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AUTOMATION SOLUTIONS AND PROTOTYPES FOR THE X-RAY TOMOGRAPHY BEAMLINE OF SIRIUS, THE NEW BRAZILIAN SYNCHROTRON LIGHT SOURCE

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Abstract

Brazil is building Sirius, the new Brazilian synchrotron light source which will be the largest scientific infrastructure ever built in Brazil and one of the world's first 4th generation light laboratory. Mogno, the future X-ray nano and microtomography beamline is being designed to execute and process experiments in only few seconds. For this reason, prototypes and automated systems have being tested and implemented in the current Brazilian Synchrotron Light Laboratory (LNLS) imaging beamline (IMX). An industrial robot was installed to allow fast sample exchange through an easy-to-use graphical user interface. Also, scripts using Python and Experimental Physics and Industrial Control System (EPICS) were implemented for automatic sample alignment, measurement and reconstruction. In addition, a flow cell for study dynamics and behaviour of fluids at the rock pore scale in time resolved experiments (4D tomography) is being projected.

MOGNO BEAMLINE

MOGNO is designed to be a micro and nano imaging beamline focused towards multi-scale analysis of the internal 3D structures of different materials and objects. The beamline will be primarily devoted and specialized in zoom-tomography where a specimen can be studied at low and high-resolution. In parallel, MOGNO will be also dedicated to enable powerful 3D imaging competences which can be extended to 4D (time-resolved) imaging through in-situ experiments. This feature will allow the researchers to observe and quantify material responses during mechanical, thermal or chemical loadings.

It will work in medium (30 keV) and high (90 keV) energies. The beamline has two main scientific drivers: Multi-scale imaging of rocks under reservoir conditions and Imaging of biological samples and tumors, however Mogno will potentially cover many different areas, such as material science, bioengineering, clay science, civil engineering, paper and wood research, chemistry and earth/planetary science, food science, paleontology, archeology and cultural heritage.

PROTOTYPES UNDER TESTING

MOGNO Beamline will have a high flux and energy, which will make possible to perform very fast measurements. Because of this, an automatic measurement system is fundamental to use all the available beamtime. As already mentioned, the fast acquisition will also allow to perform time resolved X-ray tomography (4D tomography). Two prototypes are being tested at LNLS for future application at MOGNO. An automatic measuring system and a rotational flow cell.

AUTOMATIC MEASUREMENT SYSTEM

The IMX is a microtomography beamline that has maximum sample size of 7 mm and highest resolution of 1 µm. The pink beam energy ranges from 4 to 25keV.

For hard samples (like rocks, ceramics), the acquisition time is relatively long (some seconds per projection) and, for tomography, it's necessary to take several projections (we normally do something around 1000), making it time-consuming. When the experiment is done with lighter samples (eg. biological samples), this time reduces to less than one second, what already justifies an automated exchange system. At MOGNO, the whole measurement will take only few seconds, making an automatic system not only interesting, but necessary. Another important fact is that users bring many samples (few dozens), and stay at the beamline for a short period (2 - 3 days), so, to make the most of it, continuous measurements (i.e. 24h a day) are necessary. Manually exchanging samples make obligatory the presence of one person at the beamline. With an automated system, users can organise a queue of experiments and let the system run autonomously.

This system can be separated into main parts: sample exchange robot, process control system, automatic alignment system, graphical user interface for measurements preparation, and safety system to prevent accidents

Sample Exchange Robot

The robot installed at IMX is a Mitsubishi RV-2F-D1 (controller CR-750D). Its main parameters are:

Table 1: Robot Main Parameters [1]

Degrees of freedom	6
Max velocity (mm/s)	4950
Max load (kg)	2.0
Position repeatability (mm)	±0.02

For the robotic hand set, we use a pneumatic gripper (MHZ2-10D) and two Hall effect sensors (D-M9B), to monitor the tool open/close state, both from SMC, and a solenoid valve proper to work with the robot (see figure 1).

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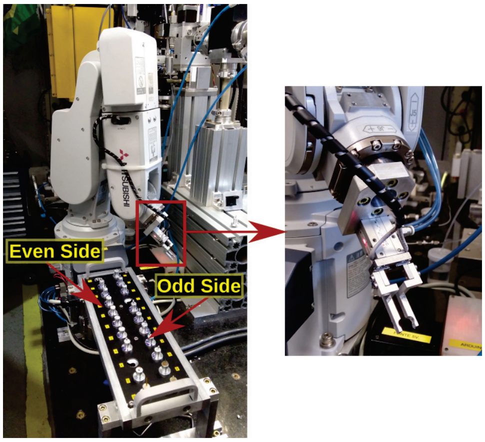

Figure 1: IMX Robot and hand mounting.

The IMX group designed a sample tray, with 22 position, and a new sample holder system (figure 2) for the beamline. The sample holder has conical base, which facilitates the handling and fitting it on the rotational stage by the robot. Two magnets are responsible for applying a small force helping to correct fitting and keep it in contact with the base.

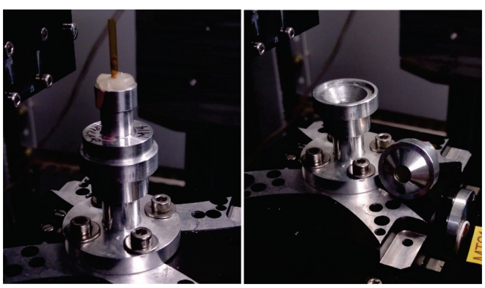

Figure 2: Conical sample holder.

In order to analyse the repeatability of the whole system on sample placement, the robot was programmed to place and remove the samples holder from the rotational stage (were the sample holder is placed for the measurement) 100 times. In between the robot movements, an image was acquired. A sphere with a very well defined diameter was used as the standard sample for the experiment (figure 3). A PCO.2000 camera, which has dimensions of 2048x2048 pixels, and a 10x objective, which gave us a pixel size of 0.82 µm were used for image acquisition. All collected images were binarized (figure 3) using the OpenCV library on Python programming language [2] for later analysis.

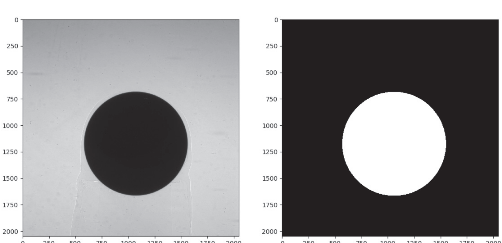

Figure 3: Sample projection (left) and binary image (right).

The next step was to calculate the centre of mass for each image. As they are binary, sample centre and image centre of mass are the same. Thus, for the X axis (transverse to the light beam), the average position obtained was the pixel 1057.1 with a standard deviation of 6.3 pixels. In this case, X-axis standard deviation is 5.15 µm, what is quite good considering that most of commercial robots, including RV-2F-D1, have repeatability worse than that. The Y-axis, the standard deviation is even smaller, 0.4 pixels or 0.32 µm. Those short deviations show the good influence of magnet and sample holder on fitting the sample to a previous defined position.

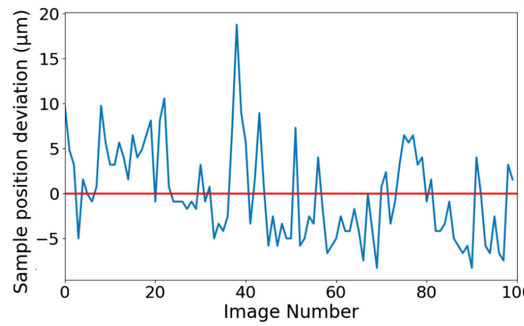

Figure 4: Positions variation on X axis, considering the mean pixel as zero position.

IMX hutch and the robot reach are small, so the robot has to be mounted over the optical table. To evaluate the vibration caused on the table, a triaxial accelerometer (10kHz acquisition frequency and 988mV/g sensor sensibility) was placed upon the rotational stage, in the same position as a sample during the tomographic measurement. The measurements were made in three different robot motion velocities: 50%, 75%, and 100% (robot velocity is set using percentage over its maximum velocity shown on table 1). Robot can take sample from even and odd side of the sample tray (1), and for each one the movement is different. So, for cover all possibilities 6 measures were made. The acceleration peaks are shown on table 2.

The critical movement can be seen on figure 5, odd side with 100% velocity.

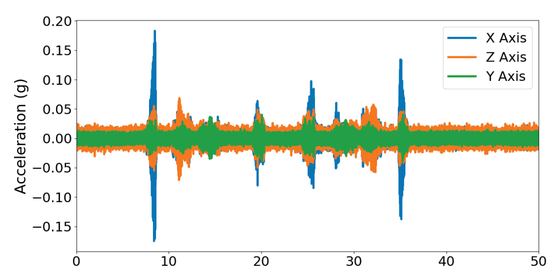

Figure 5: Sample point vibration caused by robot movement with 100% velocity and taking sample from the odd side of the tray.

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Table 2: Acceleration Peaks on Robot Movement

	Even Side		
Velocity	**50%**	**75%**	**100%**
X Axis	0,056g	0,064g	0,096g
Y Axis	0,030g	0,035g	0,045g
Z Axis	0,055g	0,059g	0,063g
Moving Time	44,9s	32,4s	27,1s
	Odd Side		
Velocity	**50%**	**75%**	**100%**
X Axis	0,066g	0,070g	0,180g
Y Axis	0,031g	0,045g	0,040g
Z Axis	0,062g	0,052g	0,069g
Moving Time	47,85.15 µms	35,9s	28,5s

From 7 to 22 seconds, the robot is placing the samples, and it is removing them from 24 to 36 s. The most important result is that the sample position vibration returns to the initial state right after the robot stops the movement, showing that placing the robot over the optical table is not a problem. It is important to note that 100% velocity is applied when the robot is moving without carrying any sample and 30% if it is loaded, to avoid damage to the users sample.

The last test was to measure the acceleration caused by the open and close action of robot gripper. Figure 6 shows the results.

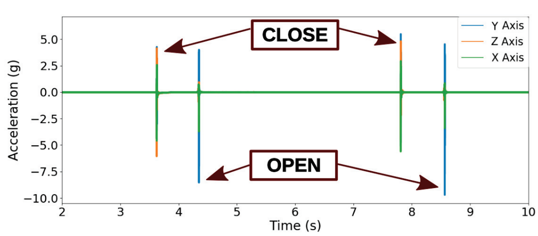

Figure 6: Vibration caused by opening and closing robot tool 5.15 µm

It was measured using the same acelerometer, which has a max acceleration measure range of 5g. Looking to the figure 6, it is possible to observe results close to 10g, which are not reliable, but an acceleration higher than 5g is expected. The gripper is pneumatic and it is controlled by a solenoid valve, which gives only close and open state. Change this tool to a electric gripper can be more interesting to MOGNO, allowing velocity, force and position control, that will reduce the chance of sample damage.

Process Control System

The CR750-D controller has an option of robot remote controling using TCP/IP connection. It also provides a vast list of remote commands [3]. So, for our application, we set up a fixed IP in the controller and implemented an algorithm using the Python programming language that connects to the controller using the Socket library [4], sends the desired commands and waits for the controller response.

Different programs can be saved on robot controller. So, we saved 46 programs: 22 for sample positioning, from the tray to the approximation position (close to the measurement position), 1 to move the sample from approximation position to the measurement point, and the others for the reverse movements.

Another important factor is that the sample holder moves, i.e. for each sample, a different positioning is required to place and aligned it in front of the beam/detector. Therefore, when robot is about to perform a sample exchange, it sends the fast-moving motors (horizontal movement motors) to a standard position. Since the vertical motion is slow, the algorithm reads the motor position using EPICS [5] and sends a command to the robot controller requesting all positions saved on the program, add the offset read to all positions, resends the positions to the controller and only then sends the execution program command.

Automatic Sample Alignment

The automatic alignment aim to place the sample fully within the field of view. To demonstrate the application a real experiment from IMX beamline user was chosen. After placing the sample in the experimental setup the alignment code is started and it first verify the angle that the rotational stage is positioned. In the chosen case the position was closer 90 or 270 degrees than 0 or 180 degrees so the algorithm initiates the iteration in those angles. Each line in the figure 7 shows an iteration. It is observed that in the two first 90 and 270 iteration both centre of rotation (COR) and the centre of sample are moved, positioning they projections collinear in relation of the centre of detector. At the third iteration the algorithm verifies that it has reached the criteria of stop. The criteria stop is less than 1 pixel of difference between the centre of sample and the centre of detector and also less than 1 pixel of COR shift relative the centre of detector.

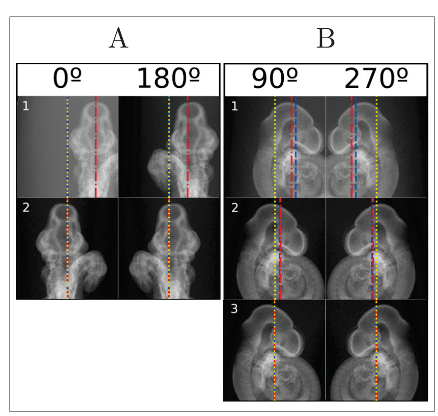

Figure 7: Iterations for sample alignment. In yellow is the centre of detector, blue is the axis of rotation and red is the centre of the sample.

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So the next step is the alignment of the sample in 0 and 180 degrees. Since the alignment at 0 and 180 degrees started with the COR aligned and both start projections are within the field of view, the process usually reaches the stopping criterion with only two iterations. Figure 7A shows the iterations steps necessary for positioning the sample centre. The whole alignment method takes about one minute without any user intervention.

Graphical User Interface

A graphical user interface (GUI) was created using Control System Studio (CSS) [6] (figure 8).

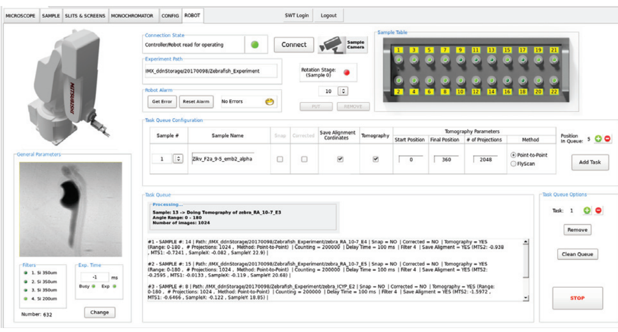

Figure 8: Graphical user interface for automatic system control.

In this interface, the user basically has two main options: use robot to place or remove a specific sample and organise a queue of measurements. When the user is going to add a task to the queue he can choose to run the automatic alignment at the moment of the measurement or before it and then add the task with the stages position saved (read motor position using EPICS). In addition, it is also possible to define which filters to use for each task, exposure time, tomography angle range, number of projections, type of tomography (point-to-point or fly-scan) and delay time between projections. Finally, the user can also choose the position on queue that he wants to add the task, delete a task that no longer is necessary and clean the entire queue, what can be done before the process starts or during movement and measurement.

All these options allow user to measure the samples in a practical and simple way, and allow measurements of completely different samples in the same bunch of experiments.

Security System

The LNLS is a multiusers facility, which means people can submit scientific proposals to use the beamlines. Users normally receive between 3 and 5 days of beamtime. To avoid accidents caused by unfamiliarity with the automated system, we implemented a security system.

The robot controller has a IO board (model 2D-TZ378) from Mitsubishi installed. With this board is possible to execute some commands sending DC voltage (from +12 to +24) to the specific port. We used the stop port to add the robot to beamline interlock. So, when experimental hutch door is open, the controller continuously receive a stop command, what disallow the robot to move. In addition, every time the Python script runs a command that puts robot in movement, it goes inside a loop that keeps checking the hutch door state, if the EPICS PV state changes to open, the algorithm stops to send "run" command and starts to send "stop" command, giving a duplicated interlock security system.

To avoid collision, we are using three kinds of sensor. Robot already comes with a force sensor that stops servo motor when robot makes a higher force than the desired. As we are dealing with fragile samples, we installed two other kinds of sensor. First is a infrared proximity sensor mounted on the rotational stage to recognise when a sample is placed in front of the X-Ray beam. The second are 22 optic-reflexive sensors installed on each position of sample tray (under the black cover on figure 9), to provide feedback on whether a sample is mounted or not. An Arduino microcontroller is being used to monitor sensor state changes and transfer this information through UDP connection, using a ethernet shield, to an Arduino IOC installed on the beamline PXI. The IOC will update the samples PV's, which will give a real time feedback to the user about the sample positions. These components were choosen because of the low cost and easy installation, considering this is only a prototype.

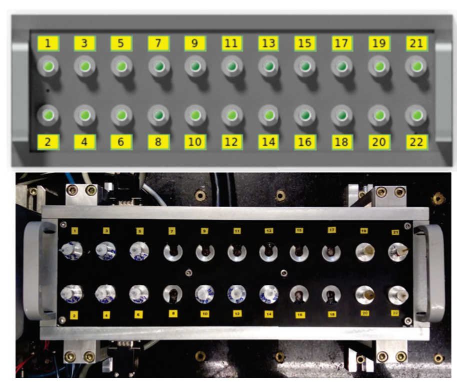

Figure 9: Graphical user interface (top) and physical sample tray (bottom).

FLUX CELL FOR TIME-RESOLVED TOMOGRAPHY

We are developing a flux cell for simultaneous inject of up to three fluids to a porous sample during continuous rotation. The cell can be seen on figure 10.

The cell is basically composed by two cylinders: the external where the three hoses come with the fluids (this cylinder is tied on top, forbidding it to rotate with the stage), and the internal with holes and ducts inside, linking to the top connection where the sample is placed using a Festo fast connection. To separate the three fluids when they are moving from the external to the internal cylinder and to allow the continuous rotation we are using Viton o'rings.

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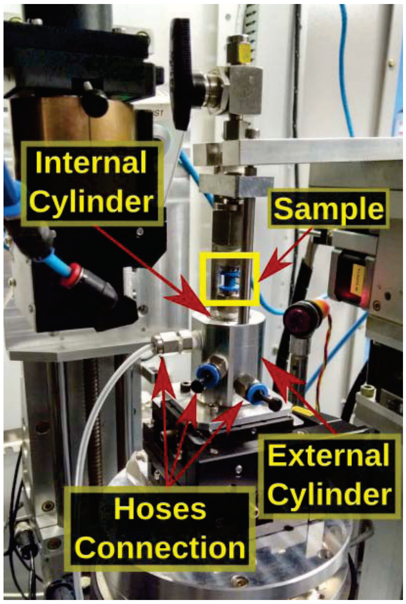

Figure 10: Flux cell mounted under IMX rotational stage.

For fluid injection, we are developing three syringe pumps to fill the three hoses. For moving the pistons, three Nema 23 motors are used. Pressure transducer give us real-time feedback and a back pressure regulator controls the outlet pressure.

As this cell can be used for different applications, all o'rings we are using are made of Viton and the cell was manufactured with stainless steel, increasing the usage fluids possibilities. For the first test a polymeric sample to simulate a porous rock was manufactured. It was necessary due to the low energy range available at IMX, what makes the measurement of a real rock nearly impossible. First test was only based on taking projections in different injection steps, to ensure there was no leakage under the desirable pressures (40 psi). We injected water and took some projections using pink beam, 200 µm Silicon filter, and exposure time of 1 second at IMX Beamline. To show the water flow we subtracted a dry sample image from each projection. The first results are shown on figure 11.

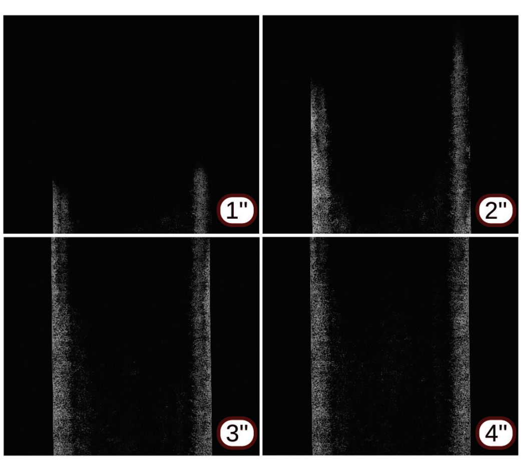

Figure 11: Per second images subtraction on injection test.

There is a preferential water path thought the sample boarder, due to irregularities on sample manufacturing. Figure 12 shows a slice for a dry sample, it is clear that the porosity is bigger all around the sample.

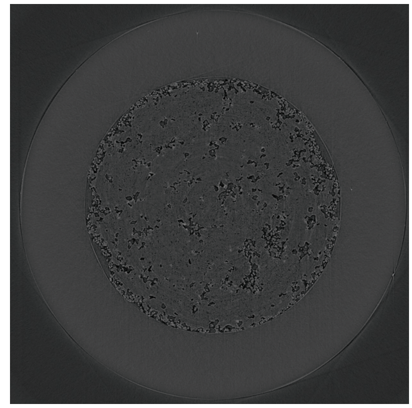

Figure 12: Slice of manufactured sample inside the hose before injection.

CONCLUSION

The automatic sample exchange system underwent several tests of stability and repeatability and the main results are: (a) the conical fit is a good solution to avoid errors on the sample positioning by the robot at IMX, however the rotation rate at Mogno is higher (360 degrees/s) and more tests are required; (b) open/close pneumatic gripper will not be considered as a possible solution for Mogno due to the instant high acceleration it causes to the sample - an electrical tool will be soon tested. Regarding the flow cell, a more homogeneous sample will be tested to verify if the preferential path disapears, and projections under different angles (i.e. rotating the cell) will be taken and the seal under this condition will be tested. Future implementation is to incorporate a temperature control to reach more subsurface conditions.

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