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CONTROL SYSTEM UPGRADE OF THE HIGH-PRESSURE CELL FOR PRESSURE-JUMP X-RAY DIFFRACTION

R. Mercado*, N. Griffin, S. Lay, P. Holloway and P. Roberts
Diamond Light Source, Oxfordshire, UK

Abstract

This paper reports on the upgrade of the control system of a sample environment used to pressurise samples to 500 MPa at temperatures between -20°C and 120°C. The equipment can achieve millisecond pressure jumps for use in X-ray scattering experiments. It has been routinely available in beamline I22 at Diamond. The millisecond pressure-jump capability is unique. Example applications were the demonstration of pressure-induced formation of super crystals from PEGylated gold nano-particles and the study of controlled assembly and disassembly of nano-scale protein cages.

The project goal was to migrate the control system for improved integration to EPICS and the GDA data acquisition software. The original control system was based on National Instruments hardware controlled from LabView. The project looked at mapping the old control system hardware to alternatives in use at Diamond and migrating the control software. The paper discusses the choice of equipment used for ADC acquisition and equipment protection, using Omron PLCs and Beckhoff EtherCAT modules, a custom jump-trigger circuit, the calibration of the system and the next steps for testing the system.

INTRODUCTION

Beamline I22 and Imperial College London built and developed a sample environment [1] used to pressurise samples for diffraction experiments. This end-station equipment has been available for several years [2].

This end-station was built using a control system based on Compact DAQ measurement and control modules (National Instruments). Its associated control was realised by the collaborators using LabView.

An initial attempt was made to integrate with EPICS but the integration was not maintained, due to LabView not being an actively supported platform for the controls group at Diamond. User feedback indicated that direct control from GDA and EPICS would make operations less prone to user error.

SYSTEM DESCRIPTION

The high pressure cell has these components (as shown in Fig. 1):

- Pressure generator. This is a motor driven high pressure pump which can generate pressures up to 700 MPa. The drive motor is a high power DC servo operated with a custom built motor controller.

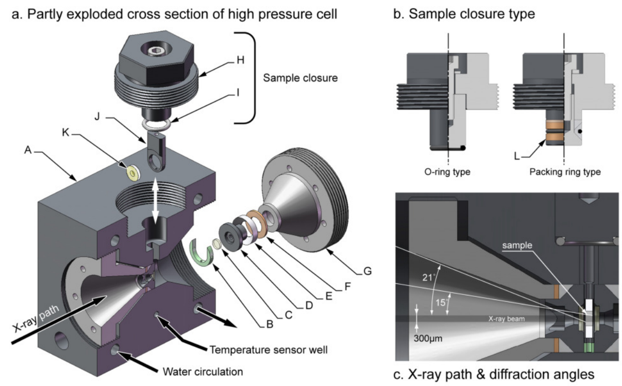

Figure 1: Detail of the pressure cell [1].

- Two remotely operated, normally closed pressure valves.
- Two remotely operated, normally open fast pressure jump valves.
- Three pressure transducers monitoring three sections of the pressure network. The sensors are 700 MPa strain gauge based transducers excited with 10 V and reading 10 mV at full scale.

The pressure network comprises three sections:

- the first section with the pressure generator,
- the second that includes an optional additional tank that can be used particularly for pressure jumps and
- the third section where the pressure cell is installed.

The control system opens and closes valves, allows the syringe pump motor to pressurise and de-pressurise the pressure network.

The pressure network has to be protected from overpressure. Valve operations are inhibited if the pressure differential is larger than 5 MPa (50 bar) between sections, with the exception of pressure jumps.

There are two jump valves, one dedicated to jumps up in pressure and another for jumps down in pressure.

HARDWARE USED IN THE UPGRADE

Diamond standardises on electronics hardware (as shown in Table 1) where possible [3, 4]. A benefit to the facility is the maintainability and support by standardising on a limited set of components.

The majority of the digital I/O signals for the system can be mapped one to one as they are standard and are based

* ronaldo.mercado@diamond.ac.uk

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Table 1: Hardware Module Types and Replacements

Function	Original module	Replacement
Cell temperature PT100 sensor	RTD Input NI-9217	Omron CJ1W-TS562
Pump piston positioner linear transducer	Analogue input NI-9201	Omron CJ1W-MAD42
Motor speed control	10V Analogue output NI-9263	Omron CJ1W-MAD42
Motor ON	24V Digital Output NI-9263	Omron CJ1W-OD212
Motor Direction	24V Digital Output NI-9263	CJ1W-OD212
Valves open/close (Solenoids)	24V Digital Output NI-9263	CJ1W-OC201
Limit switches	24V Digital Input NI-9241	Omron CJ1W-ID211
Trigger for pressure jump	High speed digital IO NI-9401	Custom jump circuit
Pressure signals	Bridge input NI-9237	Strain gauge amplifier RDP DR7DC Omron CJ1W-MAD42 (Equipment protection ADC) Beckhoff EL3702 (Fast ADC capture)

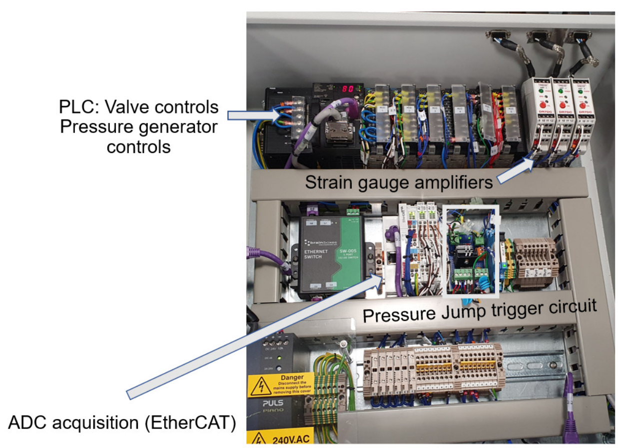

Figure 2: Electronics cabinet of the upgraded control system.

on 24 V on/off, a PT100 temperature sensor and analogue 0-10 V I/O.

Reading the strain gauge is a more specialised operation that originally used the NI-9237 module. A strain gauge amplifier was selected based on prior experience: the RDP Electronics (Wolverhampton, UK) DR7DC. The amplifier converts the millivolt strain gauge signal to the usual control range of 0-10 V. The signal is read by ADCs in parallel.

The most recent standard set of components (as shown in Fig. 2) includes a strain gauge EtherCAT module (Beckhoff EL3356), but its acquisition speed was not adequate to capture a pressure jump.

The PLC system controls the system protection, arms the pressure jumps and controls the motor for the pressure generation. The supervisory control uses EPICS. The fast EtherCAT ADC signals are captured using the ADEthercat area detector plug-in described below.

One important requirement was the desire to acquire the pressure signal during the pressure jump. An in-house electronics module was created to trigger a pressure jump on a hardware trigger signal from the beamline acquisition system. This replaced the NI-9401 high speed digital I/O module. The design was based around an inexpensive flip flop and current driver that opens a valve for a pressure jump using a TTL signal in order to synchronise with the image acquisition with the x-ray detectors. For a pressure jump acquisition the valve is "armed" so that it opens on the trigger.

During early testing the simplistic electronic design turned out to be vulnerable to noise when operating real solenoids. It took careful diagnosing and re-work of the circuit to improve the pressure jump circuit noise immunity by adding filters.

Other difficulty was signal noise from the transducers. The solution was to modify the cabling between the transducers and the strain gauge amplifiers with improved shielding and grounding.

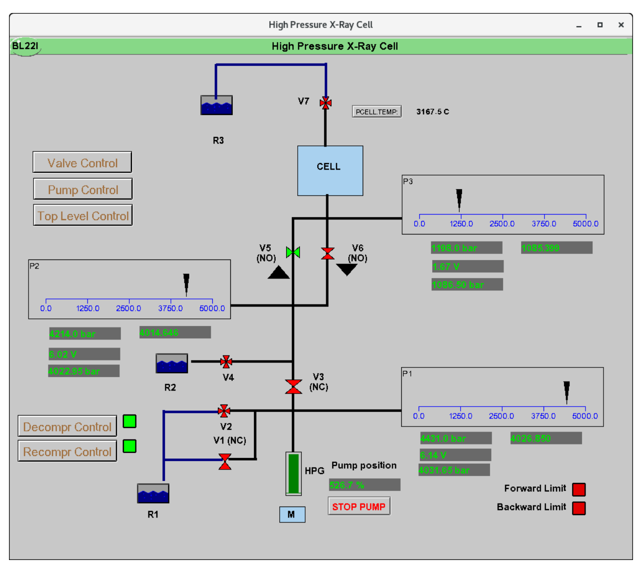

Figure 3: Top level EPICS control screen showing the pressure network.

ADETHERCAT

The pressure signals are captured at 10 kHz using the DLS EtherCAT EPICS scanner (as shown in Fig. 3). The

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ADEthercat area detector plug-in was used to capture the signal. ADEtherCAT is a variety of driver to read ADC to area detector. It was inspired by prior experience with the D-TACQ ADC (Glasgow, UK) and beam position monitor devices (e.g. quadEM [5]) that use the EPICS area detector framework to operate on 1-D signals. ADEthercat converts ADC signals from the EtherCAT master used in the Diamond ethercat EPICS driver [6] to 'image' frames. The two dimensions of the 'image' are the number of signals and 'n' samples. For the pressure cell application there are four signals captured, three for the pressures in the network and a fourth signal of the trigger value.

An immediate benefit was the ability to use a software plug-in 'NDReframe' that allows triggering on a 1-D signal as it is streamed. This solves a limitation of EtherCAT hardware that does not otherwise provide a method of triggering for data capture. The framework also adds for free the ability to write the signal to HDF5 files, which are the standard for data acquisition.

A custom pressure scaling plug-in was developed that applies the calibration method described below (Fig. 4).

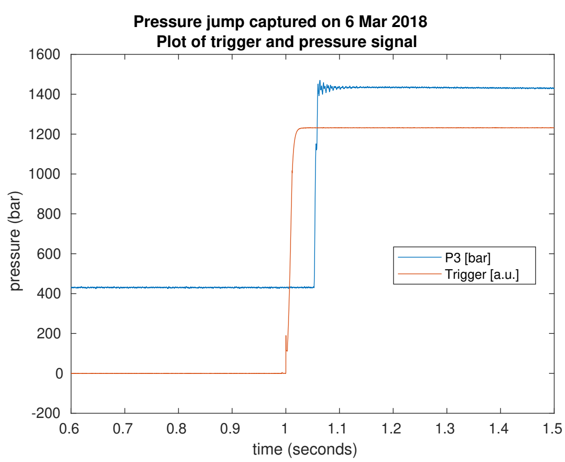

Figure 4: Plot of example pressure jump. Optimized fast valve achieves jump time of approx. 5 ms. The traces shown are the trigger (red) and pressure measurement (blue). The data capture uses the ADEthercat driver. The data scaled using a custom scaling plug-in and written to an HDF5 file using the area detector file writer plug-in.

AUTOMATED OPERATIONS

There is a mode of operation implemented at the PLC level to achieve a target pressure or a target position. It was implemented at the PLC level because it allows closer control at shorter cycle times than the supervisory control in EPICS.

There are modes of operation of higher abstraction that are implemented using a python soft IOC [7]. The operations allow setting up a pressure and setting a pressure jump.

The volume and stroke of the pressure generator are not large enough to achieve an arbitrary pressure in a single operation. Two examples are that to reach certain higher pressures the network needs to be refilled and to reach lower pressures sometimes water needs to be removed from the pressure network.

The logic allows the composition of sequences of operations to liberate the user from potentially tedious operations of pressurising the network to reach high pressures. This is more important when using the expansion tank option in the middle section of the pressure network. In tests a filling in operation needed to follow the steps to fill in and pressurise in steps for about 10 minutes with several dozen operations.

CALIBRATION

A significant hurdle to realising the project was also the calibration of the pressure signals. Calibration facilities use a calibration rig where a special oil is pressurised with known weights to reach high pressures. It was not practical or possible to operate the equipment with its full upgraded electronics read-out next to the calibrator.

Instead of this, the beamline obtained calibration certificates from ASCO Instruments (Châteaufort, France). The calibration points were linearised based on the eleven data pairs of pressure vs strain gauge voltage obtained at the calibration facility. To calibrate the electronics, the millivolt signals were input to the electronics components using a precision millivolt source (Time Electronics 1006 DC Millivolt Source Model 404S).

At lower pressures, below 50 MPa (500 bar), the calibration was changed to reflect that when opening the valves to atmospheric pressure the linearised calibration did not read zero as expected.

The result is a custom scaling mode where there are two linearisations above and below the 50 MPa threshold. This was reflected in a custom area detector pressure scaling plug-in prepared for the pressure cell and also in the PLC software.

The use of ADCs reading the amplifier signal in parallel for fast ADC capture and machine protection resulted in different voltage readings by a few millivolts after amplification. A calibration correction was adopted to account for this discrepancy that was attributed to impedance mismatch of the ADCs [8, 9].

CURRENT STATUS AND IDEAS FOR IMPROVEMENT

The system is being tested in an off-line laboratory and integration with the beamline will follow.

The project was conceived nearly five years ago. More recently it has become possible for Omron PLCs to also support EtherCAT modules that can capture ADC signals at 100 kHz. Using a higher speed CPU and the NX range of PLCs it should be possible to get the required ADC acquisition speed. The missing component is a software layer to 'stream' the ADC waveforms to area detector. This would be a welcome improvement over the current state in which limited developer effort is available to support adding more module types using the Diamond ethercat EPICS driver.

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The use of a single ADC instead of two sets of ADCs reading in parallel would also simplify the calibration.

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