEM analyses were combinedly used throughout to assess the phase composition, thermal stability, and morphological evolution of the foams. These preliminary results demonstrate the successful elaboration of anisotropic copper scaffolds with tailored porosity, suitable for subsequent iodination. Further work is ongoing to optimize the freeze-casting and iodination steps, with the aim of achieving complete and homogeneous CuI conversion, followed by evaluation of the thermoelectric properties of the resulting materials.

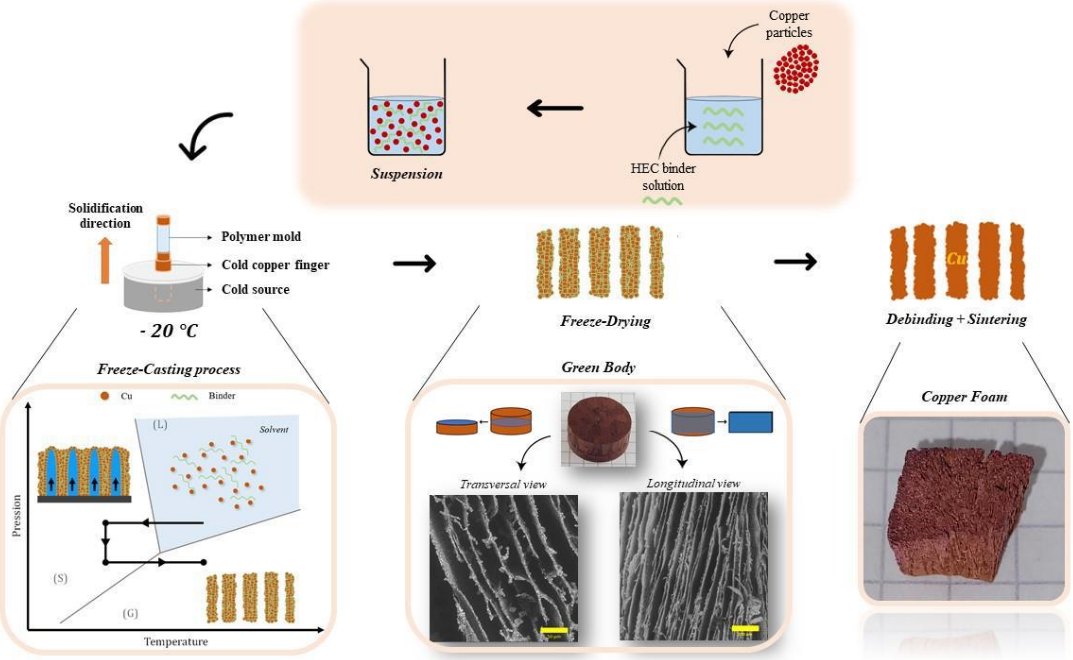

*Figure 1: Key Steps in the elaboration of Copper Scaffolds via Freeze-Casting*

*References*

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# Open-Source Desktop Binder-Jetting Printer for Researchers

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Binder jetting 3D printing is emerging as one of the most promising additive manufacturing processes, thanks to its great versatility and its ability to produce complex shapes from a variety of powders-ceramics, metals, or glasses. However, access to this technology remains limited in academia because of the high cost, large footprint, and low flexibility of commercial machines. It was in this context that our team at the Laboratory of Innovative Technologies (LTI) set out to design a 3D printer specifically tailored to research laboratories.

The primary goal of this development is to offer a solution that is affordable, modular, and suited to the experimental constraints of researchers. Thus, the printer we have designed stands out by its compact size with a build volume of 50 cm³, its low resource consumption, and its modular architecture, which allows users to make modifications according to their specific needs. At the heart of the machine is an HPC6602A thermal printhead, widely available and easy to integrate. The system is driven by free software such as Repetier or Pronterface, and benefits from an in-house slicer, Powd3r, capable of generating trajectories optimized for binder-jetting.

This approach enables users to print complex materials directly in a fully controlled environment, while promoting an open-development ethos. The machine is primarily aimed at researchers who wish to explore new powder or binder formulations. Initial tests carried out on the platform have validated the feasibility of a fully binder-jetting–based process. In parallel, the machine's hardware and software architecture have been designed for rapid evolution. Possible improvements are already under consideration, notably the integration of more ergonomic user interfaces such as the Klipper system—and the exploration of new piezoelectric printheads, better suited to a wider and more complex range of formulations.

Beyond the technological tool itself, this project is part of a broader dynamic of scientific sharing and collaboration. By making low-cost additive-manufacturing tools accessible, we hope to foster the emergence of a user community capable of advancing the process collectively, exchanging knowledge about formulations, and opening up new avenues in the field of innovative materials. In this way, this laboratory-dedicated 3D printer represents not only a technical solution but also a genuine lever for scientific exploration, education, and innovation.

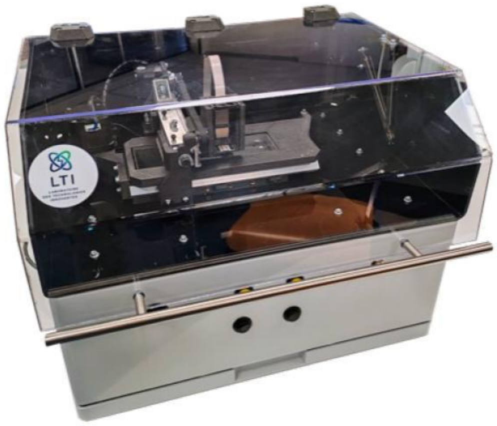

*Figure 1: The IP50 printer [1]*

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