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DEVELOPMENT OF A TELEVISION X-RAY DETECTOR SYSTEM FOR X-RAY DIFFRACTION EXPERIMENTS

by

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Development of a television X-ray detector system for X-ray diffraction experiments.

A novel powder diffraction technique using a television camera based detector to record the whole of a Debye-Scherrer pattern simultaneously has been developed. The technique has been used with a synchrotron source of x-rays to study the effect of pressure on the lattice parameters of ruby. A description of the detector is presented along with an overview of the data acquisition and analysis software. Details of the high pressure study are given and a direct comparison is made with results obtained using other powder diffraction techniques.

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1. Introduction.

The greatly reduced data acquisition times afforded by a computer based image collection (frame-grabber) board interfaced with a fibre optically coupled solid state x-ray detector and C.C.D video camera circuitry may provide an ideal platform for high pressure powder diffraction, where several images can be collected at varying pressures during the time taken for a normal diffractometer two theta scan, and also for dynamic powder diffraction studies enabling images to be taken every few seconds throughout a reaction.

With the whole of the Debye-Scherrer rings being collected it was hoped that a good statistical accuracy would be obtained by integrating round the centre of the powder rings at fixed radii to obtain the peak pattern. Analysis could then be carried out with a standard peak-fitting program, to obtain two-theta/intensity data.

The system may be driven from a wide variety of host computers. Personal computers provide both a cheap and highly portable option. The system implemented and tested at the Daresbury Laboratory Synchrotron Radiation Facility comprised a Photonic Science[1] X-ray camera, DIS3000[2] image processing frame grabber and an IBM PS/2 Model 30 personal computer with a software interface to the frame-grabber.

Ruby has applications in lasers and as a high pressure standard. Extensive studies have been carried out observing the change in the lattice constants with pressure and structure determinations have been made up to 90 kbar.[3,4] With this wealth of prior studies ruby provides an excellent choice of sample for testing the t.v. detector.

2. The X-ray Camera.

The area detector arrangement used in this experiment is based on an X-ray sensitive video camera. The video signal from the camera is connected to an image capture and frame storage system installed on a personal computer. The incoming X-rays pass through a 25 microns thick, light-tight, aluminium foil, and reach a poycrystalline layer of gadolinium oxysulphide (P43) deposited in a plastic matrix . This phosphor screen is about 100 microns thick and converts the X-rays into optical photons, which can then be registered by the video camera. The camera is configured for a 50mm diagonal, so that the input picture width of the resulting video image is nominally 40mm. The phosphor screen absorbs almost 100% of 8keV incident X-rays, while the stopping power drops to about 50% at 60keV. There will be a local minimum in the stopping power close to the Gd K absorption edge at 50keV. Taking into account photon losses through the front face of the screen and optical absorption in the screen material, approximately 80 collectable optical photons are generated per X-ray quanta. The screen itself is attached to a fibre optic plate of 1mm thickness. The plate provides a support for the screen, while having effectively zero optical thickness. The optical image generated in the screen is then passed to a high gain microchannel plate image intensifier (Mullard XX1332), which has a radiant power gain of 5000 (number of output photons per input photon at a given wavelegth). The image intensifier has a useful input diameter of 49mm and an output screen of 39mm diameter, with a typical resolution of about 20 lp/mm. Therefore approximately 800 pixels can be resolved along any diameter. Following the intensifier, there is a demagnifying fibre optic bundle which reduces the output image of the intensifier down to the size appropriate for the •" solid state image sensor (Phillips NXA1011 CCD). This CCD has been modified by Photonic Science to have a fibre optic input window. The drive electronics for the CCD camera are self-contained in the camera body itself, with an additional unit to control video gain. The intensifier operates at fixed gain. The overall size of the camera is 80mm x 130mm cross section and 250mm long.

In order to estimate the minimum discernible X-ray flux, one must first consider the performance of the CCD chip. Assuming a conservative figure for the dynamic range of about 100 and a blooming level of 250,000 electrons per pixel per frame, it follows that the minimum signal of 2500 electrons per pixel per (20msec) frame is required. Note that this is for the directly viewed video signal, without image processing or integration. Since the quantum efficiency of the CCD sensor at the wavelength emitted by the intensifier is about 25%, the minimum signal corresponds to about 10,000 visible photons per pixel per frame. Since the fibre optic taper has a transmission of 4% and the image intensifier has a radiant power gain of 5000, there should be about 50 optical photons per pixel per frame at the input of the intensifier. With 80 photons reaching the intensifier for each incident 8keV X-ray, the minimum detectable X-ray flux become 0.625 X-rays per pixel per frame. Allowing for the demagnification ratio of the camera, the pixel size and the frame time, this corresponds to about 10,000 X-rays/ mm^2 / sec at 8keV. At higher X-ray energies, the stopping power of the screen will lower. However, this will be compensated to some extent by the increased number of optical photons generated by each X-ray. Since each pixel of the CCD corresponds to an element of about 70 microns diameter on the screen, and the intensifier resolution is significantly better than this figure, the practical limit on the resolution of this X-ray camera is defined by the thickness of the conversion screen (100 microns). However, this resolution matches rather well to the cross section of the pin-hole collimators used to define the synchrotron beam.

THE X-RAY TELEVISION DETECTOR

3. The Frame-grabber.

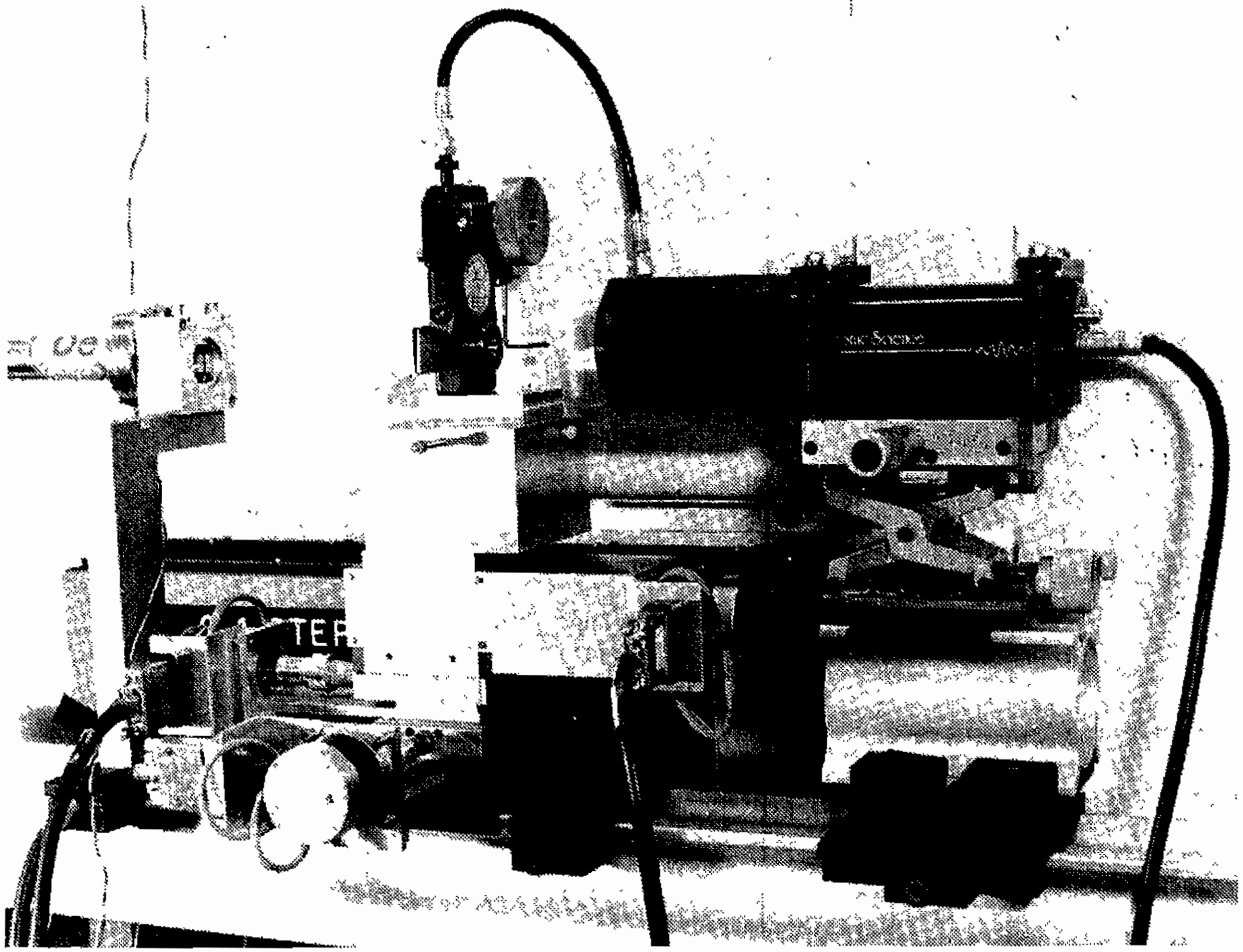
The DIS3000 image processor/frame-grabber board has two frame stores each capable of holding one 512x512 pixel image with 8 bits of depth per pixel. Images are collected at standard video rates of 40ms per frame. The unit is accompanied by a 16 bit arithmetic logic unit , this combination makes real-time integration possible to 16 bits of accuracy for periods of time from eighty milliseconds to approximately one hour.Images acquired with this system are 256 kbytes in size, making some form of mass-storage media essential, for some applications the speed at which images can be saved onto, say, a harddisk may be of importance. Software was written to allow the integration and subsequent dumping of images onto the harddisk of the host computer, on the PS/2 Model 30 this took in the region of 10 seconds, on other host computers with faster disks this time may be greatly reduced. Another option is to increase the number of frame stores on the board and to fill all the available frame store memory before dumping to disk, this will allow images to be collected one after the other, without delay, this would be ideal for dynamically observing a chemical reaction, with images being collected at various stages of the reaction.

The frame-grabber boards are housed in a personal computer sized box, with two input/output sockets, one to run to the camera and the other to a monitor for observing the powder pattern. The frame-grabber is, therefore, highly transportable between different experiments and x-ray sources.

The software simply prompts for an exposure time in seconds and then, when the integration is complete, asks you if you wish to save the image. Due to the hardware setup the resulting integrated image must be saved to 16 files, 8 lower byte files and 8 upper byte files (Each file contains the data for 64 rows of the 512 row image). If it is required to save the image an upper and lower byte file name must be specified as prompted (The 8 files for each byte have the same name, but a different extension - img, im1, im2, im3 etc). Subsequent software has been written to allow these 16 files to be read in and one output file to be created from them, allowing far easier file management.

The frame-grabber has a wide range of other image acquisition and image processing functions most of these were not tested. All further image analysis was carried out on a Microvax, to take advantage of existing powder pattern analysis programs.

Many problems were encountered which hampered the development of the DIS3000. All the technical faults found have been fixed and a new, updated ROM containing the routines that control the DIS3000 has been installed.



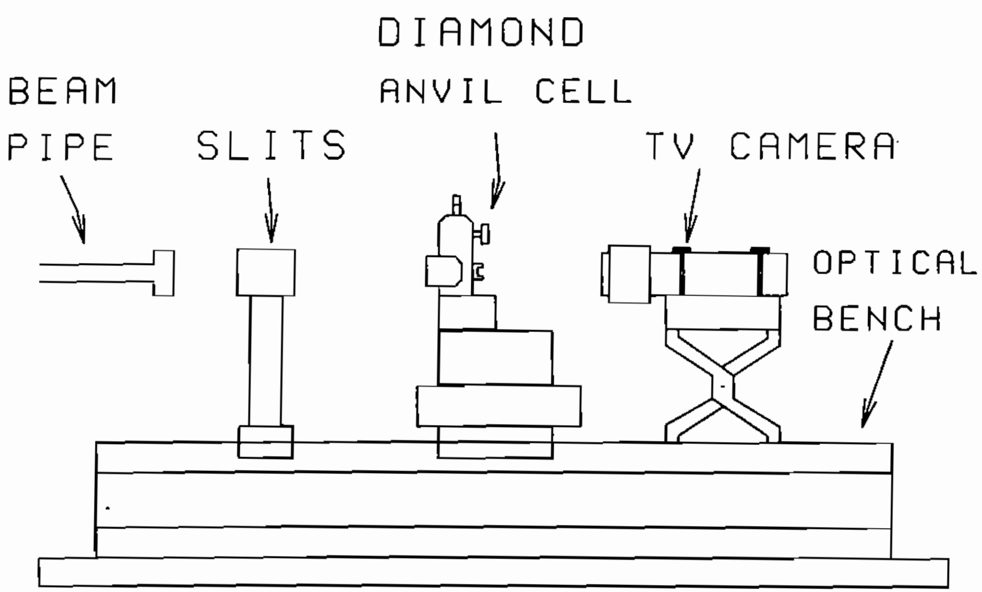
4. Experimental Setup.

The experiment was carried out with a synchrotron source of monochromatic x-rays on station 9.1 at Daresbury Laboratory.This station receives radiation from a 5 Tesla superconducting wiggler magnet with a critical wavelength of about 0.9 Å. A channel cut silicon (111) monochrometer was used to select X-rays with a wavelength of 0.55 Å. A schematic diagram of the experimental layout is shown in Figure 1. The diamond anvil cell was loaded with a mixture of finely powdered Ruby and Sodium Chloride contained by an Inconel gasket of 0.1 mm initial diameter. An X-ray beam of 0.05 mm was used and the equipment aligned so that the beam passed through the sample without scattering from the gasket. The camera was positioned close to the diamond anvil cell, to maximise the number of powder rings striking the active area of the camera, and it was also aligned perpendicular to the incoming beam. The sample to detector distance was determined by two methods: a small amount of silver powder was sprinkled on to the outer face of the diamond closest to the detector and the position of the silver diffraction lines and a knowledge of the diamond thickness were used and secondly diffraction patterns were collected at two different sample to detector distances. Images were collected at 30 and 60 second exposures at varying pressures.

CEA X-ray film exposures were also taken, for means of comparison, at low and high pressure, these were taken with a 6 hour exposure time.

The fibre-optic coupling in the x-ray camera leads to a certain amount of distortion in the image obtained. The distortion is greatest towards the edges (especially the corners) of the camera and least at the centre. Before any qualitative data can be extracted from the collected images, the detector must first of all be callibrated to remove this distortion. This was achieved by exposing the camera evenly to the x-ray source while a brass plate, with a regular lattice of 0.2mm diameter holes drilled into it, was held close to the active area of the camera. The video frames were integrated for one minute and from this resulting image a pixel to mm callibration table could be calculated for use in future analysis of powder patterns.

FIGURE 1. EXPERIMENTAL SET-UP.



5. Results and Analysis.

The first task in the analysis of the powder data obtained with the T.V camera system is to obtain a standard peak pattern from which accurate peak-fitting programs can be used to obtain the d and intensity values of the peaks.

The 16 file output of the frame-grabber is converted into a single file of pixel values for the whole of the 512x512 image. Software was written in VAX fortran to obtain the peak pattern from the image. The TV image, as seen in Figure 2, is first read in to the programs image array. It is then displayed on the terminal monitor (in outline form) and the centre of the powder rings is marked with the cursor, the centre can then be adjusted by taking a histogram cut through the assumed centre of the rings and marking two corresponding peaks on either side of the centre it can then be checked that the two radii are equal. If this step is repeated several times then a best fit for the centre can be obtained. Finally a circular integration routine moves along a diameter of the rings sampling the intensities into an array and at the same time sampling the radius of the point, from the centre of the rings, into another array, the angle of the diameter is then increased and the same radii as before are sampled and the intensities added to the previous values, this procedure is repeated through 180° (Figure 3.).

The d values are shown for both the T.V detector and film (Tables 1 and 3 respectively). Calculated and measured intensity values for the T.V detector are also shown (Table 2). A graph is also presented (Figure 4.) to show the variation of the full width half maximum of the peaks with two theta. All the data was collected at a wavelength of 0.5534Å.

The peaks were found to fit as very good gaussians (see appendix).

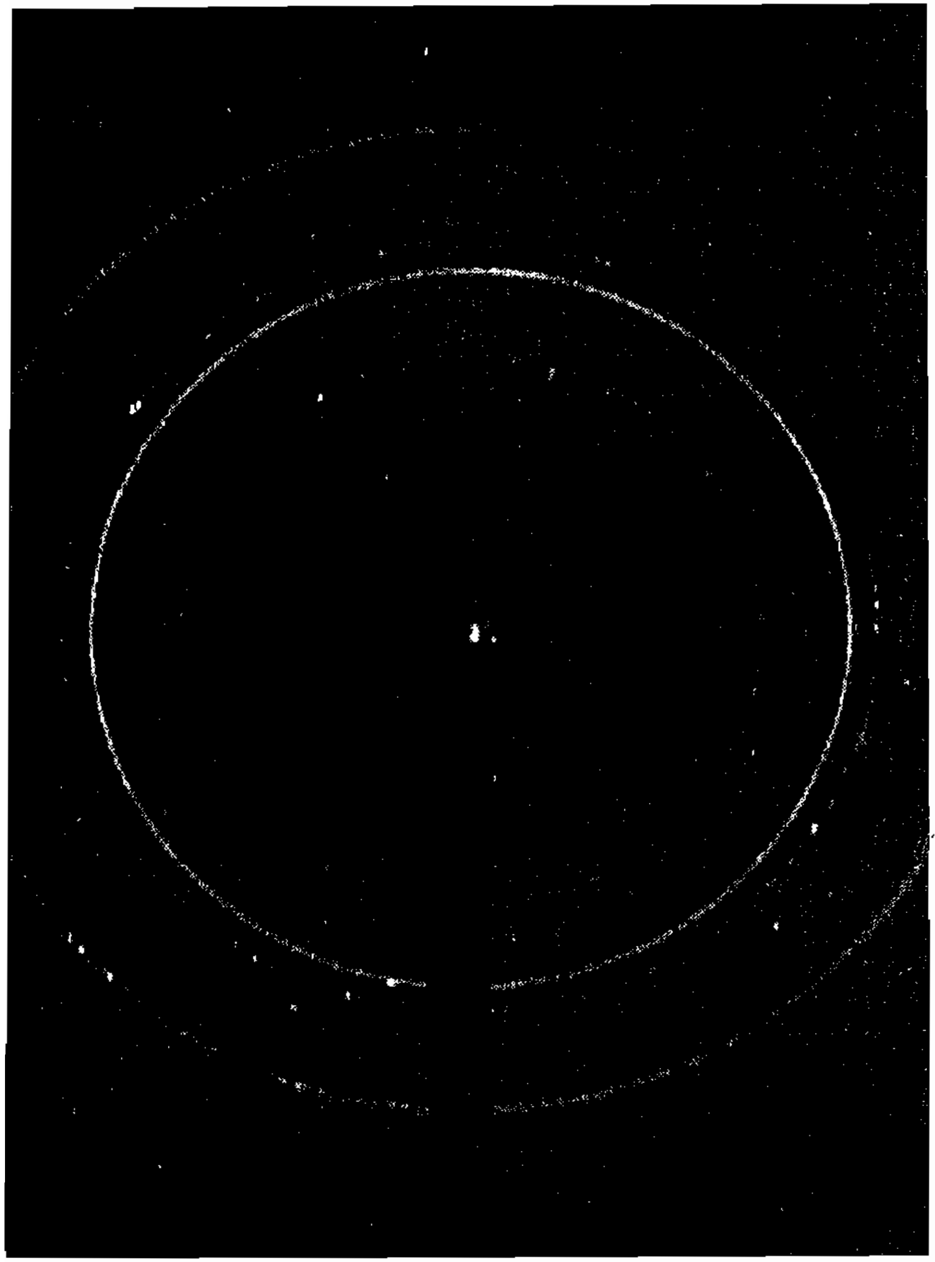


Figure 2. TV IMAGE OF POWDER DIFFRACTION PATTERN.

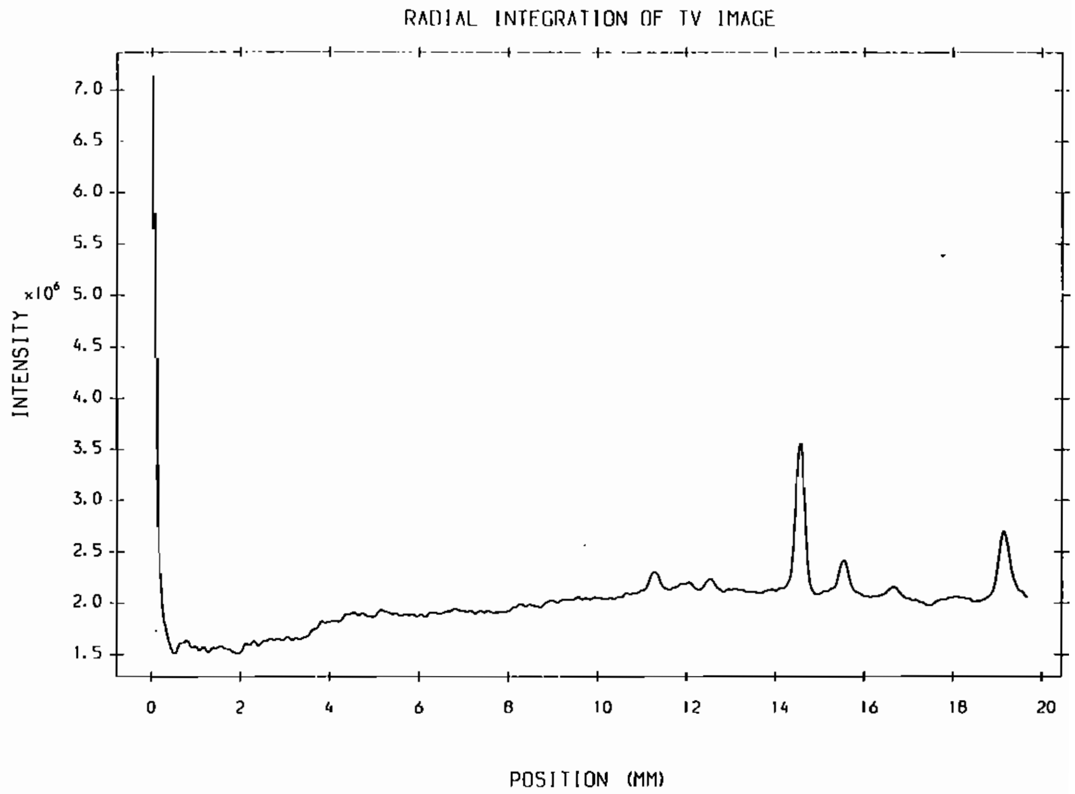


Figure 3. RESULT OF RADIAL INTEGRATION OF TV IMAGE.

TABLE 1. T.V DETECTOR RESULTS

Pressure (kbar)	d(012) calc (Å)	d(104) calc (Å)	d(012) obs (Å)	d(104) obs (Å)
43.14	3.460	2.536	3.453	2.535
42.47	3.461	2.536	3.454	2.536
42.46	3.461	2.537	3.450	2.535
41.56	3.461	2.537	3.455	2.535
40.61	3.462	2.537	3.459	2.536
39.48	3.462	2.538	3.456	2.535

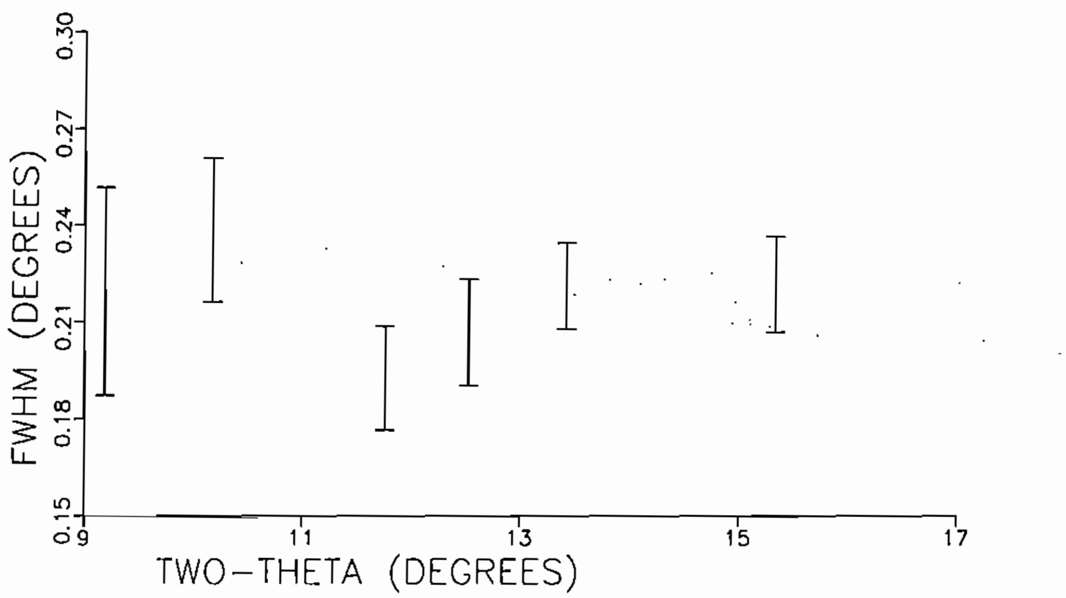
TABLE 2. T.V DETECTOR INTENSITIES

Peak (hkl)	Intensity (calc)	Intensity (obs)
012	602.7	590.9
104	1000.0	1000.0
113	550.4	332.24

TABLE 3. FILM RESULTS

Pressure (kbar)	d(012) calc (Å)	d(104) calc (Å)	d(110) calc (Å)	d(012) obs (Å)	d(104) obs (Å)	d(110) obs (Å)
1.0	3.479	2.550	2.378	3.474	2.550	2.387
39.9	3.462	2.537	2.367	3.464	2.540	2.401

FIGURE 4.VARIATION OF FWHM WITH TWO-THETA.



6. Conclusions.

The TV detector system is still in its initial stages of development , improvements are possible both in terms of hardware and software. The results obtained show that the system is usable in powder diffraction to determine lattice parameters, though in these initial tests the accuracy was about one order of magnitude less than is currently obtainable with a diffractometer, the results compared very well with those from film. Intensities were also seen to be quite accurate, the least accurate line was the ruby(113) which was shouldered on another peak so this inaccuracy was expected .

The results provide a firm basis for future development. The image processing area is one of the largest growth areas in the computer industry at the current time and video digitisers are improving rapidly.

In terms of hardware a VME based sytem would appear to be the ideal solution, allowing video memory modules to be added as required and fast access to a mass-storage device for downloading images in memory, thus making possible a wide range of dynamic diffraction experiments. Such a system would also provide the scope for carrying out the processing of data on line. Hardware improvements could also be made to the experimental setup allowing for more accurate alignment of the camera perpendicular to the incoming beam, this problem could also be addressed in software with an alignment correction[5]. Software alterations can also be made to allow a more accurate way of finding the centre of the Debye-Scherrer pattern, by collecting an image while allowing the direct beam to strike the camera for a short period without passing through the sample and then repeating with the sample in place and without moving the camera. Finally improvements can be made in the speed of the radial integration.

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Dr.P.Tomkins of Photonic Science.

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8. References.

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9. Appendix

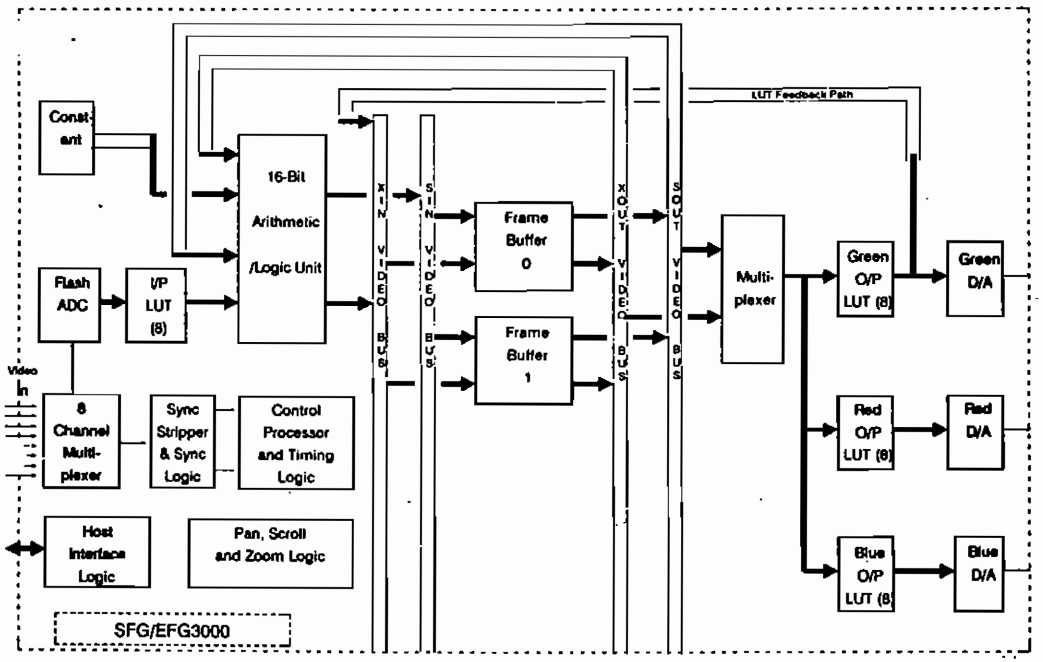

Diagram of the DIS3000 Frame Grabber

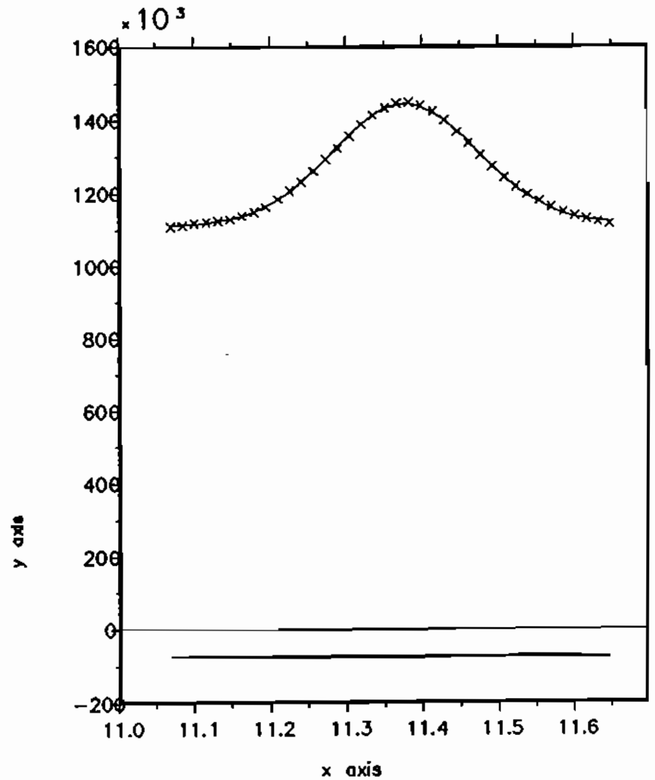

Diagram of a Gaussian Peak Fit from the TV Detector