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Development of high pressure and high temperature injection modules for 4D µCT in synchrotron environment.

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Abstract

Mogno Beamline is a high-complexity equipment which performs both Nano and Micro computed tomography (CT) scans in Sirius, the Brazilian Synchrotron at CNPEM (Brazilian Center for Research in Energy and Materials). This Beamline works with a conic X-ray beam, which allows for zoom-in tomography of samples from several areas ranging from geological to biological samples. In the experimental hutch there are two experimental stations, the Nano Station (which allows for image resolution ranging from 130 nm to 12 µm) and the Micro Station (0.5 to 52 µm of image resolution). Currently, the Micro Station is operational with a high throughput setup for dry sample exchanging with a robotic arm and magazine system, but a second experimental setup is under development, which will regard in situ porous media flow imaging under high pressure (up to 1000 bar) and high temperature (90 °C), conditions similar to those found in pre-salt oil reservoirs. With the founding of Petrobras and Equinor, this project aims to advance the study of flow behaviour in porous media by designing equipment with precision engineering techniques to guarantee precise sample positioning and stabilization, together with high-speed data gathering and post processing. This paper aims to display the main considerations needed to perform the before mentioned high complexity experiment, together with the preliminary mechanic and control design results and next steps.

Engineering, Mechatronics, Pressure, Temperature

1. Introduction

The Sirius/LNLS MOGNO (Micro and Nano tomography) beamline is dedicated to performing three-dimensional tomography experiments with synchrotron radiation, ranging from micrometric to nanometric regime. MOGNO beamline [1] is mainly divided into 3 different stations: nano, high-throughput and pre-salt. In 2024, the line incorporated a new experimental infrastructure, the high-throughput micro station, designed for three-dimensional study of representative samples of carbonate rocks from Brazilian pre-salt reservoirs. The third station, called pre-salt, is under development by the LNLS/Sirius engineering team.

The high-throughput station is designed to offer the users a spatial resolution of 500 nm and temporal resolution of seconds for tomographies [2-3], but with a simpler setup for dry samples. The current setup is under technical commissioning to verify the design parameters. Although, with this setup it was already possible to validate gantry motion capabilities (Zoom) and beamline systems operations (detectors and optics) that will be used for pre-salt setup as well.

The development of pre-salt end station aims to expand MOGNO's current experimental capabilities, offering a controlled environment, maintaining high mechanical stability for the execution of CT scans while allowing in-situ tests under controlled pressure and temperature conditions, mimicking pre-salt ones, enabling the direct observation of dynamic processes, such as fluid injection (water, oil or CO_2) and microstructural changes in geological materials. The high-energy radiation spectrum of the MOGNO beamline ensures the penetration needed to image samples with typical thicknesses up to 38.1 mm (1.5 in).

From a constructive point of view, the pre-salt micro station combines structural rigidity, sub-micrometric positioning accuracy and thermal stability, essential for tomography experiments with the fluids of interest mentioned. The mechanical system is designed to ensure fine alignment between the sample and the beam, minimizing vibrational noise and ensuring stability during prolonged acquisitions. The experimental suite includes a sample handling module (section 3.1), a pressurization module (section 3.5), fluid storage modules (section 3.4), and a sample conditioning module (section 3.3), allowing the capture of images (2D projections that, combined, make up 3D CT scans) at rates up to the kilohertz scale (section 4).

2. Main Challenges

A primary challenge of micro station pre-salt is emulating high-pressure and high-temperature flow experiments setups, typically confined to laboratory environments on benchtop CT scanners [4], into the highly constrained and vibration-sensitive setting of a synchrotron beamline. Tomographic imaging requires high mechanical stability, minimal dynamic disturbances during long acquisition times and, in our case, high acquisition speed to enable 4D imaging. Reconciling these requirements with the presence of pressurized fluids, heating systems, and auxiliary infrastructure near the sample represents a fundamental design constraint.

A second major challenge arises from the intrinsic requirement of tomographic measurements, that is the rotation between X-Ray source and sample. As in a beamline it is not possible to rotate the X-Ray source, so the sample must rotate. This configuration imposes severe constraints on the transfer of pressurized and heated fluids between static infrastructure and the rotating sample environment. Unlike electrical signals, which can be easily handled through slip-ring technologies, fluid transfer under high pressure and temperature demands robust mechanical interfaces capable of maintaining leak-tightness, chemical compatibility, and thermal stability throughout continuous rotation. The limited availability of off-the-shelf solutions suitable for these conditions demands custom approaches and careful integration between mechanical, thermal, and fluidic design domains.

Beyond hardware-related challenges, the micro station pre-salt introduces a highly complex experimental workflow that spans through sample preparation, fluid conditioning, system assembly, transport, and beamline operation. Special fluids, like live oil and carbonated water, must remain pressurized and thermally conditioned throughout the entire process, from laboratory preparation to in-situ tomography, without interruption or need for recombination. This requirement significantly increases the operational complexity, as it demands coordinated procedures, active thermal control during transportation of samples and accumulators, and strict sequencing of experimental steps.

3. Pre-salt Micro Station - Mechanical Overview

The mechanical architecture of the pre-salt micro-station was designed to manage the high complexity associated with the proposed experiment. To address all the requirements in a structured and robust way, the mechanical system adopts modular architecture, in which the overall functionality is distributed across well-defined mechanical modules with clear interfaces.

From development and operational perspective, the modular structure facilitated parallel design, and soon will facilitate independent assembly, testing and incremental commissioning of the station. Moreover, this approach improves maintainability, supports future upgrades and enhances operational flexibility, making modularization a key system-level strategy for the reliable implementation of complex in-situ experiments at the MOGNO beamline.

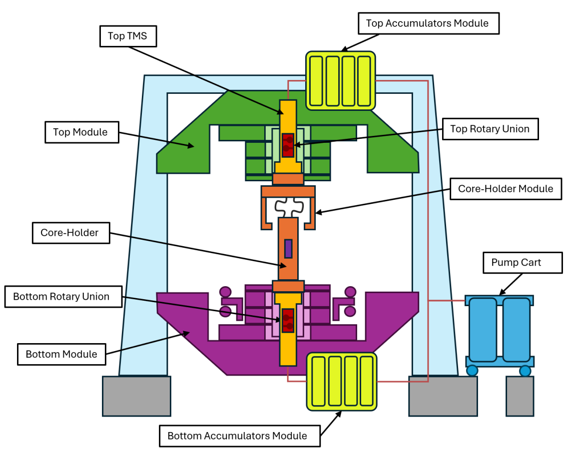

Figure 1. Pre-salt micro-station mechanical modular architecture, with module critical components highlighted.

To address the challenges outlined above, the mechanical structure of the pre-salt micro-station is organized into a set of modules as shown in Figure 1. The modules were splited in such a way to aggregate similar functions, decrease mechanical interfaces and allow a clever workflow of assembly and maintenance. The following sections provide a concise overview of the main mechanical modules and their respective functions, capacities and challenges within the overall system architecture.

3.1. Bottom Module

The Bottom Module is responsible for the precise positioning and dynamic stability of the sample during rotation, as well as for the thermal conditioning of the fluids entering or leaving the core-holder through the lower interface. This module integrates the linear positioning stages (XL and X-Z), the rotational stages (primary and auxiliary), and the thermal management system (TMS), all represented in Figure 2, forming the mechanical and functional foundation of the micro-station.

The main engineering challenge of this module lies in reconciling sub-micrometric stability (≈250 nm) and fast rotation speeds (≈1-4Hz) with the need to accommodate significant rotating masses, high-pressure fluidic interfaces, and additional degrees of freedom for region-of-interest (ROI) selection. To guarantee system target performance, an error budget was constructed with the major sources of errors being floor vibrations propagations to sample, rotary stage runout errors and rotation induced deformations and offsets. As outcome, it was identified that the major contributions to sample position error are rotation induced deformations (≈2µm) and rotary stage runout errors (≈400nm).

To address and mitigate these errors the proposed design has a separation between the primary and auxiliary rotation stages (Ry), together with the implementation of an auto-balancing system, is essential to mitigate dynamic imbalance effects introduced by sample offset during high-resolution tomographic experiments with zoom in ROI. Also, a metrology system was design to measure the primary rotation stage position in relation to the static world, in every DoF apart from the rotation itself using the multiprobe method. Its data will be used to feed the tomography reconstruction algorithm decreasing the uncertainties to levels below the target (≈200nm).

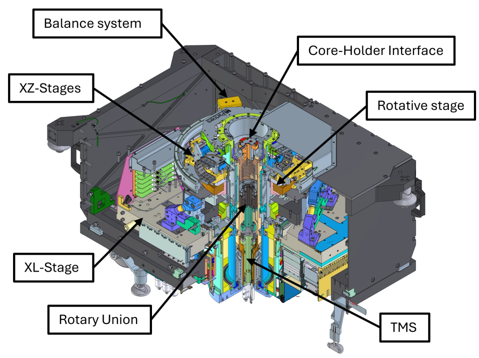

Figure 2. Bottom module global design with principal submodules highlighted.

3.2. Top Module

The Top Module is positioned above the sample and primarily supports and decouples auxiliary systems required for experiment execution, such as upper accumulators, pressure sensors and thermal management components, thereby reducing the effective inertia carried by Bottom Module primary rotation stage. In addition, this module ensures proper heating and thermal stability of the fluids entering the upper section of the core-holder, the main injection course.

From a mechanical perspective, the main challenge is the compatibility between dynamic decoupling and kinematic tracking, as the Top Module must follow the longitudinal motion of the Bottom Module without introducing parasitic loads on the core-holder. The integration of the thermal core, electrical interfaces between static and rotating domains, and thermal insulation elements makes this module have similar complexities with the bottom one, enabling the use of standard components and assemblies, decreasing design and validation efforts.

3.3. Core-Holder Module

The Core-Holder Module constitutes the experimental core of the micro-station, enabling the controlled reproduction of pressure and temperature conditions representative of pre-salt reservoirs in porous rock samples. This module, shown in Figure 3, comprises the pressure vessel (core-holder), top and bottom interface manifolds, sensor manifold to support absolute and differential pressure transducers, heat exchangers and oven, and the mechanical decoupling system based on helical tubes.

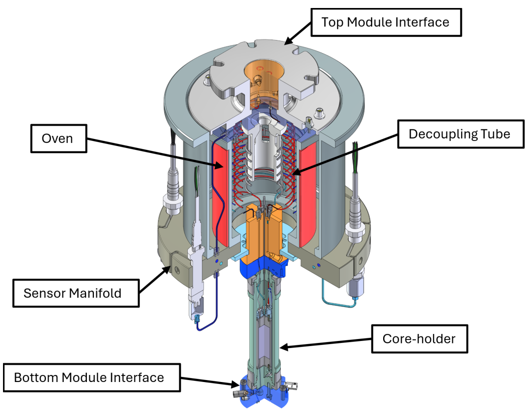

Figure 3. Core-Holder module global design with principal components highlighted.

The primary mechanical challenge is to ensure structural integrity, leak-tightness, and thermal stability under operating conditions (up to 1000 bar and 90 °C), while minimizing the transmission of mechanical loads and dynamic disturbances to the sample and bottom module during rotation. The design ensures high thermal spatial and temporal stability (±0.1 °C) around the sample, to avoid thermal induced flows that could mask the real phenomena. The use of helical pigtails and dedicated heat exchangers provides mechanical compliance and thermal conditioning, effectively decoupling the sensors mass from the sample rotating system and preserving tomographic image quality.

3.4. Accumulators Module

The Accumulators Module is designed to store and supply the fluids used in flow experiments under strictly controlled pressure and temperature conditions. Each accumulator is manufactured from titanium, typically has a volume of 500 ml and can operate independently, allowing different fluids to be used throughout the experimental sequence.

From mechanical and thermal engineering standpoints, the key challenges are individualized thermal control and operational safety. Each accumulator is therefore integrated into a dedicated heating enclosure equipped with thermal blankets, redundant temperature and pressure sensors, relief valves, and methane gas detection devices. The design ensures high thermal stability while maintaining safe-to-touch external surface temperatures, even during operation at 90 °C.

Also, due to the use of fluids with potentially flammable gases dissolved (like live oil with methane gas), the accumulator module requires dedicated exhaust systems. Active exhaust using a sensitive methane gas sensor and a high flow rate fan, ensures the safe removal of leaked or vented gases in confined environment of the oven, mitigating risks related to flammability and explosion.

3.5. Pump Cart

The Pump Cart is the control core of the injection experiment, being responsible for system pressurization, for controlling fluid injections and production during experiments. It integrates high-pressure, pulse-free pumps capable of operating in either pressure- or flow-controlled modes, automated valves, all communicating through a local double controller architecture.

The main challenge of this module lies in the integration of complex fluidic architectures, distributed thermal control and stringent safety requirements, as the Pump Cart also manages thermal control for the other subsystems of the station. A segregated control architecture, combining PLC-based control for pressure and flow with a dedicated FPGA-based system for temperature management, ensures deterministic behavior, data traceability, and operational robustness, which are critical for in-situ experiments under reservoir-representative conditions. More on this architecture will be presented in Section 4.

3.6. Auxiliar Sub-Systems

The auxiliary systems of the pre-salt micro-station include a set of transport carts designed to ensure safe handling, mobility, and operational continuity of the experimental infrastructure between laboratory facilities and the MOGNO beamline. These subsystems comprise the module transport cart and the core-holder transport cart, which together support the logistics, fluid management, and experimental readiness of the station.

A key design requirement common to all carts is the ability to preserve thermodynamic and mechanical integrity during transport. Following saturation, leak testing, and thermal conditioning in the laboratory, cooling and reheating cycles and pressure cycles are avoided to prevent fluid property variations and to reduce the likelihood of leakage during re-pressurization and reheating. For this reason, carts are conceived as active subsystems, incorporating temperature controllers, cooling circuits for thermal shields, and dedicated exhaust systems for safe handling of hazardous fluids.

The module transport cart enables the relocation of the top and bottom modules for maintenance, integration, and replacement activities, while providing adequate structural support and reproducible mechanical interfaces with the beamline and gantry.

The core-holder transport cart allows the complete sample assembly to be moved as a single unit, maintaining pressure and temperature throughout the transport path and eliminating the need for fluid reconditioning at the beamline. Although the integration of active thermal control and safety features increases system complexity, this approach significantly enhances operational robustness and beamtime efficiency once it minimizes the waiting time while X-ray beam is available.

4. Pre-salt Micro Station - Software Overview

To guarantee that all the capabilities designed into the mechanical modules previously described are easily available, the software for the pre-salt station is re-modularized by experiment functionalities (sample positioning, injection, pressurization, heating) and beamline performance exigencies (Equipment Protection Systems - EPS, Personnel Protection Systems – PPS, data compatibility). These are to be provided through a series of Graphical User Interfaces (GUIs) and monitored by the laboratory interlock systems.

GUIs are hosted in the application layer and made based on views of the injection diagram for the gantry setup, partially presented in Figure 4. To try to guarantee all experiment parameters will be thoroughly set and reviewed by the beamline staff, separate GUIs will be made to control injection pressure, flow limits, heating and tomography configuration and startup. Also, during the injection experiment, all the parameters are displayed in a monitoring GUI, to allow for easy problem identification. All variables monitored through GUIs are to be implemented in the service layer as EPICs process variables (PVs), gathered directly from the controllers allocated into the Pump Cart and hutch. In case of inoffensive behaviors, such as slight temperature or pressure variations (deemed acceptable by the user), a series of pop ups with different alarms guide the user through the equipment control without needing to interrupt the experiment.

While the experiment is ongoing, high-speed data acquisition is performed in the service layer, to be later made available fully compiled and ready for post processing to the user.

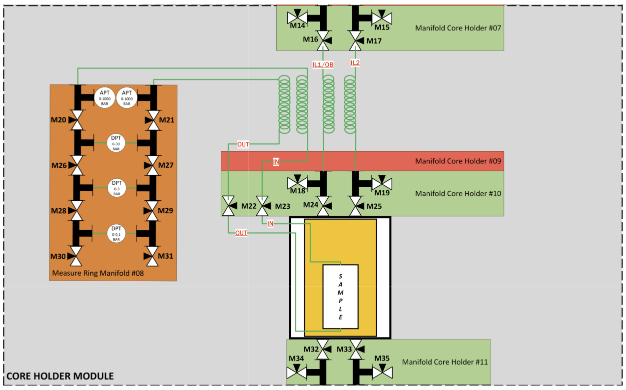

Figure 4. Core-holder injection diagram of the pre-salt experimental setup, with valves, manifolds, absolute pressure transducers (APT), differential pressure transducers (DPT) and sample.

In parallel, there is a complete equivalent system reproduced into the beamline safety interlock system, monitored by a central safety programmable logic controller (PLC). This works with direct signal readings in the device layer to guarantee fast and reliable response independently of network and third parties. The operation parameters and limits are defined to guarantee the wellbeing of humans (PPS) and integrity of the setup (EPS), disregarding experiment configuration. As the Pump Cart is designed to operate both in the beamline and the support laboratory, its internal PLC has a secondary safety monitor loop, which communicates with the main interlock PLC through Process Field Network (PROFINET) Protocol when operating outside the hutch.

All software is designed to work as stand alone, so the scientific users don't need to ponder beamline integrity and maintenance staff can monitor any equipment without needing for experiments to be performed.

5. Next Steps

The next development phase of the pre-salt micro-station focuses on the transition from detailed design to system-level integration, validation, and incremental commissioning. Ongoing activities include the completion of detailed mechanical designs, as shown in Figure 5, procurement of remaining custom components, and the assembly of individual modules, followed by subsystem-level verification and validation testing.

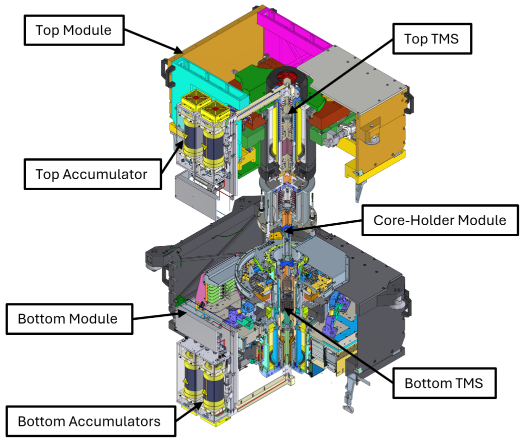

Figure 5. Global design of the beamline setup for micro station pre-salt.

A key priority is the progressive integration of mechanical, thermal, and fluidic subsystems, with particular emphasis on interfaces between static and rotating domains and on the validation of pressurized fluid handling under representative operating conditions. These activities will be supported by staged testing campaigns, initially conducted in laboratory environments and subsequently extended to beamline-compatible configurations.

In parallel, efforts will concentrate on the consolidation of operational workflows, including sample preparation, transport, system assembly, and experimental execution. This includes the refinement of procedures to ensure continuity of pressure and temperature conditions from laboratory preparation through in-situ synchrotron measurements, as well as the validation of safety and interlock strategies.

Finally, the system will undergo incremental commissioning, gradually increasing experimental complexity in terms of pressure, temperature, and fluid composition. This phased approach is intended to mitigate technical risks, validate system performance, and establish a robust experimental framework, ultimately enabling reliable in-situ tomography experiments under reservoir-representative conditions at the MOGNO beamline.

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