

Upgrade of the CATS sample changer on FIP-BM30A at the ESRF: towards a commercialized standard

L. Jacquamet,^a J. Joly,^a A. Bertoni,^b P. Charrault,^a M. Pirocchi,^a X. Vernede,^a F. Bouis,^a F. Borel,^a J.-P. Périn,^c T. Denis,^d J.-L. Rechatin^d and J.-L. Ferrer^{a*}

^aGroupe Synchrotron (GSY), Laboratoire de Cristallographie et Cristallogénèse des Protéines (LCCP), Institut de Biologie Structurale J.-P. Ebel, UMR5075 CEA–CNRS–University J. Fourier, 41 rue Jules Horowitz, 38027 Grenoble Cedex 1, France, ^bInstitut de Biologie Structurale J.-P. Ebel, UMR5075 CEA–CNRS–University J. Fourier, 41 rue Jules Horowitz, 38027 Grenoble Cedex 1, France, ^cCommissariat à l’Energie Atomique (DSM/DRFMC/SBT), 17 rue des Martyrs, 38054 Grenoble Cedex 9, France, and ^dIRELEC 20, rue du Tour de l’eau, ZAC Champ Roman, 38400 St Martin d’Hères, France. E-mail: jean-luc.ferrer@ibs.fr

An upgraded version of the sample changer ‘CATS’ (Cryogenic Automated Transfer System) that was developed on the FIP-BM30A beamline at the ESRF is presented. At present, CATS is installed at SLS (three systems), BESSY (one system), DLS (two systems) and APS (four systems for the LSCAT beamline). It consists mainly of an automated Dewar with an assortment of specific grippers designed to obtain a fast and reliable mounting/dismounting rate without jeopardizing the flexibility of the system. The upgraded system has the ability to manage any sample standard stored in any kind of puck.

Keywords: protein crystallography; automation; robotics.

1. Introduction

The first CATS (Cryogenic Automated Transfer System) has been in use since 2004 on the FIP-BM30A beamline at the ESRF (Ohana *et al.*, 2004) thanks to the various structural genomics projects that have encouraged the development of high-throughput technologies. Nowadays, CATS is used by the structural genomics groups, but even more significantly by the other structural communities that take advantage and benefit from it. The CATS developments were pursued in order to obtain a fully automated beamline. In fact, the growing demand for beam time on FIP-BM30A has made it a necessity to increase the efficiency of the experiments. As a result, the automation of the data collection process has developed throughout the years. At first the beamline alignment for different wavelengths and the optimization of the beam intensity were automated (Roth *et al.*, 2002). Today, users can also remotely center their crystals by a simple mouse click or alternatively use an automated procedure [program *C3D* (Lavault *et al.*, 2006)], recently improved by the use of a UV laser (Vernede *et al.*, 2006). Then, fully automated data collection was implemented, and data processing was automated using ‘adp’ (automated data processing) (Ferrer, 2001) and, more recently, DNA (Leslie *et al.*, 2002). Finally, the remote control of the beamline is now available *via* an NX client.

Today, several crystal mounting/dismounting robots are commercially available, or have been developed by academic laboratories at different synchrotron sources. All these robots work with prefrozen crystals stored in a loop attached to a cryo-pin mounted on a metal base. The issue raised in the course of these developments was whether or not to handle the vial of the metal base. Because of this, the existing solutions can be split into two different categories. In the category of those that do not handle the vial, as far as we know, four robotic systems have been academically developed [SAM from SSRL (Cohen *et al.*, 2002), one from ALS (Snell *et al.*, 2004; Rupp *et al.*, 2002), one from Max Planck Research Unit for Structural Molecular Biology (Karain *et al.*, 2002) and one from SPring-8 (Ueno *et al.*, 2004) (this one uses plastic sample pins instead of caps)], and one is commercially available (ACTOR from Rigaku). In the category of those that do handle the vial, to the best of our knowledge, two robotic systems have been academically developed both at the EMBL [SC3 by the EMBL Grenoble Outstation (Cipriani *et al.*, 2006) and one by the EMBL Hamburg Outstation (Pohl *et al.*, 2004)], and three are produced and commercialized by private companies (MARCSC from Mar Research, BruNo from Bruker and, more recently, CATS from IRELEC).

The main limitation of these systems is that they lack versatility, because they can only be used for one task. On the contrary, CATS presents the significant advantage of being

able to handle any kind of sample: prefrozen crystals in any kind of pucks as well as crystals in capillaries, or crystallization plates bearing interesting crystals.

Thus, we will describe here the upgraded version of CATS, as commercialized by IRELEC (<http://www.irelec.com/>), including all the new developments made thanks to a fruitful collaboration between the FIP-BM30A team, the SBT and IRELEC. This upgraded version is a highly versatile, reliable and powerful system. The upgrades from the first CATS version installed on FIP-BM30A (automated and versatile liquid-nitrogen Dewar and different grippers), as well as the various steps of the mounting and dismounting process depending on the sample type, will be presented in detail below.

2. Set-up improvement

2.1. Robot

The main component of the system is a six-axis robot from Stäubli (<http://www.staubli.com/>) (Fig. 1A). The robot features an articulated arm with six degrees of freedom for a higher flexibility. The spherical work envelope allows the maximum utilization of cell workspace. Additional benefits are the floor, wall and ceiling mount options. The robot version has been upgraded since the first CATS was put in operation (Ohana *et al.*, 2004): the RX60L version of the robot used for the first CATS has been replaced by the TX60L version. Upgrading from the RX60L to the TX60L version significantly improved the CATS performances: the load capacity increased by 0.5 kg (today the nominal load capacity is 2 kg), the working envelope went from 800 mm to 920 mm, the repeatability improved by 5 μm (30 μm instead of 35 μm) and, with the implementation of a new Stäubli CS8 controller along with new VAL3 programming software, the precision of the robot trajectories was improved.

2.2. Automated storage Dewar

2.2.1. Cryogenic design. The liquid-nitrogen Dewar was designed according to the state-of-the-art techniques in the domain of cryogenic tanks (Fig. 1A). It is made of two cylindrical stainless steel vessels, separated by a permanent-vacuum volume. The diameter of the external vessel is 650 mm. Specific

attention has been paid to the thermal insulation, which was improved by the use of multi-layer insulation (MLI) blankets (30 layers). The insulating volume is fore-pumped through a DN16CF port and closed by one all-metal manual valve. The quality of the vacuum was improved by adding a small amount of activated charcoal, which fixes some of the molecules out-gazed by the surfaces. This granulated carbon is regenerated by heating the Dewar back to room temperature and by fore-pumping the vacuum volume. This operation must be performed once a year.

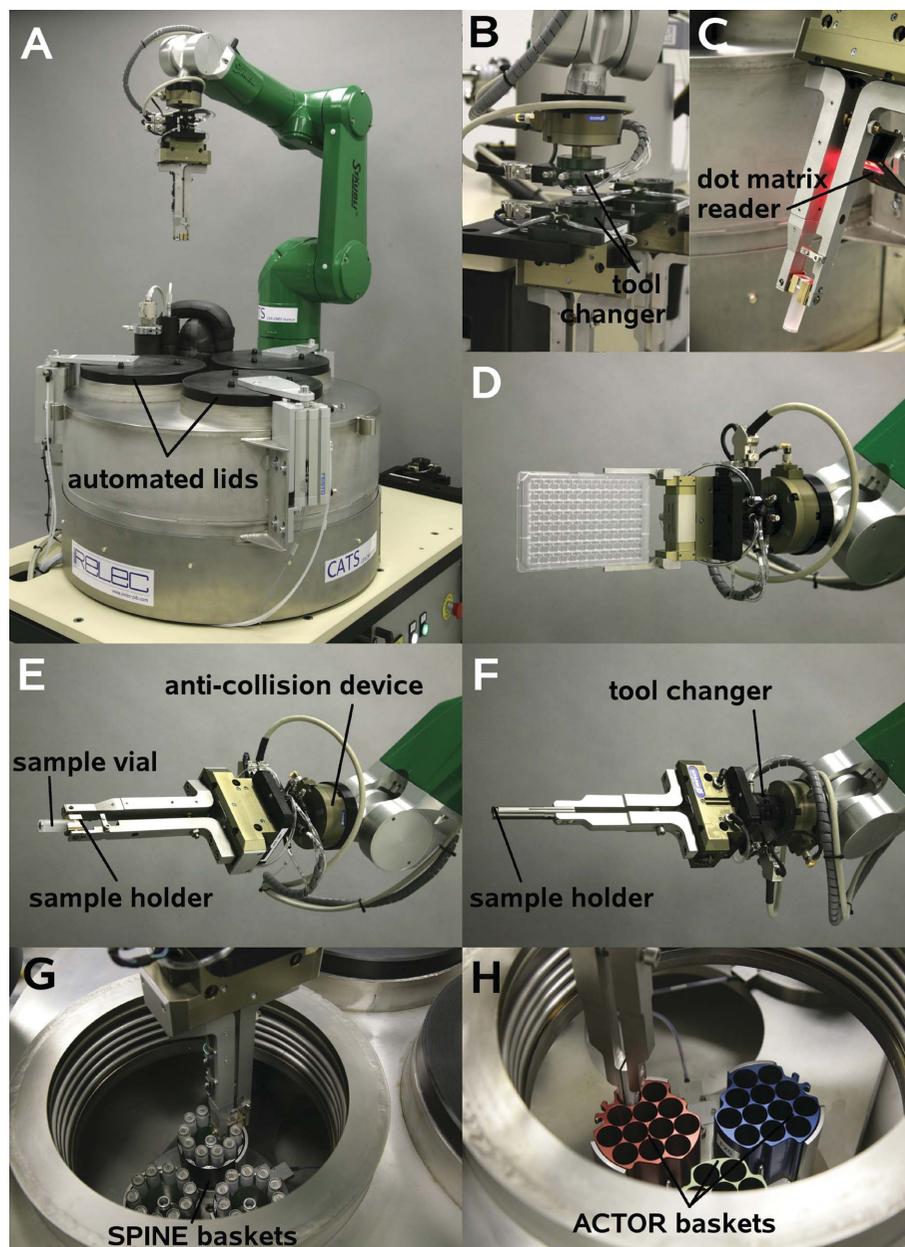


Figure 1

The upgraded version of CATS. (A) General view of the CATS system. (B) Closer view of the tool rack during a tool-changing operation. (C) Closer view of the dot-matrix reader. (D) Magnified view of the plate gripper. (E) Magnified view of the flipping gripper. (F) Magnified view of the sample-holder gripper. (G) Magnified view of an aperture of the Dewar equipped with SPINE baskets, with the gripper retrieving a sample from one of the baskets. (H) Magnified view of an aperture of the Dewar equipped with ACTOR standard baskets, with the gripper retrieving a sample from one of the baskets.

Ultra-high-vacuum (UHV) techniques were used to select the in-vacuum materials and clean the various parts. As part of the manufacturing process of the Dewar, it is thermally 'shocked' three times with liquid nitrogen and the leak tightness of the vessels is checked down to the 10^{-10} mbar l s⁻¹ leak level using an aspersions He leak test.

The top surface of the Dewar is equipped with three openings sealed by foam-insulated lids. Hydro-formed bellows are used in the three ports to link the internal cold vessel to the external one. In addition to their mechanical flexibility, these bellows were designed to increase the thermal path and therefore the thermal insulation. The internal vessel is supported by epoxy parts, which also offer a high thermal resistance.

The Dewar is equipped with a nitrogen exhaust that may be connected to the beamline exhaust. Drying of the Dewar is achieved by injecting nitrogen gas at room temperature. It can also be accelerated by siphoning the remaining liquid nitrogen (a special tool was designed for this purpose) and by heating the internal volume with an infrared lamp placed at the lid position.

2.2.2. Performance. The cryogenic tests of this Dewar show that it features a high thermal insulation. Actually, when the three lids are closed, if the Dewar is not heated only a small amount of condensation appears around the lids. No cold point is detected on the side and bottom surfaces. With the SPINE pucks, the refill cycle of the Dewar (*i.e.* the time to evaporate the liquid nitrogen between the top level and the sample-holder position) is more than 8 h. A low-power heating will be added around the lids to keep the top surface dry in order to prevent the formation of condensation when the lids are frequently opened and cold vapours surround the Dewar.

A spare volume of liquid nitrogen underneath the puck interface acts as a 'dustbin' for the liquid-nitrogen pollution. All the visible ice particles sink in this still volume, which is not disturbed by the automatic refill, and do not interfere with the samples.

2.2.3. Automatic refill. The liquid-nitrogen level is controlled by four sensors. Two of them are used for high- and low-level indication. The other two serve as alarms: they are triggered if the Dewar is overfilled or when the liquid-nitrogen level is too low to guarantee the sample integrity.

Furthermore, an additional sensor is mounted on the liquid-nitrogen inlet to detect the phase of the fluid (gaseous or liquid). As long as the nitrogen is gaseous, it is evacuated to a by-pass circuit. This solution accelerates the cooling of the feeding line, and avoids the entry of warm nitrogen inside the Dewar.

The failure of any one of the sensors is detected, and stops the automatic refill. If the high-level alarm is triggered, all the valves are immediately closed. Opening of the lids can be forbidden when the low-level alarm is triggered.

2.2.4. Automatic lids. The lids are actuated by double-effect pneumatic pistons, which combine a linear and a swivel movement. These actuators were chosen in order to limit the circulation of air and the entry of moisture inside the Dewar

during the opening movement. The lid opening is controlled by proximity sensors.

2.2.5. Configuration and teaching. The configuration of the Dewar can be easily modified at room temperature. It can be equipped with various interfaces corresponding to the various puck standards (Figs. 1G and 1H). Each of the three Dewar openings can be associated with one puck standard. Today, two interfaces have already been designed for SPINE baskets and for 12-position baskets, in which the sample holders are stored without the vials. Additional interfaces can be designed for other standards, such as the American Uni-Puck Project (http://smb.slac.stanford.edu/robosync/Universal_Puck/).

Each interface can receive three baskets of the same standard. For instance, the Dewar can be configured with six cassettes of the first standard in lids 1 and 2, and three of the other one in lid 3. Consequently, depending on the cassette standard (10 or 12 sample holders per cassette), the total capacity of the Dewar varies from 90 to 108 samples. When the Dewar is full of liquid nitrogen, the cassettes are loaded using suitable manual grippers. A future improvement of the sample changer will be loading of the cassettes by the robot arm. Electrical switches are used to check that the magazines are correctly positioned inside the Dewar.

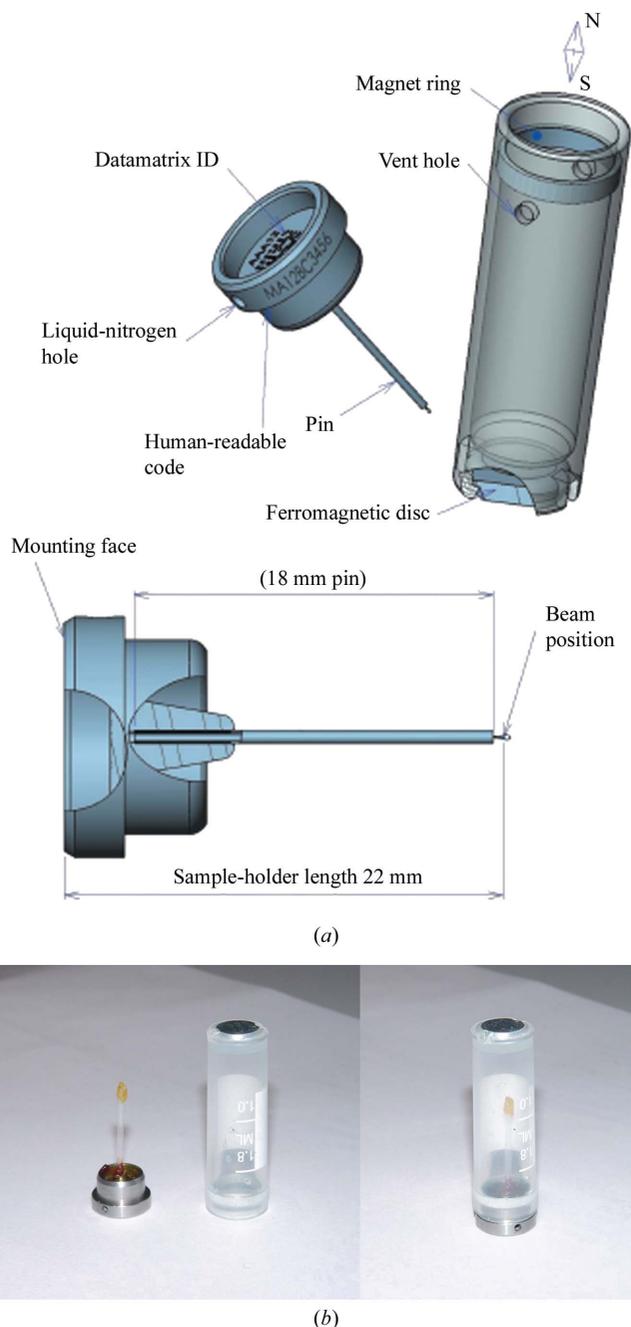
Teaching of the Dewar frame is performed by pointing the three central points of the three lids using the robot gripper. For this operation the gripper is equipped with a teaching pin. This procedure must be performed again with each new gripper. Then, the position of all the samples is automatically calculated for each standard. The reproducibility of the interface positions is mechanically guaranteed. Consequently, the teaching of the Dewar frame has to be performed only once. The taught frame is then kept in the system memory, whatever the Dewar configuration, and is reloaded when the configuration is specified by an API command.

At cryogenic temperatures the teaching is extrapolated by using the specific geometry of the Dewar. Indeed, the Dewar has been specifically designed with a central fixed point, which guarantees a cylindrical symmetry of the thermal contraction and a radial displacement of the puck's interfaces.

2.3. Tools

2.3.1. Sample standard. A lot of effort has been put into the standardization of sample holders of macromolecular crystals (Cipriani *et al.*, 2006). One of the results of these efforts is the SPINE standard, as illustrated in Fig. 2(a). However, there are still two different ways to mount/dismount the sample. One way consists of using both the sample holder and the vial; the other consists of handling the sample holder alone. This is why there are two different categories of robotic systems for mounting/dismounting the sample, as was previously described in the *Introduction*.

2.3.2. Tool changer. One of the biggest improvements in the reliability of CATS is the use of a tool changer [from Schunk (<http://www.schunk.com/>)] (Fig. 1B), which enables the system to automatically change grippers, depending on the sample standard, with a positioning accuracy of 10 µm. Three


Figure 2

(a) The standard sample holder and its vial standardized by SPINE. (b) A modified sample holder for non-frozen samples with a capillary instead of a cryo-pin.

possibilities are offered: (i) when both the sample holder and the vial are handled at the same time, as is the case for the SPINE standard (Fig. 1E), a flipping gripper is used; (ii) when the sample holder is to be handled alone (Fig. 1F), the system uses an overlapping gripper; (iii) when the *in situ* analysis of crystallization drops is to be performed (Jacquamet, Ohana, Joly, Legrand *et al.*, 2004; new manuscript in preparation), the system uses specific grippers that can handle crystallization plates.

2.3.3. Flipping gripper. A rotating gripper has been developed for mounting/dismounting the sample holder and the vial

(Fig. 1E). Since the sample holder is stored face-up in the Dewar, the robot picks up the sample holder and the vial from the top of the vial. In order to mount the sample holder onto the goniometer, the system has to rotate the sample holder. In the first version of CATS, the robot puts the sample holder and the vial on external and intermediate grippers (clamps manufactured by BioFisher). Then, it flips around these grippers and grabs the sample holder and the vial from the side, and finally mounts the sample holder on the goniometer. Since it was found that these external grippers were slowing down the mounting/dismounting processes, it was decided to take advantage of the motion from the Dewar to the goniometer to proceed to the sample-holder rotation. In other words, the sample-holder rotation was to be realised in masked time. In order to do this, the FIP-BM30A team studied a new gripper and machined it in-house. This gripper includes a rotating system driven by a DC motor. It allows a 180° rotation of the sample holder during the transfer from the Dewar to the goniometer. This ‘flipping gripper’ is mounted on a pneumatic system (from Schunk) that enables its opening/closing (this is the object of patent submission EP07013874.8, entitled ‘Flipping grip system for the transfer of macromolecule crystals’).

2.3.4. Sample-holder grippers. When the robot has to deal with the sample holder alone, as is the case with the ACTOR pucks or the Universal pucks, the sample holder does not need to be rotated. However, since the vial is no longer present to preserve the sample integrity in these cases, the grippers have to do it. Special grippers have been studied and machined for this purpose (Fig. 1F). Special care has been taken to maintain the sample integrity and avoid icing problems.

2.3.5. Crystallization-plate grippers. Crystallization-plate grippers are also available as an option in the commercialized CATS systems (Fig. 1D). These grippers grab the plate on its side. They were specifically designed and machined for the SBS plate standard. A description of these grippers, of the related services, and of the new scientific results obtained with the *in situ* analysis will be reported in detail in another manuscript (in preparation).

2.3.6. Heating device. A heating gun needs to be installed in the vicinity of the robot in order to heat the different grippers after each mounting/dismounting process. The heating time depends on the type of gripper. Fifteen seconds are necessary to dry the flipping gripper, while the sample-holder grippers require 60 s to dry.

2.3.7. Barcode reader. To identify and track samples as well as crystallization plates, a barcode reader is provided. Reading for both types of samples is done just after the first grab (Fig. 1C). At the same time, a metal detector (not visible in Fig. 1C) is used to check the conformity of the vial with the SPINE standard.

2.3.8. Graphical user interfaces. The various mounting/dismounting sequences are written in the robotic programming language VAL3. Tcl/Tk graphical user interfaces (GUIs) are available for users to control the handling of one particular sample placed in a carousel in the liquid-nitrogen Dewar, by simply clicking on the sample number (Figs. 3a and 3b).

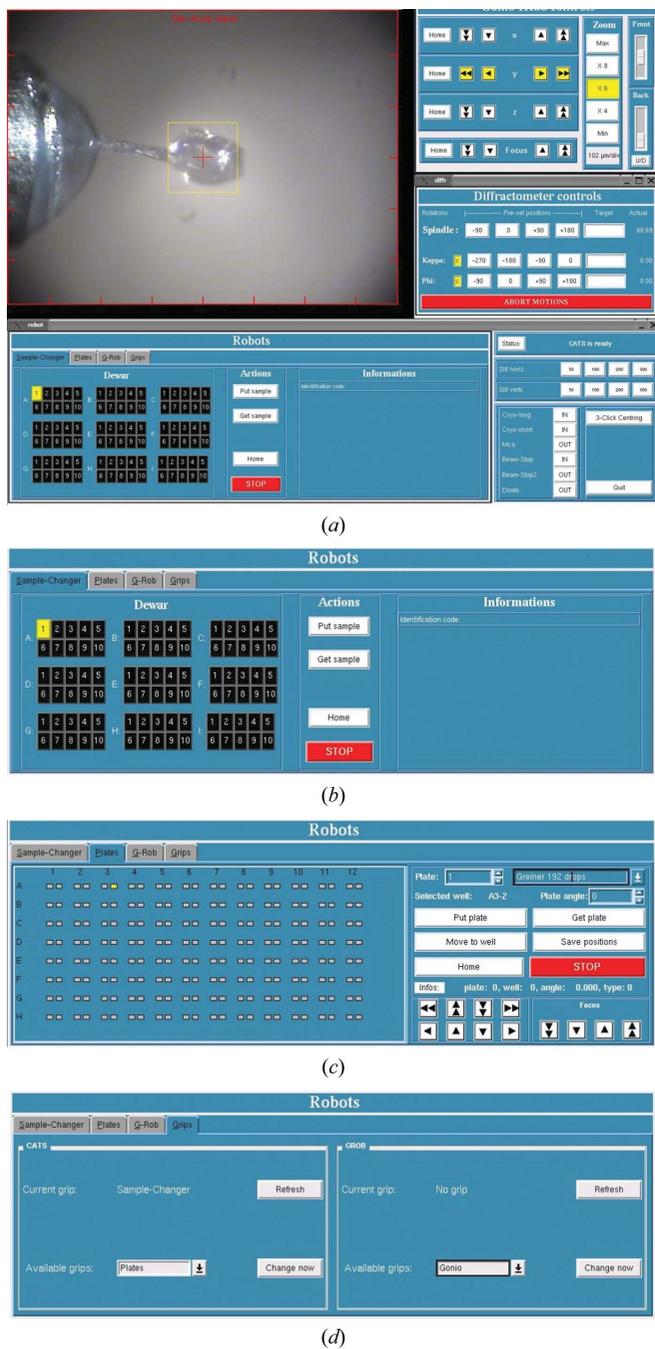


Figure 3
 The Tcl/Tk GUI of CATS for mounting and dismantling samples. (a) Overall view of the interface in the ‘sample changer’ mode. (b) Magnified view of the sample selection window. (c) Magnified view of the plate manipulation window. (d) Magnified view of the tool-changing window.

3. Crystal mounting/dismounting processes

The mounting/dismounting processes depend on the sample type to be mounted. Four different processes have been carefully elaborated, depending on where the crystal is stored: (i) on the sample holder in the vial, (ii) on the sample holder alone, (iii) inside crystallization plates and (iv) inside capillaries. By means of the tool changer, the system automatically mounts the grippers corresponding to the sample to be mounted.

3.1. Crystal stored on the sample holder in the vial

In this case the system first mounts the flipping gripper, if necessary. Then the Dewar lid corresponding to the selected sample is automatically opened.

During the mounting process the sample is grabbed by the gripper and placed in front of a barcode reader in order to be identified. Since the SPINE sample holder is stored inside the Dewar (which is automatically closed after the sample has been grabbed) with the cap at the top, it has to be flipped over by 180° in order to be placed on the goniometer. This rotation is ensured by the internal mechanisms of the flipping gripper (patent submission EP07013874.8, in progress), and is performed during the time required to move from the Dewar to the goniometer. The rotation inside the flipping gripper is synchronized with the robot rotation in order to keep all of the liquid nitrogen inside the vial. As a result, more than three-quarters of the vial is still full of liquid nitrogen, right before the sample is placed horizontally on the goniometer head. This last motion was carefully designed in terms of speed and trajectory in order to make sure that one-third of the liquid nitrogen remains inside the vial when it is back in the vertical position. Then the vial is returned to its original position. This last motion is synchronized with the automatic opening of the lid of the Dewar. Then the flipping gripper is warmed up in front of a heat gun for a time between 15 and 25 s.

The various sequences of the dismantling process are strictly identical to those described for the mounting process. The presence of one-third of the liquid nitrogen inside the vial when the system retrieves the sample holder ensures the absolute integrity of the crystal and a highly reliable dismantling process. Details of the different cycling times are given in Table 1(a).

A special dismantling/mounting procedure has been designed for high-throughput screening. In this case the mounting process follows directly the dismantling, without warming the gripper or the Dewar closing/opening steps. During the screening process of a series of samples, the CATS cycle can be split into two parts: (i) a dead-time of 25 s (no sample mounted on the goniometer), and (ii) a ‘user’ time of 65 s (warming of the gripper *etc.*) including sample centering and exposure to the X-ray beam. On FIP-BM30A this ‘user’ time matches quite well with the time needed for centering the sample [about 35 s when done automatically using the *C3D* program (Lavault *et al.*, 2006), and between 30 and 45 s when done manually by the user] and collecting two diffraction frames of 15 s each, in two orientations, at a 90° angle. Thus, the resulting screening rate is of 40 samples per hour, *i.e.* 90 s between samples. Optimization of the ‘user’ time may be necessary on intense beamlines where the exposure time is much shorter, and will certainly be necessary when reliable and fast automated sample centering processes are available.

3.2. Crystal stored on the sample holder with no vial

First, the system mounts the sample-holder grippers, if necessary. Then the Dewar lid corresponding to the selected sample is automatically opened.

Table 1

(a) Cycle times: flipping gripper.

Time (s)	State/action	Comments/events
Sample mounting		
0	Home position	'Put sample' command sent
15	Transfer of the sample from the Dewar to the goniometer Sample holder mounted	Sample ready for centering
25	Transfer of the empty vial back to the Dewar Vial back in the Dewar Closing Dewar, warming of the gripper (15 s)	
55	End of the trajectory	Ready for a 'Get sample' cycle
Sample dismounting		
0	Home position	'Get sample' command sent
15	Transfer of the empty vial from the Dewar to the goniometer Sample holder covered by the vial Transfer of the sample back to the Dewar	
25	Sample holder in the vial back in Dewar Closing Dewar, warming of the gripper (15 s)	
55	End of the trajectory	Ready for a 'Put sample' cycle
Sample mounting immediately after the dismounting		
0	Home position	'Get/put sample' command sent
15	Transfer of the empty vial from the Dewar to the goniometer Sample holder covered by the vial Transfer of the sample back to the Dewar	
25	Sample holder in the vial back in the Dewar Transfer of the sample from the Dewar to the goniometer	Starting of a 'Put sample' cycle
40	New sample mounted	25 s between the removal of the first sample and the mounting of the new one
50	Transfer of the empty vial back to the Dewar Vial back in the Dewar Closing Dewar, warming of the gripper (25 s)	
90	End of the trajectory	Ready for a new cycle

(b) Cycle times: sample-holder grippers (for all the following processes the chilling time is 40 s and the warming time is 60 s).

Time (s)	State/action	Comments/events
Sample mounting		
0	Home position	'Put sample' command sent
57	Chilling of the gripper and transfer of the sample from the Dewar to the goniometer Sample holder mounted Gripper moving back for warming up	
130	End of the trajectory	Ready for a 'Get sample' cycle

Table 1 (continued)

Time (s)	State/action	Comments/events
Sample dismounting		
0	Home position	'Get sample' command sent
50	Chilling of the gripper and moving to the mounted sample Sample holder covered by the grippers Gripper moving back to the Dewar	
60	Sample holder back in the Dewar Closing Dewar and warming up of the gripper	
135	End of the trajectory	Ready for a 'Put sample' cycle
Sample mounting immediately after the dismounting		
0	Home position	'Get/put sample' command sent
50	Chilling of the gripper and moving to the mounted sample Sample holder covered by the grippers Gripper moving back to the Dewar	
60	Sample holder back in the Dewar Transfer of the new sample from the Dewar to the goniometer	
80	New sample holder mounted	30 s between the removal of the first sample and the mounting of the new one
155	Gripper moving back for warming up End of the trajectory	Ready for a new cycle

During the mounting process, the grippers are first cooled down for 40 s (which is the time needed to stop the boiling) in the liquid nitrogen contained in the Dewar. Then the sample holder is grabbed by the grippers and placed in front of a barcode reader in order to be identified and recorded. Since the sample holder is stored inside the Dewar with the cap at the bottom, the system can place it directly onto the goniometer. The grippers are then placed in front of the heat gun and 60 s are necessary to warm up and completely dry the grippers.

The various sequences described for the dismounting process are strictly identical to those described for the mounting process. To retrieve the sample the grippers grab the sample holder. They have a sufficient heat capacity to preserve the sample integrity during transfer back to the Dewar. Details of the different cycling times are given in Table 1(b).

Using the dismounting/mounting procedure described above during the screening process of a series of samples, the CATS cycle can be split into two parts: (i) a dead-time of 30 s (no sample mounted on the goniometer), and (ii) a 'user' time of 125 s (chilling/warming of the gripper *etc.*), including sample centering and exposure to the X-ray beam. However, in this case this second phase is likely to be longer than the necessary time for centering and collecting one or two diffraction frames, and therefore slows down the screening process. The screening rate is then only 23 samples per hour, *i.e.* 155 s between samples, at best. This makes a significant

difference in cycle time compared with the case of a sample holder transferred with its vial. This is a consequence of the need to bring the sample-holder grippers to the temperature of liquid nitrogen before each sample manipulation.

3.3. Crystal stored inside crystallization plates

The system has the unique ability to place the crystallization plates directly in the X-ray beam for data collection; feasibility tests were published in 2004 (Jacquamet, Ohana, Joly, Legrand *et al.*, 2004). Today, an *in situ* analysis service is open to academic and industrial users on beamline FIP-BM30A. As long as the crystallization plate fits the SBS standard, the plates, stored in a ten-magazine hotel at room temperature, can be mounted inside the grippers, and the corresponding crystallization drops can be viewed simultaneously by a CCD camera and by the X-ray beam. Since 2004, efforts have been made to obtain a reliable system. Tests on different crystallization plates have been successfully carried out ever since. New interesting scientific results have been obtained recently and will be published soon (manuscript in preparation).

3.4. Crystal stored inside capillaries

Finally, the system has the ability to mount crystals even if they have been placed inside a capillary. The only requirement is the standardization of the capillary holder. On FIP-BM30A, capillaries are fixed on caps that are placed in vials (Fig. 2b) and stored on a rack. Upon user request, the system grabs the flipping gripper and mounts/dismounts capillaries for X-ray diffraction experiments. Academic and industrial users can use CATS to perform capillary experiments on FIP-BM30A.

4. Results

4.1. CATS: a reliable system

The reliability of the CATS system has already been proved and described in detail (Ohana *et al.*, 2004). The upgraded version, as described here, is even more reliable. It is now a commercial product with two new grippers. The grippers have been developed with the knowledge and the background of previous grippers. As a consequence, their functionalities and efficiency have been improved. However, the complete reliability of the system in normal operation is difficult to evaluate, as a large part of the failures observed in normal operation on beamline FIP-BM30A are a consequence of user error (vial with no cap, sample outside of the accepted standard *etc.*). In the last few months about 10% of sample mounting/dismounting cycles failed (for between 500 and 1000 cycles per month of operation) owing to user error, most being associated with the use of non-SPINE standard vials. Failures with no user error detected represented less than 1% of the cycles. The intrinsic reliability of the process was also assessed by a separate evaluation of the different steps through normalized acceptance tests. The flipping gripper, which involves a delicate mechanism, was first tested at the factory (no failure for 10000 cycles in a row at room temperature), then on the FIP-BM30A beamline (no failure for 500 cycles in a row, each cycle

consisting of retrieving a frozen sample from the Dewar, flipping it back and forth, bringing it back to the Dewar, and drying the gripper). Another acceptance test for the grippers was designed to guarantee that no ice appeared on the samples: ten mounting/dismounting processes were performed on various crystals, and the diffraction patterns recorded after each cycle were carefully inspected for ice rings. No ice formation was detected for any of these tests. The main reason for this is the presence of liquid nitrogen around the crystal on both grippers during the mounting/dismounting.

4.2. CATS: a versatile system

The versatility of the CATS system comes mainly from the choice of robust industrial components, the first of them being the Staubli robotic arm. This high degree of versatility was established in the first version (Ohana *et al.*, 2004; Jacquamet, Ohana, Joly, Borel *et al.*, 2004; Jacquamet, Ohana, Joly, Legrand *et al.*, 2004), was greatly improved in the newly upgraded system, and will continue to be steadily improved in the future. As an example, on one of the CATS systems installed at SLS the robot will be installed upside down to fit into the restrained volume of the mini-hutch.

Compared with the existing systems, CATS has the unique ability to manage crystals no matter how they are stored. CATS is the only system that can ensure the mounting/dismounting of crystals stored on crystal holders in vials, on crystal holders alone, inside capillaries and inside crystallization plates.

However, it is important to note that in the case of the prefrozen crystal mounting/dismounting the use of the vial ensures a real gain of cycling time (see Table 1). As a consequence, only the process using the vial should be considered as a high-throughput process. CATS can furthermore be interfaced with any available control software, including EPIC, SPEC and TANGO, and can provide hardware signals to other devices present in the robot vicinity (goniometer, detector *etc.*).

Therefore, CATS is a highly versatile and powerful system that can also be easily modified and adapted to any new user requirement or any new standard developed in the future by the X-ray crystallography community.

5. Conclusion

CATS is a highly reliable, versatile and powerful system developed on the FIP-BM30A beamline at the ESRF to mount and dismount any kinds of sample (prefrozen crystals, crystals in capillaries *etc.*).

Compared with the other robotic systems available, CATS is an evolutive and adaptive system that is not limited by the present sample standards. CATS can easily be adapted to any new sample standard or experimental procedure.

The upgrade and commercialized version is the fruit of a successful collaboration between the FIP-BM30A team, the

SBT and a private company, IRELEC. We would like to thank all the FIP-BM30A users for their extensive tests of the CATS system, and for having shared with us their experience in that field.

References

- Cipriani, F. *et al.* (2006). *Acta Cryst.* **D62**, 1251–1259.
- Cohen, A. E., Ellis, P. J., Miller, M. D., Deacon, A. M. & Phizackerley, R. P. (2002). *J. Appl. Cryst.* **35**, 720–726.
- Ferrer, J.-L. (2001). *Acta Cryst.* **D57**, 1752–1753.
- Jacquamet, L., Ohana, J., Joly, J., Borel, F., Pirocchi, M., Charrault, P., Bertoni, A., Israel-Gouy, P., Carpentier, P., Kozielski, F., Blot, D. & Ferrer, J. L. (2004). *Structure*, **12**, 1219–1225.
- Jacquamet, L., Ohana, J., Joly, J., Legrand, P., Kahn, R., Borel, F., Pirocchi, M., Charrault, P., Carpentier, P. & Ferrer, J.-L. (2004). *Acta Cryst.* **D60**, 888–894.
- Karain, W. I., Bourenkov, G. P., Blume, H. & Bartunik, H. D. (2002). *Acta Cryst.* **D58**, 1519–1522.
- Lavault, B., Ravelli, R. B. G. & Cipriani, F. (2006). *Acta Cryst.* **D62**, 1348–1357.
- Leslie, A. G. W., Powell, H. R., Winter, G., Svensson, O., Spruce, D., McSweeney, S., Love, D., Kinder, S., Duke, E. & Nave, C. (2002). *Acta Cryst.* **D58**, 1924–1928.
- Ohana, J., Jacquamet, L., Joly, J., Bertoni, A., Taunier, P., Michel, L., Charrault, P., Pirocchi, M., Carpentier, P., Borel, F., Kahn, R. & Ferrer, J.-L. (2004). *J. Appl. Cryst.* **37**, 72–77.
- Pohl, E., Ristau, U., Gehrman, T., Jahn, D., Robrahn, B., Malthan, D., Dobler, H. & Hermes, C. (2004). *J. Synchrotron Rad.* **11**, 372–377.
- Roth, M., Carpentier, P., Kaikati, O., Joly, J., Charrault, P., Pirocchi, M., Kahn, R., Fanchon, E., Jacquamet, L., Borel, F., Bertoni, A., Israel-Gouy, P. & Ferrer, J.-L. (2002). *Acta Cryst.* **D58**, 805–814.
- Rupp, B., Segelke, B. W., Krupka, H. I., Legin, T., Schäfer, J., Zemla, A., Toppani, D., Snell, G. & Earnest, T. (2002). *Acta Cryst.* **D58**, 1514–1518.
- Snell, G., Cork, C., Nordmeyer, R., Cornell, E., Meigs, G., Yegian, D., Jaklevic, J., Jin, J., Stevens, R. C. & Earnest, T. (2004). *Structure*, **12**, 537–545.
- Ueno, G., Hirose, R., Ida, K., Kumasaka, T. & Yamamoto, M. (2004). *J. Appl. Cryst.* **37**, 867–873.
- Vernede, X., Lavault, B., Ohana, J., Nurizzo, D., Joly, J., Jacquamet, L., Felisaz, F., Cipriani, F. & Bourgeois, D. (2006). *Acta Cryst.* **D62**, 253–261.