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FAST X-RAY SHUTTER FOR LAUE PROTEIN CRYSTALLOGRAPHY

by

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Fast X - Ray Shutter for Laue Protein Crystallography.

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Abstract

The two x-ray fast shutters used on the Laue diffraction stations 9.7 and 9.5 of the Daresbury SRS are described. By using two types of shutter, opening times in the range of 50μs upwards can be achieved. Data have been collected using both types of shutter, and some typical diffraction patterns presented.

Contents

1) Introduction

In Laue crystallography, a polychromatic x ray beam from a storage ring (0.2Å to 2.5Å) strikes a stationary crystal and the resulting diffraction pattern is recorded on a subsequent detector (usually multiple films). In this way a diffraction pattern from a "typical" protein can be recorded in a few hundred milliseconds. A fast shutter "solenoid" was developed at Oxford University (ref 1), and later refined at Daresbury to give continuously variable opening times greater than 20 ms, in order to achieve these short exposures.

The new Laue diffraction station at the SRS (Station 9.5, ref 2) offers focussing optics and shortened exposure times in Laue mode by up to a factor of 60 (ref 3), and recent developments in detector technology (image plates, CCD's) are further shortening exposure times (in the case of the image plate by up to a factor of 20 dependent on wavelength). Thus required exposure times for a typical protein sample under the above conditions on station 9.5 can be around 1ms, and are down in the few hundred microsecond range for strongly diffracting "small" molecule crystals. A new very fast shutter "rotating cylinder" that can reproducibly isolate a 50µs X ray pulse is described. The shutter has been designed to be completely user transparent, although a command is required from the user when switching between shutters. It is driven via a software package which allows continuously variable exposure times between 50µs and 20 seconds (overlapping with the range of usefulness of the fast shutter).

2) Fast "Solenoid" Shutter

The shutter consists of a mechanical body and an electronic firing unit. The body (fig 1) has two solenoids on an aluminium mount (a) with a brass slide mechanism (b) for two Tungsten Carbide plates (c). Each plate contains a 6mm aperture which aligns with a window on the body when the solenoids are energised. The solenoids are modified to obtain the correct pull in and drop out characteristics needed to achieve required opening times.

The firing unit is contained in a metal case (300*200*100mm) and is designed to accept either timed TTL or open collector type inputs. There is also a manual override switch fitted to open the shutter for manual alignment. Input pulses of the required opening times are fed into the box by means of a single pin LEMO connector. The input stages use transistors and pull up resistors to arrange the correct logic for the inverting input of the Operational Amplifier (fig 2). The Op-Amp working at unity gain then acts as a fast switch for a reed relay. The relay is held permanently on under normal conditions (i.e. shutter closed) and released when opened. The coils are permanently connected to +74 Volts on one side and the other to the collector of a power transistor via a Resistor-Capacitor (RC) network.

When the relay contacts open the transistor is turned hard on and therefore allowing current to flow through the solenoid coils to ground. The coils being rated at 24 Volts energise very quickly and would burn out if held at this voltage. So due to the values of the RC network the full voltage of +74 Volts remains across the coils for only approximately 10 ms. The voltage then drops to a holding voltage of just under +24 volts so as to protect the coils from burning out.

3) Very Fast "Rotating Cylinder" Shutter

This very fast shutter consists of two slotted tungsten carbide cylinders mounted in opposition to one another (fig 3). Two DC motors are used to drive the cylinders, each having an encoder attached for feedback control. Each cylinder is referred to as "open" when its slots are aligned so as to allow radiation through. These motors and cylinders are mounted in a body that sits vertically on an optical bench. One motor, known as the fast motor, is meant for driving continuously at fast speed. The other motor, known as the slow motor is required to reliably and rapidly accelerate to what are relatively slow velocities. The fast motor drives the larger outer cylinder and the slow motor drives the inner cylinder (fig 4). Both motors produce a single 'index' pulse once per revolution when in known positions. Drive cards for the motors and the microprocessor control systems are mounted in a standard rack mounted 19" case. The control system (fig 5) uses two Motorola 68008 16 bit microprocessors connected on a common bus , optimising maximum control of both motors.

Programming of the shutter is achieved through an RS232 port which is set at 9600 baud, 7 data bits, 1 stop bit and even parity. Each motor has its own unique address which is placed at the beginning of instructions intended for a specific motor. Instructions are further composed of two character mnemonics followed by optional values. Verification of commands executed successfully, returned values or error conditions, are reported back along the RS232 line. Commands are sent to the control system from software, written in Fortran, presently running on a DEC LSI-11 computer.

Figures 7 and 8 show a model of how the system works. Circles represent processes within the system, squares are external items, solid lines are data flows and dashed lines are control flows. Unlabelled control flows represent the activation of processes. The solid horizontal bar in figure 8 represents the transformation of control for the system and is decomposed into figure 9, the state transition diagram. This last diagram shows states the system can be in and pairs of conditions and actions between them. The conditions are the requirements to go between states and the actions are those that take place as a result. Figure 6 shows an extract from the data dictionary explaining some of the data and control flows in more detail. The figures 7, 8 and 9 should be read in conjunction with the "order of events" as described below.

For exposure times below 5ms the fast motor is driven continuously at constant velocity and the slow cylinder is flipped through a single open position. The starting position and velocity of the slow cylinder are calculated so that the time it takes to reach its fully open position is the same as that the fast shutter will take from its index position to fully open. These values also dictate a single opening of the fast cylinder whilst the slow cylinder is open. For opening times greater than 5ms the slow cylinder is held stationary in its open position whilst the fast cylinder is flipped half a revolution through one open position.

Having received the 'exposure required' signal from the user, the system calculates the motor set up and prepares the motors for an exposure. This will involve setting such things as motor accelerations, deccelerations, velocities and positions. The system is now ready for an exposure and requires the shutter enable trigger pulse. This open collector pulse is a latched output from the fast trigger hardware, set from the software by a fast CAMAC output driver pulse. This latch is held at its trigger level whilst waiting for the index from the fast motor. When both pulses are present at the correct level, the slow motor is moved. Exposure times less than 5ms also require the fast index pulse to allow the slow motor to flip. For all exposures the trigger pulse is reset on the falling edge of the fast motors index pulse, ready for the next exposure as shown in fig 10.

4) Test results

Fast Shutter "Solenoid"

Tests using a bright white light as the source and standard photographic camera for the detector, showed that the minimum exposure time lay somewhere between 16ms and 33ms. From the photograph (fig 11), it can be seen that at 16ms the shutter was not fully open.

When tested using a red laser and photo diode coupled up to an oscilloscope, a more accurate measurement was achieved, giving a minimum opening time of 18ms (Fig 12). The results were later confirmed when used on station 9.5, where by an ionisation chamber and a fast rise time current amplifier were used to find the minimum exposure time.

Very Fast Shutter "Rotating Cylinder"

Initial tests were performed using a red laser, photo diode and shaping amplifier, which gave square pulses for ease of measurement. This provided experimental confirmation that isolated pulses could be achieved as shown in fig 13, and that the calculated speeds, start positions and acceleration rates were also correct. These values being crucial to the operation of the shutter along with the index positions. Each index is set with respect to opening, which is defined as the shutter zero offset position.

The useful range of the shutter is far greater than that of the table, the upper limit being 20 seconds which is governed by the 'step' rate of the motors.

These results were later confirmed using the X-ray beam on station 9.5 using a fast rise time amplifier and ionisation chamber to measure opening times. Some typical Laue exposures are given in figs 14 and 15.

5) Conclusions

The fast "solenoid" shutter has been used successfully on both stations 9.7 and 9.5 of the Daresbury SRS for the last 2 years, but is likely to be superceded by the new very fast "rotating cylinder" shutter on station 9.5, where by the additional flux from the focussed optics, and the use of an image plate will make exposure times very much shorter.

A second rotating cylinder shutter is to be designed in order to reduce the minimum exposure time still further.

References

[1] J. Hajdu (Oxford University) Unpublished

[2] R.C. Brammer, J.R. Helliwell, W. Lamb, A. Liljas, P.R. Moore, A.W. Thompson and K.A. Rathbone et al **Nucl.Instrum.Meth** A 271 (1988) 678-687

[3] J.R. Helliwell et al **Daresbury Laboratory Internal Report** In preparation

Figure Captions

Fig 1. Photograph showing the solenoid shutter with labels a, main body, b, slide mechanism for the shutter plates and c, Tungsten Carbide plates.

Fig 2. This figure shows the circuit diagram of the solenoid shutter firing unit. One of two possible input signals turns off transistor TR2, this in-turn switches a fast OP-Amp which fires a reed relay. The contacts of the relay then turn on transistor TR1 which provides a path for current flowing through the coils of the solenoids attached to the outputs.

Fig 3. Photograph showing the rotating cylinder shutter with labels a, fast cylinder, b, main body and c, slow cylinder.

Fig 4. The figure shows various characteristics of the cylinders used in the very fast shutter.

Fig 5. A block diagram of the rotating cylinder shutter control system is given in this figure.

Fig 6. This figure gives a more detailed explanation of data and control flows used in figures 7, 8 and 9.

Fig 7. Context diagram for very fast shutter system, showing the whole software system in it's environment.

Fig 8. Decomposed dataflow diagram for very fast shutter system, showing decomposition of the system from figure 5.

Fig 9. State transition diagram for the very fast shutter system, showing transfer motion of control in the system.

Fig 10. Timing diagram for the very fast shutter, showing the fast index, CAMAC output and trigger output pulses.

Fig 11. The photograph shows that when given a 16 ms pulse from the camera x-sync output the solenoid shutter is only part open.

Fig 12. This plot shows the solenoid shutter producing an 18 ms pulse onto a photo diode connected to an oscilloscope. The light source used was a red laser which was positioned approximately 1 metre away, with the shutter in the centre.

Fig 13. This figure shows three traces of the rotating cylinder shutter when opened for 50 us using a red laser and a photo diode with fast amplifier. Distance between the source and detector was approximately 1 metre and the laser beam reduced at source to approximately 0.5 mm diameter. The top trace shows repeated 50 us pulses on a 4 ms oscilloscope time base, the middle an isolated 50 us pulse on a 5ms time base and the bottom trace showing a magnified pulse on a 50 us time base.

Fig 14. This figure shows a diffraction pattern from a crystal of the protein concanavlin-A, and was recorded using the solenoid shutter on station 9.5 of the Daresbury SRS in 20 ms (beam current 132 mA)

Fig 15. The same crystal as in Fig 14 was used to record this diffraction pattern (900 us, 200 mA)

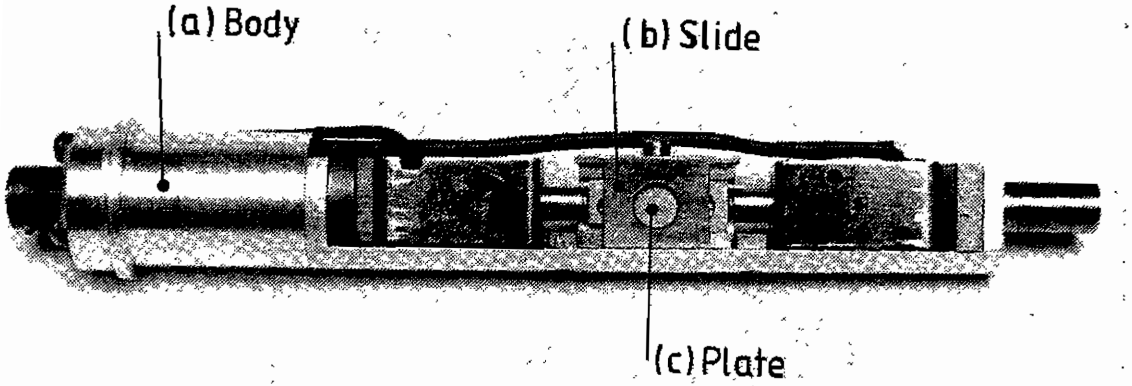


Fig. 1

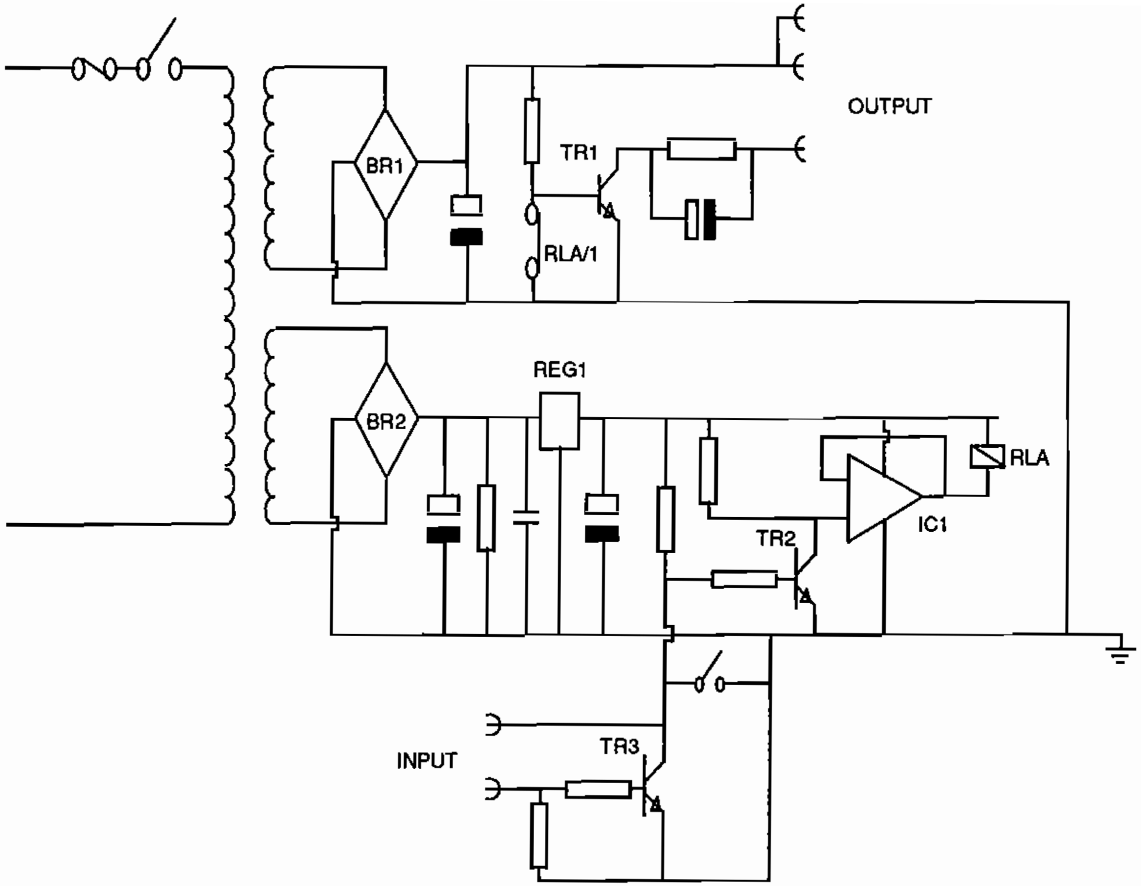


Fig 2.

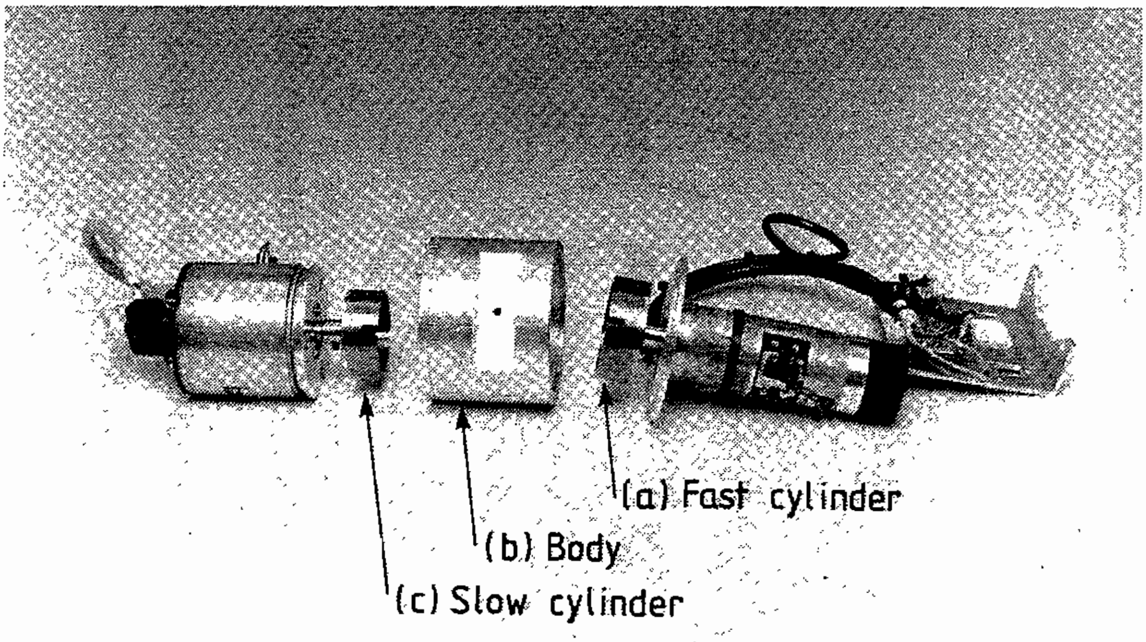
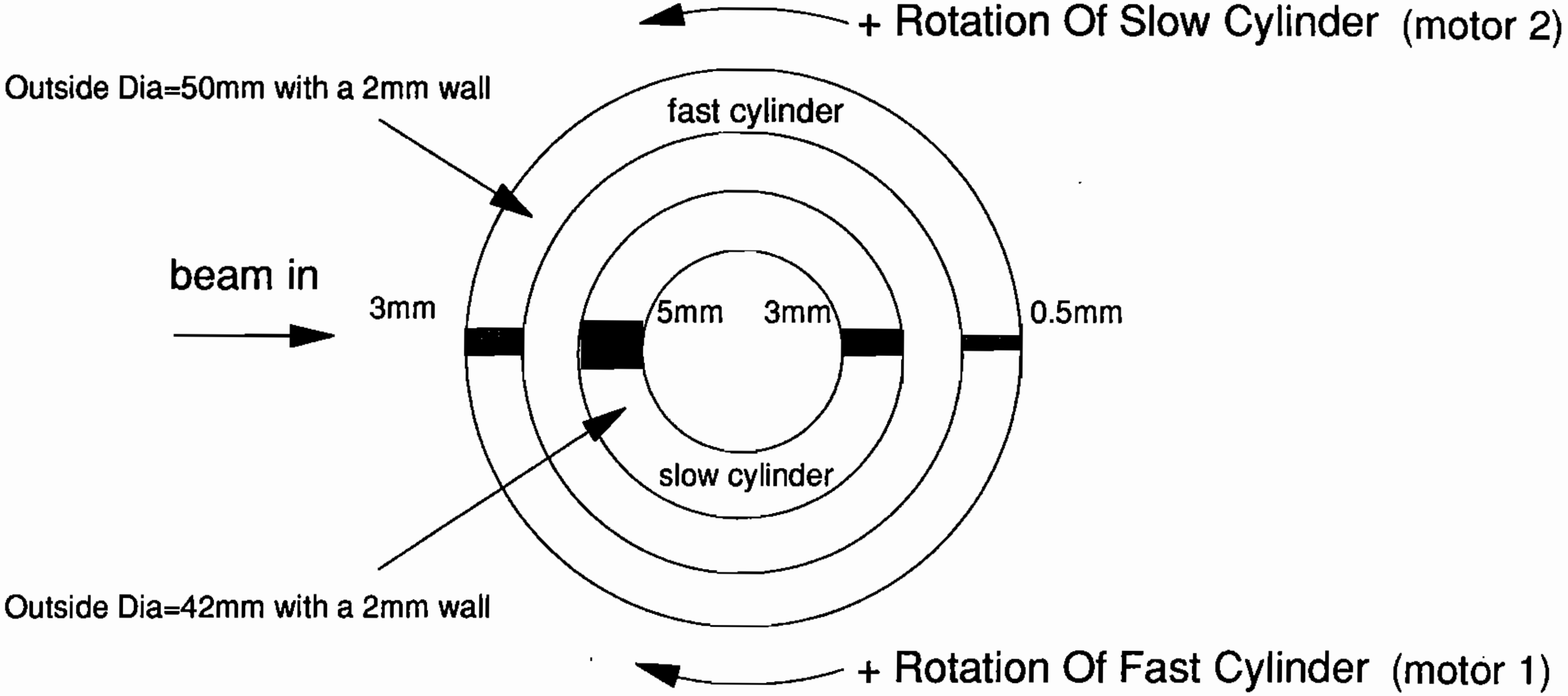


Fig. 3



Top View Of Cylinders

Cylinders are aligned in the zero position

There are 2000 encoder steps per revolution

Fig 4.

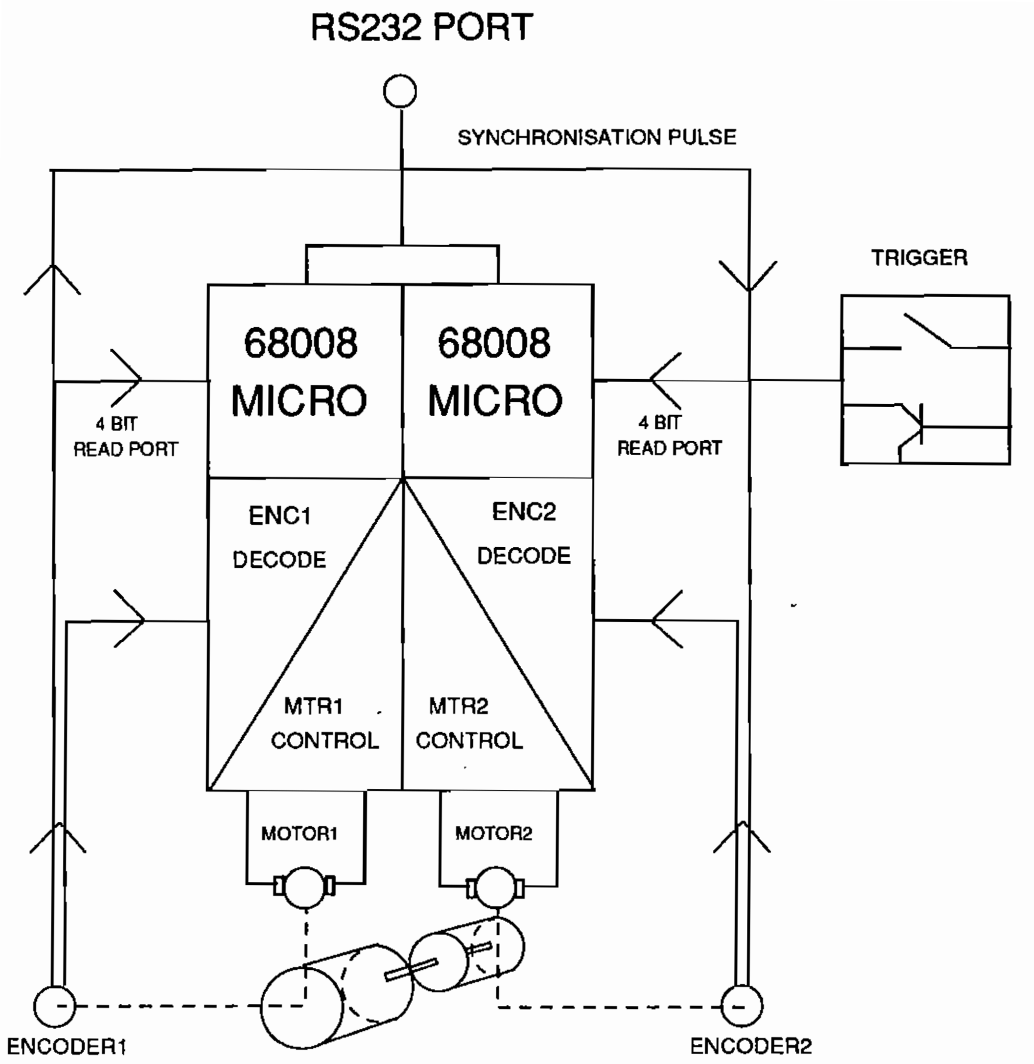

RS232 PORT
SYNCHRONISATION PULSE
TRIGGER
68008
MICRO
68008
MICRO
4 BIT
READ PORT
4 BIT
READ PORT
ENC1
DECODE
ENC2
DECODE
MTR1
CONTROL
MTR2
CONTROL
MOTOR1
MOTOR2
ENCODER1
ENCODER2

Fig 5.

StP Data Dictionary Report - Data Elements

Element Name: **fast_index**

A pulse given from the fast motor one per revolution when the fast shutter is in a known position.

Element Name: **fast_motor_command**

A sequence of characters representing a command, addressed to the 'fast' motor and optionally containing a value, sent to the motor control system over RS232.

Element Name: **fast_motor_reply**

A sequence of characters, possibly containing a value, representing the motor control system's response to a command executed by the 'fast' motor. Received over RS232.

Element Name: **motor_setup**

A number of parameters required to position and program both motors for an exposure.

Slow velocity.
Fast velocity.
Encoder offset of motor to be flipped from fully open.

Element Name: **short_time_mode**

A signal indicating whether the shutter open time is short enough to necessitate driving the fast shutter continuously and flipping the slow shutter (TRUE), or it will be ok to keep the slow shutter open and flip the fast shutter (FALSE).

Element Name: **shutter_enable**

A signal generated (TRUE) by the 'fast shutter trigger interface' either on receipt of a CAMAC pulse generated by the control software or a front panel button.

Element Name: **shutter_parameters**

A number of parameters which remain constant during a run of the program but are required to control the shutter system. There may be reason to change these however and this can be done by editing the disk file they reside in.

Widths of all four slits in the cylinders.
Diameters of cylinders.
Number of encoder steps per revolution of cylinders.
Encoder value at index positions of each cylinder.
Accelerations of motors.
Time barrier between the two methods of exposure.
Fixed offset of fast shutter for long exposures.
Positional accuracy applied by motor control system.
Settling time applied by motor control system.
Creep speeds applied by motor control system.
Creep distances applied by motor control system.

Element Name: **slow_motor_command**

A sequence of characters representing a command, addressed to the 'slow' motor and optionally containing a value, sent to the motor control system over RS232.

Element Name: **slow_motor_reply**

A sequence of characters, possibly containing a value, representing the motor control system's response to a command executed by the 'slow' motor. Received over RS232.

Fig. 6

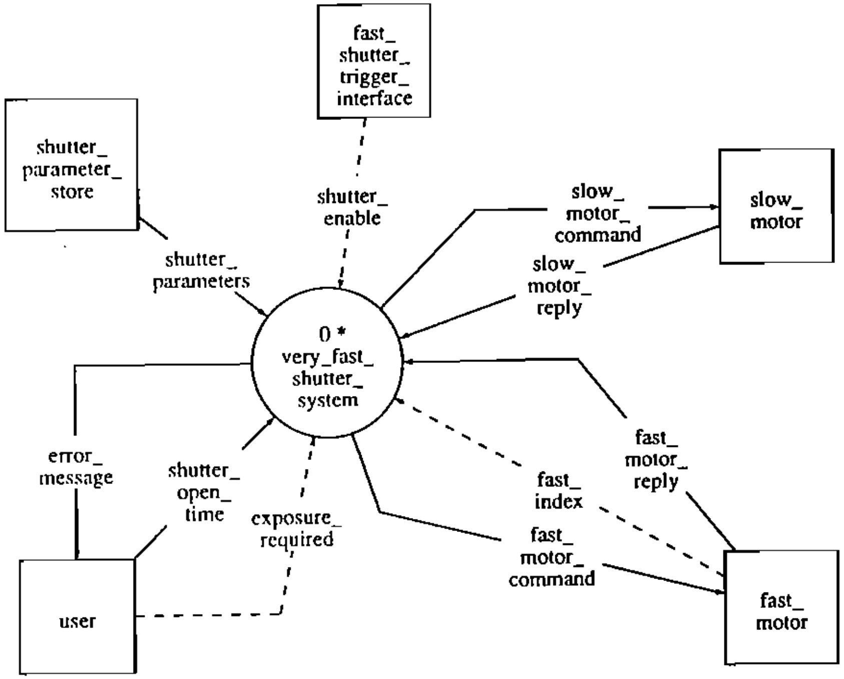

fast_
shutter_
trigger_
interface
shutter_
parameter_
store
shutter_
enable
slow_
motor_
command
slow_
motor
slow_
motor_
reply
shutter_
parameters
0 *
very_fast_
shutter_
system
error_
message
shutter_
open_
time
exposure_
required
fast_
index
fast_
motor_
reply
fast_
motor_
command
user
fast_
motor

Fig. 7

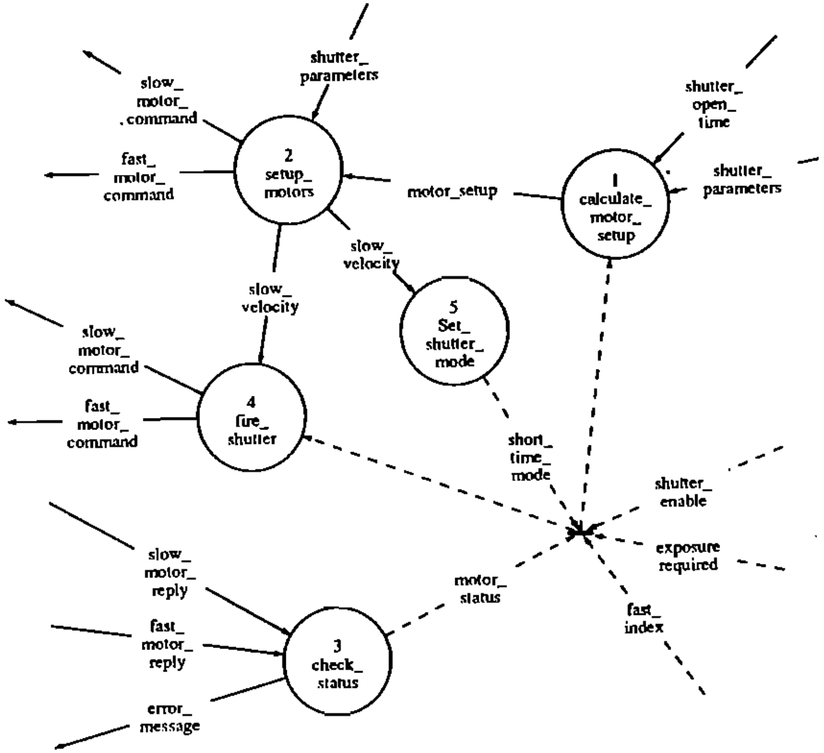

shutter_
parameters
slow_
motor_
. command
2
setup_
motors
shutter_
open_
time
fast_
motor_
command
motor_setup
1
calculate_
motor_
setup
shutter_
parameters
slow_
velocity
slow_
velocity
5
Set_
shutter_
mode
slow_
motor_
command
4
fire_
shutter
fast_
motor_
command
short_
time_
mode
shutter_
enable
exposure
required
slow_
motor_
reply
motor_
status
fast_
index
fast_
motor_
reply
3
check_
status
error_
message

Fig.8

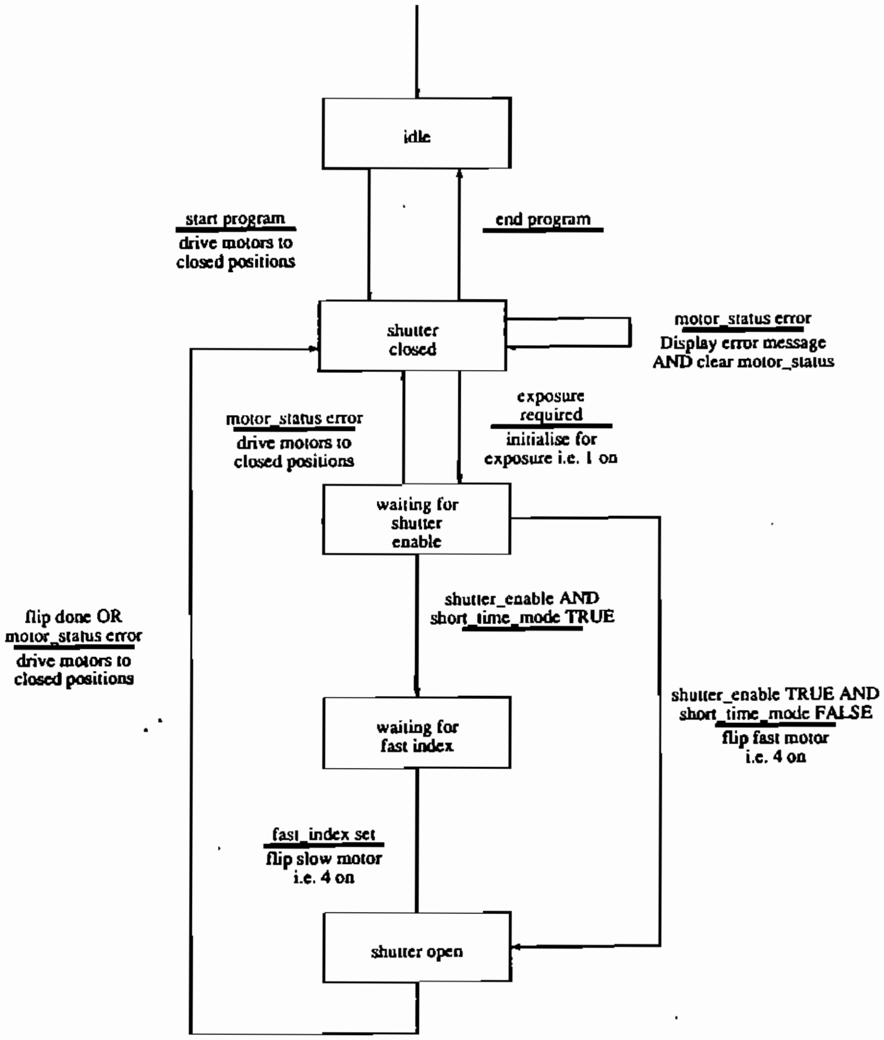

idle
start program
drive motors to closed positions
end program
shutter closed
motor_status error
Display error message AND clear motor_status
motor_status error
drive motors to closed positions
exposure required
initialise for exposure i.e. 1 on
waiting for shutter enable
shutter_enable AND short_time_mode TRUE
flip done OR motor_status error
drive motors to closed positions
waiting for fast index
shutter_enable TRUE AND short_time_mode FALSE
flip fast motor i.e. 4 on
fast_index set
flip slow motor i.e. 4 on
shutter open

Fig. 9

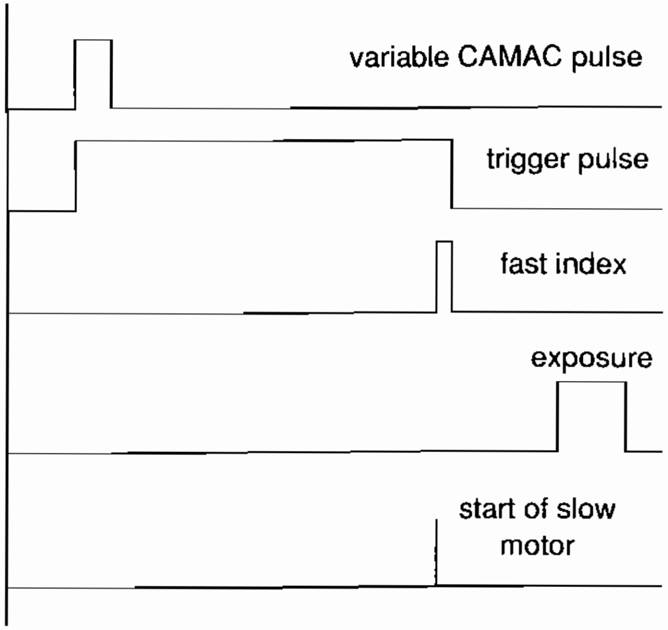

variable CAMAC pulse
trigger pulse
fast index
exposure
start of slow motor

Fig 10.

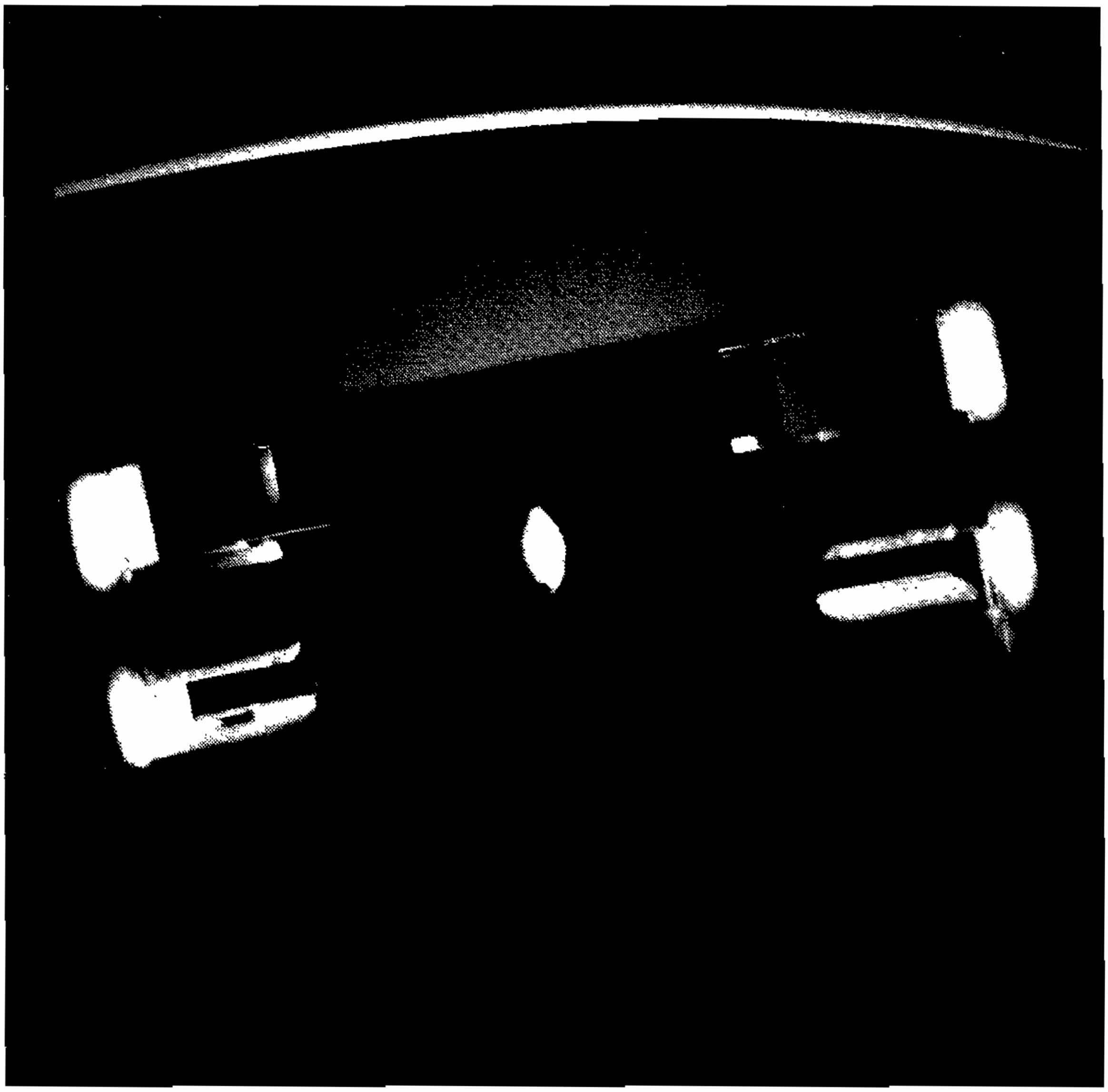


Fig.11

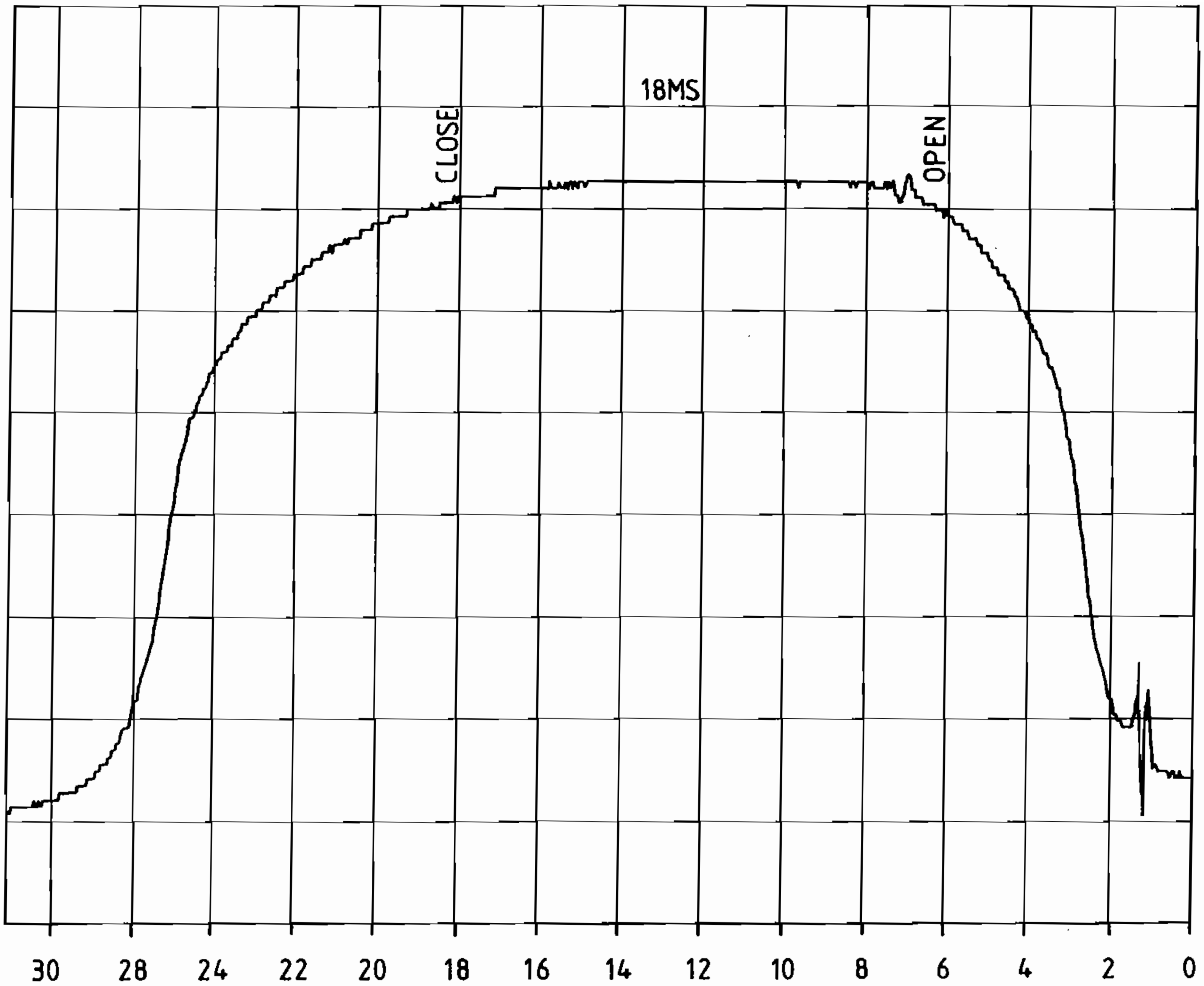
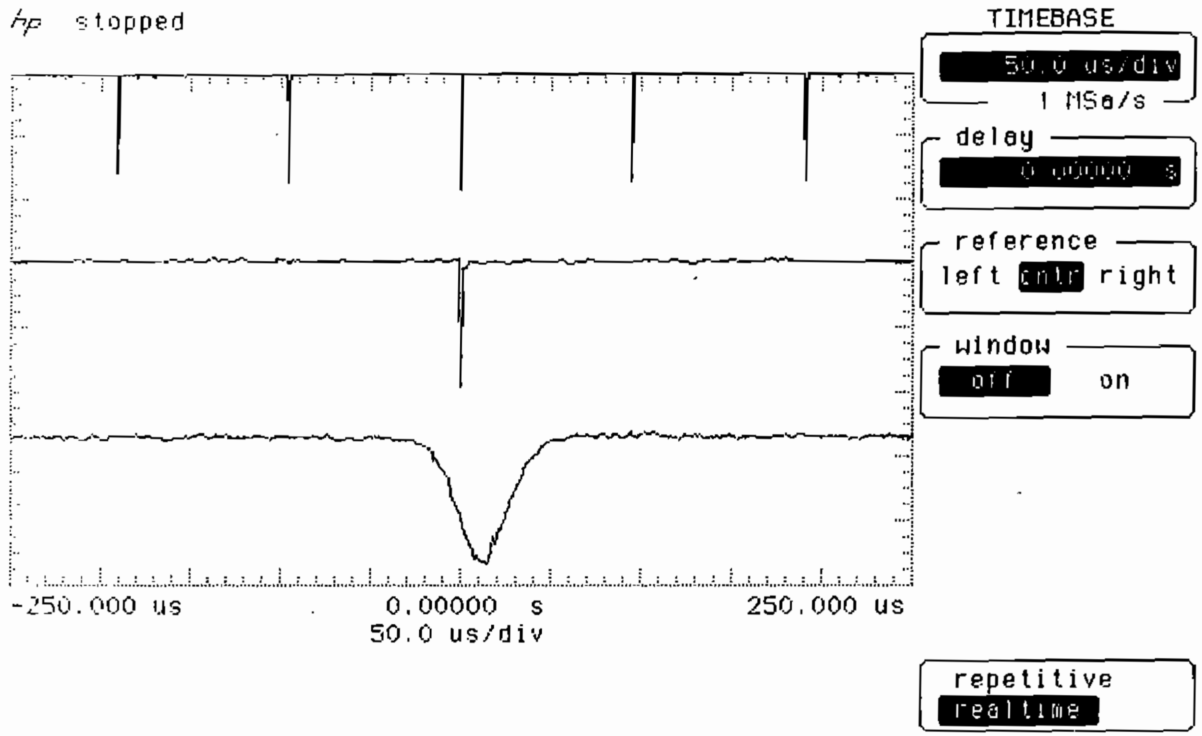


Fig.12



TIMEBASE 50.0 us/div
1 MSa/s
delay 0.00000 s
reference left [✓ cntr] right
window [✓ off] on
repetitive [✓ realtime]

```
             Sensitivity      Offset         Probe         Coupling
Channel 1    500  mV/div    -2.00000  V    1.000 :1      dc (50 ohm)
Memory 1     500  mV/div    -2.00000  V    MemSweep -   4.00 ms/div
Memory 2     500  mV/div    -500.000 mV    MemSweep -   5.00 ms/div
Memory 3     500  mV/div     875.000 mV    MemSweep -   50.0 us/div


      Trigger mode : Edge
On Negative Edge Of Chan1
Trigger Level
  Chan1 = -250.000 mV (noise reject ON)
Holdoff = 40.000  ns
```

Fig.13

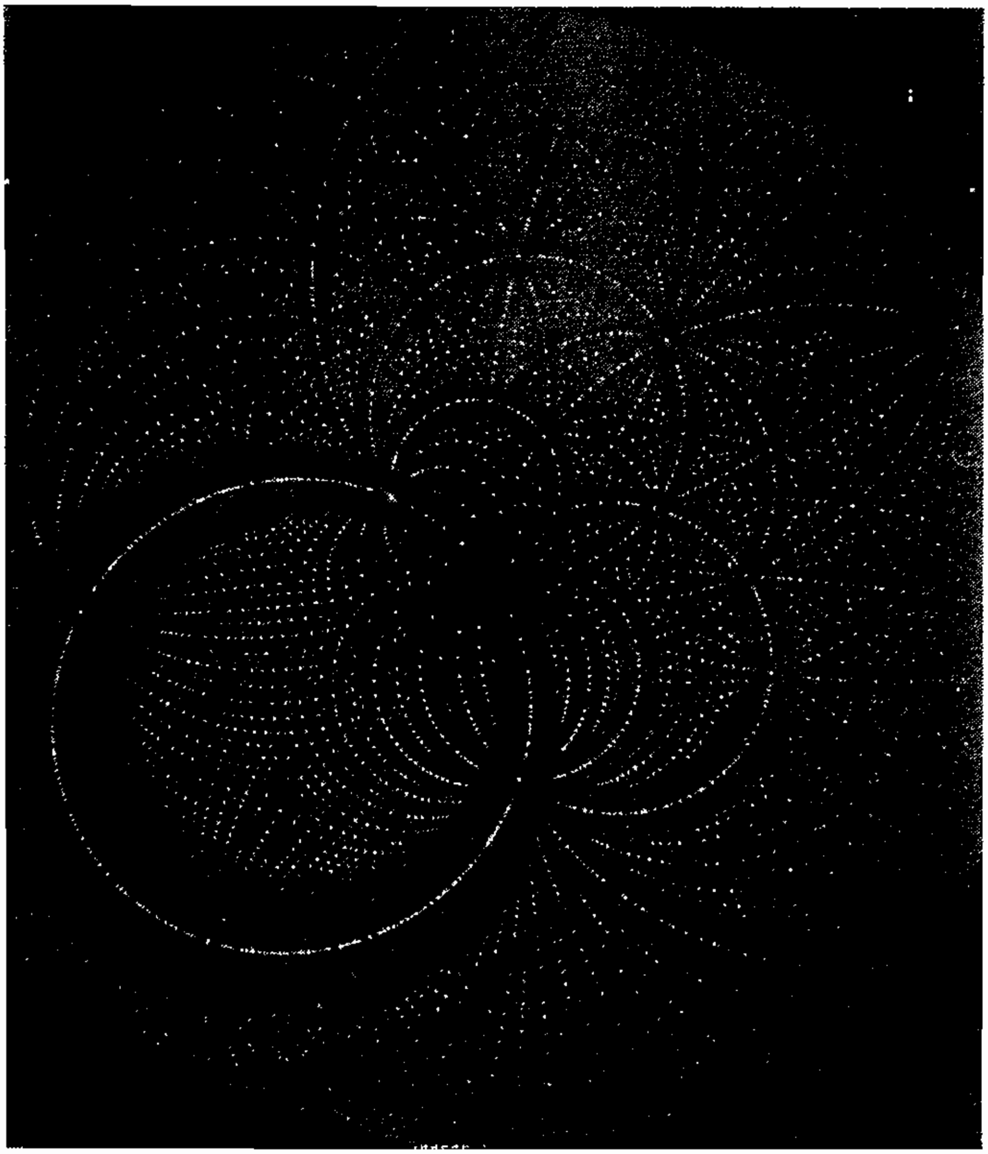


Fig.14

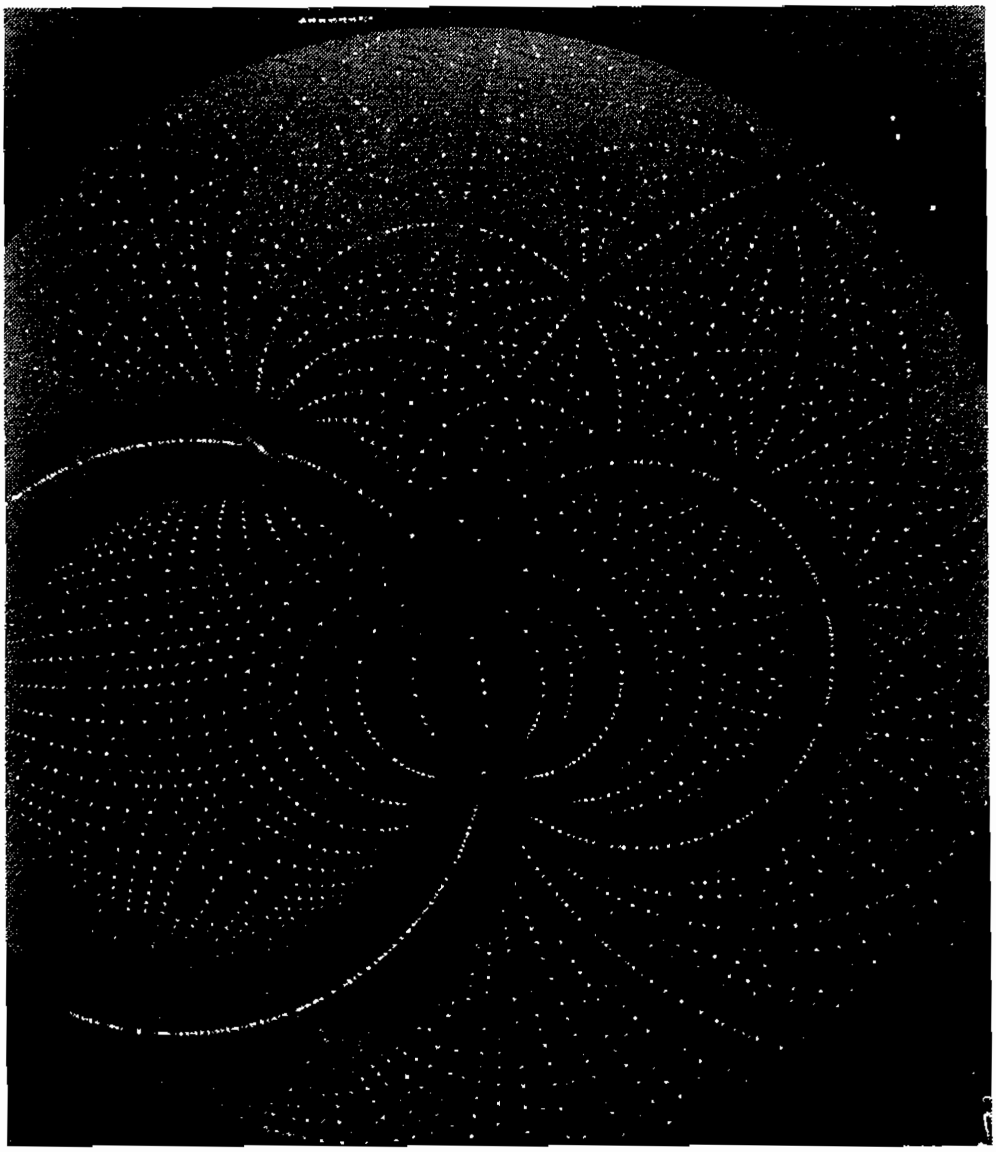


Fig. 15