



# ANNUAL REPORT | 2023



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# Foreword

Polifab is the place where brilliant scientific ideas meet cutting-edge technologies. A definition borrowed from someone who knows well one of the top research labs of our university.

These are the words used by its Director a few years ago when Polifab entered the 2.0 phase with the expansion of the clean room, at the very core of its activities. When, together with STMicroelectronics, Politecnico di Milano gave birth to a leading center for studies and applications on advanced materials and MEMS.

The history of Polifab has deep roots firmly planted in the ground. Since July 3, 2015, the date of its opening alongside Pirelli, for almost a decade this "21st century workshop" has represented the contact point between advanced research and business as a central topic for the development of our country. As a key element for the growth of human capital and career advancement of young researchers. As an attractive value to increase investments, increase competitiveness and operate in an international perspective.

This description fits perfectly into the overall mission and vision of Politecnico di Milano: a university that is open and dynamic, that is based on long-term cooperation agreements with innovative companies, that looks to the future. In fact, our goal is to further focus on joint initiatives, on recruiting talent programs, on increasing spaces and modern equipment that let us compete with the best European research institutes.

Polifab's Annual Report matches the goals for the upcoming years, which are increasingly aimed at converging and multidisciplinary research areas, such as those represented by electronics and digital, micro and nano technologies, photonics and materials science. An essential step for a technical university that intends to anticipate change.



**Prof. Donatella Sciuto**  
Rector of Politecnico di Milano



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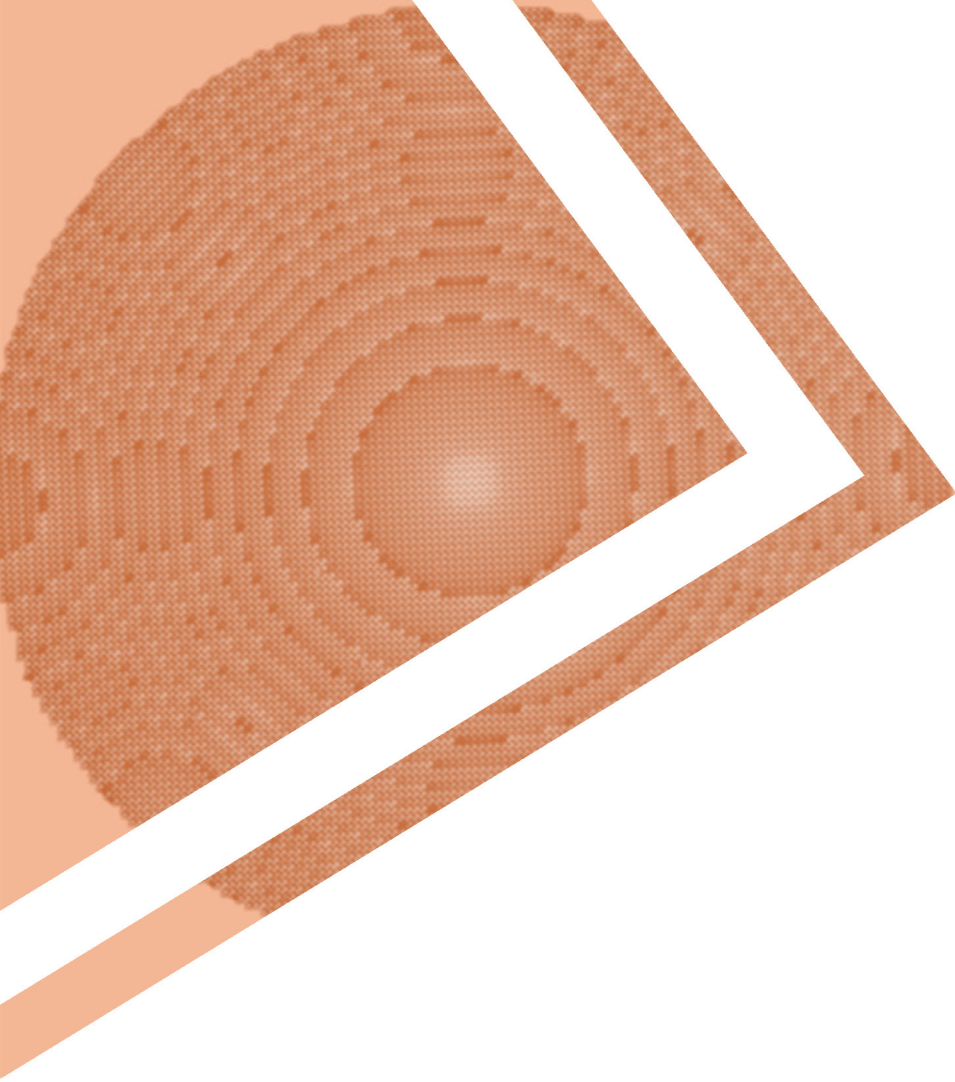
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# Introduction



## Director's Message

The 2023 has been a year of major growth in Polifab, in terms of tools, machines and processes, multidisciplinary skills and competencies, new relationships, new users in addition to the consolidation of collaborations, and new spaces and staff personnel. This is reflected in the major publications that have recently recognized Polifab as one of the enabling factors for the achieved results. The growth concerns the three pillars on which Polifab operates: fundamental research, applied research and education.

Thanks to the constant care and the discrete guidance of the highest Boards of the Politecnico, the Rector and her delegates, the General Director and the Director of Research, Innovation and Corporate Relations, Polifab is acquiring a solid reputation and a pivotal role in the internal, national, and international panorama. Our proactive Staff, always ready to intervene technically and participate scientifically, is the flywheel providing the necessary momentum to many projects, users, and everyday life.

2023 is a year that shaped the future technology capability of Polifab. Many new strategic tools and instruments have been acquired and will be installed soon, mainly thanks to the long-lasting collaboration with STMicroelectronics, our strategic partner operating through the Joint Research Program STEAM. These new tools, in addition to the renewal of some historic machines that were important workhorses in the first years of our adventure, cement Polifab's status as one of the best multidisciplinary academic micro- and nano- technological facilities in Italy and Europe.

2023 is a year that set the stage for our upcoming expansion into the "Parco dei Gasometri" in Bovisa, the Politecnico Campus in the northern part of Milano. This will move Polifab into a rich area of public and private research and innovation facilities, a veritable citadel of innovation, where the copresence with STMicroelectronics, Luxottica and other leading companies will allow a continuous exchange of knowledge and experience thus creating a fertile ecosystem of research, innovation and creativity.

Beside projects and collaborations with large companies, consolidated research groups, spin-off companies and Research Institutes, Polifab has a particular emphasis on the education and development of young researchers, who find in Polifab an opportunity to add an experimental flavour to their research and gain practice with the physical level, from materials science to prototyping devices. This year, for the second time, Polifab assigns a "Starting Grant" to support two groups of early-stage researchers waiving the cleanroom access fee for two years. As occurred for the previous grant, we hope to encourage and boost the passion of young researchers and PhD students and enrich their knowledge, preparing them to launch a brilliant research career. In return these researchers, as well as all the others working in our environment, leave as legacy to Polifab new processes, new materials, new ideas and applications that inspire each other and the new generations.

A micro- and nanotechnology centre with an ISO06/08-class clean room such as Polifab can imply a high environmental impact related to factors such as high electricity consumption, special waste management, and local heating induced by air handling units. Polifab's



growth is managed with a strong focus on sustainability and environmental impact. In 2023, the cleanroom lighting was replaced by LED lamps, reducing the thermal load on the thermalisation system; in the new part of the cleanroom, forced air recirculation for filtering and thermalisation is carried out with new ceiling-mounted Fan Filter Units, which allow great energy savings and the reduction of air expulsion; a high-efficiency refrigeration unit and two high-efficiency cascade heat exchangers have been installed. These interventions are estimated to have reduced the specific energy consumption related to the cleanroom by a factor of 2.5.

2023 has also seen an increase of the Academic courses that exploit Polifab for the education of students at both the Laurea Triennale and Magistrale levels on micro- and nano-technologies. More than 150 students per year gain exposure to our research facility through lab Experiences, specific courses on fabrication, and periodic visits. It is amazing seeing the reaction of young students in Computer Science when they perceive the physical structures that are behind their software code! I'm glad to see the increasing educational value of Polifab for students at the Politecnico.

The research groups that take advantage of the Polifab facilities are outstanding and produce first-class research. Looking through the list of publications, I am struck by the broad range of fields we contribute to, and it is rewarding to see our contributions to journals such as Nature and Science and others setting the new state of the art in their respective fields.

This second Polifab Annual Report will be distributed during another traditional event, the Polifab User Meeting. It is our tradition to have a time of sharing where our Users community can meet and discuss outside the cleanroom and consider the vision of speakers with prominent international role. We would like to stimulate even more interaction among the groups that bring our clean room facilities to life, but also open the doors to new researchers interested in our facility. The User meeting is an ideal situation to present the perspectives of Polifab to our actual Users but also to researchers interested in the new enabling technologies that will be available at Polifab in the coming years.

I wish all the Users, the Polifab Staff, the Departments, collaborators, and the entire Politecnico a fruitful, gratifying, and pleasant 2024, rich with professional successes and personal growth, looking to the future with positivity and optimism.



**Prof. Andrea Melloni**  
Director of Polifab



# Mission & Vision

The main mission of Polifab is to provide technology infrastructure, high-tech tools and know-how to support research from proof-of-concept in materials science and devices to rapid prototyping for industrial applications.

We envision Polifab as an aggregation center for scientists from universities, research centers and industries, promoting world-leading interdisciplinary research and education, as well as the development and transfer of key enabling technologies.

The activities are well balanced between fundamental research, applied research and university education to create the substrate of multidisciplinary knowledge that can drive technology transfer from the university to companies, research centers, start-ups and universities.

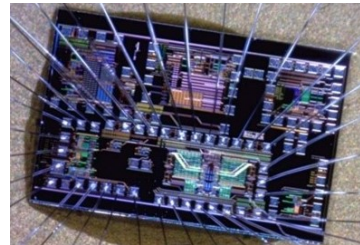
## Foundamental Research

Basic, fundamental research and material science for low TRL activities take advantage from the large number of tools for deposition of a large class of materials and characterization. Most of the tools are the capable of operating from small samples to 8" wafers.



## Applied Research

Some Users and typically Start-ups and companies aim for a higher TRL processes and access Polifab either independently or taking advantage of the Service provided by the Polifab Staff. The backend facility will be further enriched in a short future with processes compatible with industrial needs.



## Education

Polifab supports 3 courses for Laurea Magistrale of Politecnico with hands-on activities. This year more than 120 students from PoliMi courses and other institutions visited Polifab. Specific Training on processes and machines are regularly organized and given by Polifab Staff for our Users. These courses authorize the User to access independently to tools and processes.



## Polifab at a Glance

Polifab is an infrastructure created at Politecnico di Milano in 2015 and dedicated to micro and nanotechnology.

Polifab provides the highest technological standards for different fields of research and applications including photonics, micro and nanoelectronics, spintronics, MEMS, biotechnology, advanced materials and nanotechnology in general.

Since its establishment, it has well-defined access policies in terms of users and fees, training on equipment and safety. Master's and Ph.D. students, researchers, faculty and even personnel from companies can enter the cleanroom at Polifab and operate the tools under the guidance of our Staff.

This access model attracts talented researchers and students, satisfying their needs and passion for technology and is creating a new generation of young researchers who can give an innovative impetus to micro and nanotechnology in the region.

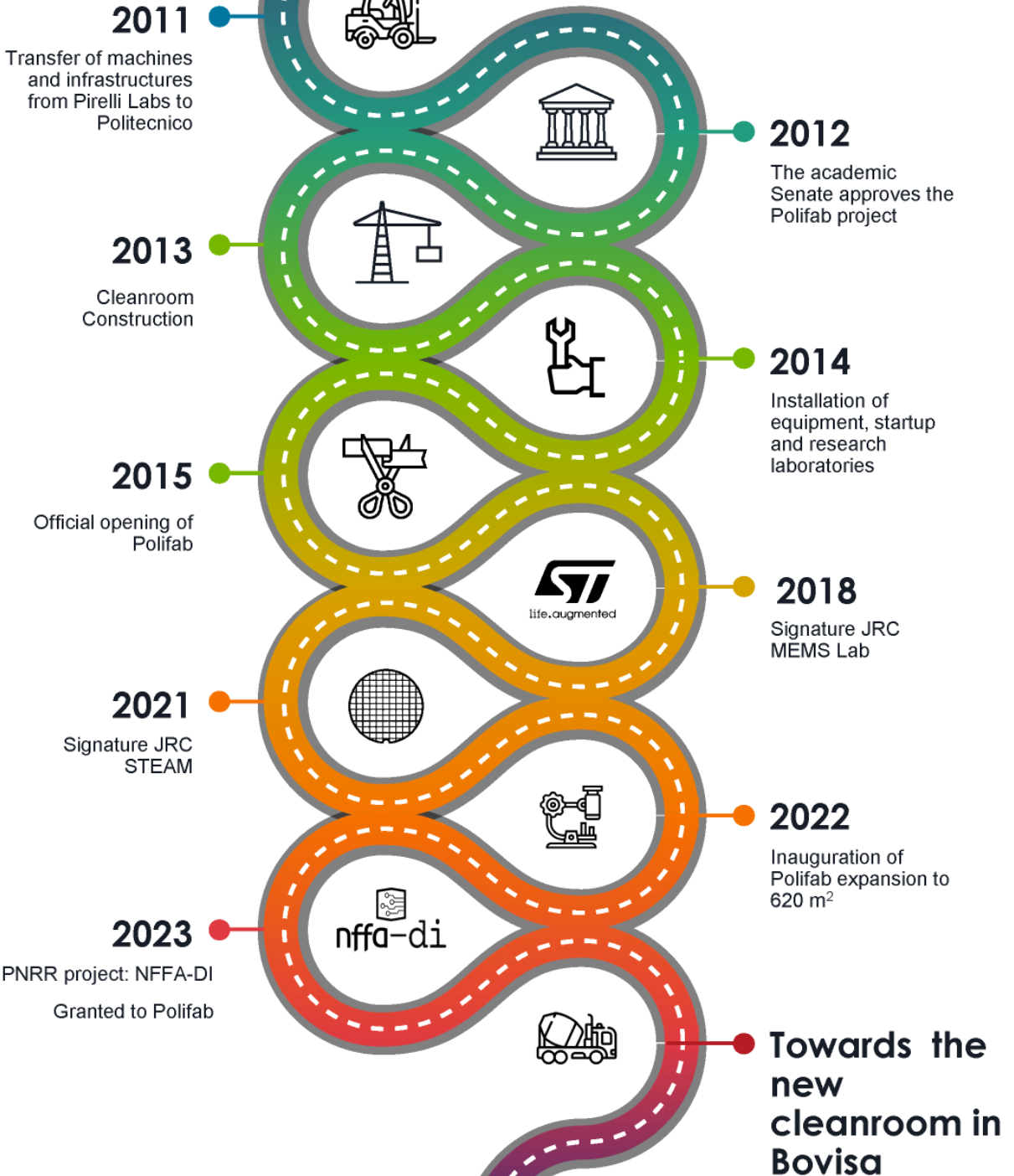
At the same time Polifab can also work with external partners, acting as a pure third party.

Around 30 research groups in different areas operate in Polifab with about 40 basic and industrial research projects and 6 ERC, generating outstanding scientific results in an international environment. In 2023 alone, more than 30 papers with results obtained in Polifab were published, some in journals with high impact factor, while we count more than 200 papers since 2015.

The list of available equipment, which is constantly enriched, is detailed on our website and ranges from lithography to thin film deposition, from metrology to back-end. We can process samples from pieces to 8" wafers and several times during the last few years we have been working with companies setting up, in our facility, processes compatible with industrial needs. In 2023 several new tools have been installed in our cleanroom, improving our capabilities: a four-chambers cluster for sputtering, an advanced system for high resolution optical lithography, dedicated tools from spin coating and development, a mapping system for film thickness measurement and a probe station for electrical characterization that can operate both at cryogenic temperatures and in high vacuum. Next year 2024 will be quite crucial, as various important tools will become available, further improving the number and the quality of our processes.

Our community of users grows in synergy and contributes to the enrichment of the portfolio of available materials and technological processes. Polifab has become a reference facility for micro and nanofabrication in Italy with an expanding user community and a growing interest from high-tech industries such as STMicroelectronics, Technoprobe, Huawei, Luxottica, and many others (<https://www.polifab.polimi.it/partners>).

# Polifab Timeline

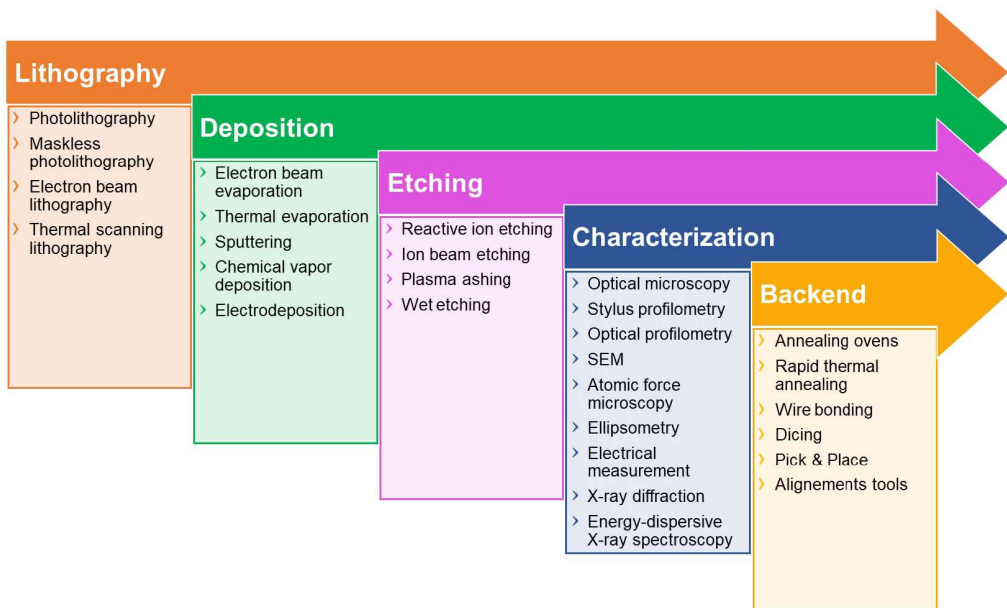


# Cleanroom Equipment

Polifab is based on a 620m<sup>2</sup> cleanroom (330m<sup>2</sup> ISO06 and 290m<sup>2</sup> ISO08) plus annexed laboratories for materials and device characterisation. The core of Polifab is a pilot line for micro- and nano-fabrication capable of processing different formats, from small samples to 200 mm wafers. It includes facilities for:

- lithography (mask aligner, laser writer, e-beam lithography, thermal scanning probe lithography)
- thin films deposition (e-beam and thermal evaporation, PECVD, sputtering, electroplating, MBE and PLD)
- direct printing (ink aerosol jet printer)
- etching (wet, RIE, IBE)
- metrology (stylus and optical profilometer, SEM+EDX, AFM, probe station, spectroscopic ellipsometry, XRD)
- back-end (thermal treatments, dicing saw, ball bonder, automatic alignment, packaging machines).

More information on our facility and equipment can be found on our recently renewed website: <https://www.polifab.polimi.it/cleanroom>.



## New Installations

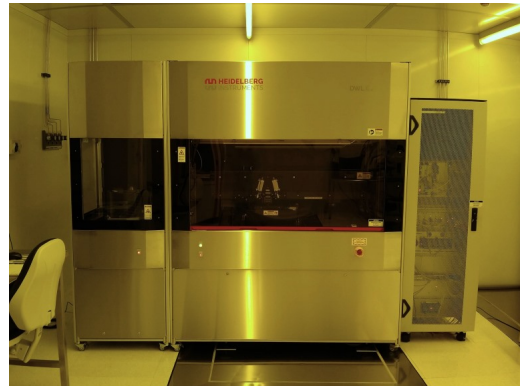
### Evatec Clusterline® 200E

A cluster tool designed for the deposition of thin films using magnetron sputtering on 200 mm SEMI standard silicon substrates. It allows for soft etch processes, growth of metal layers, and two of its chambers are dedicated to research and development of ceramic piezoelectric thin films in the framework of the Joint Research Center with STMicroelectronics.



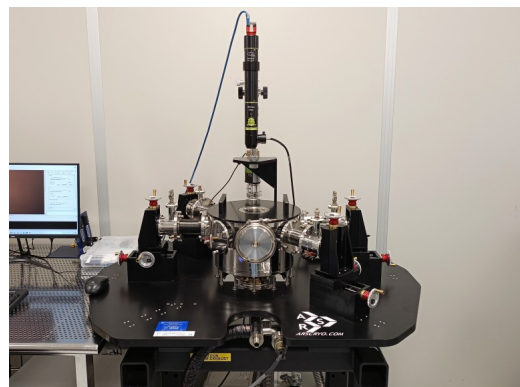
### Heidelberg Instruments DWL 66+

A highly versatile, high-resolution pattern generator for direct writing lithography and low-volume mask making. It features different switchable Write Modes – minimum feature size of  $0.3\ \mu\text{m}$  – as well as backside alignment (BSA), optical autofocus, vector exposure and gray-scale exposure mode for complex 2.5D structures in thick photoresist. It features an automatic loading system for handling of masks up to 7" and wafers up to 8".



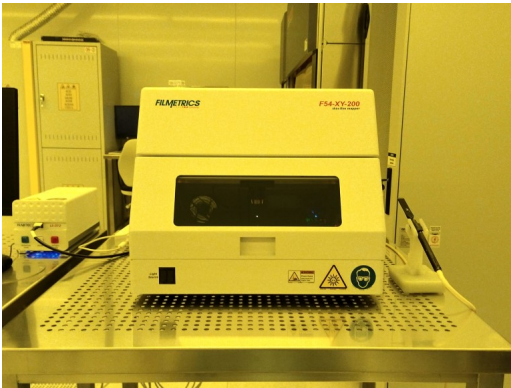
### ARS PS-L Flow Cryostat Probe Station

An electrical four-probe measurement tool with vacuum, cryogenic and heating capabilities. It can be used with liquid Helium or liquid Nitrogen. The vacuum chamber is made out of welded stainless steel and the radiation shield is made out of nickel-plated OFHC copper. Its applications are MEMS, Nanoscale Electronics, Superconductivity, Quantum Dots/Wires, and non-destructive device testing in general.





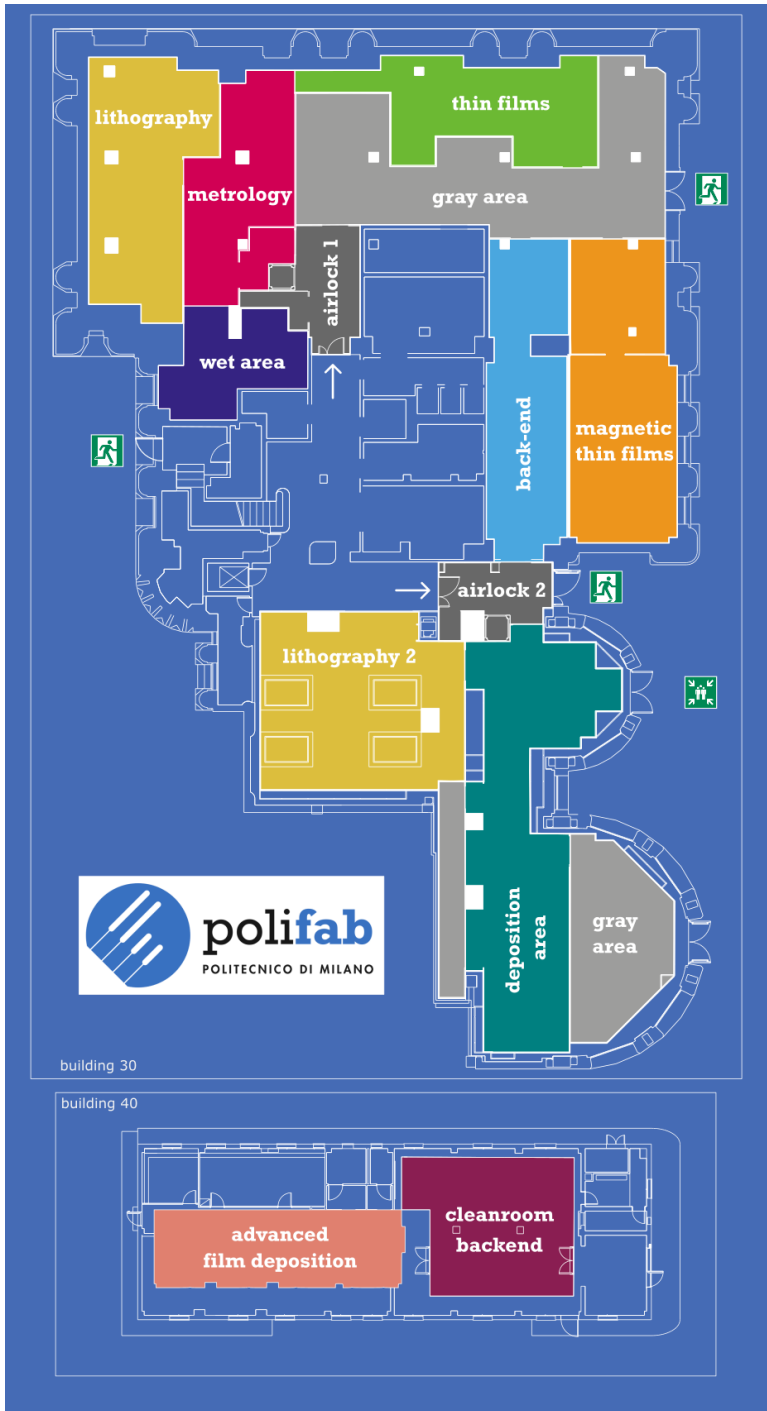
**SAWATEC SM-200/HP-200duo & SMD-200**  
The Spin Module SM-200 allows for semi-automatic coating of thin layers, with automatic dispensing of different media and manual loading/unloading. The HP-200 hotplate is suited for soft and hard bake processes in lithography, MEMS and similar applications, its temperature ranging up to 250 °C. The SMD-200 is designed to clean and to develop wafers up to 8" (200mm) or substrates up to 6"x6".



**Filmetrics F54-XY-200-UVX**  
An automated thin-film thickness mapping system, measuring thin-film thickness between 4 nm and 115 μm in different specified locations on samples up to 200 mm by 200 mm. It features a motorized X-Y stage for automated positioning and an integrated microscope with live video camera for exact monitoring of the measurement spot for patterned samples and rougher materials.



# Cleanroom Map





## Scientific Board



Director  
**Andrea Melloni**  
Dept. of Electronics,  
Information and  
Bioengineering



Deputy Director  
**Giovanni Isella**  
Dept. of Physics

## Scientific Committee



**Marco Boccione**  
Director of the Dept. of  
Mechanical Engineer



**Luca Magagnin**  
Dept. of Chemistry, Materials  
and Chemical Engineering



**Stefano Mariani**  
Dept. of Civil and  
Environmental Engineer



**Laura Castoldi**  
External member  
ST Microelectronics



**Giorgio Rossi**  
External member  
IOM-CNR, UniMi

# Polifab Team



Cleanroom Manager  
**Claudio Somaschini**



Technologist  
**Marco Asa**



Technologist  
**Andrea Scaccabarozzi**



Backend Processist  
**Stefano Bigoni**



Technician  
**Stefano Fasoli**



Process Engineer  
**Chiara Nava**



Process Engineer  
**Elisa Sogne**



Process Engineer  
**Gianluca Cannetti**



Technologist  
**Matteo Villa**

Administration  
**Giuseppina Maggioni**

## Team Talk: Conversation with our Staff

Dive into Polifab's cleanroom with the team! Explore access, inside work, and collaborations. First, let's talk to Chiara Nava, a Physics Engineering graduate from Politecnico di Milano and a member of the Polifab team since 2021, specialising in optical lithography.

**Q1: Dear Chiara, what makes the case of Polifab interesting and unique is the fact that among the different users we find many young researchers, including those in their first laboratory experience, master's students, and PhDs. What is the standard access procedure for these kinds of users?**

**Chiara:** Yes, a significant percentage of our users are indeed represented by undergraduate students who access Polifab to carry out research work for their master's thesis, and by PhD students who use the cleanroom to fabricate devices within the framework of their doctoral projects. Typically, these are users who do not have prior cleanroom experience, so the Polifab Staff guides them through all the necessary steps until they can work independently. We begin with a general introduction on how to work in a cleanroom, placing a strong focus on safety aspects. Users must pass a safety test before entering the lab, and then they also have to be trained by the Staff on every tool needed for their research.

**Q2: How does the Staff support the users accessing the lab for their research?**

**Chiara:** Following the initial general training and specific sessions on different tools, new users are allowed to work independently. However, especially for those who are just starting their activity, we also provide day-by-day support in cleanroom. Our users are aware that the Staff is always present in the lab to help them solve problems they may encounter with tools or processes. Additionally, we organize a coaching activity: a Staff member is assigned to each research group and acts as a technology consultant, offering advice and suggestions to guide users toward achieving their research goals.

**Q3: What are the challenges and advantages of an "open infrastructure" like Polifab?**

**Chiara:** Working in a cleanroom is not easy and requires a lot of attention, along with special care due to environmental and safety risks. This awareness does not develop quickly, and until the process is complete, users need to be carefully guided. At the same time, having an open infrastructure makes it easy for people from different fields to access the world of technologies, fostering the contamination of knowledge, new ideas and making the cleanroom a vibrant and exciting experience.

Marco Asa, Ph.D. in Physics from Politecnico di Milano, has been part of the PoliFAB team since 2018, specializing in thin film deposition and surface characterization. Let's have a chat with him.

**Q1: Dear Marco, several external institutions collaborate with Polifab; what is the typical customer of Polifab?**

**Marco:** In recent years, we have witnessed a continuous growth in the number of external customers collaborating with Polifab. In addition to our longstanding collaboration with ST Microelectronics and close relationship with external research institutions such as IIT and CNR, we also engage with various startups. This is not surprising, as these users often lack their own technological facilities but require one to demonstrate the feasibility of their ideas. Furthermore, we collaborate with many small and medium-sized enterprises seeking to test new ideas or concepts in a dedicated research environment outside their own company.

**Q2: Polifab offers access to the cleanroom but can also work as a pure third-party partner. What is the most common approach with external customers?**

**Marco:** The approach depends largely on the type of customer. For research institutions, we strongly encourage researcher to access the lab, undergo training, and work independently on their projects —this also applies to startups. In the case of the companies, however, we are typically requested to collaborate as a third-party partner. In this scenario, the Staff takes care of the work in the cleanroom, and we then share the results with the customer.

**Q3: Which are the main differences between working with research and industrial partners?**

**Marco:** Polifab provides support to both research institutions and private companies. When supporting academic researchers, we often deal with cutting-edge projects at the forefront of the state of the art. This means that frequently, we have to invent new methods, techniques, and push the technology to its limits. With companies, however, the focus is more on the reliability of the processes, the timeliness of the responses, and the flexibility of in-house characterization techniques. Both worlds are extremely stimulating, and we always strive to extract the best from both approaches.

## Polifab - ST: a unique opportunity for MEMS R&D

The Polifab represents an ambitious R&D commitment for ST in partnership with the Politecnico di Milano. It provides users with a MEMS R&D cleanroom, tools and resources that can perform rapid prototyping, exploration of new materials and recipes and ultimately create innovative products which can be scaled to volumes by ST as an industrial partner.

At the same time, it provides students with a unique opportunity to approach the fascinating world of MEMS through a hands-on experience, together with professors from the Politecnico and R&D engineers from ST, a world leader in MEMS. This is particularly important, since the Politecnico has launched in 2023 with ST's contribution a new Masters course in "Micro and Nano Systems", accessible to students from the departments of Mechanical Engineering and Physics.

During the course of the year 2023, several important milestones have been reached, among them the exploration of a lead-free material piezo material "KNN" of relevance in terms of environmental sustainability, as well as other activities aimed at exploring new recipes and materials for innovative MEMS devices.

Also, during 2023, several new tools were installed, expanding further the capabilities of the MEMS line. Another important milestone was the groundbreaking ceremony of the new Bovisa campus of the Politecnico, the venue to which the Polifab will relocate upon completion of the new campus in 2026.

Our collaboration with the Politecnico of Milano remains a cornerstone of ST's global MEMS R&D strategy. It represents a unique opportunity for ST and the Politecnico, but is also open to 3<sup>rd</sup> parties, with a need for innovative sensor or actuator products, who are looking for partners to help them develop such devices.



**Anton Hofmeister**

STMicroelectronics | AMS Group | Group Vice  
President – General Manager R&D and Strategy



## Polifab: dynamic intersection of industry and research

The Politecnico di Milano has in its DNA a natural inclination towards relations with local companies, institutions and other research centres. The University has undertaken this commitment since its establishment, which is demonstrated by its ability to attract major investments from national and international companies through **strategic partnerships**. The aim of these partnerships is to foster wide-ranging **interdisciplinary** research on research, innovation and development issues, with both a high experimental and innovative content of common interest and substantial and long-term investments. In short, it is an **investment for the future**.

These agreements are not made overnight and do not necessarily start from a specific scientific problem; rather, they involve sharing strategic objectives, willingly and jointly tackling **transformations that have an impact on everyday life**, acknowledging a limit and - at the same time - an opportunity: complexity requires us to work together to overcome the technological challenges distinguishing the current era by pooling the expertise of universities and businesses alike. In a nutshell, this is **the culture that has developed around Politecnico di Milano's** innovation ecosystem.

It is from this very culture that the exemplary experience of Polifab, a long-standing collaboration with STMicroelectronics, emerges. Joint labs are the most concrete and valuable spin-off from this perspective. We 'build' them and maintain them over time, together with enlightened companies that make substantial, long-term investments, testifying to the symbiotic relationship **between the academic and industrial worlds**, with clear benefits for both. Not only is Polifab a laboratory of Politecnico di Milano, but it is also available to companies and research institutes throughout the territory. It is an integral and key part of a network that brings together researchers, funding companies and 'client' companies, a synergy that in turn shapes the perfect ecosystem where new ideas can develop. The connection with our lively academic environment has 'side' effects of no small importance for the industrial fabric, bringing our academic partners in direct contact with the Politecnico's expert researchers and high-quality students working in the cleanroom, who are drivers of innovation and cultural development. On the other hand, our exposure to the industrial environment and proximity to the vibrant Lombardy region has positive repercussions on education and the development of an applied research approach typical of the Politecnico world.

There is one further thing I'd like to stress. Today, Politecnico di Milano tops the rankings of Italian and international universities for its ability to **attract research funding from the European Commission**. This is especially true for basic research geared towards scientific excellence. One of the important criteria for accessing these resources is the possibility of relying on research laboratories. The large University laboratories, such as Polifab, are thus not only indispensable facilities for many researchers, but also an asset allowing us to work better and better and to promptly respond to calls from the European Commission.

It is, in essence, a meeting place: where the worlds of technology and research, business and institutions converge to forge and grow ideas and people, with a positive impact for all.

**Federico Colombo**

Director of Research, Innovation and Corporate Relations Area

## Partners

In the last year we collaborated with the following companies, start-ups and universities:





# Users contributions



# Thermal Phase Shifters for Femtosecond-Laser-Written Universal Photonic Processors

M. Gardina<sup>2</sup>, A. Rajan<sup>3</sup>, A. Caime<sup>3</sup>, F. Ceccarelli<sup>1,2</sup>, and R. Osellame<sup>1,2</sup>

<sup>1</sup>*Istituto di Fotonica e Nanotecnologie (CNR), Piazza Leonardo da Vinci 32, 20133 Milano, Italy*

<sup>2</sup>*Ephos S.r.l., Piazza Vetra 17, 20123 Milano, Italy*

<sup>3</sup>*Dipartimento di Fisica – Politecnico di Milano, Piazza Leonardo da Vinci 32, 20133 Milano, Italy*

Programmable photonic integrated circuits (PICs) are widely employed for classical communications and today have become a leading platform also for optical quantum information processing and computation. Among programmable PICs, universal photonic processors (UPPs) are of particular interest because they can implement any unitary transformation among  $N$  input/output optical signals. UPPs are typically implemented by a mesh of programmable Mach-Zehnder interferometer (MZI) cells, in which reconfigurability can be achieved by means of thermal phase shifters, i.e. resistive micro-heaters able to locally tune the optical properties of the circuit by exploiting the thermo-optic effect.

Among the many technological platforms available nowadays, femtosecond laser writing (FLW) on glass substrates displays excellent features for quantum applications. Despite the plethora of advantages, the complexity of FLW-UPPs is limited to six optical modes by the phase shifter fabrication technology<sup>1</sup>, based on the laser ablation of a single metal film, shared between micro-heaters and interconnections.

A novel photolithography-based phase shifter fabrication technology, recently developed and demonstrated on single MZI cells<sup>2</sup>, has been purposely optimized for the fabrication of FLW-UPP with higher number of modes.

As a matter of fact, the integration density of FLW-UPPs can be increased by using chromium, highly resistive and thermally stable, for micro-heaters and copper, highly conductive and compatible with wire-bonding, for interconnections. The use of two metals requires a photolithographic approach, made possible thanks to the

maskless aligner available in the clean room facility of PoliFAB.

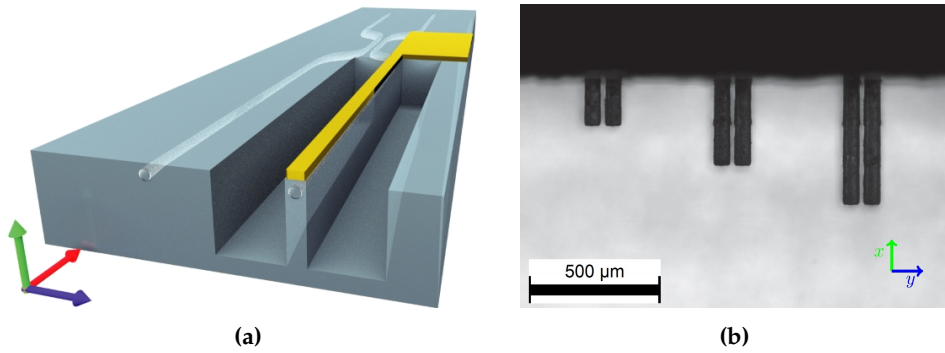
Chromium is first evaporated with an e-beam evaporator. A dry-film photoresist, which provides a uniform coverage on thermal isolation structures (Figure 1a), is patterned via a maskless aligner. Chromium is therefore etched with a commercial acid solution. The copper film is evaporated along with a thin titanium adhesion layer. After the patterning of a new photoresist mask, copper is etched with a custom solution of hydrochloric acid and hydrogen peroxide, whereas titanium is etched with buffered oxide etch (Figure 1b). The process is completed with a vacuum annealing at 400 °C for one hour in order to prevent the restructuring of the chromium film happen during operation and to enhance the contact between metals. Metal oxidation, and the consequent resistance upward drift, is prevented with a silicon nitride passivation layer, deposited by PECVD. The packaging finalizes the fabrication process (Figure 2).

The fine optimization of this fabrication process allowed the achievement, along with a monolithic generator of Greenberger-Horne-Zeilinger (GHZ) states, of an 8-mode UPP, featuring 112 micro-heaters within a 83 mm long chip, state of the art in FLW. The final experimental characterization showed an average fidelity of 98.650% over 100 random Haar matrices. The processor is now part of a quantum sampling machine deployed in Rome at Università Sapienza.

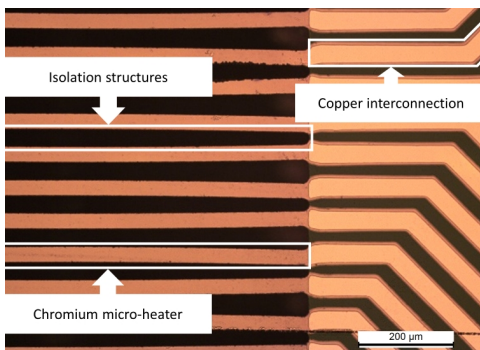
This result, combined with the fine optimization of the process resolution – down to the nominal photoresist one – enables the use of compact configurations, thus opening the perspective of a 20-mode UPP within a 12 cm chip.

<sup>1</sup> C. Pentangelo *et al.*, High-fidelity and polarization insensitive universal photonic processors fabricated by femtosecond laser writing arXiv, 2310.19718 (2023)

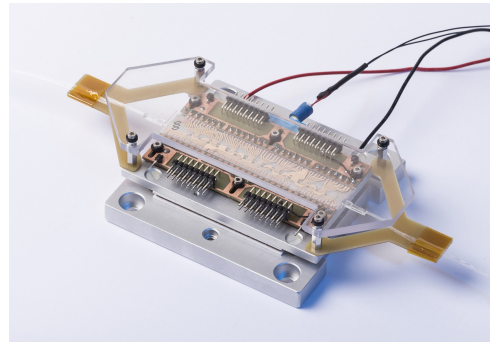
<sup>2</sup> R. Albiero *et al.*, Toward higher integration density in femtosecond-laser-written programmable photonic circuits Micromachines, 13(7), 1145 (2022)



**Figure 1:** (a) Scheme of the trench isolation structures. Waveguides are isolated on the sides; (b) Cross-section microscope image of the trench isolation structure, fabricated by femtosecond laser micromachining.



**Figure 2:** Chromium micro-heaters and copper interconnections patterned on curved isolation structures. Micro-heaters are fabricated on each MZI arm, the photo represents a portion of a column of a 20-mode device.



**Figure 3:** Packaged 8-mode UPP. The chip is mounted on an aluminum chip-holder between electrical PCBs. A Peltier cell is sandwiched between the chip-holder and a heat sink for temperature stabilization. Input/output fiber pigtailed are sustained by Y-shaped supports.

# The Biophysical Properties of Breast Cancer Circulating Tumor Cells and Clusters as a Proxy of Disease State and Therapy Response

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Metastasis begins with the invasion of primary tumor cells into the surrounding tissues, subsequent intravasation of tumor cells into the circulation followed by arrest or traverse of cells through capillary structures where they extravasate and form metastases.<sup>1</sup> Primary solid tumors, particularly in the case of carcinoma, are composed of collective units and clusters of tumor cells, which experience confined environments and compressive stresses generated by the growing tumor. These stresses can lead to intravasation favoring collective cancer invasion over single cell invasion, as the cancer cells expand into the surrounding extracellular environment. High deformability for both the cell and its nucleus offers significant advantages for tumor cell dissemination through micron sized matrix pores and endothelial vessel walls.<sup>2</sup> Collective modes of migration have been linked with increased E-cadherin cell-cell adhesions, clustering of cancer cells and heightened proliferation capacity.

Intravasation into the bloodstream can be assisted by immune cells and lead to the formation of circulating tumor cells (CTCs) or CTC clusters. Clusters may originate from the primary tumor, or consist of single cells that divide and aggregate during metastasis. This aggregation and division is unlikely to occur in the circulation, but rather in host niche environments or within narrow vessels. Clusters are not simply aggregated tumor cells, but are often composed of stromal cells (fibroblasts), endothelial cells and myeloid cells.<sup>2</sup>

CTC clusters display increased metastatic po-

tential and worse patient prognosis but are rare, difficult to count, and poorly characterized biophysically. The circulating clusters are usually small (2-50 cells) but they possess up to 50-fold increased metastatic potential compared to individual CTCs.<sup>3</sup>

In this study, we have developed a microfluidic device that captures single CTCs and CTC clusters according to their size, deformability and epitope expression level.

Cancer cells are labeled with magnetic nanoparticles conjugated to epithelial cell adhesion molecule (EpcAM), and then introduced into the device at a constant flow rate. Micropillars of varying gap dimensions capture clusters and larger single cells according to their size (diameters ranging from 10-200  $\mu\text{m}$ ) and deformability. In the last two zones, an external magnet is placed under the device and CTCs and small clusters are captured due to a combination of size and magnetic forces. The number of nanoparticles bound to the CTC is directly proportional to the levels of EpcAM.

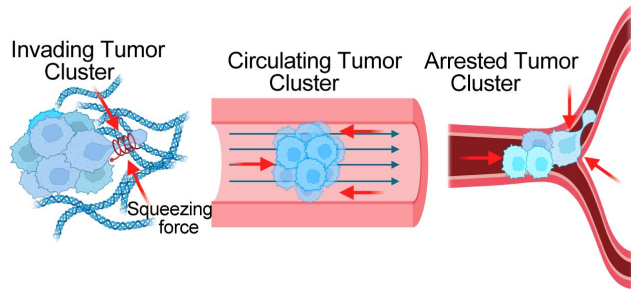
CTCs and clusters are profiled in the device using blood samples collected from metastatic breast cancer patients. The patients have histological diagnosis of invasive ER<sup>+</sup> / PR<sup>+</sup> / HER2<sup>-</sup> breast cancer.

Using the microdevice combined with confocal imaging, we identified an average of 26 single CTCs and 3 CTC clusters per ml of whole peripheral blood. This validation represents a proof of principle that could be applied towards tumor diagnosis and treatment monitoring.

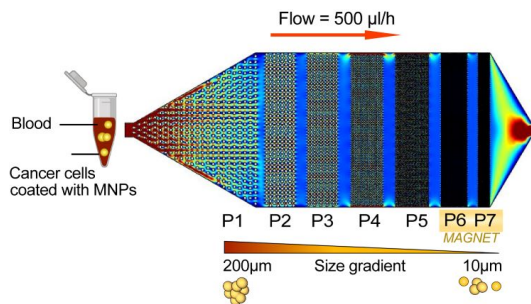
<sup>1</sup> C. Alix-Panabieres *et al.*, Circulating tumor cells: liquid biopsy of cancer. *Clin Chem*, 59 (1), 110-8, (2013)

<sup>2</sup> N. Aceto *et al.*, Circulating tumor cell clusters are oligoclonal precursors of breast cancer metastasis. *Cell*, 158 (5), 1110-1122, (2014)

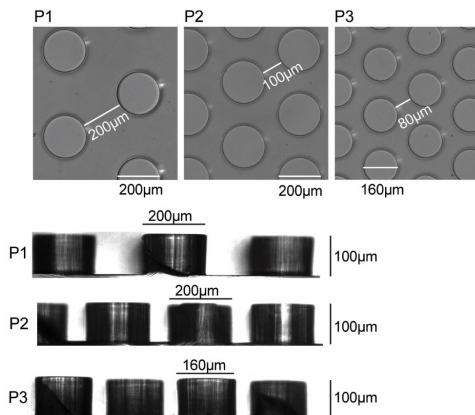
<sup>3</sup> S.H. Au *et al.*, Clusters of circulating tumor cells traverse capillary-sized vessels. *Proc Natl Acad Sci USA*, 113 (18), 4947-52, (2016)



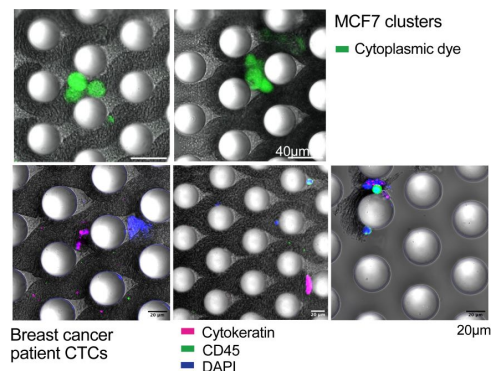
**Figure 1:** Intravasation of circulating tumor cell clusters into blood vessels depends on biophysical properties such as size and deformability.



**Figure 2:** Consol fluid modeling of the microfluidic device. Whole blood is introduced into the device at a constant flow rate. Single CTCs and clusters are captured in the fluidic channels according to their size, deformability and EpCAM levels.



**Figure 3:** Representative microfluidic pillar dimensions. Top view and side view are shown.



**Figure 4:** Confocal images of fluorescent MCF7 breast cancer clusters and patient-derived breast cancer CTCs captured in the microfluidic device.

# Bio-reconfigurable Differential Impedance Electronic Platform for High Sensitivity Multiplex Detection of Viral Respiratory Pathogens

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Respiratory viruses such as coronaviruses are unfortunately taking the stage of public concern. In this light, it is becoming essential to revamp current diagnostic approaches for these diseases by using novel technological strategies, merging the most sophisticated biochemistry recognition mechanisms into an advanced electronic miniaturized platform. The detection sensitivity of our platform has demonstrated unprecedented levels, reaching down to a few single biomolecules<sup>1</sup> resolution.

With this objective in mind, at PoliFAB we are working on the development of a biosensor system that relies on the impedance changes among microelectrodes when capturing the complete target pathogen, using either virus-specific antibodies or specific oligonucleotide DNA sequences. The biosensor core, made of a borosilicate chip with microelectrodes (shown in Figure 3), is integrated into a microfluidic path and electronically accessed to perform multichannel impedance detection. Multiple sensing sites in parallel will be addressed, first targeting DNA sequences (as shown in Figure 1 and Figure 2) and later detecting specific antigen-antibody binding. Real-time comparative analysis on different targets from a single clinical sample will be performed, innovating the current multiplexing diagnostic tools state of art.

The interdigitated gold microelectrodes have fingers of 3  $\mu\text{m}$  width, 90  $\mu\text{m}$  length, and 3  $\mu\text{m}$  spacing. Dimensions were optimized using numerical simulations performed in COMSOL Mul-

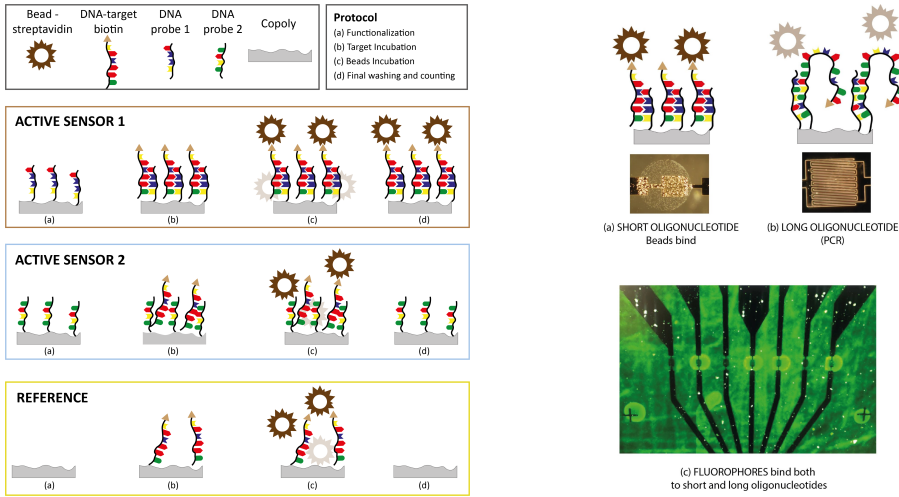
tiphysics, maximizing the impedance variation due to a single bead binding (800 nm diameter). The overall area of the microelectrodes is  $90 \times 90 \mu\text{m}^2$ , matching the minimum spotting size achievable by the functionalization machine; these design choices result in a dynamic range of about 1 part over  $1.3 \times 10^4$ , given by the full coverage of the sensing area by a layer of beads. The outreach of the project will be a portable multichannel electronic platform of unprecedented sensitivity for laboratory or point of care multiplex respiratory infection diagnosis, as well as a detailed protocol scheme of high affinity ligands for specific molecular investigation.

The flexibility of the proposed microsystem in addressing different pathogens by modifying the biosensor functionalization with various bioprobe, makes this platform of durable application and very practical industrial interest, yet reaching clinically breakthrough results. This strategy will bring advantages in terms of reduced sample handling and processing (less contamination and no loss of viral components) resulting in a reduction of time and, especially, cost of the analysis.

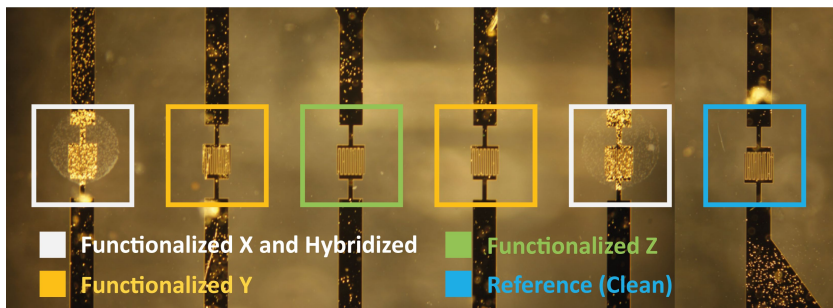
The platform has proven to reach nanoparticle resolution of few tens of target molecules. A limit of detection (LOD) below 100 pg/ml has been demonstrated and has been proven with the system operating in a real clinical setting using human serum samples from Dengue positive individuals, while the ongoing experiments involve tests on COVID DNA sequences and specific antigen-antibody complex<sup>2</sup>.

<sup>1</sup> P. Piedimonte *et al.*, *Biosensors and Bioelectronics*, Vol.202, 113996, ISSN 0956-5663, (2022)

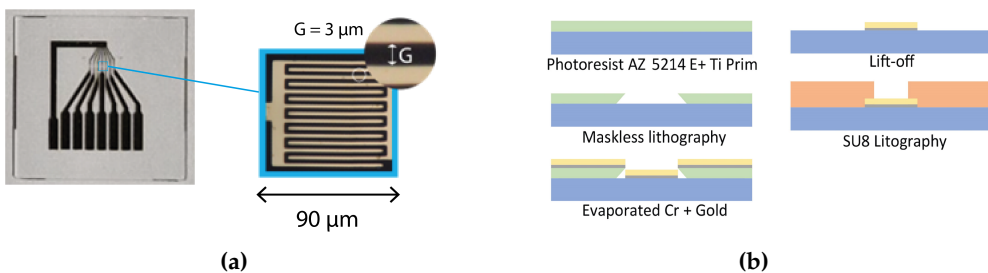
<sup>2</sup> P. Piedimonte *et al.*, *Bio-Reconfigurable Differential Impedance Electronic Platform for Multiplex Biomarker Detection*, *Proceedings of BIOCAS* (2023)



**Figure 1:** Scheme of the protocol of the assay, subdivided into Functionalization, Incubation and Beads counting phase. On the top right are shown picture from experiments performed with short oligonucleotide sequences and effective beads binding, or longer sequences (resulting from COVID PCR amplification) not showing beads binding. The reason has been imputed to steric hindrances, since smaller particle like fluorophores can successfully bind to the biological complex.



**Figure 2:** Image showing the optical results of a multiplexing experiment with 3 different oligonucleotides sequences, with beads concentrated exclusively over the electrodes functionalized with the probes complementary to the target in solution.



**Figure 3:** (a) Multiplexing chip shape, made of gold evaporated over a borosilicate substrate, with fabrication process shown in (b).

# Tunable Synaptic Working Memory with Volatile Memristive Devices

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Filamentary switching memories is class of RRAMs which rely on a metallic filament to change the electrical properties, where high mobility metal ions migrate from one electrode to the other creating a conductive bridge<sup>1,2</sup>. Silver-based RRAMs exhibit spontaneous disruption of the metallic conductive filament with a lifetime ranging from few microseconds to several seconds, thus by controlling and predicting the filament lifetime, devices can be engineered for a wide range of applications, such as those which resemble the biological systems to emulate tasks and functions of the human brain. Figure 1(a) shows the quasi-static response of Ag-based RRAMs, together with a sketch of the iconic bridge across the electrodes. When a positive bias is applied to the Ag electrode, the electric field leads to the Ag ions to migrate across the oxide and the resistivity drops down<sup>1</sup>. As the voltage decreases, the filament spontaneously disrupts, resulting in an abrupt increase of the resistance. Because of the spontaneous disruption of the filament<sup>1,2</sup>, it is important to study the temporal evolution of the devices, by switching on the memory and then monitoring the state until it switches off. The time window in which the filament remains stable is called retention time. Figure 1(b) collects the cumulative distribution curves of the retention time as a function of the maximum current reached during the switching<sup>2</sup>, which is limited by the saturation region of transistors. The devices result to be sensitive also to the pulse amplitude<sup>2</sup>, meaning that for small amplitude the devices do not switch on while for large values (> 3 V for example) the devices always switch on (Figure 1(c)).

Here we proposed a neuromorphic system which emulates the short-term memory, a primary concept in human brain, since it is respon-

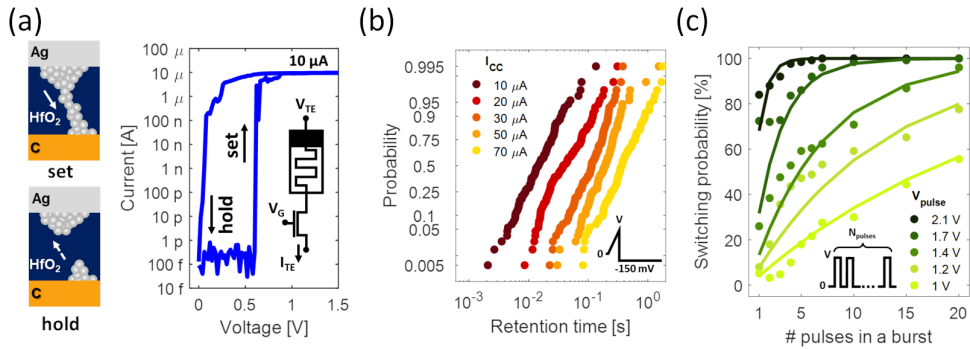
sible of the storing for the storage and processing of the sensory information. The system relies on the switching probability, tunable with the voltage, and the temporal dynamics which are adaptable according to the compliance current. The system has two main features: storing the information in the memory and later recognizing it, as depicted in Figure 2(a). The circuit is implemented only using 5 different devices (in Figure 2(b)) where the total current is summed together. Each device receives is sent a signal which is calibrated to have a specific switching probability (PON). After storing a pattern (the green symbol in the example of Figure 2(c)), when the true pattern arrives (marked with a dot) the system is triggered and recognizes it. Different experimental parameters, in terms of pattern rate, delay and amplitude, are tested, thus allowing to identify the best condition when the switching probability is low, and the refresh is high (Figure 2(d)). This condition is in good agreement with what happens to the human brain during the advertisement: all the spots have a small relevance, but when a good one is on the tv, the attention rises up, thus we can distinguish what we like from the other spots. Differently, when the spot is less broadcasted (small spike rate, in Figure 2(e)) the information is lost, and it is more difficult to recognize it. On the other hand, the volume also plays a crucial role, because our attention changes drastically. For great PON (so large volume) the system easily changes the information stored and thus is not able anymore to recognize the previous one.

**Acknowledgment:** This work was supported in part by the European Research Council (ERC) through the European's Union Horizon Europe Research and Innovation Programme under Grant Agreement No. 101054098 and from the European Union's Horizon 2020 research and innovation program, Grant Agreement No. 824164 and 899559.

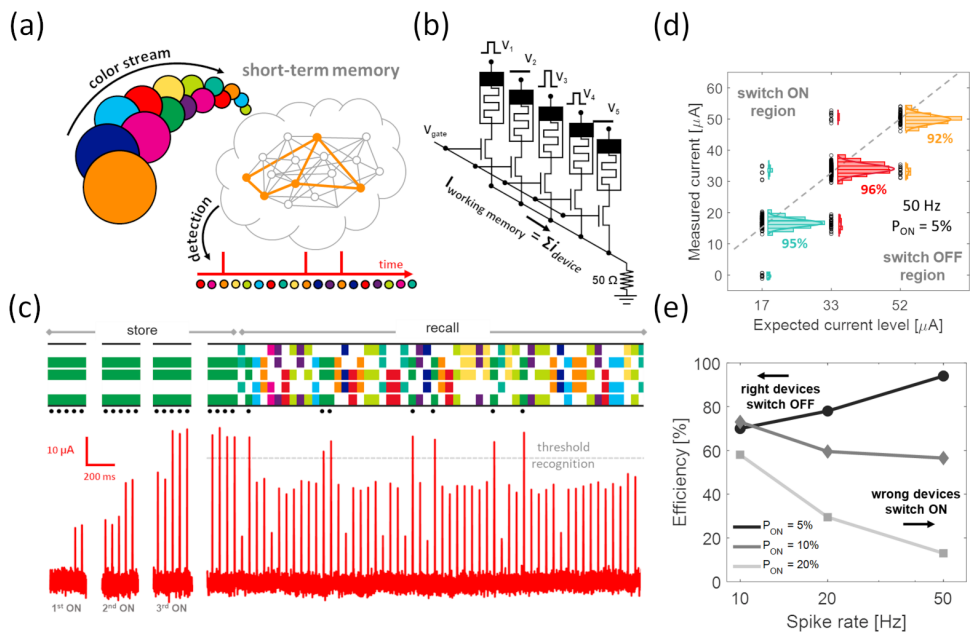
<sup>1</sup> S. Ricci *et al.*, Decision Making by a Neuromorphic Network of Volatile Resistive Switching Memories, 29th IEEE International Conference on Electronics, Circuits and Systems (ICECS), (2022)

<sup>2</sup> S. Ricci *et al.*, Tunable synaptic working memory with volatile memristive devices. In Neuromorphic Computing and Engineering (Vol. 3, Issue 4, p. 044004). IOP Publishing, (2023)





**Figure 1:** Ag-based volatile 1T1R RRAM electrical characterization. **(a)** Quasi-static I-V sweep. **(b)** Retention time distributions for different maximum current. 3 V and 1 ms triangular pulse are applied to set the device, and a constant  $-150$  mV bias voltage is applied to monitor the status of the device. **(c)** Impact of the number of pulses applied. Considering a group of pulses, the probability of finding the device in the ON state increases with the number of applied pulses. Adapted from [2]



**Figure 2:** Sketch of the memristive implementation of a working memory circuit emulator. **(a)** After an object (a colour for example orange) is stored in the mnemonic architecture, a stream of objects is sent to the system. When the true object arrives, the system is refreshed and triggered. **(b)** Real implementation using 5 Ag-based volatile 1T1R RRAMs. The devices are connected in parallel to sum the currents and share the same gate voltage to have the similar electrical responses. **(c)** Ex-ample of an experimental trace. In the store phase the same pattern is sent multiple times to switch ON the right devices, until all the 3 RRAMs are in the ON state. In the recall phase random pattern are sent. The current is discretized in four levels, according to the number of ON devices. A suitable current threshold is used to discriminate when the true pattern arrives. **(d)** Correlation plot of the best experimental parameters to check the accuracy of the system. **(e)** Behaviour of the memristive architecture by changing experimental parameters. The best results are achieved when the system is frequently refreshed. Adapted from [2]

# Lithographic processes on emerging controllable materials

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As high data rate wireless services become increasingly available and affordable, telecommunication networks need to strive to offer flexible and ubiquitous coverage. In consequence, an unprecedented effort is needed to investigate novel filters and phase shifters supporting tunable behavior beyond 20 GHz. Significant research is being devoted to passive materials exhibiting intrinsic tunability of dielectric properties<sup>1</sup>. The activity carried out with Polifab covered manufacturing of test structures on emerging tunable materials. Two distinct manufacturing issues have been addressed:

- multilayer manufacturing with backside alignment;
- lithography on “hostile” materials.

## Multilayer manufacturing

Electrical characterization of wafer-level components normally involves a probe station setup<sup>2,3</sup>, where the wafer is placed on a vacuum table and the top face exposes the necessary metal pads for power supplies and RF inputs and outputs. The transmission line most suited for such measurements is the coplanar waveguide, with Ground-Signal-Ground pattern and pitch compatible with the probe tip. The coplanar arrangement allows to achieve tight gaps, across which even a low DC voltage can establish high local electric fields, allowing to strongly bias electro-optic materials located into the gaps.

Some components instead require microstrip lines, where the signal line lies flat upon the ground plane, separated by an insulating material: here the silica substrate is used as main thick layer between ground plane (mostly large areas of copper patterned on the backside) and top signal plane (finely patterned copper traces on the front side). The available MLA100 machine

however natively enables fiducials alignment through its main top-view cameras only. However, the precision camera for standard alignment has a very close-range focus, not reaching the backside. Instead, the overview camera shows the backside rather clearly through the transparent 0.5 mm silica substrate. The resulting alignment is shown in Figures 1 and 2, and the  $\sim 100$   $\mu\text{m}$  offset in  $x$  and  $y$  appears to be a systematic error; a manual correction could be introduced to compensate for it and achieve a good backside alignment even using a top-view-only machine through a transparent thin substrate.

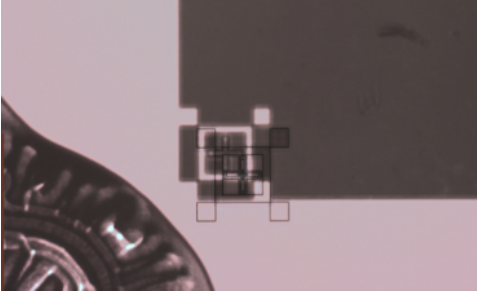
## Lithography on “hostile” materials

Another part of the project has been dealing with lithography on organic materials of rather unknown properties. Such organic materials, unfortunately, have been found to be sensitive to plasma cleaning and are corrupted by AZ726MIF (and similar developers of the same family) and by NI555. Instead, these materials are compatible with PGMEA-based developers. As a consequence, SU8 has been selected as suitable photoresist. Although the process has been reasonably optimized on a pure silica substrate, when repeated on a uniform layer of these organic materials produced wavy lines and severely warped shapes (Figure 3). Most structures were hence unusable except for a couple of lines, and the rest of the lithographic process was carried out evaporating copper and concluded by NMP liftoff, whose duration and conditions were experimentally determined, yielding the result of Figure 4. However, the same process repeated on other similar wafers removed all copper from the wafer and still created severely warped shapes. A full lithographic process compatible with such hostile yet sensitive organic materials is still to be determined and research is still ongoing, together with a first-of-its-kind electrical characterization of the successful samples.

<sup>1</sup> A. Giere *et al.*, Characterization of the Field-Dependent Permittivity of Nonlinear Ferroelectric Films Using Tunable Coplanar Lines, *IEEE Microwave and Wireless Components Letters*, vol. 17, no. 6, pp. 442-444, (2007)

<sup>2</sup> J. Rammal *et al.*, Comparison of different techniques of microwave characterization of high loss dielectric materials. 15th International Conference on Microwave and High Frequency Heating, Crakow, Poland, (2015)

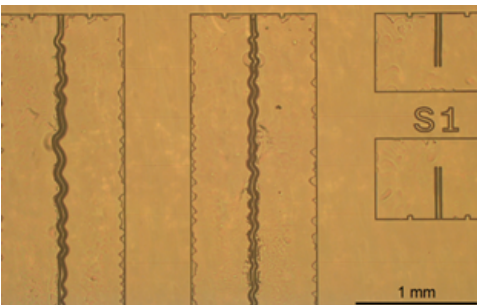
<sup>3</sup> M. Ouaddari *et al.*, Microwave characterization of ferroelectric thin film materials, *IEEE Transactions on Microwave Theory and Techniques*, vol. 53, no. 4, pp. 1390-1397, (2005)



**Figure 1:** Fiducial alignment through backside alignment via the MLA100 overview camera (low resolution).



**Figure 2:** Lithographic test pattern showing the  $\approx 100 \mu\text{m}$  offset achieved through the back-side alignment via overview camera.



**Figure 3:** Deformations of shapes after SU8 development with SU8-developer (PGMEA-based).



**Figure 4:** Developed shapes after NMP liftoff, with evident residuals. Those lines are however still acceptable for electrical characterization.

# Synthesis and characterization of thin film silicon oxycarbide for integrated photonics

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Silicon oxycarbide (SiOC) has emerged in integrated optics framework as an exciting material<sup>1</sup> due to its transparency over the IR wavelength range, the possibility to tune the refractive index, its CMOS compatibility, and its high thermo-optic coefficient  $2.5 \times 10^{-4} \text{ }^\circ\text{C}^{-1}$ . SiOC is a glass compound that results from the chemical incorporation of oxygen atoms in the silicon carbide network. SiOC thin films can be synthesized through different deposition techniques such as sol-gel, CVD, PVD, and polysiloxane pyrolysis. The thermo-optic coefficient has already been demonstrated to be one order of magnitude higher than other glassy materials used in photonics and 30% higher than silicon<sup>2</sup>, which makes it perfect for the actuation of the optical fields in integrated photonics chips. Integrating silicon oxycarbide in an integrated photonic platform is promising to exploit the high thermo-optic coefficient for efficient thermal actuator applications. The presence of SiOC on a waveguide changes the propagation of the confined electromagnetic wave, which can be used to control the guided light's phase efficiently. In this context, we present an experimental study to synthesize, characterize, and integrate PVD sputtered SiOC thin films onto a silicon nitride waveguide platform photonic chip.

Our work at Polifab consists of developing a recipe for a repeatable deposition of SiOC thin films by reactive (RF) magnetron sputtering (Leybold LH Z400) from a 4-inch diameter SiC target using argon and oxygen as plasma and reactive gases, respectively. Our target is a dense SiOC film that can be deposited on specific sections of pre-patterned silicon nitride waveguides on silicon substrates. In Figure 1 we show the cross-sectional image of our optimized films taken with a scanning electron microscope. We can see a

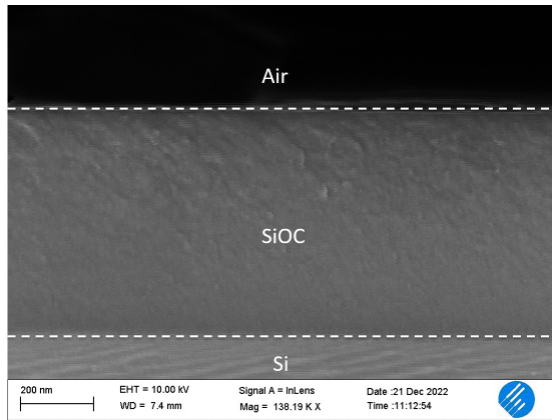
dense and compact film free of defects. Our target optical properties are a refractive index close to that of silicon nitride ( $n \approx 2$ ) and low absorption losses in the telecommunications wavelength range. With this scope in mind, we use Polifab's Spectroscopic Ellipsometer (J.A. Woolam VASE) to measure the SiOC film's optical properties after every deposition, confirming the repeatability of our process. Our deposition process involves considering the thermal budget of our substrates. We have successfully deposited SiOC films on photonic chips even with polymeric cladding layers, as seen in (Figures 2 and 3), where we show a selectively SiOC-clad silicon nitride waveguide.

We characterized our final devices by measuring the transmitted optical power through the fabricated waveguides for the telecommunications C-band wavelength range. This information allows us to measure our platform's propagation losses and the enhanced thermo-optic coefficient of the silicon nitride waveguide in contact with the SiOC film. We measured  $0.08 \text{ dB}/\mu\text{m}$  propagation losses at the wavelength of 1550 nm with a strong interaction of the guided mode with the overlying SiOC film. Additionally, the experimental spectra allow us to calculate a thermo-optic coefficient of  $2.6 \times 10^{-4} \text{ }^\circ\text{C}^{-1}$  for the deposited SiOC film, a value that agrees with the literature.

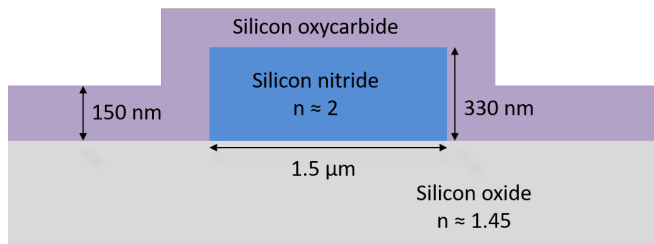
In conclusion, we have shown the integration of SiOC thin films on a standard SiN waveguide platform compatible with traditional processing of integrated photonic chips. We highlight the trade-off between propagation losses and thermo-optic efficiency, which needs to be considered for the next generation of devices. We acknowledge the invaluable support of Polifab's staff, especially from Dr. Marco Asa.

<sup>1</sup> F.A. Memon *et al.*, Silicon oxycarbide platform for integrated photonics. 38(4):784–791, (2020)

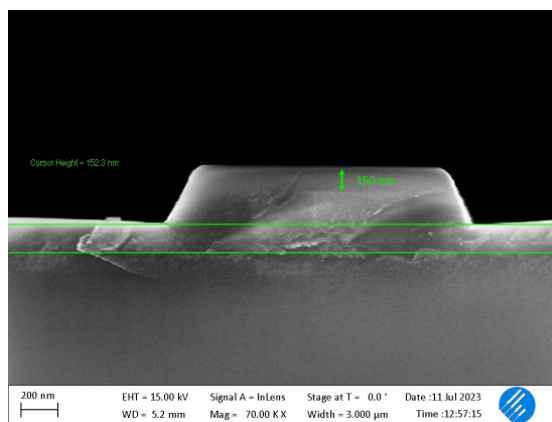
<sup>2</sup> F.A. Memon *et al.*, High thermo-optic coefficient of silicon oxycarbide photonic waveguides. 5(7):2755–2759, (2018)



**Figure 1:** Cross-sectional SEM image of the deposited SiOC film on a flat silicon substrate.



**Figure 2:** Schematic cross-section of our silicon nitride + SiOC waveguide platform.



**Figure 3:** Cross-sectional SEM image of the deposited 150 nm SiOC film directly on top of a silicon nitride waveguide.

# ZnS antireflection coating integrated in TMOS sensors: lift off and cleaning strategy

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Fabrication of infrared (IR) radiation detectors compatible with CMOS technologies is very challenging. TMOS is an IR detector based on a distinctive and innovative CMOS-SOI MEMS technology, manufactured at STMicroelectronics. It consists in an uncooled, thermally isolated MOS transistor operating at subthreshold. As the embedded and suspended sensitive area absorbs infrared radiation the transistor temperature is varied; this temperature's change is transduced in a voltage or current signal<sup>1</sup>.

The presented sensor is intended to work at room temperatures or slightly higher ones, corresponding to a radiation in the range of wavelengths of 8-12  $\mu\text{m}$ . To improve TMOS performances, in particular to reduce the reflection at air - silicon interface, an antireflective coating (ARC) is integrated in the device's structure, as displayed in Figure 2. The ARC coating consists in a quarter wavelength thick film of ZnS deposited on the silicon top cap of TMOS sensor and an adhesion layer of  $\text{Al}_2\text{O}_3$ , thick 20  $\text{nm}^2$ . Figure 2 shows the ARC and TMOS structure.

The depositions of ARC stack,  $\text{Al}_2\text{O}_3$  and ZnS, were performed in Polifab using the available e-beam evaporator machine Evatec BAK 640. It's important to underline that this deposition method allows to realize room temperature process. During the past year ARC layer deposition has been tested on different TMOS sensors layout, to verify if different geometric features require changes in the ARC deposition parameters or composition. The three layout masks of wafers processed are presented in Figure 1 and they're intended for different applications. Previously

designed ARC and deposition process<sup>2</sup> resulted suitable for the respective masks.

Once that deposition's yield was verified, lift off step has been performed. Lift off step is required because wafers to be processed arrive with a designed pattern of aperture through which ARC must be deposit. As it can be seen in Figure 1 in TMOS structure there are some trenches with high aspect ratio. The resist removal from these trenches it's particularly hard. For this reason the lift off and cleaning steps must be optimized to achieve a sufficient cleaning level that will allow to introduce the processed wafer into STMicroelectronics production line. Various trials have been performed exploiting several techniques as Ultrasonic Bath, High Pressure Cleaning and Megasonic Bath. The latter one is the standard procedure in MEMS fabrication with high aspect ratio features. After several tests, in which we experiment and vary all the possible parameter and combination of the different techniques, we have found that the most promising procedure involves 1h bath in DMSO at 71 °C, to melt the most of the resist present on the wafers' surface. Then a step of UTA bath in DMSO at 57 °C for 20 minutes must be performed. Sometimes for the mask in Figure 3(a) an additional step of High Pressure Cleaning with deionized water or DMSO, could be necessary. Some of the wafers processed with this cleaning recipe have been sent back to ST for inspection. The results showed an inspection cleaning yield of  $\sim 94\%$ . One of these examples is depicted in Figure 3(b).

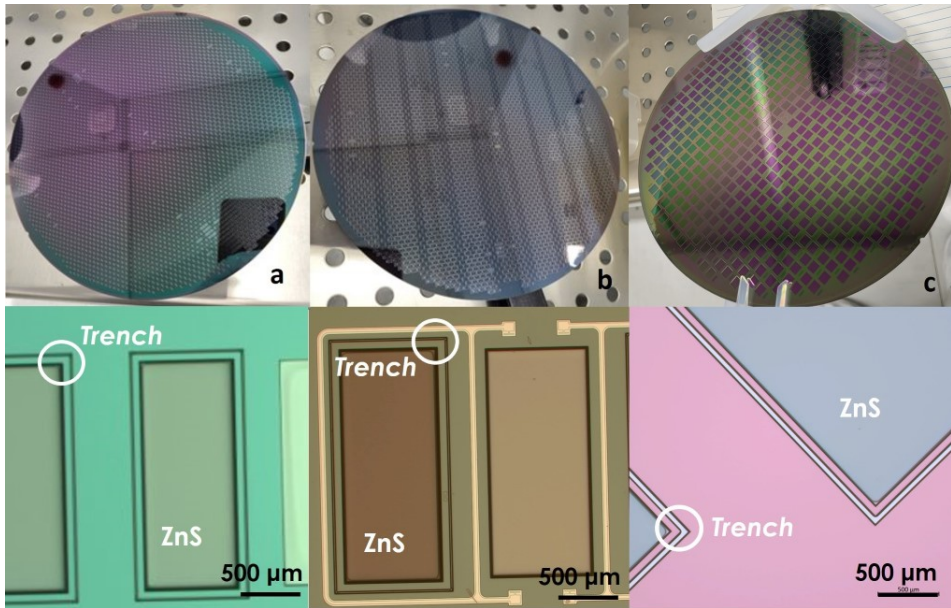
From the first trials, activity started in 2018, 282 wafers have been processed in collaboration between STMicroelectronics and Polifab.

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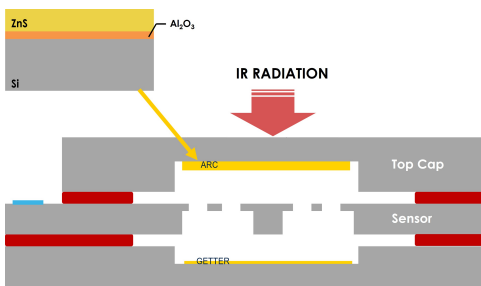
The work is carried out within the Joint Research Centre MEMS and STEAM agreement between STMicroelectronics and Politecnico di Milano.

<sup>1</sup> E. Moisello *et al.*, High Responsivity Thermopile Sensors Featuring a Mosaic Structure. *Micromachines* (Basel), 11;13(6):934, (2022)

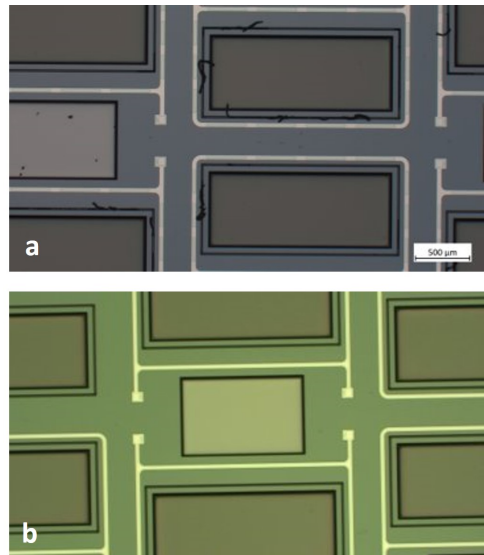
<sup>2</sup> C. De Vita *et al.*, Room-temperature deposition of ZnS antireflection coatings for MIR-LWIR applications, *Opt. Mater. Express* 12, 272-283, (2022)



**Figure 1:** TMOS different layout masks processed in Polifab. (a) standard cap mask (b) cap a ground mask (c) modified cap mask. For all three layout the “Trench” area is underlined.



**Figure 2:** TMOS structure in the ST process flow. Detail also on the ARC stack film.



**Figure 3:** Comparison between two different lift off + cleaning procedures on cap a ground mask. (a) Non optimized procedure: resist residues left in the trench (b) Optimized cleaning procedure: no resist residues.

# Germanium based Quantum Well Infrared Photodetector for Mid Infrared applications

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In recent years, mid-infrared integrated photonics has raised an increasing interest due to the presence of unique vibrational fingerprints of many molecules in the wavelength range  $\lambda = 5$  to  $15\ \mu\text{m}$ . A photonic integrated device (PIC) such as an on-chip biological and gas sensing spectroscopic detector (Figure 1) operating in the MIR spectral range could find a variety of applications for environmental monitoring for safety and security purposes and in the health-care field. The silicon-on-insulator (SOI) and silicon nitride (SiN) based technologies, operating at wavelengths  $\lambda < 4\ \mu\text{m}$  have already reached a significant technology readiness level. By leveraging the maturity of the SOI technology and taking advantage of the high index contrast between Si and SiO<sub>2</sub> many functionalities such as low-loss waveguiding, modulation and frequency comb generation have already been demonstrated. Nevertheless, due to the strong multiphonon absorption of SiO<sub>x</sub>, SOI-based and SiN-based devices can not address the operational wavelengths toward the mid and long-wave infrared region ( $4\ \mu\text{m}$ - $12\ \mu\text{m}$ ).

In this context the Ge-on-Si and SiGe-on-Si material platforms seem particularly promising. Taking advantage of the wide transparency range of Ge, low-loss waveguides operating up to  $\lambda = 11\ \mu\text{m}$  have been recently demonstrated, as well as a whole set of passive photonic components including Mach-Zehnder interferometers, resonators and spectrometers. Also, electro-optic modulation based on plasma effect has been recently reported<sup>1</sup>. However, an open challenge consists in the implementation of key functionalities such as wavelength conversion, photodetection, and high speed optical modulation.

A promising platform to tackle this problem is represented by Ge/SiGe quantum wells. Adopting a well-developed approach in III-V materials, it is possible to exploit intersubband optical transitions (ISBT) in the valence band of these heterostructures for light detection, high speed optical modulation through quantum confined Stark effect (QCSE) and for wavelength conversion through second harmonic generation in asymmetric coupled quantum wells (ACQW) (Figure 2).

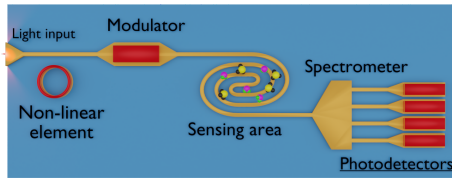
Regarding light detection, we recently modeled a heterostructure to implement quantum well infrared photodetectors working at  $\lambda = 8\ \mu\text{m}$ . The Ge/SiGe quantum well stack has been grown by low-energy plasma-enhanced chemical vapor deposition (LEPECVD)<sup>2</sup> (Figure 3) on top of a graded buffer providing, not only a compositionally matched substrate for the quantum wells, but also a suitable platform for a future waveguide integration of the device, due to the grading of the refractive index along the growth direction. After a careful optimization of the growth parameters by HR-XRD measurements, we demonstrated the ISBT in a prism-cut sample by FTIR measurements, and we are now moving toward waveguide integration. A fabrication process for a vertically illuminated devices has been already optimized and realized (Figure 4), and we are currently developing a procedure suitable to fabricate rib waveguides with challenging requirements on vertical depth and lateral smoothness.

<sup>1</sup> M. Montesinos-Ballester *et al.*, Mid-infrared Integrated Electro-optic Modulator Operating up to 225 MHz between 6.4 and 10.7  $\mu\text{m}$  Wavelength, ACS Photonics 9(1), 249–255 (2022)

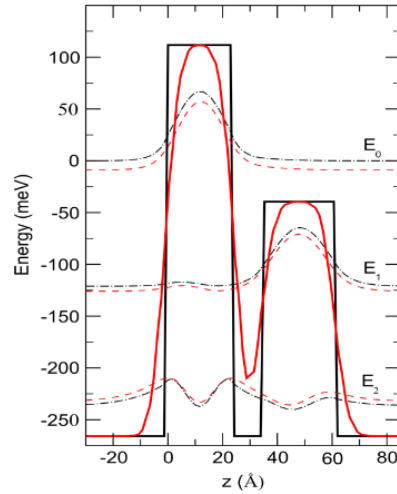
<sup>2</sup> G. Isella *et al.*, Low-energy plasma-enhanced chemical vapor deposition for strained Si and Ge heterostructures and devices. Solid-State Electron, 48, 1317– 1323 (2004)

<sup>3</sup> J. Frigerio *et al.*, Modeling of second harmonic generation in hole-doped silicon-germanium quantum wells for mid-infrared sensing, Opt. Express 26, 31861-31872 (2018)

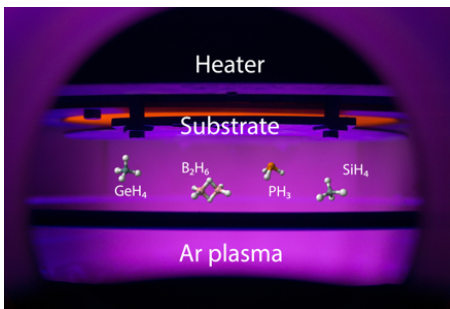




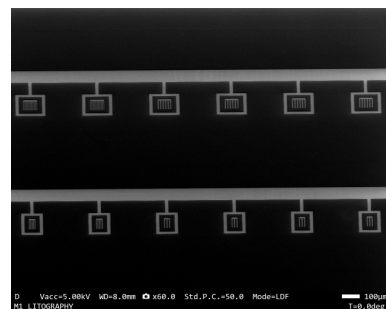
**Figure 1:** Schematic of a possible sensing device. The key active elements, highlighted in red, can be addressed by intersubband transitions in Ge/SiGe quantum wells.



**Figure 2:** ACQW band structure modeling. An ad-hoc tight binding modeling has been developed to address the complexity of the SiGe valence band<sup>3</sup> The intermixing induced smoothing of the compositional profiles has been taken into account.



**Figure 3:** LEPECVD growth chamber. Leveraging an Ar plasma to grow out of thermodynamic equilibrium, this technique features unique growth conditions respect to deposition rates and temperature control.



**Figure 4:** SEM image of the QWIP devices fabricated in Polifab. The finger design of the metallic contacts enables spatially resolved detection of MIR light.

# Research Activity of PhyND group

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The research activity carried out at Polifab has been primarily focused on three topics.

## Direct Laser Writing (DLW) of Magnonic Crystals in YIG:

The objective of this project was to demonstrate a novel patterning approach, “phase micro- and nano-engineering”<sup>1</sup>, applied to magnetic materials for magnonics. We utilized the laser of the NanoFrazor Explore at Polifab to locally modify the structural and magnetic properties of 1  $\mu\text{m}$  and 100 nm thick Yttrium Iron Garnet (YIG) crystals. Magnetic Force Microscope (MFM) characterization, micro-Kerr, and micro-Raman characterization were performed (in collaboration with V. Russo and A. Li Bassi, POLIMI). By tuning the laser power, various changes can be achieved, with a minimum feature size of the patterns below 100 nm. Below a certain threshold, a slight decrease in the magnetic contrast measured by MFM in the patterns is observed without any significant change in the domain configuration (figure 1a). Beyond the threshold, the period of the stripe domains halves, and the magnetic contrast increases (figure 1b). This is consistent with a change in the effective anisotropy constant. Importantly, these magnetic changes are associated with a negligible change in the YIG composition, indicating a partial rearrangement of the oxygens. Through micro-Brillouin Light Scattering measurements (in collaboration with Perugia University), we demonstrate that these changes can modify the dispersion and localization of the spin waves<sup>2</sup>, creating a magnonic crystal. Currently, we are optimizing the patterning of thin YIG films via Thermal Scanning Probe Lithography, with the aim of achieving a resolution down to the 10 nm range.

## DLW of Skyrmion Crystals:

We used the Laser of the NanoFrazor Explore to demonstrate the magnetic patterning of Py/IrMn (in collaboration with O. Boulle, SPIN-TEC) and CoFeB/IrMn multilayers with perpendicular magnetization. By exploiting the combined effect of the laser and the external magnetic field, we were able to pattern spin textures with different topologies (skyrmions, skyrmionium, and worm domains) without affecting the sample’s composition, structure, and topography. These purely magnetic patterns were then imaged via MFM (figure 2). By tuning the laser power and exploiting the tunability of exchange bias, we demonstrated that the written patterns are reconfigurable via external magnetic fields. We were able to pattern skyrmion lattices with deterministic topology and geometry and a minimum diameter of about 200 nm, which are currently under investigation via Brillouin Light Scattering (in collaboration with Perugia University) to measure their dynamic properties.

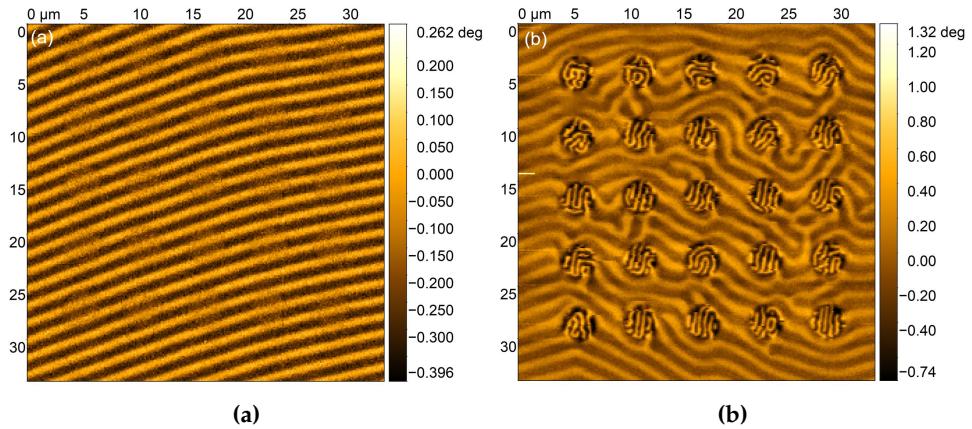
## DLW of Heusler Alloys for Magnetocalorics:

We applied phase micro- and nano-engineering to pattern NiMnGa films, with a minimum feature size below 500 nm. These materials exhibit both magnetocaloric and shape memory effects, undergoing a phase transition from a magnetic martensitic phase at room temperature to a nonmagnetic austenitic phase. By patterning these films with different laser powers, we demonstrated the ability to tune their phase transition temperature and phase hysteresis loop (figure 3) without significant changes in their magnetic properties<sup>3</sup>. Various applications for microscale cooling systems, actuators, and energy harvesting devices are envisaged.

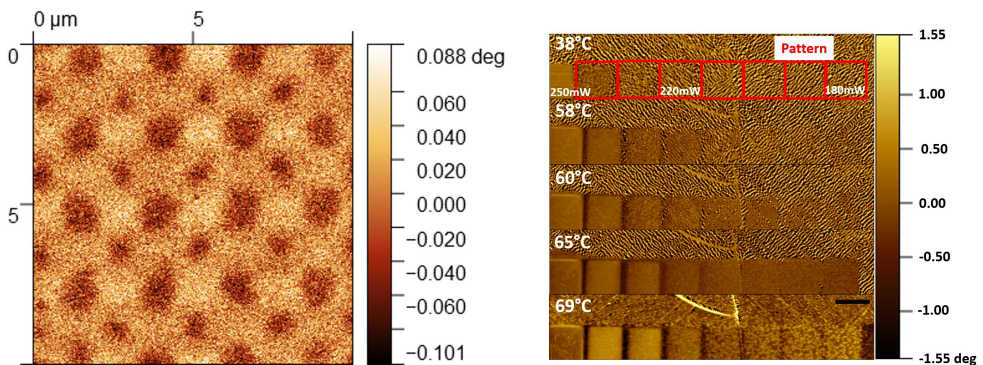
<sup>1</sup> V. Levati *et al.*, *Advanced Materials Technologies* 8 (16), 2370075 (2023)

<sup>2</sup> V. Levati *et al.*, in preparation

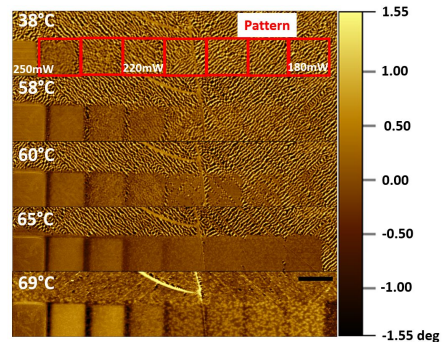
<sup>3</sup> N. Pellizzi *et al.*, in preparation



**Figure 1:** Magnetic force microscope (MFM) image of a lattice of dots with a diameter of  $2\ \mu\text{m}$ , patterned via direct laser writing on a  $1\ \mu\text{m}$  - thick YIG crystal. Two different regimes are visible: **(a)** under a certain power threshold the dots exhibit only a change in magnetic contrast. **(b)** Beyond the threshold the micromagnetic configuration changes and, the magnetic contrast increases.



**Figure 2:** MFM image of a centered cubic lattice of skyrmions. The central skyrmion is written at a different laser power. Applying an external magnetic field transforms the centered cubic lattice into a simple cubic one.



**Figure 3:** MFM image of  $10 \times 10\ \mu\text{m}^2$  squared patterns at various temperatures. The squares were written at different power levels. The stripe domains in the patterns disappears at different temperatures. This demonstrates the ability to tune the phase transition temperature through the patterning. Scale bar  $5\ \mu\text{m}$ .

# 3D PDMS micropatterned substrates form to observe the cellular response to substrate stiffness: SU8 and SI molds

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Cells composing tissues, in in-vivo environments reflect the contribution of nano- and micro-topographies they are exposed to, in the tissue organization, macroscale topography, and sub-cellular architecture. The interaction with Extracellular matrix (ECM) and/or neighboring cells, affects various biological functions such as activation of signaling pathways, cell differentiation, migration, and proliferation<sup>1</sup>. Since stiffening of the biological environment, for example, tissue fibrosis and extracellular matrix (ECM) stiffening, happen and promote tumor progression<sup>2,3</sup> and it is typical in pathological conditions is interesting to replicate such "stiffening" in in-vitro complex culture. Microfabrication offers several approaches to build up substrates for cell culture. A very well-known silicone-based material, Polydimethylsiloxane (PDMS, Dow Sylgard 184) shows a Young elastic modulus that, on a microscale pseudo-pillar structure, may offer, to cells in culture, a substrate stiffness comparable to the physiological/pathological tissues.

Micropillar arrays size allows single cells to spread on multiple pillars. The stiffness of pillars can be modulated by the shape of the pillar section, and the aspect ratio of the pillars. PDMS substrates are obtained by molding on dedicated molds, down to the singular micron scale. In Polifab we performed two different molds fabrication to obtain substrates composed of micropillars: an optimization of standard photolithography, to obtain SU8 molds and a work-in-progress RIE to obtain molds on silicon substrates.

SU8 enables the realization of micro pillar patterns on multilayered structures, of hundreds of microns thickness, that are often useful in the fabrication of microfluidic devices for cell cul-

ture and biological applications. On the other side molds in Si, by RIE, (Figure 1) enable better control of the shape of the structures and a better repeatability of processes. Since the actual height of the final PDMS structures depends on the thickness of the molds, but also on the efficacy of filling, measurements on final PDMS structures are necessary. We need to optimize both the mold production process and the PDMS casting and the curing.

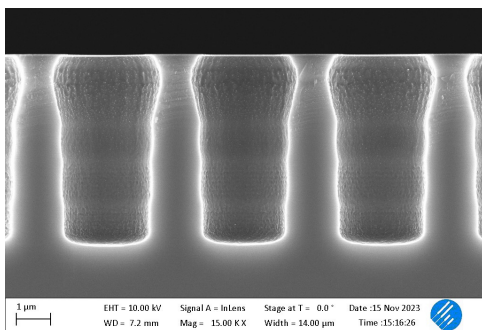
The first sets of micropillars were fabricated on SU8 molds, showing a height range from 2 to 5 μm, Ø 3 to 4 μm at a spatial density of 4 μm to 5.5 μm while on SI, based on previous biological results we aim at a single pitch and diameter, producing pillars of different heights. The electrical insulating properties, the translucent aspect, micron-scale size, and the of both PDMS forced us to introduce a step of surface metallization on a test pillars sample, to measure heights and check the aspect by SEM (figure 2-3). PDMS pillars are then exposed to plasma treatment, sterilized, and prepared with a protein coating. Cell samples (H5V murine endothelial cells) were seeded on PDMS micropatterned samples, in a 96-multiwell plate. As a preliminary result, cells (24h of incubation) showed different grades of elongation on PDMS patterns of different stiffness. (figure 4). Si seems to allow better repeatability of total processes, but processes still need to be optimized to get a more cylindrical and close-to-standard shape.

This work is part of the Accelerator Award n° A26815: "Single-cell cancer evolution in the clinic" funded by Cancer Research UK and Fondazione AIRC (n° 22790).

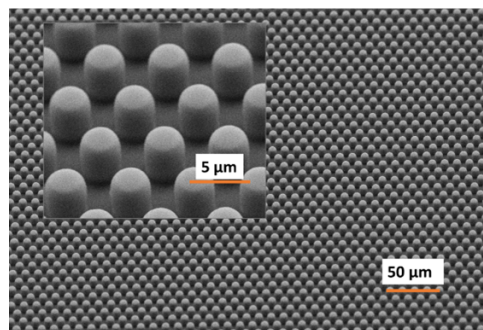
<sup>1</sup> H. van Hoorn *et al.*, The nanoscale architecture of force-bearing focal adhesions, *Nano Letters*, vol. 14, pp. 4257-4262, (2014)

<sup>2</sup> L. E. Dickinson *et al.*, Endothelial cell responses to micropillar substrates of varying dimensions and stiffness, *Journal of Biomedical Materials Research*, vol. 100, pp. 1457-1466, (2012)

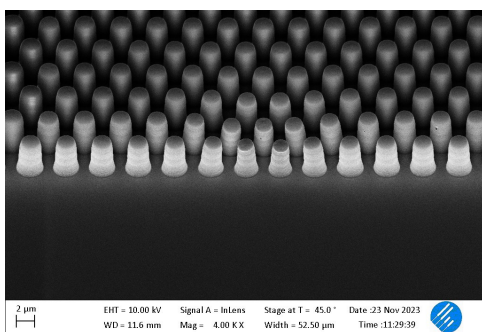
<sup>3</sup> B. Wu *et al.*, Stiff matrix induces exosome secretion to promote tumour growth. *Nat Cell Biol.* 25(3):415-424, (2023)



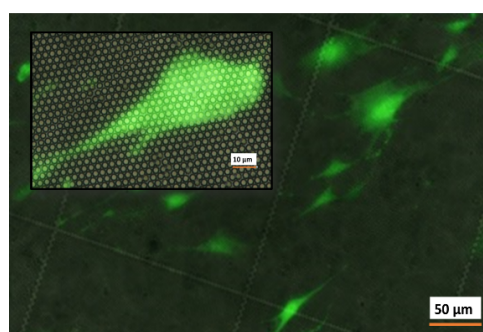
**Figure 1:** Molds in made of Si.



**Figure 2:** PDMS micropatterned surface obtained on SU8 molds.



**Figure 3:** PDMS micropatterned surface obtained on from SI molds



**Figure 4:** Phase-contrast images in brightfield of the micropillar substrate and a fluorescence image of H5V cells, merged (20X.)

# Functional materials for MEMS and magnonics

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Functional materials, such as piezoelectric or magnetic materials can enable new possibilities in the design and fabrication of new MEMS devices such as actuators, sensors or electronic components<sup>1</sup>. During these years, we have been working at the development of two class of materials: magnetic materials and lead-free piezo/ferroelectric materials for MEMS applications.

## Magnetic materials

We have set up a sputtering process to deposit relatively thick magnetic layers (1  $\mu\text{m}$ ) of rare-earth-based magnetic materials (SmCo) which can exhibit very large coercivity ( $H_C > 3.6\text{T}$ ) (Figure 1(e)). Following the optimization of these materials we have worked towards new patterning techniques for the fabrication of micro-magnets. The first technique is based on ion-beam-etching, which allows the definition of micro-magnets just using a photoresist masks, leading however to a roughening of the surface. The second technique is based on lift-off, using a home-made undercut bi-layer of dense/mesoporous sol-gel-derived  $\text{SiO}_2$ , that can withstand the high temperatures occurring during the sputtering deposition process (Figure 1).

## Application of permanent magnets

Permanent magnets (PM) at the microscale enable new applications. One example is the integration of PM and magnonic elements. Magnonic is a branch of magnetism which studies the generation and propagation of spin-waves (SW) to transmit and process information. One of the limits of magnonic is the need of an external magnetic field. In M&MEMS project, we are exploiting micromagnets to control the propagation of SW. In this year we have successfully integrated permanent magnets and magnonic conduits, using a combination of the previously mentioned

technique and planarization of topography using inorganic planarization layer. We show a sketch of the final device in Figure 2. We are now moving towards new techniques to couple magnets and magnonic, such as flip-chip bonding of different chips (Figure 3).

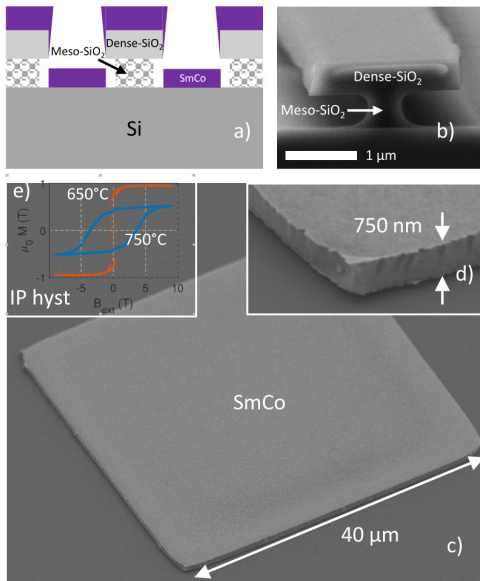
## Lead-free piezoelectric

Lead zirconate titanate (PZT), a perovskite material, is nowadays the most used piezoelectric. However, lead is toxic, prompting a search for its replacement with other lead-free piezoelectric materials.  $\text{K}_{1-x}\text{Na}_x\text{NbO}_3$  (KNN) is a solid solution of potassium niobate and sodium niobate. Like PZT, KNN is a perovskite material, but it does not contain toxic elements and it is biocompatible. Within the joint project STEAM, Polifab and ST Microelectronics are working together on the development of KNN films deposited on 8" wafers by confocal sputtering with an industrial EVATEC Clusterline-200 machine. We have developed individual recipes for deposition of KNN films of controlled (001) fiber texture<sup>1,2</sup>, potassium niobate (KN), and sodium niobate (NN) from single targets. We have also worked on developing recipes for confocal deposition of KN and KNN to produce preferentially-oriented KNN solid solutions with tailored stoichiometries around polymorphic phase boundaries (PPB). Theoretical approaches like Density Functional Theory (DFT) and Machine Learning have also been used to guide the material development activities, as well as to discover other useful perovskite piezoelectrics. Parallely, we have developed Pt/ZnO/SiO<sub>2</sub>/Si 8" templates for enhanced crystalline growth of KNN-based films, with reduced inter-diffusion of defect-producing elements. In the short term, we aim at interface-tuning<sup>3</sup> and at incorporating doping elements into KNN to achieve a better control of its electrical properties to reduce leakage currents.

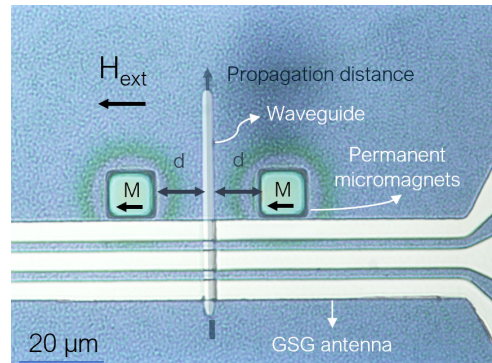
<sup>1</sup> F. Maspero *et al.*, Magnetism meet microelectromechanical systems, IEEE International Magnetic Conference - Short Papers (INTERMAG Short Papers), (2023)

<sup>2</sup> C. Groppi *et al.*, Spontaneous pattern of orthogonal ferroelectric domains in epitaxial KNN films, J. Appl. Phys. 134(20), (2023)

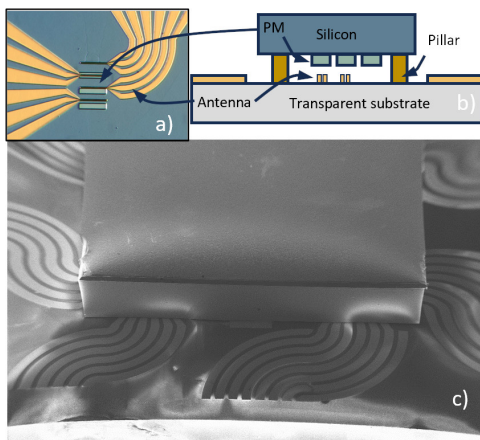
<sup>3</sup> C. Groppi *et al.*, Electrode-dependent asymmetric conduction mechanisms in  $\text{K}_{0.5}\text{Na}_{0.5}\text{NbO}_3$  micro-capacitors, Mater. Sci. Semicond. Process. (2023)



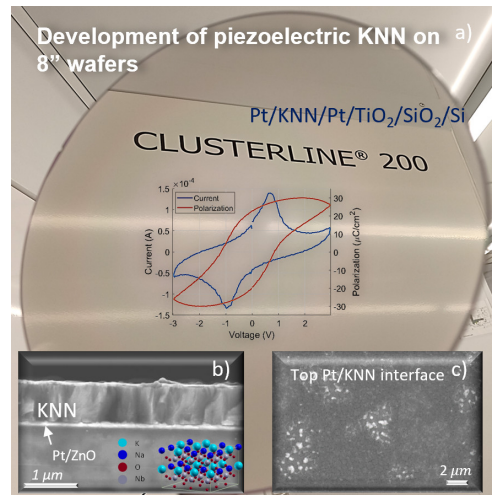
**Figure 1:** a) to d) Lift-off-defined micromagnets of SmCo obtained with SiO<sub>2</sub> bilayer b). To date, we demonstrated 750 nm-thick micro PM with a well-defined definition d) and up to 1.3 μm-thick magnets when embedded in Si substrate.



**Figure 2:** Permanent magnets are coupled to a magnonic waveguide for the control of spin-waves propagation.



**Figure 3:** Flip-chip bonding between two chips. a) view through the transparent substrate, note that features are on two different substrates; b) scheme of flip-chip; c) SEM image of the two bonded chips.



**Figure 4:** a) 200 mm wafer of KNN; b) Cross-section of thick KNN and inset showing DFT simulation; c) Study of segregation in KNN.

# CMOS-based pH and DNA sensors for biomolecular tests

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In our research, we are investigating the use of CMOS floating-gate transistors as a platform for the detection of charged analytes in a liquid solution.

According to the material deposited on top of the floating gate, different analytes can be selectively bound to the interface. As a result of the interaction between the (charged) analytes of interest and the material on the floating gate, an accumulation of charge develops on the interface, which can be detected by the transistor through field-effect (figure 1). One of the analytes we are interested in is H<sup>+</sup> ions, whose concentration in solution is related to pH. In order to selectively capture H<sup>+</sup> ions, a wide variety of materials can be used, normally oxides (e.g. Al<sub>2</sub>O<sub>3</sub>, Ta<sub>2</sub>O<sub>5</sub>, Si<sub>3</sub>N<sub>4</sub>, etc.). We have developed a prototype in CMOS 0.35 μm technology for a preliminary characterization of this type of device. As sensitive materials, we have used Al<sub>2</sub>O<sub>3</sub> and Si<sub>3</sub>N<sub>4</sub>, as they could be found in the CMOS process (native oxide layer of aluminum pads and passivation layer, respectively), and thus did not require a post-processing of the chip.

In figure 2 we report calibration curves related to the two different sensitive materials, for layers of different geometries (15 μm × 15 μm, 30 μm × 30 μm, 60 μm × 60 μm). The response of the Si<sub>3</sub>N<sub>4</sub> sensors (figure 2a) shows a linear behavior between pH 2 and 10. The different slopes are associated to the capacitive partition between the capacitance associated to the sensitive layer and that seen from the gate of the transistor. The Al<sub>2</sub>O<sub>3</sub> sensors (figure 2b) show a narrower range, outside which instabilities in the measured sig-

nal arise. The different slopes in this case are not justified by the capacitive partition, but we hypothesize that it is due to variability between the different native oxide surfaces.

The biggest limitation for this type of solid-state pH sensor is the long term drift, associated with the very slow hydration of the sensitive layer<sup>1</sup>. In order to mitigate this effect, other materials, deposition techniques and thickness of the sensitive layer can be investigated. With this scope in mind, we have designed a second integrated circuit in standard CMOS 0.35 μm, prioritizing flexibility (figure 3). Namely, the design is such that the material for the sensitive layer and deposition technique can be defined later on a post-process of the chip, which will be performed in Polifab.

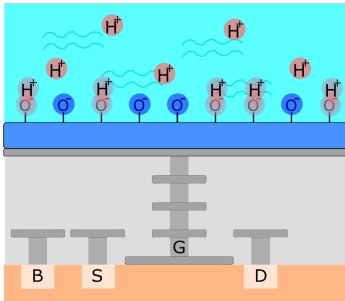
The strategy to obtain this flexibility is as follows<sup>2</sup>: in the design phase, a large aperture in the passivation layer (1.91 mm × 0.5 mm) is defined, exposing the aluminum top metal layer. Single tungsten vias are positioned under the top metal layer and connected to the respective read-out circuit. In the post-process, the aluminum is etched away, leaving independent single vias exposed on a very planar surface. The material for the sensitive layer is then deposited on top of the vias (figure 4a-c).

This degree of flexibility allows us to comprehensively investigate different sensitive layers to improve the sensing of H<sup>+</sup> ions, but also to target different analytes. In particular, we have introduced in the chip novel read-out circuits for DNA detection.

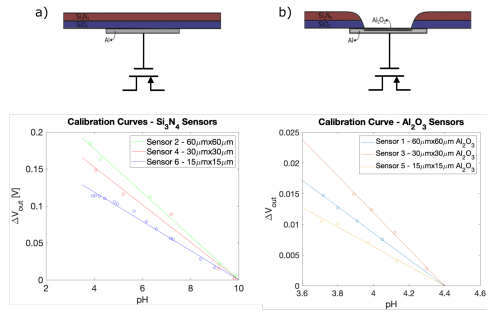
<sup>1</sup> X. Jin *et al.*, Steady-State and Transient Performance of Ion-Sensitive Electrodes Suitable for Wearable and Implantable Electro-Chemical Sensing, *IEEE Transactions on Biomedical Engineering*, (2022)

<sup>2</sup> A.Y. Wang *et al.*, A Multimodal and Multifunctional CMOS Cellular Interfacing Array for Digital Physiology and Pathology Featuring an Ultra Dense Pixel Array and Reconfigurable Sampling Rate, *IEEE Transactions on Biomedical Circuits and Systems*, (2022)

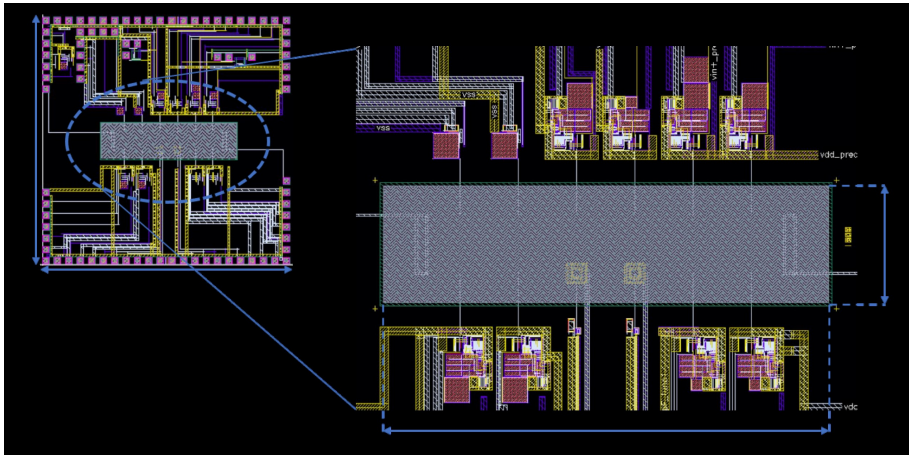




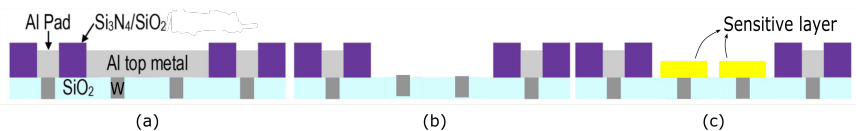
**Figure 1:** Schematic view of a CMOS floating-gate transistor with an oxide layer as sensitive layer for  $H^+$  ions sensing.



**Figure 2:** Calibration curves of the  $Si_3N_4$  **a)** and  $Al_2O_3$  **b)** sensors, for different geometries of the sensitive layer:  $15\ \mu m \times 15\ \mu m$ ,  $30\ \mu m \times 30\ \mu m$ ,  $60\ \mu m \times 60\ \mu m$ .



**Figure 3:** Layout of integrated circuit designed for pH sensing and DNA detection. The chip will go through a post-process in Polifab in order to etch away the aluminum top metal layer and define the electrodes.



**Figure 4:** Schematic view of the post-process to remove the aluminum top metal layer **(a)**, leaving independent single vias connected to their respective read-out circuit **(b)**, and deposition of sensitive layer on top the vias.

# Biosensors

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Electrolyte-Gated Field-Effect Transistors (EGFETs) have emerged as a potential breakthrough in biosensing applications, demonstrating the ability to transduce minute changes in potential into significant alterations in current. Operating with a low working voltage ( $< 1$  V) in aqueous environments is made possible by the high capacitance of the electrical double layer formed at various interfaces. Their direct and efficient interaction with biological samples, coupled with compatibility with cost-effective large-area manufacturing processes, positions them as excellent candidates for affordable point-of-care diagnostics<sup>1</sup>.

The research activity focuses on two primary objectives:

1. Designing highly sensitive biosensors for the diagnostic and health monitoring of biomarkers in physiological fluids.
2. Developing a bioelectronic recording platform to facilitate real-time monitoring of the electrical activity of electrogenic cells.

## Cardiomyocytes' Action Potential Recording

The amplitude, frequency and duration of Action Potentials (APs) encapsulate vital information about cell viability, enabling an exploration of cardiac pathologies and the effects of emerging pharmaceutical products. Current methodologies predominantly rely on invasive approaches like patch clamp techniques or intricate 3D nanostructured electrodes, often coupled with electro/opto-poration. This study's primary aim is to propose a straightforward and cost-effective de-

vice capable of non-invasively recording the electrical activity of in-vitro cell cultures. Within this investigation, we achieved a groundbreaking milestone: the spontaneous recording of intracellular action potentials in human induced pluripotent stem cells-derived cardiomyocytes (hiPSC-CMs). This was accomplished through the direct plating or seeding of cells on the channel of our printed, planar Electrolyte-Gated Field Effect Transistor (EGFET).

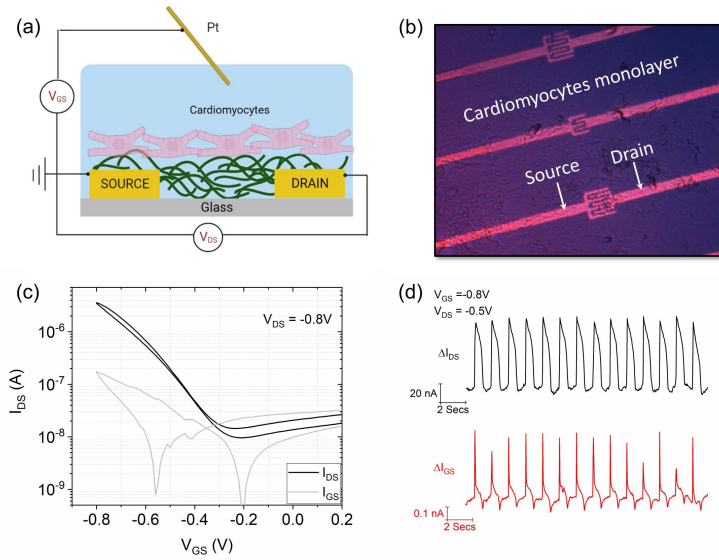
## Multiparametric detection of metabolites by transistor-based sensors

The objective of this project is to design and implement a new system for convenient sweat analysis in non-centralized healthcare facilities and personalized diagnostics<sup>1</sup>. A portable system for multi-parameter recording using Electrolyte-Gated Transistors (EGTs) as transducers and a microfluidics sample collection system will be developed to enable highly accurate and continuous monitoring of metabolites. The flexibility introduced by the extended-gate architecture allows the simultaneous multiparametric analysis of different metabolites, such as glucose, ions, such as chloride, or physiological conditions, such as pH levels<sup>2</sup>. Encouraging preliminary results showed an appreciable sensitivity and good performance, enhancing the reliability and integrity of the extracted information and its medical relevance. To fabricate these devices, we employed a photolithographic image reversal process (AZ5214E photoresist) to transfer the desired pattern onto substrates such as glass and plastic (PEN) using a maskless aligner (Heidelberg MLA100). Subsequently, gold electrodes were deposited via thermal evaporation (Moorfield MINILAB-080), followed by a lift-off step.

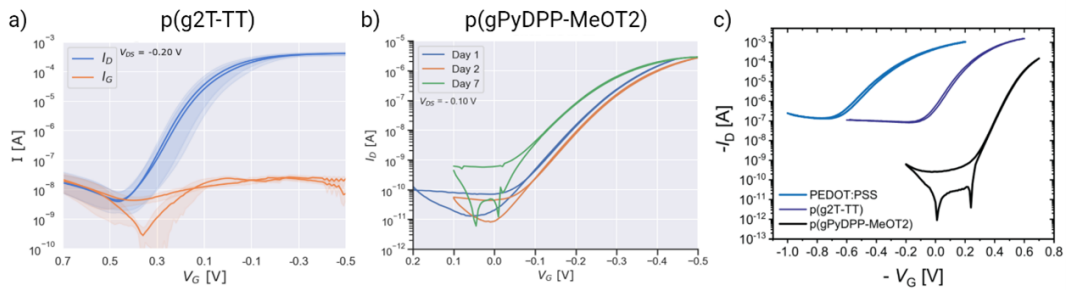
<sup>1</sup> F. Torricelli, *et al.*, Electrolyte-gated transistors for enhanced performance bioelectronics. *Nature Reviews Methods Primers*, (2021)

<sup>2</sup> S. White *et al.*, Operating and Sensing Mechanism of Electrolyte-Gated Transistors with Floating Gates, *Journal of Physical Chemistry*, (2016)

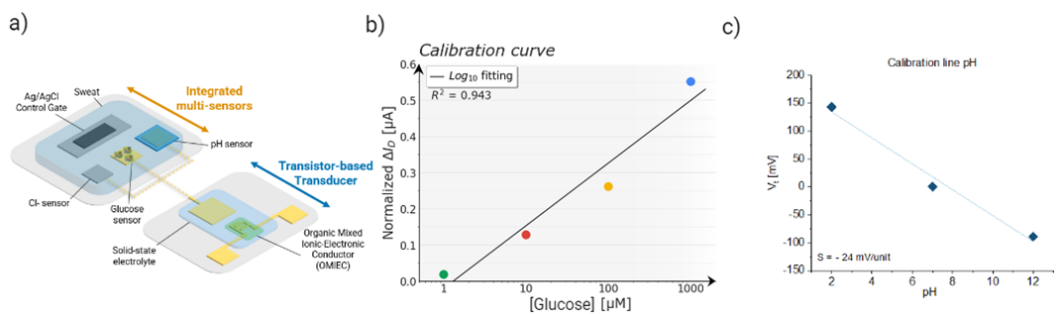
<sup>3</sup> A. Sharma *et al.*, Non-invasive, ultrasensitive detection of glucose in saliva using metal oxide transistors, *Biosensors and Bioelectronics*, (2023)



**Figure 1:** Device schematics and micrograph (a,b); Semiconductor characterisation (c); Action Potential Recording of Cardiac Cells (d).



**Figure 2:** Optimization of Organic Electrochemical Transistors.



**Figure 3:** Device schematic and preliminary results.

# Spin transport and spin-charge interconversion for next generation low power electronics

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The digital revolution we have witnessed over the past 50 years has been covertly driven by synergic improvements in microelectronics fabrication and silicon-based devices. However, a paradigm shift is necessary to drive the transition towards next-generation electronics able to cope with the demand for better performances, lower power consumption and enhanced scalability. Rather than limiting the attention to charge-based transport, we add the spin degree of freedom in conventional metals and in more innovative materials with unique spin-orbitronics properties as well. The common denominator is the *exploitation of materials with a significant spin-orbit coupling*.

Part of our activity is devoted to the study of spin-orbit torque (SOT) memories and spin-orbit logic, the latter pointed out by the Exploratory Integrated Circuits division of Intel as one of the most promising technologies for beyond-CMOS transistors.

Polifab offers state-of-the-art equipment for the *fabrication of nano and micrometric devices using conventional spin-orbit metals* such as Pt or Ta and ferromagnetic materials like iron, cobalt and their alloys. In the laboratory of Nanomagnetism of the Department of Physics annexed to Polifab, we can fully characterize the spin-transport properties of stacks and devices by magneto-electric and optical methods. We can quantify the magnitude of different spin-to-charge current conversion (SCC) mechanisms such as the spin Hall, anomalous Hall and anomalous Nernst effects. Such know-how opened up the possibility of establishing both national (e.g. Istituto Nazionale di Ricerca Metrologica - INRIM - di Torino / CNR-ISM, Rome<sup>1</sup>) and international (Université of Aix-Marseille, Marseille, France) collaborations on related topics.

Common materials are studied alongside

emerging *multifunctional materials*. In particular, we continued our investigation of chalcogenides (e.g. GeTe, SnTe, GeSnTe) as promising members of the class of *ferroelectric Rashba semiconductors*. Those compounds allow the electrical control of the generation and flow of spin currents in semiconductors, paving the way to logic-in-memory non-volatile devices exploiting ferroelectricity and spin-to-charge current conversion<sup>2</sup>. Such compounds can already be grown by *molecular beam epitaxy* in a cluster tool operating in ultra-high vacuum, although a new *state-of-the-art commercial system working on two inches* will be available from the end of next year. Overall, we were able to successfully grow GeTe and tune its ferroelectric and Rashba properties via chemical alloying with either In or Sn; additionally, we assessed the spin-transport and ferroelectric properties of the grown substrates and worked to achieve innovative spintronics devices for in-memory computing. At Polifab, we characterize the *chemical and structural properties of thin films by in-situ X-rays and UV photoemission spectroscopy* and ex-situ X-ray diffraction.

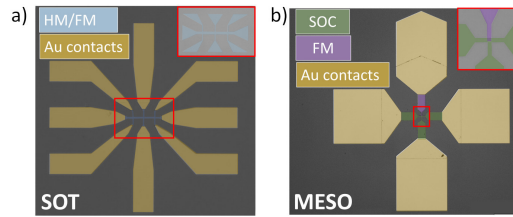
We have a well-established know-how in the advanced characterization of ferroelectric materials, that we use to electrically control magnetism and spin transport properties. Piezo-response force microscopy is especially useful to investigate the local piezoelectric/ferroelectric properties, while electrical methods (PUND, Sawyer-Tower, electro-resistance) allow to study of larger areas. Exemplarily, these techniques were fundamental for a fruitful investigation of 2D lamellas of GeSbTe<sup>3</sup> furnished by co-worker S. Cecchi from the University of Milano Bicocca (see Fig. 4).

In synergy with Polifab, these activities might bring novel spintronic devices with a significant reduction in the energy per single operation towards the attojoule regime.

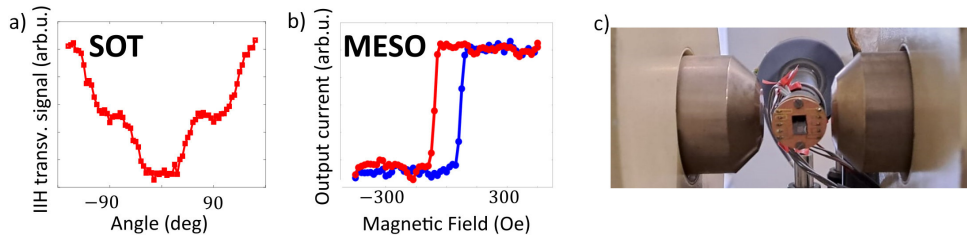
<sup>1</sup> M. Hassan *et al.*, Co/Pd-based spin-valves with perpendicular magnetic anisotropy on flexible substrates. Direct deposition vs transfer-and-bonding approaches, Applied Surface Science 635, 157740 (2023)

<sup>2</sup> S. Varotto *et al.*, Room-temperature ferroelectric switching of spin-to-charge conversion in germanium telluride, Nature Electronics 4, 740-747 (2021)

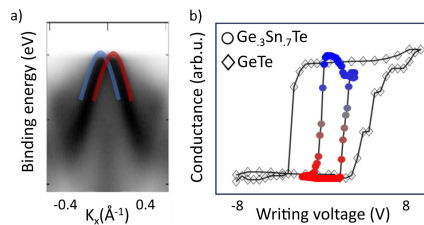
<sup>3</sup> S. Cecchi *et al.*, Thick Does the Trick: Genesis of Ferroelectricity in 2D GeTe-Rich (GeTe)<sub>m</sub>(Sb<sub>2</sub>Te<sub>3</sub>)<sub>n</sub> Lamellae, Advanced Science (2023)



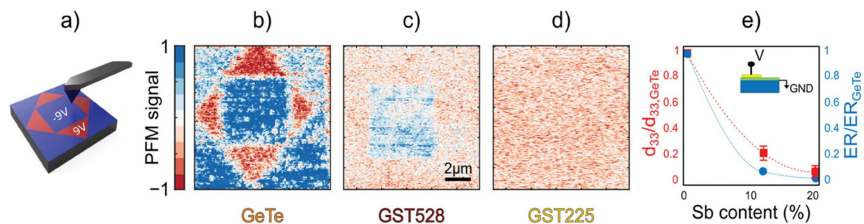
**Figure 1:** Optical images of spin-orbit torque (SOT) devices a) and magneto-electric spin-orbit (MESO) devices b). The devices were realized by optical or electron-beam lithography.



**Figure 2:** The functionalities of both spin-orbit torque Hall bars a) and magneto-electric spin-orbit devices b) were proven by measuring the electrical response versus the applied magnetic field in a custom setup developed in the Nanomagnetism lab c).



**Figure 3:** a) Rashba bands detected on the  $\text{GeSnTe}$  thin films grown by molecular beam epitaxy. The red and blue curves correspond to bands with opposite spin. b) Ferroelectric hysteresis loops of  $\text{GeTe}$  (diamonds) and  $\text{GeSnTe}$  (coloured circles) obtained by electro-resistance measurements. The coercivity is controlled with the chemical composition of thin films.



**Figure 4:** b) c) d) Ferroelectric patterning on  $\text{GeTe}$ ,  $\text{Ge}_5\text{Sb}_2\text{Te}_8$  and  $\text{Ge}_2\text{Sb}_2\text{Te}_5$  obtained by poling the sample with the conductive tip of the atomic force microscope, according to the sketch in (a). The concentration of Sb in the ternary alloy has an important impact on the ferroelectric response, as evident from the trend of the piezoelectric coefficient versus concentration reported in e).

# Control and characterization of dewetting instability in semiconductor thin films

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Dewetting is a ubiquitous phenomenon in nature; many different thin films of organic and inorganic substances (such as liquids, polymers, metals, and semiconductors) share this shape instability driven by surface tension and mass transport. In the framework of micro- and nanoelectronic devices, the dynamics of solid-state dewetting has been extensively investigated in the past 60 years: in analogy with metal films, thin semiconductors solid films break into small islands upon annealing (even at temperatures lower than the inherent melting point). These materials share an evolution guided by surface diffusion – limited kinetics: The presence of intrinsic defects in the thin layers or of ad hoc created edges is, upon annealing, the starting point of mass transport<sup>1</sup>. In this process, mass is accumulated in a thick, receding rim at the film edges, which, in turn, evolves under the action of other instabilities and finally breaks into islands featuring a scarce spatial organization and a relatively large size dispersion. The average size and interparticle distance are set by the initial thickness of the layer, which determines the period of the underlying Rayleigh-like instability. Thus, dewetting has been regarded for a long time as a major drawback, de facto limiting the further size reduction of electronic devices. Moreover, for advanced devices in microelectronics and photonics where a high level of control over size, shape, and position of the structures is the major requirement, the intrinsic randomness of the dewetted islands limits the practical exploitation of this method for micro- and nanopatterning.

Here, we show that the dewetting of thin Si or Ge films and SiGe alloys on silica films can be perfectly controlled to form a variety of monocrystalline nanostructures with high fidelity over hundreds of repetitions and extremely large

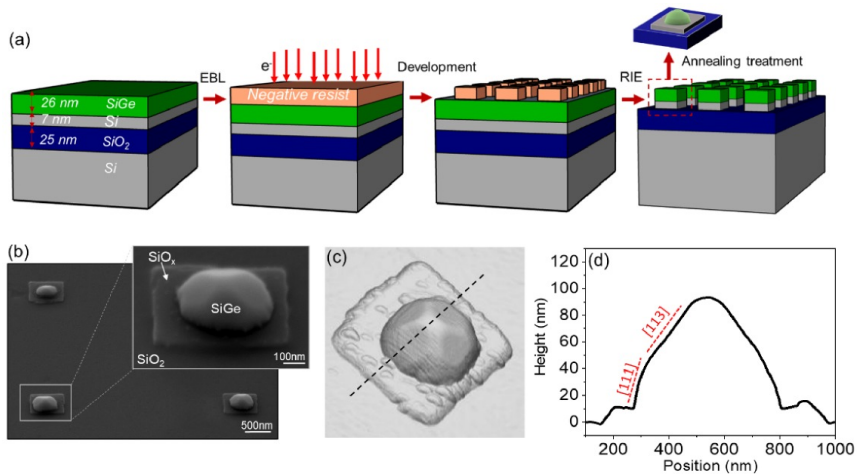
scales (5 mm) using electron-beam lithography (EBL) and reactive-ion etching (RIE). The rim evolution can be engineered to deterministically form a plethora of complex nanoarchitectures of connected islands and wires, with fluctuations of the main structural parameters as low as a few percentages. The optical properties of self-assembled dewetted nanostructures have been investigated in the near-field<sup>2</sup> and in the far-field<sup>3</sup>, highlighting how Mie resonances sustained by the dewetted nanoantennas under tilted illumination, can generate radiation patterns in different directions.

Our samples consist in pure Si (12 nm), pure Ge (25 nm) or Si<sub>0.8</sub>Ge<sub>0.2</sub> (26 nm) arrays of islands obtained through a hybrid top-down/bottom-up approach by means of templated solid state dewetting. Squared SiGe patches are fabricated by means of EBL and RIE (Fig. 1(a)). Upon annealing, they undergo a solid-state dewetting process forming self-assembled monocrystalline faceted islands, directly formed on the 25 nm layer of SiO<sub>2</sub>. The resulting resonators (Fig. 1(b)) exhibit clear faceting on their surface conductible to [001], [111] and [113] crystalline planes as confirmed also by the 3D island (Fig. 1(c)) and profile (Fig. 1(d)) reconstructed via atomic force microscopy (AFM). Fig. 2 reports the normalized DF spectra of two representative Si<sub>80</sub>Ge<sub>20</sub> dewetted islands with diameter of 330 nm and 530 nm, positioned atop a SiO<sub>2</sub> layer of thickness  $t = 25$  nm and a semi-infinite Si bulk substrate: the resonance frequency is governed by size, shape and composition and thus constitutes a precise probe of the dewetted material's homogeneity. These linear optical properties make the dewetted islands a suitable platform for light manipulation at the nanoscale and for integrated semiconductor devices.

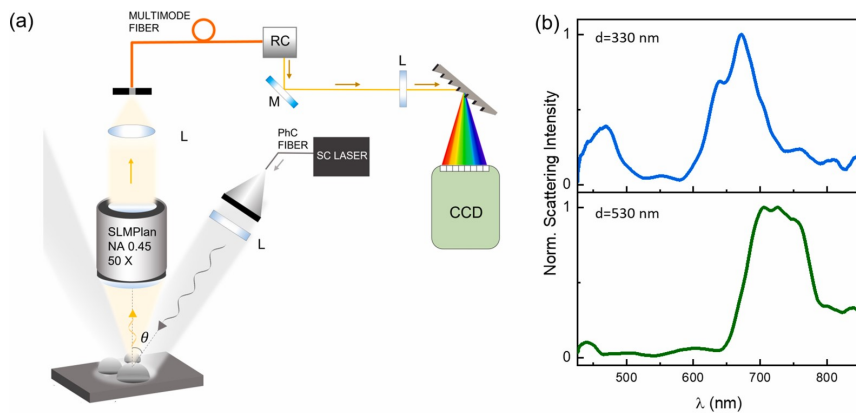
<sup>1</sup> M. Naffouti *et al.*, Complex dewetting scenarios of ultrathin silicon films for large-scale nanoarchitectures, *Science advances* 3, eaao1472 (2017)

<sup>2</sup> N. Granchi *et al.*, Near-field hyper-spectral imaging of resonant Mie modes in a dielectric island, *APL Photonics* 6.12 (2021)

<sup>3</sup> L. Fagiani *et al.*, Linear and nonlinear optical properties of dewetted SiGe islands, *Optical Materials*, 13, 100116 (2022)



**Figure 1:** (a) Schematic sequence of the fabrication steps to realize ordered dielectric dewetted nanoparticles: the SiGe film is deposited over SOI wafer, and, after the spin coating of AR-N 7520.07 negative resist, the surface is exposure by EBL. The exposed areas are developed by TMAH and the SiGe/Si layer is etched by RIE. (b) SEM characterization of SiGe resonator with crystalline facets formed on SiO<sub>x</sub> pedestal film. (c) 3D reconstructed AFM image of a single crystalline dewetted island. (d) AFM profile of the island obtained by the transversal section on (c) highlighted with the black dashed line.



**Figure 2:** Dark Field micro-spectroscopy characterization of dewetted Si<sub>80</sub>Ge<sub>20</sub> islands. (a) Sketch of the DF setup. (b) Normalized DF spectra of Si<sub>80</sub>Ge<sub>20</sub> dewetted island with  $d = 330$  nm (above) and  $d = 530$  nm (below).

# A compartmentalized microfluidic platform to investigate immune cells cross-talk in rheumatoid arthritis

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Rheumatoid Arthritis (RA) is a chronic autoimmune disease affecting joints, causing disability and reduced life expectancy<sup>1</sup>. It involves immune cell infiltration in joints, initially affecting the synovium, leading to inflammation, hyperplasia, cartilage, and bone destruction, causing severe joint swelling and pain<sup>2</sup>. The disease arises from immune system dysregulation, particularly involving interactions between macrophages and CD4<sup>+</sup> T helper type 1 (Th1) cells<sup>3</sup>. Despite its global prevalence and severe impacts, the precise causes and molecular mechanisms driving immune imbalance and joint damage are poorly understood. Understanding these complex interactions and early cause-effect relationships among immune cell types and their communication could pave the way for targeted therapies, improving patient outcomes and reducing side effects<sup>1</sup>. Organ-on-chip technology holds promise for modeling the disease, replicating human tissue functions in microsystems, and facilitating the co-culture of different immune cell types.

Here we developed a microfluidic compartmentalized platform, integrating an innovative technology, named sieving valves, which allows for precise confinement of circulating immune cells in organ-on-chip, with minimal waste of biological material. Moreover, the platform enables to co-culture different immune cell types (e.g. macrophages and Th1), having the possibility to stimulate them separately, and to assess their cross-talk at desired time points, upon opening of central communication valves. Microfluidic devices were fabricated through photolithography and soft-lithography. The design features three layers: a valve layer, featur-

ing central communication valves and sieving valves, a culture chamber layer for 3D culture of macrophages-laden fibrin gel and Th1 cell suspension (Figure 1), and an unpatterned membrane. The chambers are 150  $\mu\text{m}$  high while the microgrooves underlying the sieving valves are 5  $\mu\text{m}$ -high. Firstly, master molds were fabricated through standard two-layer photolithography of SU-8 on silicon wafers. Microfluidic devices were then produced by soft lithography of PDMS on master molds. Briefly, PDMS was poured on silicon wafers at a pre-polymer to curing agent mixing ratio of 10:1 (w/w) and cured at 65 °C for 3 h. Cast PDMS was peeled-off the molds, trimmed and through-holes were punched to obtain inlets for valve functioning (1.5 mm diameter), medium reservoirs (5 mm diameter) and gel injection ports (1 mm diameter). Finally, PDMS microfluidic layers were plasma bonded and stored until use. The device was technologically characterized, assessing sieving valve and central communication valve functioning (Figure 2). Furthermore, the platform was successfully used to co-culture human macrophages and Th1 cells. M0 macrophages were stimulated and polarized towards a pro-inflammatory state M1 (Figure 3), Th1 seeding conditions were optimized. Upon central valve opening, their interaction was assessed in terms of Th1 cells migration in the device (Figure 4).

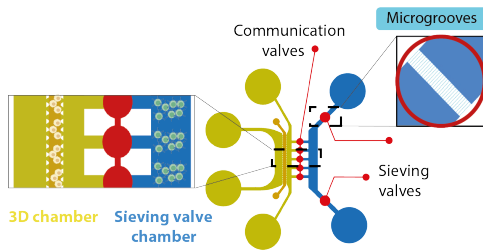
Future steps will involve integrating additional joint tissues and cells into the platform to create a more realistic in-vitro model mimicking RA joints. This will aid in further understanding the molecular mechanisms underlying RA and facilitating the discovery of novel therapeutic strategies.

<sup>1</sup> J. S. Smolen *et al.*, Rheumatoid arthritis, *Nat Rev Dis Primers*, 4, 18001, (2018)

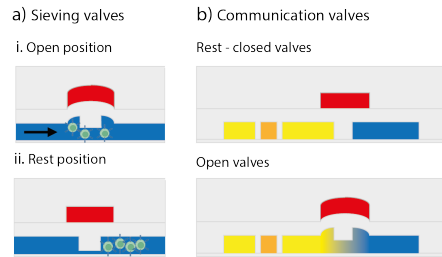
<sup>2</sup> C.Y. Wu *et al.*, Anti-Citrullinated Protein Antibodies in Patients with Rheumatoid Arthritis: Biological Effects and Mechanisms of Immunopathogenesis, *Int J Mol Sci*, 21, 4015, (2020)

<sup>3</sup> C. A. Roberts, *et al.*, The Interplay Between Monocytes/Macrophages and CD4<sup>+</sup> T Cell Subsets in Rheumatoid Arthritis, *Front. Immunol.*, Volume 6, (2015)

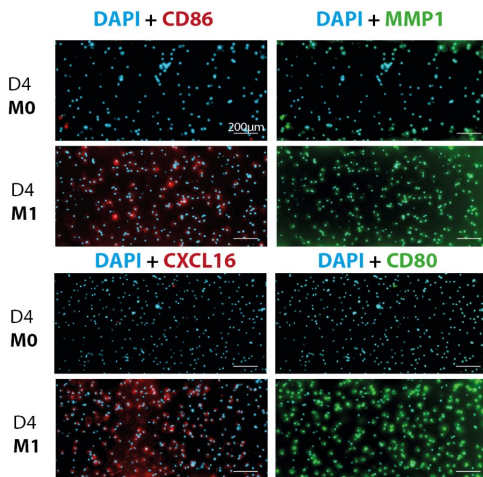




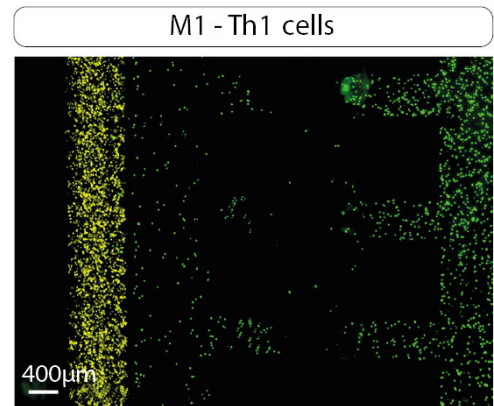
**Figure 1:** Illustration of the 3D microfluidic device with the 3D chamber hosting macrophages in fibrin gel and the Sieving valve chamber hosting Th1 cell suspension.



**Figure 2:** Schematic representation of a) sieving valve working mechanism both in open and rest position and of b) central communication valves.



**Figure 3:** Immunofluorescence staining of cell nuclei (DAPI, in light blue), CD86 (in red) and MMP1 (in green) on top and CXCL16 (in red) and CD80 (in green) on the bottom on the fourth day of culture.



**Figure 4:** Th1 cells (in green) migration towards pro-inflammatory macrophages M1 (in yellow) assessment after 4<sup>th</sup> hour upon valve opening.

# Bone marrow on chip unravelling the Mesenchymal and Hematopoietic stem cells crosstalk

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## Introduction

Bone marrow (BM) is a connective tissue where the process of haematopoiesis takes place allowing a continuous cellular renewal; in particular, hematopoietic stem cells (HSCs) have the capacity to self-renew and progressively differentiate into lineage-specific progenitors of each major immune and blood lineage<sup>1</sup>. Hematopoietic homeostasis is guaranteed by distinctive characteristics of the BM niche; first, HSCs reside in close association with different cellular populations that interact through a distal communication mediated by soluble factors. MSCs (mesenchymal stem cells) have a pivotal role in HSCs (hematopoietic stem cells) regulation since they help in preserving their stemness and the balance between self-renewal and differentiation. Such crosstalk is also involved in pathological events: as an example, BMSCs can sense peripheral tumour and, by the secretion of IL1 $\beta$ , alter HSC transcription factors activation and guide related differentiation.

Organs-on-chip (OoC) field is based on the design of microfluidic bioreactors able to finely control the cell culture environment. Such technology can be considered an effective strategy to overcome 2D culture and animal models limitations in modelling such BM niche, by reproducing a highly controlled microenvironment that preserves niche properties in terms of HSCs stemness and differentiation potential. The aim of this study was the development of a functional BM on chip to study the interaction of MSCs on HSCs in a 3D in-vitro microenvironment and at the same time create a model of endosteum niche including multipotent and differentiated MSC.

## Materials and methods

A microfluidic device was designed to include three channels hosting separately MSCs and HSCs. Channels were separated by rows of trapezoidal pillars which enabled the confinement of cell-laden hydrogel constructs while allowing a proper contact for the diffusion of nutrients and chemical factors. The device was produced by means of soft-lithography techniques. Briefly, a mould was fabricated through photolithography: 4-inch Si wafer was coated with SU8 negative photoresist and MLA100 was used to selectively expose the substrate and create the microstructures. The micropatterned wafer was used to produce multiple PDMS replicas of the device. HSCs and MSCs were isolated, suspended in a fibrin-based hydrogel and cultured in the device for 5 days. BM on chip model was tested in terms of cell viability and functionality through gene expression and immunofluorescence analysis.

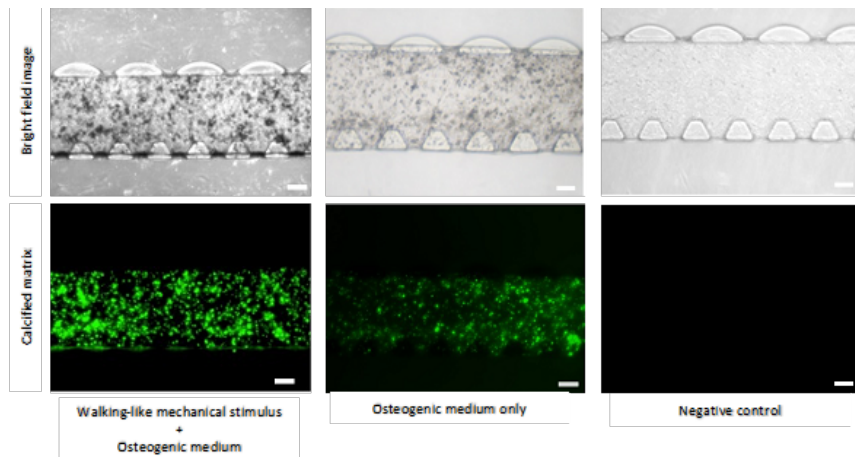
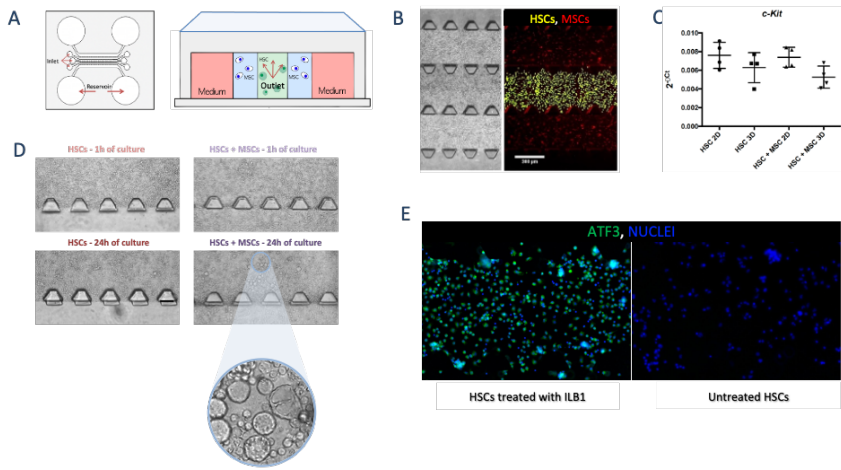
## Results

The developed device could support MSCs and HSCs 3D co-culture preserving HSCs stemness and differentiation capacity as compared to 2D controls and single HSC 3D culture. The designed model was also able to reproduce HSCs activation in response to MSCs IL1 $\beta$  tumour-induced production (Fig. 1). The device was also modified to include a pneumatic chamber for mechanical stimulation which was proved to be pivotal for the recreation of an endosteum niche with MSC differentiated toward osteoblasts (Fig. 2). Overall, the designed OoC represents a step forward in understanding mesenchymal-hematopoietic crosstalk, opening new opportunities in the BM and HSCs research.

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<sup>2</sup> G. B. Adams *et al.*, Stem cell engraftment at the endosteal niche is specified by the calcium-sensing receptor, *Nature*, vol. 439, no. 7076, pp. 599–603, (2006)

<sup>3</sup> M. Perrone *et al.*, ATF3 Reprograms the Bone Marrow Niche in Response to Early Breast Cancer Transformation, *Cancer Res*, vol. 83, no. 1, pp. 117–129, (2023)



# MOoC for the development of immunotherapies against colorectal cancer (CRC) metastases

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Colorectal cancer (CRC) is a prevalent malignancy globally, characterized by neoplastic growths in the colon, rectum, and a high propensity for metastasis<sup>1</sup>. CRC leads to various symptoms including altered bowel habits, blood in stools, weight loss, and in advanced stages, liver and lung metastases<sup>2</sup>. The complexity of CRC is attributed to the interplay of genetic, epigenetic, and environmental factors. Currently, there is no definitive cure for advanced CRC, and treatment options are often limited to palliative care. This is partly due to the absence of comprehensive CRC models that accurately mimic human colorectal tissue and its interaction with metastatic sites.

In this context, organ-on-chip technologies emerge as promising tools for CRC research. We are developing a novel microfluidic system that allows for the co-culture of colorectal organoids and lung tissue models<sup>3</sup>. This system aims to replicate the colorectal microenvironment and its interaction with common metastatic sites.

Microfluidic devices were fabricated through photolithography and soft lithography. The design (Figure 1) involves two different compartments: the first one (Figure 1(a)) features a central channel intended to host the tumoral spheroid embedded in a fibrin hydrogel, a lateral channel to provide medium and an other lateral channel intended to host the endothelial cells that should vascularize the spheroid and connect this first compartment to the second one (Figure 1(b)). Besides the vascular channel connected to the tumoral compartment, the lung compartment features a central channel where the fibrin gel hosts a hollow tubule (120  $\mu\text{m}$  diameter)

made of alveolar epithelial cells. Firstly, master molds were fabricated through standard two-layer photolithography of SU-8 on silicon wafers. Microfluidic devices were then produced by soft lithography of PDMS on master molds. Briefly, PDMS was poured on silicon wafers at a prepolymer to curing agent mixing ratio of 10:1 (w/w) and cured at 65 °C for 3 h. Cast PDMS was peeled-off the molds, trimmed and through-holes were punched to obtain cell-laden gel access ports (1 mm diameter) and medium reservoirs (5 mm diameter). The first layer was then plasma bonded to the tank layer under the microscope. Finally, PDMS microfluidic layers were plasma bonded to glass cover slides and stored until use.

The microfluidic platform was initially divided into two compartments for independent study: 1) the development of a consistent, healthy alveolar epithelial model (Figure 2), 2) the formation of a colorectal cancer spheroid (Figure 3), and 3) the vascularization process fostered by endothelial cells in response to chemical stimuli (Figure 4).

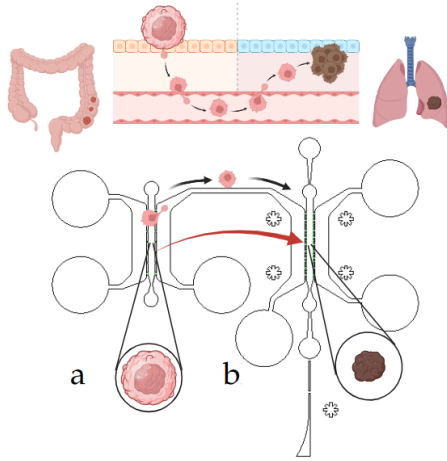
Future steps will involve co-culturing cancer spheroids with endothelial cells to model a vascularized tumor. Further, the platform will be validated by studying the migration process of cancer cells from the colorectal compartment towards the healthy lung tissue.

In the end, by integrating multiple cell types and replicating the tumor microenvironment, this innovative platform promises to advance CRC research, offering new insights and aiding in the development of more effective treatments.

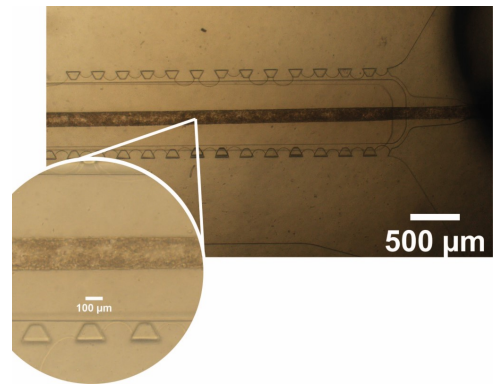
<sup>1</sup> P. Beckers *et al.*, Pulmonary metastasectomy in colorectal carcinoma. *Journal of Thoracic Disease* vol. 13 2628–2635, (2021)

<sup>2</sup> G. Golshani *et al.*, Advances in immunotherapy for colorectal cancer: a review. *Therapeutic Advances in Gastroenterology* vol. 13 (2020)

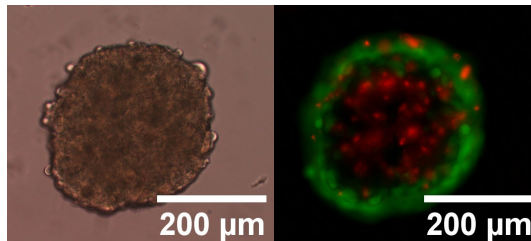
<sup>3</sup> Q. Wu *et al.*, Organ-on-a-chip: Recent breakthroughs and future prospects. *BioMedical Engineering Online* vol. 19 (2020)



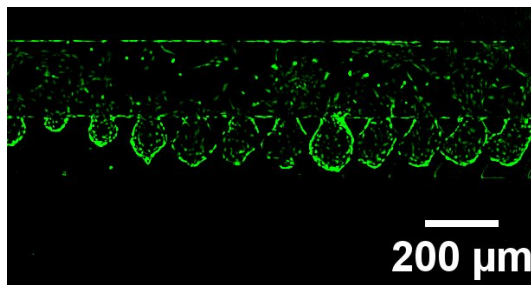
**Figure 1:** Illustration of the microfluidic device designed to model the metastatic process, featuring two compartments: the left one (a) housing a colorectal cancer spheroid, and the right one (b) resembling a healthy lung targeted by the metastasis.



**Figure 2:** Brightfield image of the lung compartment showing a circular hollow channel within a fibrin gel, which is lined by alveolar epithelial cells.



**Figure 3:** Brightfield (left) and live/dead assay (right) showing the colorectal spheroid before the injection.



**Figure 4:** Fluorescent image (using Alexa Fluor™ 488) displaying endothelial cells sprouting from a lateral endothelial channel into a fibrin gel, culminating in the formation of new vessels.

# Development of an Organ-on-Chip based model to study the role of mechanical stimuli on cartilage development and pathological onset

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Biomechanics plays a pivotal role in articular cartilage development, maturation and pathophysiology. In early embryo, mechanical factors regulate chondrogenic differentiation of stem cells and foster limb morphogenesis. In adult cartilage, mechanical and shear forces resulting from joint loading regulate homeostatic balance between matrix protein deposition and remodeling<sup>1</sup>. However, abnormal loading could contribute to the initiation of cartilage degeneration and the onset of pathologies such as osteoarthritis (OA)<sup>2</sup>. In this scenario, this work aims to design and develop an organ-on-chip (OoC) platform to investigate the role of mechanical stimuli in cartilage embryogenesis and to elucidate their involvement in the onset of OA-induced cartilage degradation.

The OoC platform comprises two layers, one on the top hosting three cell culture chambers and one on the bottom designed to provide the mechanical stimulation (Figure 1a). Each culture chamber comprises a central channel to host the 3D cell model and two lateral channels for medium supply (Figure 1b). The chambers are 150  $\mu\text{m}$  high. The three channels are delimited by two rows of T-shaped hanging posts (Figure 1c). The gap beneath them dictates the strain magnitude transferred to the cellular model (i.e., 14  $\mu\text{m}$  for 10% compression, 43  $\mu\text{m}$  for 30% compression). The bottom layer comprises three rectangular chambers 50  $\mu\text{m}$  high, connected by single channel. To fabricate the microfluidic platform, master molds of the two layers were firstly fabricated through photolithography of SU-8 on silicon wafers. Each layer was then produced by soft lithography technique. Briefly, PDMS pre-polymer was mixed to curing agent in a ratio of 10:1 (w/w), poured on the silicon wafer and

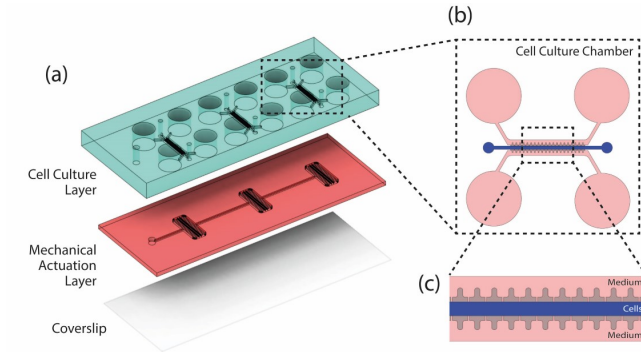
cured at 65 °C for 2 h. The PDMS was peeled-off from the molds and through-holes were punched to obtain culture chamber reservoirs (5 mm diameter), cell channel inlets/outlets (1 mm diameter) and access port for the mechanical stimulation (1.5 mm diameter). Finally, the top layer was plasma bonded to the bottom layer, and the PDMS layers were plasma bonded to glass cover-slide.

Following a previously established protocol<sup>2</sup>, the microfluidic platform was successfully used to culture human primary articular chondrocytes (hACs) in PEG under static condition for 14 days and then mechanically stimulated for the next 7 days under 30% compression to induce OA onset. Preliminary results with RT-qPCR confirm the generation of OA cartilage model both looking at the expression of OA-relevant genes and recently OA-correlated genes (Figure 2). To study the role of mechanical cues in cartilage embryogenesis, the same device was also used to culture human primary mesenchymal stem cells (hMSCs) in fibrin gel under dynamic condition (i.e., mechanically stimulated, with both 10% and 30% compression) either with or without the support of biochemical cues for 14 days. As evidence by morphological analysis, imposing hMSC-based microtissue to compression led to different morphological organization, similarly to that of AC embedded in gel (Figure 3).

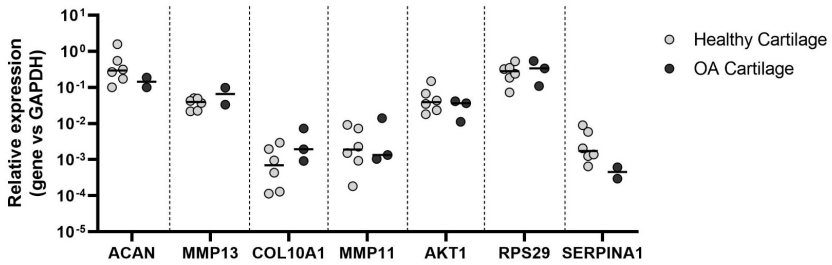
Next steps concern the deepening of the relationship between mechanical overload and OA onset also at the level of signaling pathways activated during the pathogenesis and furthermore the chondrogenic differentiation of the hMSCs mesenchymal will be verified with flow cytometry and genes expression analysis.

<sup>1</sup> D.J. Responde *et al.*, Biomechanics-driven chondrogenesis: from embryo to adult. The FASEB Journal, 26(9), 3614–3624. (2012)

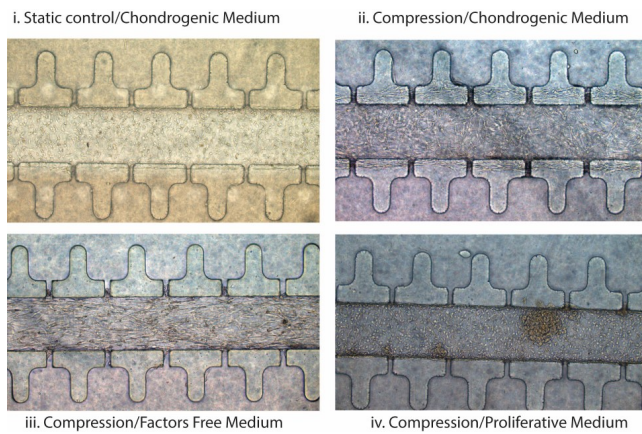
<sup>2</sup> P. Occhetta *et al.*, Hyperphysiological compression of articular cartilage induces an osteoarthritic phenotype in a cartilage-on-a-chip model. Nature Biomedical Engineering, 3(7), 545–557, (2019)



**Figure 1:** (a) Exploded view of the microfluidic platform. (b) Layout of the cell culture chamber in the device. (c) Zoom on the channels and the hanging posts.



**Figure 2:** Results of RT-qPCR on OA samples vs healthy controls.



**Figure 3:** Morphological analysis of mechanically stimulated hMSCs vs static control, with and without biochemical cues.

# Design and development of a mechanically active beating heart-on-chip integrating force-sensing capabilities

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Organ-on-chip aims at mimicking the native cellular milieu by recapitulating fundamental organ-specific dynamic conditions in microfluidic cell culture devices. In the case of heart-on-a-chip, the goal is to create advanced in vitro platforms that foster the development and maturation of cardiac tissue. These devices have the potential to function as a human model for predicting drug toxicity. In doing so, they could contribute to reducing the risk of drug-induced ventricular arrhythmia associated with new compounds<sup>1</sup>.

In our lab, a mechanically active heart-on-chip with electrical readouts has been extensively used and validated<sup>1</sup>. However, for more advanced insights, a chip system integrating electrophysiology and contractility readouts is highly desired. This enhancement would elevate the heart-on-chip's potential in predicting cardiac toxicity, thereby mitigating the persistent occurrence of adverse reactions in later stages of the drug discovery pipeline<sup>2</sup>. For this project, we have developed a set of designs to integrate force-contractile readouts in our platform using two parallel posts to foster the formation of an engineered heart tissue (EHT)<sup>3</sup>.

In Figure 1 it is shown the working principle behind the use of EHT to measure the contractile force in our chips. Briefly, when cardiomyocytes (CMs) form a muscle bundle around two posts and contracts it induces a bending that can be calculated through tracking softwares. Then, the contractile force is computed through beam theory.

Microfluidics devices were fabricated through

photolithography and soft-lithography. The chambers are 150  $\mu\text{m}$  high and the posts and pillars separating the media and central channel are 100  $\mu\text{m}$  high. Firstly, master molds were fabricated through standard two-layer photolithography of SU-8 on silicon wafers. Microfluidic devices were then produced by soft lithography of PDMS on master molds. Briefly, PDMS was poured on silicon wafers at a pre-polymer to curing agent mixing ratio of 10:1 (w/w) and cured at 65  $^{\circ}\text{C}$  for 3 h. Casted PDMS was peeled-off the molds, trimmed and through-holes were punched to create the medium reservoirs (5 mm diameter) and inlet and outlet seeding site (1 mm diameter). Finally, PDMS microfluidic layers were firstly plasma bonded to a mechanical actuation layer and then to a glass cover slides.

The heart-on-chip successfully promoted the formation of an EHT around the posts after a previous BSA 2% (w/v) coating to reduce protein adsorption and cell attachment to the walls (Figure 2). This step is critical to promote hydrogel compaction around the posts. To assess the cell viability within the muscle bundle a Live/Dead assay was performed showing a high cell survivability after 7 days of culture (Figure 3). Proper cell alignment and cardiac biomarker expression were evaluated through immunostaining imaging (Figure 4).

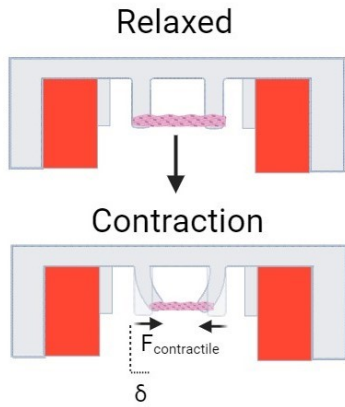
As future steps, it will be necessary to correctly detect the displacement of the posts after cell contraction to measure their contractile force. Furthermore, it would be of great interest to use the developed platform to study diseases that leads to muscle weakness, such as Duchenne muscular dystrophy, and compare the results with healthy CMs.

<sup>1</sup> R. Visone *et al.*, Micro-electrode channel guide ( $\mu\text{ECG}$ ) technology: an online method for continuous electrical recording in a human beating heart-on-chip. *Biofabrication*, 13(3), 035026, (2021)

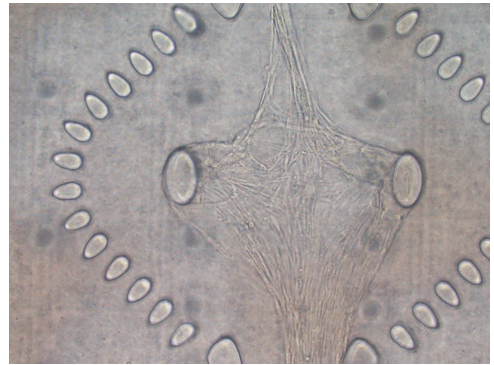
<sup>2</sup> J. Kim *et al.*, In situ biosensing technologies for an organ-on-a-chip. *Biofabrication*, 15(4), 042002, (2023)

<sup>3</sup> K. Ronaldson-Bouchard *et al.*, Engineering of human cardiac muscle electromechanically matured to an adult-like phenotype. *Nature protocols*, 14(10), 2781-2817, (2019)

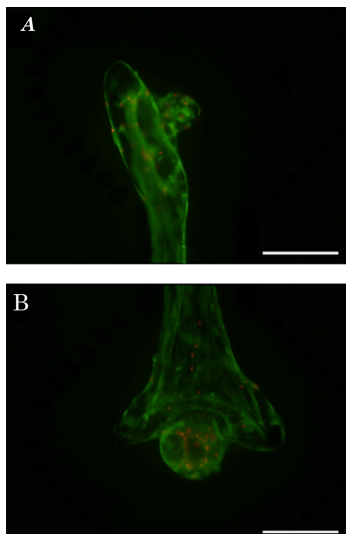




**Figure 1:** Schematic sketch showing the working principle of using an EHT for contractile force readouts inside a heart-on-chip.

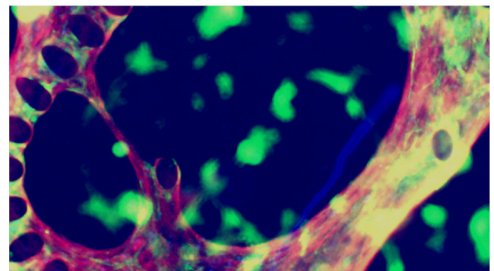


**Figure 2:** EHT formed around two inside the developed microfluidic heart-on-chip.



**Figure 3:** Live/Dead images showing live (in green) and dead (in red) CMs in the chip after 7 days of culture.

DAPI - TROPONIN I -  $\alpha$ SMA



**Figure 4:** Immunofluorescence staining of an EHT in the microfluidic platform, showing nuclei (in blue), Troponin I (in red) and  $\alpha$  smooth muscle actinin (in green) after 7 days of culture.

# MEMNONE-Micro Electro Mechanical System Nonlinearities Exploitation for Filtering and Sensing Applications

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Nonlinear phenomena in MEMS devices have been largely investigated, nevertheless exploiting nonlinearities for practical applications is still a major challenge. Within this setting, we leverage innovative Model Order Reduction (MOR) techniques we recently developed to design and optimize MEMS structures to operate in a fully nonlinear regime. The outcome of this project is to design and model an innovative class of resonators that exploits nonlinear phenomena for enhanced performance.

During the last year, the MEMNONE group designed a MEMS ring resonator. In Figure 1, a schematic view of the mechanical structure is reported. An outer ring is connected through arched suspension springs to a central circular anchor. At a fixed gap with respect to the outer ring, a set of electrodes is also designed to allow the electrostatic actuation/readout. Additional auxiliary fixed structure (light green in Figure 2) are designed in between the different arched suspension springs to guarantee a better uniformity in terms of gaps during the fabrication process, but do not play any mechanical role.

In operation, the ring resonator is kept in oscillation according to one of the  $0^\circ$ -theta or  $45^\circ$ -theta modes reported in Figure 2. In particular, the mechanical structure will be kept at a DC voltage, while an AC-voltage will be applied to the driving electrodes to provide an electrostatic actuation. The natural frequencies computed in COMSOL Multiphysics by considering the nominal geometric dimensions reported in Figure 1 read 67093 Hz and 71257 Hz for the  $0^\circ$ -theta and  $45^\circ$ -theta modes, respectively. The discrepancy between the natural frequencies of the two

modes ideally identical is due to the anisotropic behavior of the adopted single-crystal silicon.

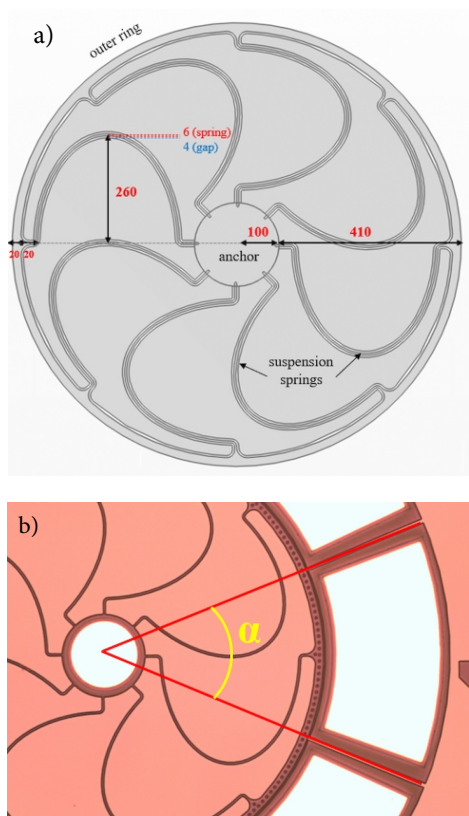
In the literature<sup>1</sup>, it has been shown that ring resonators like the one reported in Figure 1, show an intriguing nonlinear behavior. The intrinsic mechanical coupling between the two modes shown in Figure 2, leads indeed to an autoparametric resonance that can be exploited in the functioning of the device as high-performance gyroscope. The goal of the MEMNONE group is then to fabricate the device in order to experimentally prove such complex nonlinear behavior already demonstrated numerically thanks to the reduced order models.<sup>2,3</sup>

During the last months, the fabrication of the device took the vast majority of the MEMNONE group effort. The process is based on silicon-on-insulator (SOI) having a layer of metal on top to form ohmic contacts. The contacts are defined through a combination of photolithography and wet-etch of the top metal layer (Figure 3a). The MEMS structure is defined through a second lithography, however due to the large size of the structure and the small features involved, adhesion issues might arise (Figure 3b). The photorealist preparation step has been further addressed and test structures have been designed to this purpose to obtain good adhesion and definition (Figure 3c). Finally, the etching of silicon is performed using reactive ion etching. First attempts showed excessive isotropic etching, (Figure 3d), the optimization of this step is currently ongoing, but preliminary results show better anisotropy (Figure 3e). After reactive ion etching, the device is finally released using vapor HF to fully suspend the moving elements.

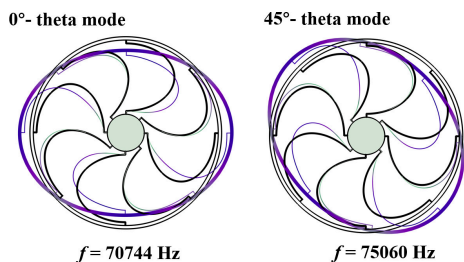
<sup>1</sup> G. Gobat *et al.*, Reduced Order Modeling of Nonlinear Vibrating Multiphysics Microstructures with Deep Learning-Based Approaches. *Sensors*, 23(6):3001, (2023)

<sup>2</sup> G. Gobat *et al.*, Modelling the Periodic Response of Micro-Electromechanical Systems through Deep Learning-Based Approaches. *Actuators*, 12(7):278, (2023)

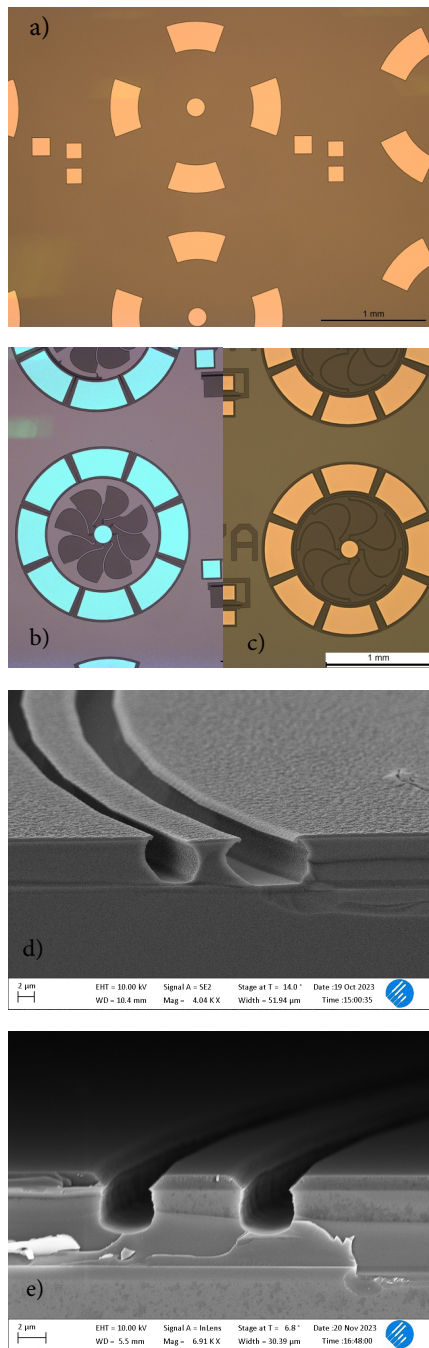
<sup>3</sup> A. Frangi *et al.*, Reduced order modelling of the non-linear stiffness in MEMS resonators, *International Journal of Non-Linear Mechanics*, 116:211-218, (2019)



**Figure 1:** (a) Schematic view of the proposed MEMS ring resonator. Geometric dimensions are reported in microns. (b) Close-up view of one electrode located outside the outer ring for the capacitive actuation/readout.



**Figure 2:** Modal shape-functions of the 0°-theta and 45°-theta modes together with their natural frequencies. The contour of the displacement field is shown in color.



**Figure 3:** Fabrication steps: (a) electrodes patterning (b-c) MEMS lithography, in b part of structure is missing due to adhesion problems of the photoresist (d-e) reactive Ion Etching, showing isotropic (d) and anisotropic etching (e).



# Publications & Patents



# Journals

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A word cloud centered around 'Politecnico Polifab Milano', featuring various terms related to technology, engineering, and research. The words are in different sizes and shades of blue.

spintronics nanotechnology  
Process-flow groups MEMS research  
micromechanics Lithography Plasma  
optics Politecnico european microfluidics  
know-how micro facilities wafer  
equipment nanomagnetism Etching support processes industries  
Vacuum Thin-film laboratories electronics  
university users partners chip devices photonics Milano innovative Silicon  
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