Implementing Remote Control of a Spectrometer on a Laser Heating Table

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Abstract

Continuous and Pulsed Laser Heating have become indispensable tools in the study of materials under extreme conditions. It is used to heat samples to a high temperature in order to see changes in their structure and chemistry. In order to improve the experience of users on the extreme conditions beamline P02.2 at DESY it is was necessary to incorporate measurement of laser heating temperatures into the workflow. The implementation of the remote control of the spectrometer is outlined in this paper featuring the control of the hardware using different software tools, including Tango control systems.

Contents

1 Introduction

1.1 Background

Petra-III is a 3rd generation Synchrotron radiation source of circumference 2.3km located at the DESY site in Hamburg, Germany. The beamline P02.2 is located in the Max Von Laue experimental hall and is one of 20 beamlines across 3 experimental halls at DESY. The beamline investigates the behaviour and structure of materials under extreme conditions using X-ray diffration techniques. The beamline offers access to X-ray diffraction in combination with high pressure diamond anvil cell technique.

1.2 The Beamline

The beamline can probe samples at high pressures while also at ambient, low (cryostat) or high temperatures (resistive heating, laser heating). The high pressure is created by compressing a small sample between two flat tipped diamonds as seen in figure 1.

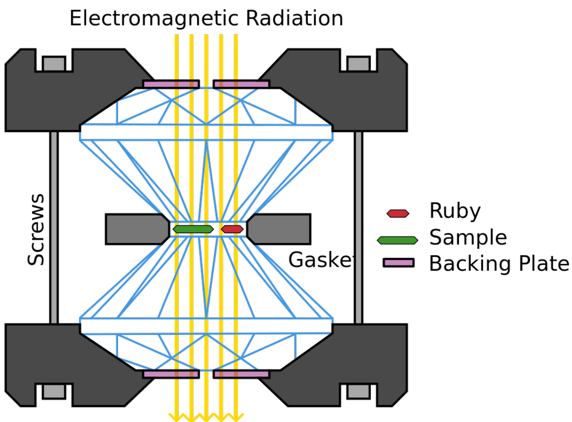

Figure 1: A diagram of the cross section of a diamond anvil cell. The rubies are often used for calculating the pressure in the sample chamber. One can also use diffraction standards in combination with published equations of state to calculate pressure for a given temperature.

The area of the tips or 'cullets' of the diamond are very small, spanning from 20μm to several millimeters, allowing a high pressure to be created by a given force according to the relation

$$P = F/A \tag{1}$$

where P is pressure, F is force and A is area. Diamond is transparent to a broad electromagnetic spectrum, including visible and infrared light. Light of a broad bandwidth can pass through the diamonds without losing considerable intensity of incoming light or scattered signal. This intrinsic property is used by various modern techniques[1] and allows direct sample monitoring through a microscope.

The temperature of the sample can also be controlled and measured. Low temperatures can be achieved using a liquid nitrogen or helium cryostats while high temperatures are reached using resistive or laser heating. Laser heating makes use of a Near InfraRed (NIR - 1070nm) or Far InfraRed (FIR - 8000-10000nm) to heat the surface or the volume of the studied material to extreme temperatures. The temperature of the sample is conventionally measured using a spectrometer. The combination of diamond anvil cells with laser heating opens access to extreme conditions similar to the conditions of deep planetary interiors, i.e. Earth's Inner core and beyond [2].

Experiments under these conditions have applications not only in fundamental physics but also in fields such as planetary science, Earth science and chemistry [2]. The high pressures and temperatures created simulate the conditions and processes of the deep planetary interiors, including planetary mantles or cores. Scientist also simulate conditions similar to asteroid impacts and their influence on mineralogical assemblies as signatures for specific pressure-temperature conditions to understand the history of our planet.

The inherent chemistry of materials can be substantially altered under extreme conditions and can possibly lead to the discovery of new functional materials. This was seen by Zhang *et al* in 2013 when they showed that NaCl, or rocksalt, can have different stoichiometries at high pressures [3]. Under these high pressures and after high temperature stimulation new compounds were formed with structures of Na and Cl impossible at ambient conditions. High pressure can also lift the critical temperature for superconductivity as was shown by Drozdov *et al.* In this study at around 170 gigapascals the critical temperature for superconductivity of lanthanum hydride was about 250k which is one of the highest temperature ever reached [4]. This was a significant step toward ambient temperature super conductivity not only important as a fundamental study, but the one paving a way to more efficient electronics of the future.

2 The Summer Project

Laser heating is an essential tool of the extreme conditions beamline P02.2. Many groups visit the beamline and make use of the laser heating table. A significant portion of the experiments focus on exploration beyond the limits of current knowledge in the fields of chemistry, geoscience, physics and etc.

The presence of diamond as anvils in the diamond anvil high pressure technique enables the external heating of the sample by lasers, but at the same time, the properties of diamonds enable sample characterization and even temperature measurements with spectroscopic techniques (Planck radiation function). Temperatures of the samples can be measured by special spectrometers such as the Andor Shamrock SR-303 located at P02.2.

In order to provide the user of the beamline with the best possible experience the process of measuring the temperature of the sample on the laser heating table has to be improved from different points, including the automatization of the data collection workflow. The software used to control the spectrometer, Andor Solis (Oxford Instruments, Andor) is efficient, but at the same time too complicated for a general user (see figure 2. The task of the project was to implement remote control of a spectrometer via its standard software in order to make data collection quicker and easier for users of the beamline.

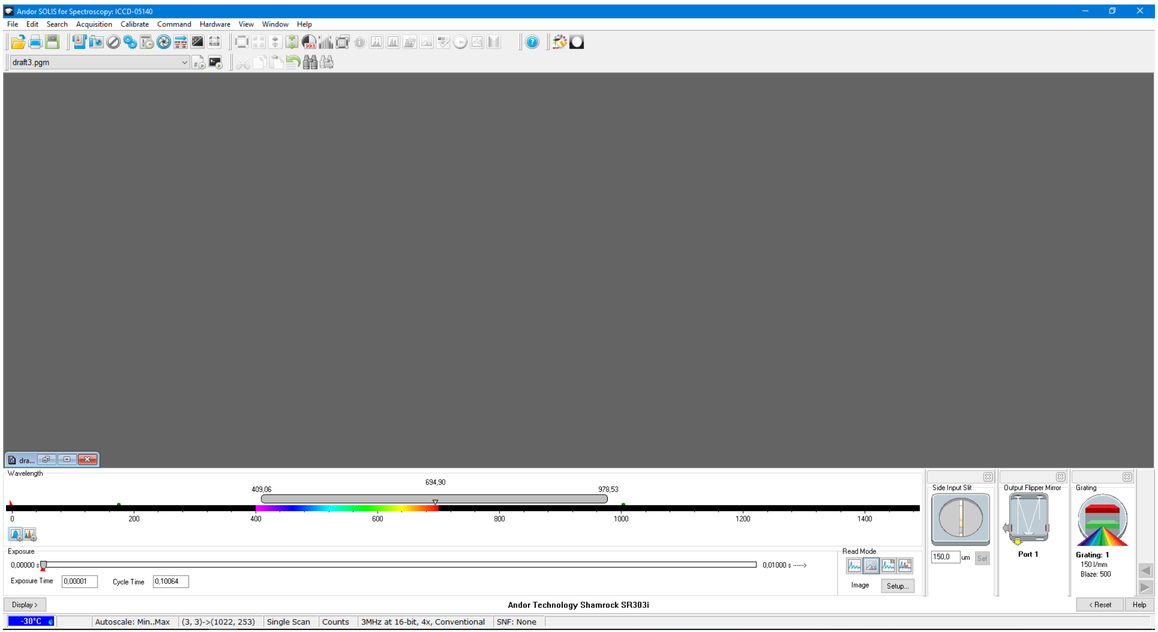

Figure 2: A screenshot of the Andor Solis software showing the extent of tools available to the user.

2.1 Overview

Commands issued by the user located somewhere in the network had to end up being read by the spectrometer. In order to enable network communication we use the implementation behind Tango Control system and its infrastructure. A custom implementation of a Tango server receives input from a user. We define a user as a client which is built from a series of python scripts that communicate certain actions to the Tango Server. Tango server receives the input from a client and transforms it to the commands which would be processed by Andor Solis. The latter is achieved using the interface of a file system (communication via a file) and the interface of AndorBasic. The overview of the implementation can be seen in figure 3.

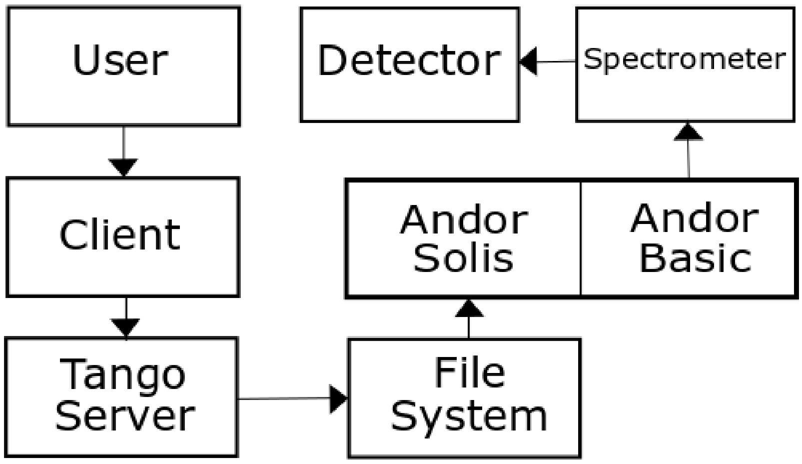

Figure 3: The diagram showing the route the commands take from user to the spectrometer

2.2 Continuous and Pulsed Laser Heating

Laser heating was introduced by Ming and Bassett in 1974 as an alternative to resitive heating [5]. Laser heating has advantages over restive heating such as it allows only specific parts of the sample chamber to be heated. Over decades the laser heating technique was enhanced supplementing the original continuous laser heating with pulsed laser heating techniques. In continuous heating a laser of constant power is focused onto a point on the sample and transfers the energy of the light into heat.

The sample temperature increases due to conduction as described by Fourier's law of heat conduction [6].

$$\frac{dQ}{dt} = -k\nabla T \qquad (2)$$

where Q is heat, t is time, k is thermal conductivity and T is temperature. Due to the temperature gradient the diamond anvil cell will also be heated under this process which could eventually lead to the failure of diamonds [5]. In pulsed laser heating the sample is hit with laser light for repeating short periods of time. The heat is able to dissipate from the sample before the next laser pulse arrives [5]. This prevents substantial heating of the diamond anvil cell and the accompanying issues. Pulsed laser heating is able to reach higher temperatures then continuous laser heating with a laser of the same average energy. A pulse could be shaped with the peak power having a higher concentration of energy [7]. This means more energy is transferred to the sample during the period of the peak then in the same period during continuous heating. This then leads to a larger increase in the temperature of the sample. The shape of the pulse can vary and be set to different shapes for different experiments. As the heat dissipates from the sample the temperature profile of the sample varies in time as can be seen in figure 4. Measurement of sample properties in the pulsed laser regime is more complicated as it requires more effort on synchronization of the signal collection.

Both the continuous and pulsed laser heating have their uses, advantages and disadvantages. The laser heating table at P02.2 uses a single infrared laser and series of lens, polarizers and

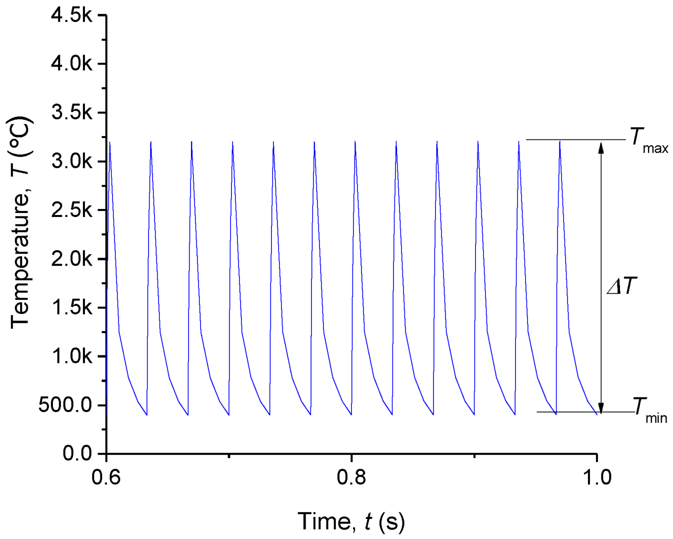

Figure 4: A diagram representing temperatures of a sample against time during pulsed laser heating [8]

beam splitters to allows both sides of the sample to be heated either individually or simultaneously. The development of simultaneously heating allowed the sample to be heated more evenly.

2.3 Temperature Measurement

Using a spectrometer the temperature is measured through a technique know as spectral pyrometry. This allow the temperature to be measured using black-body radiation curve without knowing the emissivity of the sample [9]. A section of the radiation spectral curve is measured by separating the light into its spectral components via a diffraction grating and measuring the spectral radiance at each wavelength.

$$B_\nu(\nu, T) = \frac{2h\nu^3}{c^2}\frac{1}{e^{\frac{hc}{k_B T}}-1} \tag{3}$$

Equation 3 represents the Planck radiation function and provides a relationship between the spectral radiance *B* at a particular wavelength ν and temperature T [10]. The values of h, c and k_B are that of the Planck's constant, the speed of light and the Boltzmann constant, respectively. After measurement the spectrum can be transformed into the Wien region giving a straight line where the gradient depends on the temperature and is independent of emissitiviy. Refer to [9] for a complete description of this method.

The spectrometer installed at P02.2 station has two available detectors for temperature measurement from Oxford Instruments which shall be referred to as ISTAR and IDUS (see appendix for more details). The IDUS detector is slower than the ISTAR detector but it has higher quantum efficiency, and, thus, it is more sensitive. Each detector has its advantages for collecting data under different conditions. For example, the ISTAR detector has Digital Delay Generator (DDG) mode allowing the temperature to be measured fast and efficiently during repetitive mode of pulsed laser heating.

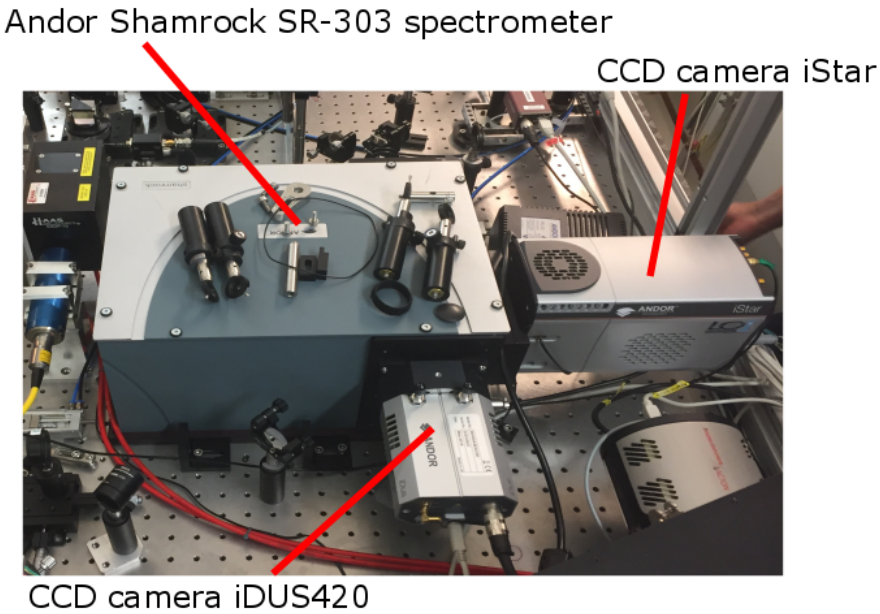

Figure 5: Photo showing the spectrometer and the two available detectors.

3 Project Implimentation

In order to have the best possible functionality for the user the code was designed so they only had to set the essential information. This included setting the file name, file index and folder path for the data to be saved into. The file index counts up with each new data acquisition while the file name and folder stay the same until they are changed. The user is also able to set whether to use the ISTAR or IDUS detectors. When the camera is changed all the necessary changes to the settings are made and the background is retaken without any input from the user. The commands to collect the back ground and collect single and multiple measurements are also available to the user. These both require a user input of the exposure time to be set and will then collect and save the data. In multiple measurements the number of acquisition is also set. This set of inputs and commands are the minimum requirement for the user to be able to take singular or multiple temperature measurement.

3.1 Andor Solis

Andor Solis is the software package used to control the spectrometer Shamrock 303 installed on the beamline. It has its own macro language, AndorBasic, that allows execution of Andor

Solis internal commands responsible for data acquisition, analysis and display. Within Andor-Basic a script was written to facilitate functionality with an emphasis on data collection. The synchronization between the script of the Andor Basic and the Tango Server was also implemented as a part of this project and is done via file system. Firstly, the Andor Basic script looked for the existence a special file (operator file created by a Tango server) and try to read out commands. A timestamp is used as a unique indicator for a new set of commands. The commands and their values are then read line by line off the file and set by Andor Solis. The client side can request updates on the state of the command execution within AndorBasic and wait until the end this process before issuing a new set of commands. The debugging information displayed during the execution of the AndorBasic script as well as the one available in the framework of Tango Server allows the operator to control the process of measurement.

3.2 Python implementation: Controllers and Classes Responsible for Communication

A comprehensive set of scripts was written, including the backend libraries and the frontend implemented as a Tango Server. The backend libraries were written to take the input of the user and transform the incoming commands into the commands which would be understood and processed by the Andor Basic script. Our implementation is focused on the following steps of the workflow, namely, the background collection, signal collection and control of the data saving protocol (file name, index and folder). Additional features have been implemented enabling switching between different detectors and collection modes (single frame vs multiframe data collection). The software was written in a way allowing easy expansion for additional functionality.

3.3 Tango Control System and current implementation

Tango is a free, open source and hardware independent toolkit for developers to make control systems. It can be written in either C++, Java or Python and as Python has been used here it is known as Pytango. In its simplest form Tango is a software proxy communicating inputs from the user to the software controlling the hardware. It can also be used as a 'mailbox' from one piece of software to another. Tango is built around the idea of devices and device classes and about an idea of a simple homogeneous network interface of communication with all sorts of devices. In this case the spectrometer is represented by a device which is an instance of a device class. These devices are contained in a device server that connects them to the network allowing them to be accessed and controlled from anywhere joined to the network. A device server can host many devices at once to create more complicated control systems and makes tango completely scalable. Within the device class the control of the device is implemented through attributes, commands, properties and pipes. A diagram of the tango system control system can be found in figure 6.

Attributes can be read only or read and write and hold a quantity held by the device. File name, file index and folder path are held in read and write attributes while exposure time is held in a read only attribute.

Commands are associated with an action that can be performed and can take inputs as well as give outputs. Setting the detector, collecting the background and collecting a measurement were all implemented as commands.

Properties contain a configuration parameter of the system important for the initialization of the startup phase. They are stored in a Tango Database alongside other run-time information. The location of the files important for the inter-script communication (file system) are stored as properties.

Each device also has a state that reflects the internal state of affairs of the system. It could, for example, represent if a certain piece of hardware is on or off. In our implementation, certain commands and attributes can become unavailable depending on the state of the system.

The tango server written for the remote control of the spectrometer is run by a series of classes behind the scenes. This includes a configuration class to hold all the variables associated with both the commands to be implements and those needed for the running of the tango server. The exposure time in this classes is only ever updated after having been successfully passed to Andor Solis in a command. In addition, there is a worker class running in a separate thread that uses the command classes and controller passing the user input to the background job of the worker class.

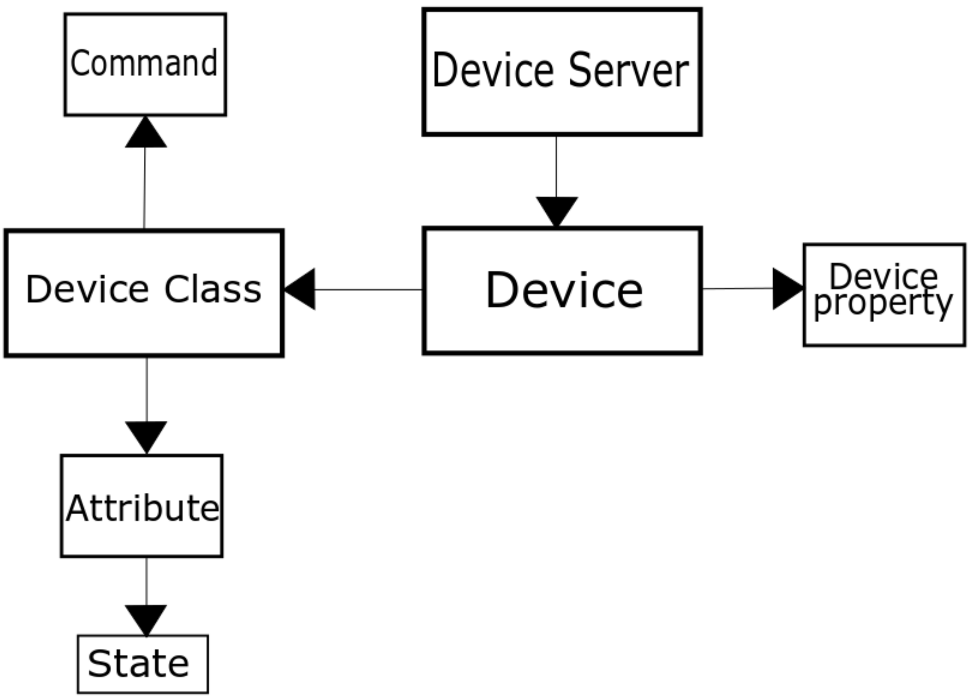

Figure 6: A diagram detailing the structure of a single device tango system

3.4 Future Extensions

There are the foundations within the code to increases the control level the user has over the spectrometer. The script in AndorBasic has the ability to change more parameters and the python backend has been written for all of them. With a few modifications to the controller and Tango server additional functionality could be implemented. For example, DDG mode of the spectrometer could be controlled with better precision with a command setting output delay, gate pulse delay, gate pulse width and gate step. In addition, other commands of the spectrometer or the CCD cameras could be easily made available for the user. Any new implementations would be very quick and simple.

4 Acknowledgements

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5 Appendix

5.1 Equipment

Spectrometer:

- Andor Shamrock SR-303 spectrometer

Detectors:

- CCD camera iDUS420 model DU420A-BEX2-DD
  - Pixel matrix: 1024 x 255
  - Pixel size: 26 μm
  - Minimum dark current: 0.0004 e^{-}/pixel/sec
  - Spectra per second: 88 μm (10 row crop mode), 75 (full vertical bin), 65 (open electrode, full vertical bin)
- CCD camera iStar DH320T
  - Pixel Matrix: 1024 x 265
  - Pixel size: 26 μm
  - Minimum dark current: 0.1 e^{-}/pixel/sec
  - Spectra per second: 323 sps (FVB), 3571 sps (ROI)

For the complete specification of the detectors visits https://www.oxinst.com/

Software:

- Andor Solis (version 4.28.30039.0)
- Python 3.7
- JetBrains PyCharm Community Edition 2018.3.7 x64

5.2 Python Backend

A diagram detailing the backend library. For each command class there was an a parallel function in Andor Solis to implement it.

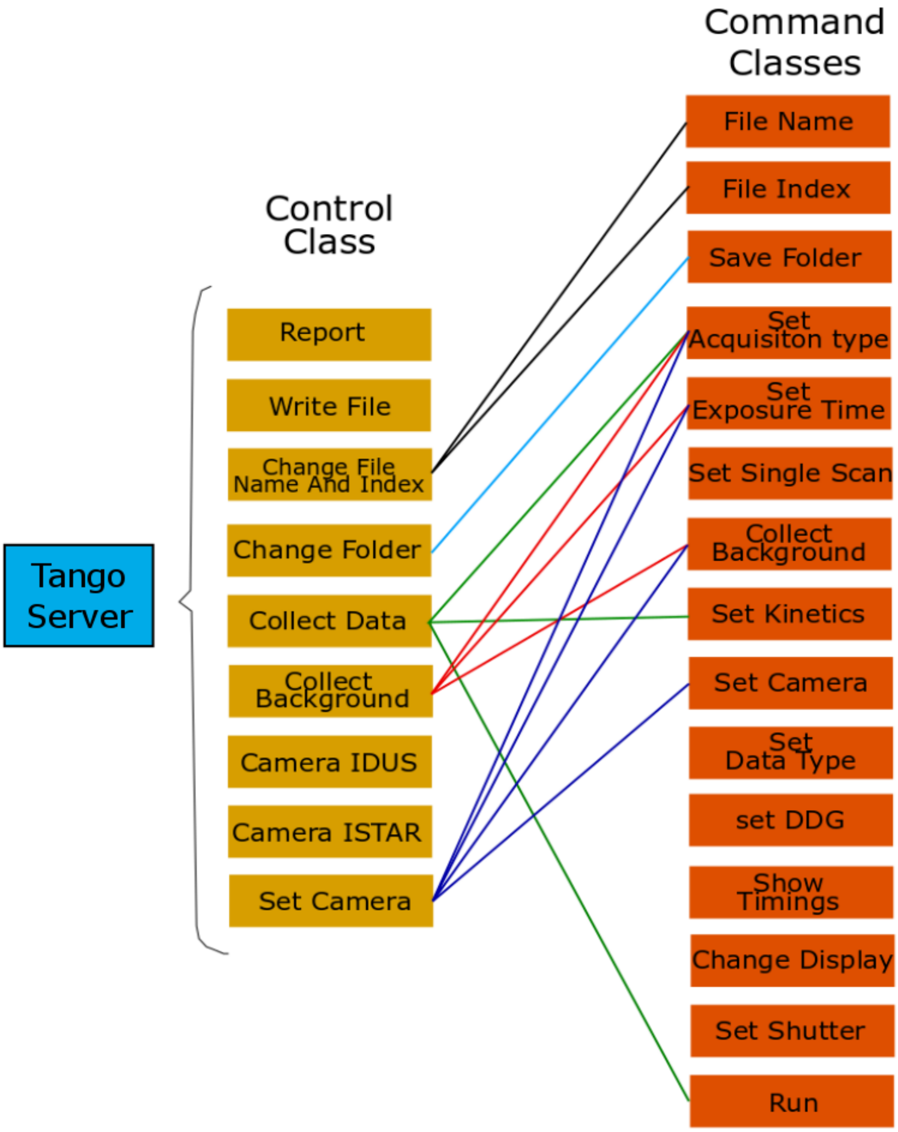

Figure 7:

References

[1] M.Eremets. *High Pressure Experimental Methods*. Oxford University Press, 1996.

[2] N.Subramanian V.Struzhkin A.Kolesnikov M.Somayazulua R.Hemleya A.Goncharov, J.Montoya. Laser heating in diamond anvil cells: developments in pulsed and continuous techniques. *Journal of Synchrotron radiation*, 16:769–772, 2009.

[3] Weiwei Zhang, Artem R. Oganov, Alexander F. Goncharov, Qiang Zhu, Salah Eddine Boulfelfel, Andriy O. Lyakhov, Elissaios Stavrou, Maddury Somayazulu, Vitali B. Prakapenka, and Zuzana Konôpková. Unexpected stable stoichiometries of sodium chlorides. *Science*, 342(6165):1502–1505, 2013.

[4] V.Minkov S.Besedin M.Kuzovnikov S.Mozaffari L.Balicas F.Balakirev D.Graf V.Prakapenka E.Greenberg D.Knyazev M. Tkacz M.Eremets A.Drozdov, P.Kong1. Superconductivity at 250 k in lanthanum hydride under high pressures. *Nature*, 528, 2019.

[5] C.Barthel S.Rekhi J.Tempere I.Silvera S.Deemyad, E.Sterer. Pulsed laser heating and temperature determination in a diamond anvil cell. *Review of Scientific Instruments*, 2005.

[6] David Sands. *Pulsed Laser Heating and Melting*. IntechOpen, 2011.

[7] I. Kupenko S.Linhardt A.Laskin D.Vasiukov V.Cerantola E.Koemets C.McCammon A.Kurnosov A.Chumakov R. Ruffer N. Dubrovinskaia L. Dubrovinsky G.Aprilis, C.Strohm. Portable double-sided pulsed laser heating system for time-resolved geoscience and materials science applications. *Review of Scientific Instruments*, 2017.

[8] Y.Su M.Zhang Z.Tong C.Cui Y.Wang, G.Huang. Numerical analysis of the effects of pulsed laser spot heating parameters on brazing of diamond tools. *Metals*, 612, 2019.

[9] A. N. Magunov. Spectral pyrometry (review). *Research Institute of Advanced Materials and Technology, Moscow State Institute of Electronics and Mathematics*, 2009.

[10] J.Allday; S.Adams. *Advance Physics*. OUP Oxford, 2000.