# Scientific Highlights 2024



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#### **EDITO**

#### HIGHLIGHTS 2024 Laboratoire 3SR

February 2024 Grenoble, France The 3SR laboratory is a Joint Research Unit (UMR 5521) between Université Grenoble Alpes, CNRS and Grenoble INP - UGA Institute of Engineering and Management. With a staff of around 110, 3SR conducts cutting-edge research on the mechanics of solids, materials and structures, for a wide range of applications (civil engineering, environment, energy, transport, defense, health). To this end, its researchers develop a multi-scale approach (to investigate phenomena ranging from quasi-static to ultra-fast across scales from nm to decam) and a multi-physics methodology combining experimental mechanics (including materials or structures coupled with 2D/3D imaging, field measurements, etc.) as well as theoretical and numerical modeling (homogenization, constitutive laws, simulations of nano/micro/meso/macro structures using finite elements, finite volumes, discrete elements, molecular dynamics, probabilistic methods, etc.).

2024 was an eventful year for 3SR, with a new management team in place as of January 1, 2024; the appointment of one of its members to the IUF, and the award of two prestigious prizes to the laboratory for its groundbreaking work on in operando scientific imaging using X-rays and neutrons (Cristal collectif CNRS and Prix du logiciel opensource).

The 3SR's lab. management is delighted to present the first in a new series of annual collection of highlights of our research through 8 research initiatives that illustrate some of the laboratory's research activities for the year 2024.

Our warmest thanks to Mehdi BOUZID, the editor of this inaugural issue of our highlights, and we hope you enjoy reading it.



**3SR MANAGEMENT TEAM** Director : Gaël COMBE Deputy director : Lucie BAILLY Administrative and financial manager : Shéhérazade MEZENNER

#### **DIRECTOR OF PUBLICATION** Gaël COMBE

**EDITOR** Mehdi BOUZID

**LAYOUT** Tim CATINAT

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## **AWARDS and EVENTS**



Cristal collectif CNRS – Plateforme TomoMéca P. Charrier (3SR), O. Stamati (3SR), N. Lenoir (3SR), A.-C. Hladky (DAS CNRS)



SPAM: Software for Practical Analysis of Materials. E. Roubin (3SR), E. Andò (EPFL), O. Stamati (3SR). Prix science ouverte du logiciel libre de la recherche, Ministère de l'éducation nationale et de la recherche



A. Tengattini (3SR), nommé membre junior à l'IUF. Cérémonie du 18 oct. 2024



Inauguration de la chaire industrielle M2-Brittle's Codex, P. Forquin (3SR), C.Martin (SIMAP), Saint GOBAIN et la Fondation UGA, 18 Sept. 2024.

### **AWARDS and EVENTS**



Gunnar Rugheimer Prize 2024 - Best Poster Award - 15th Congress of the PanEuropean Voice Conference, Spain. "On the development of a mechatronic avatar capable of articulating speech and singing", coll. GIPSAlab/3SR, PhD Mounib Tlaidi.



Gustavo PINZON, PhD Prize 2024 ED I-MEP2, UGA



ALERT Geomaterials Ioannis Vardoulakis PhD Prize 2024 : I. VEGO



Biomim'Challenge 2024 - 3rd Prize - "Bio-inspired approach to save water, energy natural resources : Lightweighting paper-based packaging material by printing patterns", coll. LGP2/3SR, PhDs Clément Turpin (LGP2/3SR), Maxime Legay (LGP2), Léopold Oudinot (3SR/LGP2).

# Paper: a model material to study the rupture of heterogeneous materials

Statistical analyses of the behavior of paper mate in traction highlight two regimes, depending on whether the rupture is due either to the propagation of a crack or to a more diffuse process. These results could lead to an improved modeling of fracture risks in heterogeneous materials such as concrete, rock, or composite materials.

Understanding and modeling the failure risks of heterogeneous quasi-brittle materials, like concrete, composites and some rocks, is a major challenge in mechanics. Their fracture, under the action of a mechanical load, results from a progressive microcracking in a damage zone where finally a main crack propagates through the material. However, an inherent difficulty in studying the fracture of such materials lies in their opacity to visible light, which limits direct observations of the phenomenon and its understanding. Therefore, paper material has been chosen as a quasi-brittle model material, as its microstructure and damage mechanisms can be easily revealed by optical transmission.

Tensile tests were carried out on paper samples until complete rupture in order to analyze in a original way the force evolution. As in various papers, the force reached a peak before decreasing. During this decrease, due to the heterogeneity of the material, load fluctuations were observed. The statistical distribution in amplitude of the fluctuation is analyzed in order to find the signature of the heterogeneities. The study shows that for samples whose failure is the consequence of significant crack propagation, the measured force drops are statistically distributed according to two distinct regimes. When failure is more diffuse (without significant crack propagation), a single statistical distribution regime was observed. The presence of two regimes in the distribution of force drops, a quantity easily measured

Further reading: Villette, F., et al. International Journal of Fracture 245.1 (2024): 25-35. Villette, F., et al. Acta Mechanica 234.9 (2023): 4197-4215. Fundings: ED IMEP2 in mechanical tests, is therefore a statistical signature of crack propagation.

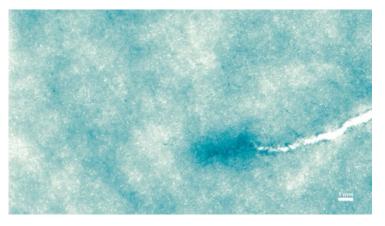


Figure: Optical observation of crack propagation and fracture process zone

The link between force drops and the size of the damage zone is also highlighted based on optical analysis during the mechanical tests. It therefore seems possible to obtain information on the damage zone based on statistical analysis of force drops, even for opaque materials in which crack propagation cannot be observed visually. Taken together, these results could lead to improved modelling of quasifragile materials, to better anticipate the risk of fracture.

#### **Contacts:**

julien.baroth@3sr-grenoble.fr frederic.dufour@3sr-grenoble.fr jean-francis.bloch@3sr-grenoble.fr sabine.rollandduroscoat@3sr-grenoble.fr

# A chair IUF focused on coupled process for the energy and the environment

Multiple environmental challenges are ultimately driven by multi-physical processes occurring at the microscale. This IUF project focuses on three particularly important ones: energy storage in reservoirs, fire safety in low carbonfootprint materials and carbon-neutral cementitious materials. Despite their disparate nature they share similar hydro-thermo-chemo-mechanical couplings. New understanding is made possible by 5D imaging, which allows a new generation of micro-inspired models to be built.

The 2023 Climate Change Report of the IPCC confirms the connection between human activities, greenhouse gas emissions and climate change. It details its adverse impact on food and water security, human health, and society. The report proposes a diversified strategy towards Net Zero emissions.

This project directly **tackles three of these critical environmental and energetic challenges**: the storage of energy into underground reservoirs, which relies on an improved understanding of hydro-mechanical couplings. The adoption of lower carbon footprint construction materials and their fire-safety, which is driven by thermo-hydro-mechanical couplings. The reduction of the carbon footprint of cementitious materials through the re-injection of CO2 in recycled aggregates, which is driven by chemo-thermo-hydromechanical couplings.

These three, diverse challenges share **key**, **highly heterogeneous thermo-hydro-chemo-mechanical couplings** occurring at the micro-scale. Novel understanding of these driving mechanisms is made possible by **5D imaging**: the simultaneous and synergic use of X-ray and neutron tomography (in 3D + time). This powerful approach has only now been made possible by the unique instrument NeXT, whose development I have spearheaded for the last years at the Institut Laue-Langevin, and which has recently undergone a major upgrade.

These micro-scale measurements are the missing brick to

build and improve the **micro-to-macro constitutive models**. They also underpin the development of multi-physics, **multi-scale "digital twins"**, a joint experimental/finite element modeling approach.

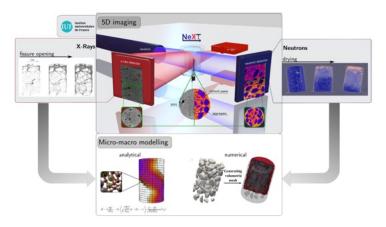


Figure: Schematic representation of the project.

Beyond these main applications: the approach developed to tackle these three challenges, and notably **5D imaging**, is **even directly applicable** to the study of other sustainable energy challenges and notably solid-state **lithium batteries** and **fuel cells**. This project relies on **truly unique** new data made possible by NeXT-Grenoble and takes advantage of them to create new micro-inspired analytical and numerical models which will be validated at multiple scales.

Further reading: IUF

#### **Contacts:**

alessandro.tengattini@univ-grenoble-alpes.fr Fundings: institut Universitaire de France

# «Neutron tomography analysis of permeability-enhancing additives in refractory castables»: a paper awarded the ACerS 2024 Rising Stars prize.

Understanding the drying mechanisms of refractory castables is crucial for several applications. These are often used in high-temperature industrial applications, for example to line furnaces, incinerators, and reactors due to their excellent heat resistance, thermal shock resistance, and structural integrity at elevated temperatures. One of the main issues they suffer from is indeed spalling upon first heating, which significantly affects their safety, and implementation cost.

Understanding their drying response allows for the optimization of the initial drying processes, and thus allows the minimisation of the risk of defects like cracking or spalling. A better understanding also makes it possible to develop more efficient and cost-effective drying additives, which are used to enhance permeability and mitigate the risk of explosive spalling, but whose exact driving mechanisms remains unclear. A deeper experimental understanding of these mechanisms is also crucial to improve our numerical models, which are essential for predicting the behavior of refractory castables during drying and service.

The research conducted at Laboratory 3SR in collaboration with ILL and Sao Carlos University (Brazil) investigated the effects of a variety of additives (polypropylene, PP, polyethylene, PE, and cellulose fibers) on water transport in refractory castables using rapid neutron tomography. This technique allowed for real-time visualization of water distribution, drying front advancement, and moisture accumulation patterns. The observations revealed that PE and cellulose fibers accelerate drying fronts compared to PP. However, PE exhibited larger moisture accumulation, residual moisture behind its drying front, and a slower drying rate at higher temperatures despite its early water removal initiation. In contrast, cellulose demonstrated superior performance due to a swelling-shrinkage mechanism.

Neutron tomography constitutes a unique tool to unravel the complex and dynamic influence of fibers on permeability

Further reading:

Understanding the drying mechanisms of refractory and lead us to a deeper understanding of these underlying cables is crucial for several applications. These are of- mechanisms.

For our contributions to this field, our research was awarded the ACerS (American Ceramic Society) « Rising Stars» prize as well as the cover of volume 187 of the Journal of the American Ceramic Society



Figure: Cover Photograph: Residual moisture in refractory castables can lead to explosive spalling. Understanding water transport mechanisms is key to improving the longevity of these critical manufacturing tools. In-situ visualization using neutron tomography shows both drying fronts (dark red) and internal water accumulation (dark blue) for a castable containing cellulose fibers.

#### **Contacts:**

alessandro.tengattini@3sr-grenoble.fr stefano.dalpont@3sr-grenoble.fr

Moreira, M. H., et al. Journal of the American Ceramic Society (2024).

# Bio-cementation of granular media: micro-mechanical characterisation of contacts to predict macroscopic properties

Bio-cementation is an eco-friendly alternative to traditional reinforcement techniques, using bacterial activity to precipitate calcium carbonate that forms solid bonds in the materials, increasing its strength. We have characterized the strength of these bonds that governed the efficiency of the reinforcement through micromechanical experiments and X-ray µtomography in synchrotron. These results were used in a discrete elements model to predict the behaviour of the material and compared to in-situ triaxial test.

Bio-cementation, also known as MICP (Microbially Induced Calcite Precipitation) is an alternative process for classical reinforcement techniques that tends to be more environmental-friendly than the ones based on the injection of synthetic products. It uses bacterial activity to reinforce cohesionless soils, fabricate construction materials, as well as preserve heritage and monuments. The bacterial activity triggers the precipitation of calcium carbonate crystals that create solid bonds in the material, increasing its strength.

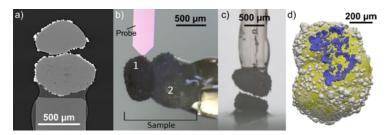


Figure: a) Cross section of two cemented grains glued on a capillary before mechanical test. b) Shear test. c) Traction test. d) 3D image obtained with X-ray micro-tomography of a sand grain (yellow), covered by calcite crystals (white and blue) after mechanical test. The blue crystals were identify as active before the test, that means there were bonding the two sand grains before the test, participating to the strength.

Previous studies have shown that only the presence of around 2-3% of crystals significantly enhance the mechanical properties of cohesionless sandy soils, and that the macroscopic mechanical behaviour depends on the

#### **Further reading:**

- 1. Dadda, A. Doctoral dissertation, Université Grenoble Alpes, 2017.
- 2. Sarkis, M., et al. Acta Geotechnica, vol. 19, pp. 1555–1570, 2024.
- 3. Sarkis, M., et al. Computers and Geotechnics, vol. 149, p. 104860, 2022.

micromechanical properties of the cemented contacts [1]. Thus, in order to be able to predict the mechanical behaviour of biocemented sand, we have performed a micromechanical study to characterize the mechanics of the biocemented contacts. Pairs of biocemented grains were characterized in tension [2] and shear using our new micromechanical machine Femtotools FT-MTA03. They were observed before the test using high-resolution synchrotron X-ray tomography in order to compute with accuracy the contact surface areas between the active crystals and the sand grains. After the tests, scanning electron microscopy coupled with energy dispersive X-ray spectroscopy were performed to identify the failure mode. The results show that the failure occurred at the interface between the calcite crystals and the sand grains, and could happen on both grains. Thanks to these experiments, the tensile and shear strengths of the biocemented bond were measured to be respectively equal to 2.76 ± 1.79 MPa and 5.81 ± 1.99 MPa on average. Then, these values were used in numerical simulations based on the discrete elements method, using the open source software YADE, to scale up and predict the macroscopic behavior of the material [3]. In parallel, triaxial tests were performed under X-ray tomography. Comparison between the simulations and triaxial tests showed good agreement, validating this approach.

This project continues by focusing on the durability of this reinforcement process, a key property for engineering applications.

#### **Contacts:**

antoine.naillon@3sr-grenoble.fr fabrice.emeriault@3sr-grenoble.fr christian.geindreau@3sr-grenoble.fr marilyn.sarkis@esrf.fr Fundings: IDEX UGA, ANR Micromechabio, PEPS CNRS and Soletanche Bachy

# Anchor pile for floating wind turbines under multidirectional cyclic loading

The aim of this project is to increase the knowledge on pile behaviour under lateral cyclic loading, including loads with changing direction, so as encountered for shared anchor piles for floating wind turbines. The experimental phase took place in the frame of the H2020 GEOLAB project. A medium-scale model pile, embedded in a dense sand mass in an indoor pit, was laterally loaded by actuators connected to the pile head. Different loading sequences were applied and the system's response was monitored using a range of sensors, including fibre optic sensing within the soil mass around the pile.

SAM-WT is the acronym for "Shared Anchor under Multidirectional cyclic loading for floating Wind Turbines". It is a GEOLAB transnational multi-partner project led by the 3SR Laboratory and hosted by the Geotechnical Institute of the Technical University of Darmstadt in Germany, where the experiments took place in an indoor test pit between April and June 2024.



Figure: Experimental set-up: pile head equipped with a twocylinder loading system.

Using open-ended steel piles is one of the solutions to anchoring Floating Offshore Wind Turbines (FOWTs) in the seabed via catenary lines. These piles must withstand complex loading conditions caused by the metocean environ-

#### **Further reading:**

ment. Moreover, the option of shared anchors is currently being considered as a solution for foundation optimisation, with each pile connected to several FOWTs, thus leading to the application of cyclic lateral loading on the piles, with significant change of direction. The prediction of the pile behaviour is crucial for future designs and for the analysis of the techno-economic interest of shared anchor. To understand the key parameters involved in the response of this geostructure and to collect relevant experimental data for numerical model development, a test campaign was conducted on an instrumented model pile of 325 mm in diameter and 2 m embedment depth in a dense sand mass, mimicking a real anchor pile at a reduced scale of approximately 1/5. A series of lateral cyclic loading packages was applied at the pile head, using a pair of actuators connected to the pile with a mechanical articulated system, specifically designed for this project. A range of sensors was used to measure the pile's response. Additionally, an innovative fibre optic sensing set-up was implemented to assess the deformations within the soil mass around the pile. The post-experiment analysis of the abundant data collected led to a comprehensive understanding of the soil-pile interaction behaviour and highlighted the effect of multidirectional versus monodirectional cyclic loading. These experimental results constitute a database for the development of advanced numerical models to perform more detailed parametric studies on the behaviour of piles subjected to multidirectional cyclic lateral loading.

#### **Contacts:**

orianne.jenck@univ-grenoble-alpes.fr christophe.dano@3sr-grenoble.fr **Fundings:** European Union's Horizon 2020 Research and Innovation Programme GEOLAB and ED IMEP2

Chalhoub R., et al. 5th Int. Symp. on Frontiers in Offshore Geotechnics, Nantes, France, 9-13 June 2025.
GEOLAB

The DéFI project "Dynamic Fragmentation of Ice": Development of a constitutive model to describe the fragmentation process in hydrogen ice by analogy with water ice.

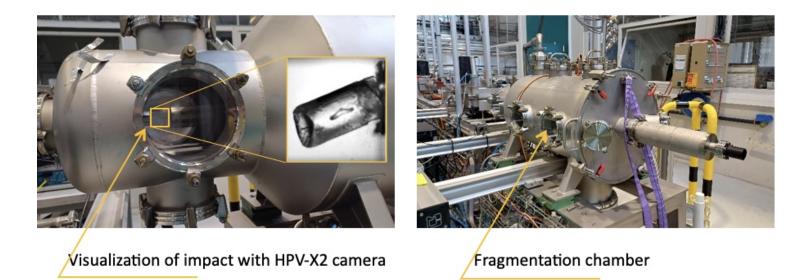


Figure: Cryogenic ice injection test bench for analyzing fragmentation processes using a Hopkinson bar impact test (IRIG/DSBT) instrumented with vibrometer (3SR) and high-speed cameras (3SR, IRIG/DSBT).

The phenomenon of disruption (plasma instability) in high-performance tokamaks such as ITER is a major concern. This risk can be minimized by injecting a cloud of millimeter and submillimeter fragments made of hydrogen, deuterium, neon, argon, etc... To do this, cylindrical hydrogen ice samples called 'pellets' are accelerated to a speed of several hundreds of m/s using a cryogenic chamber coupled to a gas launcher. Next, fragments are injected into the torus by impacting a wall or an inclined structure. However, given the temperature (of the order of 15K) and vacuum conditions to be achieved (10-6 mbar), the distribution of fragments generated by impact remains difficult to analyze and model.

It is the reason why, in the DéFI project, an approach based on the analogy between water ice and hydrogen ice was developed. First, dynamic mechanical tests under confined compression and dynamic tensile loading were carried out

**Contacts:** 

pascal.forquin@univ-grenoble-alpes.fr fabien.souris@cea.fr on water ice at 3SR.

Thanks to these tests a constitutive model describing the increase in strength with loading-rate and confinement pressure was tuned. Once implemented in a numerical code, this modelling was validated by a series of impact tests of water ice specimen impacting a Hopkinson bar instrumented with strain gauges and laser velocimetry (3SR). Secondly, impact tests of hydrogen ice pellet against a Hopkinson bar (Fig. 1) were carried out at CEA Grenoble (IRIG/DSBT). The fragmentation process was analyzed using a Shimadzu HPV-X2 (3SR) ultra-high-speed camera. These tests, which are still in progress, are used to refine the numerical modelling of dynamic fragmentation process considering different microstructures of hydrogen, deuterium, neon or argon ice. This work should lead to optimization of the fragments production process that will be injected into the tokamaks.

# Pressurized foams: understanding fatigue to extend service life

Do the soles of your favorite running shoes wear out and lose their effectiveness after just a few months of use? What if there was a way to make them last longer while maintaining their cushioning and comfort? A recent study took a closer look at the microstructures of the foams used in these shoes to understand how and why they degrade over time. By using advanced 3D imaging techniques, researchers uncovered fascinating details about the mechanisms behind the deterioration of these materials, raising hopes for more durable sports shoes in the future.

(winner of the regional "My Thesis in 180 Seconds" competition), conducted in collaboration with Decathlon Research Center (Dimitri Ferré Sentis), researchers from the 3SR Laboratory (Lucie Bailly, Sabine Rolland du Roscoat, Laurent Orgéas) investigated the micro-mechanisms of fatigue in closed-cell elastomeric foams used in running shoes. Their results reveal interesting insights into how these materials degrade over time and under cyclic loading, thereby providing valuable information for designing more durable sports footwear.



Figure: trail nunning shoe with a zoom on the 3D cellular structure of its midsole before (left) and after (right) running a 150km trail.

**Further reading:** 

Aimar, C., et al. Polymer Testing 128 (2023): 108194. Aimar, C., et al. Footwear science (2024): 1-11. Fundings: Décathlon

More specifically, as part of Clara Aimar's PhD thesis To achieve this, Clara developed continuous and interrupted cyclic compression tests on samples of ethylene-vinyl acetate (EVA) foams, a cellular material commonly used in the midsoles of running shoes due to its excellent energy absorption properties. Despite its widespread use, the degradation process of EVA foams under repeated stress remains poorly understood, especially with respect to the relationship between mechanical fatigue and changes at the cellular wall level.

> This methodology allowed her to demonstrate and understand how this structure evolves under loading, providing a clearer view of the mechanisms responsible for fatigue: plastic bending and partial tearing of the cellular walls. The mechanical properties of EVA foams continuously degrade during cycling, with progressive softening of the foam occurring primarily during the first 5,000 cycles. This rapid degradation is followed by a slower, steady decline, leading to irreversible loss of the initial desired functions of these materials. Clara showed that these changes were closely related to the initial microstructural defects observed, which became more pronounced as the number of cycles increased. Furthermore, Clara's work highlighted the importance of rest periods during fatigue testing of EVA foams. Samples subjected to interrupted cycles exhibited partial recovery of their shape and mechanical properties after each rest period. This recovery is attributed both to the viscoelastic nature of the foam and to the pressure of the gas trapped inside the cells. These results suggest that the ability of foams to partially recover, through a memory effect between cycles (or between running or training sessions of small to moderate intensity), is crucial for their long-term performance.

#### **Contacts:**

laurent.orgeas@3sr-grenoble.fr lucie.bailly@3sr-grenoble.fr sabine.rollandduroscoat@3sr-grenoble.fr The study aimed to test hypotheses by analyzing the bounces of modern balls on old floor tiles from various historical game rooms. Collaborating with archaeologists and historians, we found that floor types did not affect ball rebounds. However, ball characteristics significantly influenced the playing style and mindset of court games played by kings and possibly related games like tennis.

Some researchers in the GéoMécanique group focused on natural hazards and aimed to develop skills and tools for analyzing impact mechanisms [1]. Using an interdisciplinary approach, the research combined experimental archaeology, trajectology, ballistics, and sports science [2].

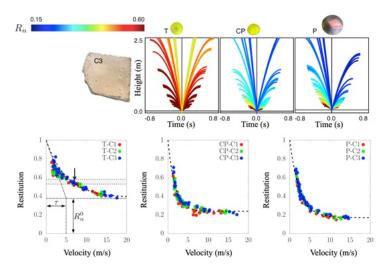


Figure: (Top) Bounce heights of the balls T, CP and P as a function of time for impacts with vertical trajectories on the horizontal tile C3. The curves are colored according to the value of normal energy restitution. (Bottom) Restitution versus normal impact velocity for each ball colliding each tile.

In a study of historical pavements, three floor tiles were tested: a 20 cm terracotta tile (C1) from Versailles, a 30 cm limestone tile (C2) from Louis XIII's game room at Versailles, and a 60 cm limestone slab (C3) from the Château Royal de Villers-Cotterêts. Three contemporary balls were used: a court-paume ball (CP), a pasaka ball (P), and a homologated tennis ball (T). The CP and P balls were crafted by skilled artisans, while the T ball was chosen for its standardized characteristics, being homologated by the IFT. Software was developed to analyze high-speed camera data from rebound

**Further reading:** 

tests, reconstructing ball trajectories before and after impact to identify restitution patterns.

The study examined how tile nature, size, and surface appearance affect play style by conducting direct impact tests for all ball/tile pairs at various energies. The top plots display bounce heights over time, with curves colored by restitution coefficient  $R_n$  values for easy differentiation. Pressurized T balls generally showed higher rebounds and better trajectory clustering than CP and P balls. The bottom plots show  $R_n$  against impact velocity for all pairs. At low velocities,  $R_n$  strongly depended on impact velocity, but approached a limit at higher velocities, more notably for CP and P balls. The trends were influenced by the ball type rather than the tile type.

The study revealed that the differences in bounce behavior of the balls are primarily attributable to their nature and manufacture, rather than the type of tile. The evolution of balls from a P wool ball to a more dynamic CP ball and then to a T pressurized ball has been the main factor influencing restitution. The P and CP balls have a high restitution only at low velocities, while the T ball maintains a high energy restitution over a wide range of speeds. This impacts the game itself, with players preferring slower balls for better control and predictability. The significant loss of deceleration of the P or CP ball at each impact makes the game less spectacular, which may explain the emphasis on volley play and returning balls before the first bounce during the Renaissance period. This is still evident in Basque pelota, where play before the rebound is presented as more lively and true to the spirit of the game. This study highlights the importance of considering multiple disciplines to understand how ancestors were playing. It allowed us to gain insights into the historical context of play and the role of technology in shaping the game.

#### **Contacts:**

Vincent.Richefeu@univ-grenoble-alpes.fr Pascal.Villard@univ-grenoble-alpes.fr

<sup>1.</sup> Richefeu, V. and Villard, P. Modeling gravity hazards from rockfalls to landslides. Elsevier (2016).

<sup>2.</sup> Dufour, J-Y, et al. "Archéologie et histoire des jeux de paume en France." (2024): 419-p.

# Laboratoire 3SR (UM5521)

Bâtiment Galilée 1270 rue de la piscine 38400 Saint Martin d'Hères - France



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