A Short-Range Order Diffuse Scattering Measurement System : I. Micro-Computer Controlled Data Collection System

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A Short-Range Order Diffuse Scattering Measurement System
I. Micro-Computer Controlled Data Collection System*

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Synopsis

A rotating anode X-ray generator and a four-circle diffractometer have been combined to form a diffuse scattering measurement system which has the potential for the rapid collection of intensity data of high resolution from short-range ordered crystals. The system is automatically controlled by a micro-computer, which is linked to TSS computing facility through paper tapes. A description of the whole composition, particularly in detail of the micro-computer system, is given.

I. Introduction

For the determination of short-range ordered (SRO) structure of solid solutions, it is necessary to explore an intensity distribution of X-ray diffuse scattering over a volume of reciprocal space and, if a separation of atomic displacement modulation intensity by a Borie-Sparks method[1,2] is required, the number of the sampling points amounts to as many as a few thousands. Moreover, if a high resolution measurement is made in order to reveal a fine structure of SRO diffuse maxima, the number increases markedly.

This kind of measurement is only possible when all the procedures are automatically controlled. An LSI micro-processor nowadays commercially available can be used for this purpose. Since the intensity of diffuse scattering is generally weak, a high power X-ray generator is indispensable to collect intensity data within a reasonable period of

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time. Rotating anode-type X-ray generator with a small focal spot size can emit a beam intense enough to obtain high counting rate even for diffuse scattering.

In the present paper description is given on the construction and performance of a specially designed, micro-computer controlled diffuse scattering measurement system consisting of rotating anode X-ray generator and four-circle diffractometer. It has been shown that, if properly used with the aid of TSS computing facility, a micro-computer exhibits great capability than one simply expects. A typical measurement procedure intended for the use of the SRO diffuse scattering experiments is also described.

II. Description of the System

A block diagram of the system is illustrated in Fig. 1, and its pictures are given in Fig. 2(a) and (b). The system consists of four blocks, i.e., X-ray generator and goniometer, electronic circuit panel for detecting X-rays, TSS data analysis softwares and micro-computer panel. In this section, characteristics of the parts composing the system will be described.

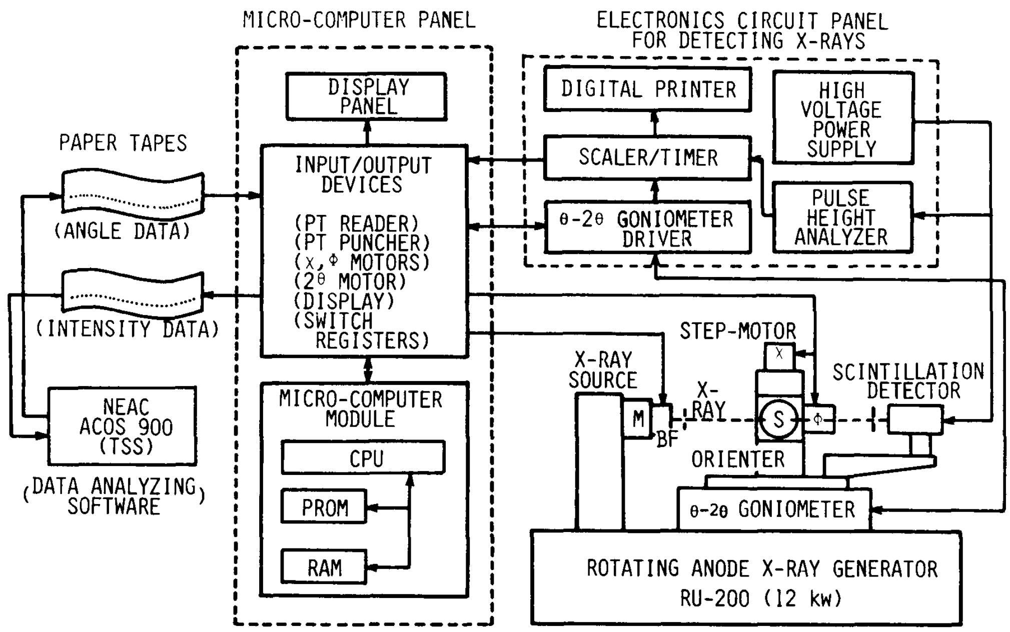
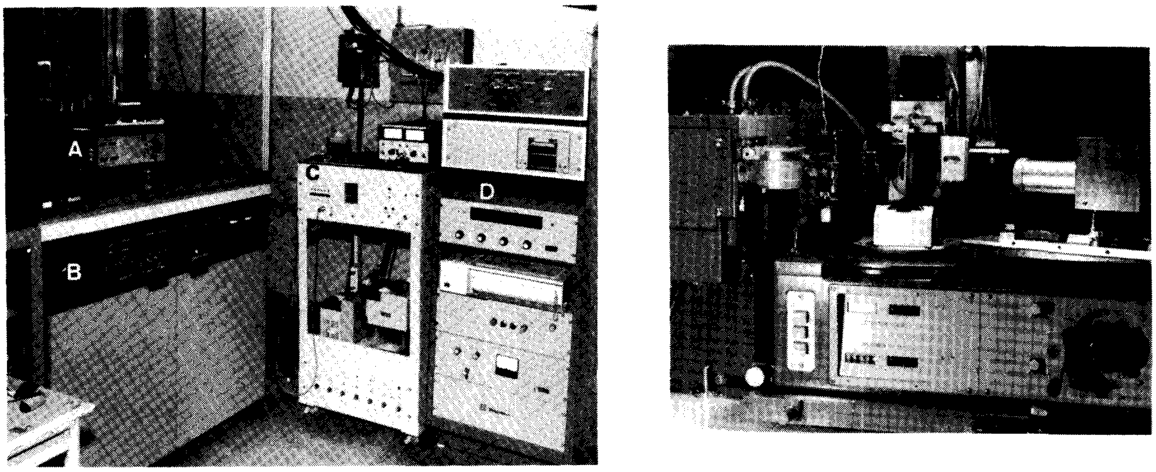


Fig. 1. Block diagram of the X-ray diffuse scattering measurement system using a micro-computer.



(a) (b)

Fig. 2(a). Instrument for the diffuse scattering measurement. A:goniometer, B:X-ray generator, C:micro-computer panel, D:electronic circuit panel.
(b). Goniometer.

1. X-ray generator

The Rigaku Rotating-Anode X-ray Generator RU-200 is provided. The maximum power of this generator is 60 kV and 200 mA with Cu anode. The effective focal spot size was measured to be 0.53 (vertical) $\times$ 0.78 (horizontal) mm^2 at 50 kV, 180 mA and bias voltage 70 V. Fig. 3 shows the intensity distribution of the effective focus measured as an image through a pin hole. The stability is held within ± 0.1 % for input power variation of ± 10 %, and within ± 0.1 % over many hours of operation.

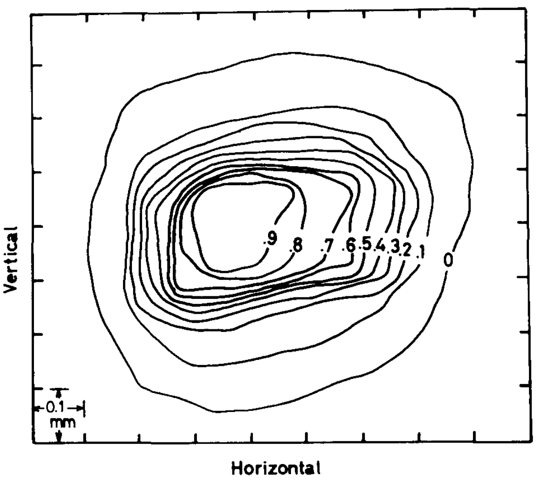


Fig. 3. Intensity contours on the effective focus of X-ray source.

2. Monochromator with balanced filters

A monochromator is placed at the distance of 110 mm from the X-ray source (M in Fig. 4). As a monochromator crystal, pyrolitic graphite with a flat surface parallel to the (0002) plane is employed. The d_{0002} spacing is 3.355 Å, and $2\theta_m$= 26.56° for CuKα radiation. The λ/n components of the characteristic radiation will be

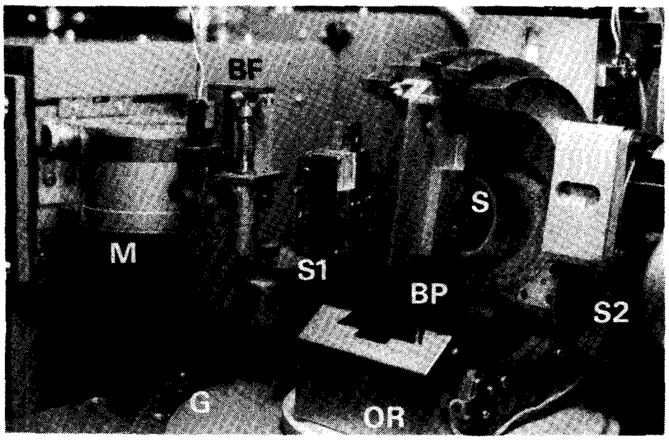


Fig. 4. X-ray optical system. M:monochromator, BF:balanced filter exchanger, S:sample, OR:orienter, G:θ-2θ goniometer(SG-7).

diffracted as the higher order reflections at the same 2θ setting of the monochromator crystal. Most part of them can be discriminated by a pulse-height analyzer, except λ/2 component. Ni and Co filters[3], which are balanced strictly at one-half the wavelength of the CuKα radiation, are prepared in the present system, and are set just after the monochromator (BF in Fig. 4). The filter holder can be moved up and down by an electromagnetic shutter. X-ray beam is attenuated by the filters at the rate tabulated in Table 1, where reduced intensities are normalized with respect to that measured without a filter. The

Radiation / Filter	$\lambda_{CuK\alpha}$	$\lambda_{CuK\alpha}/2$
non	1.0	1.0
Ni	0.61	0.56
Co	0.014	0.56

Table 1. Intensities of filtered radiation relative to that without a filter.

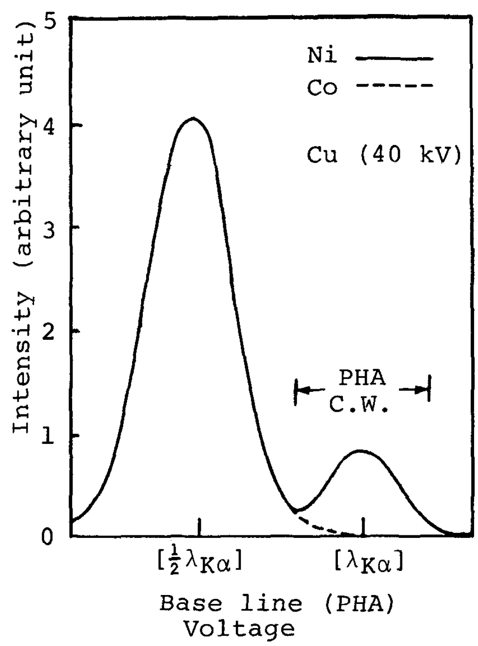


Fig. 5. X-ray spectra of the 100 scattering from the SRO CuAu using Cu radiation filtered with Ni, Co foils, observed with a scintillation counter probe, which has an intrinsic low energy resolution. Practical channel width (PHA) for SRO intensity measurement is indicated.

intensity to be used for the analysis can be obtained by taking the difference between two measurements of the intensity. As an example, at the 100 reciprocal lattice point (diffuse intensity peak position) for the disordered Cu-Au alloy, the X-ray spectra with Ni or Co filter placed in position are shown in Fig. 5.

3. Single crystal orienter with a baffle plate

Fig. 6 shows a quarter-circle single-crystal orienter which allows a specimen crystal to do χ and ϕ motion using stepping motors. Stepping motors 2CPH3A-6 and 2CPH-034 manufactured by Oriental Motor are used for χ and ϕ circles, respectively. The body of this orienter is fixed to the specimen axis (θ axis) of the θ-2θ goniometer. For χ and ϕ motion, one step represents an angular displacement of 0.01° and 0.05°, respectively. Maximum slewing speed is about 2.6°/sec for χ, and 13°/sec for ϕ.

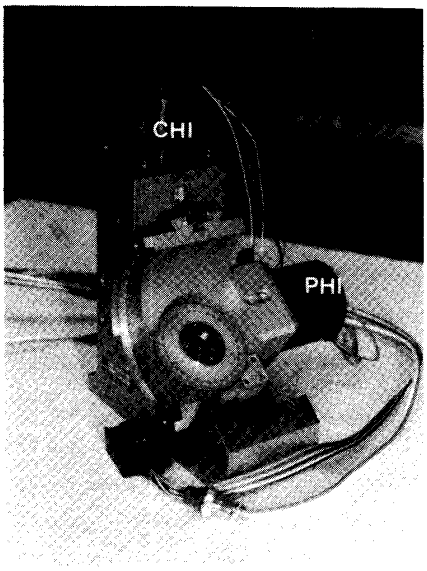


Fig. 6. Quarter-circle single-crystal orienter.

A baffle plate (BP in Fig. 4) for eliminating the air scattering is designed so as to be fixed to the χ axis. Distance between the plate edge and the surface of the specimen is 4 mm. Data on the air scattering will be given in the later section (IV. 2). This baffle is designed also to remove the air scattering using He atmosphere.

4. θ-2θ goniometer

The single-crystal orienter mentioned above is set on the Rigaku Wide Angle Goniometer SG-7 equipped with the Step Scan Controller. This controller is used as a driver of the θ-2θ goniometer and the scaler and timer in the present system. This goniometer generates an active low TTL pulse at an interval of 0.04° (2θ). Maximum slewing speed is 4°/min. The θ and 2θ shafts are independently rotated by manual operation. In the case where only the symmetric reflections from a sample surface are observed, the shafts can be left coupled during the measurement.

5. Electronic circuit panel for detecting X-rays

This consists of a detector, pulse-height analyzer, scaler and timer, ratemeter and recorder, and digital printer. As a detector the Rigaku Scintillation Counter Probe (NaI[Tl]) is used. This is suitable for detecting X-rays of short wavelength range from 0.3 to 2.5 Å. The pulse-height analyzer (PHA) can eliminate most of the λ/n components from a monochromator, except weak contribution of λ/2, and also reduce electronic noise. The noise level can be reduced to 0.04 cps on a certain optimum condition of PHA (for example, channel width as indicated in Fig. 5). With scaler and timer, counting capacity in fixed time method is 8×10^6 counts and maximum counting time in fixed count method is 1×10^4 sec. The counted values are indicated by 6 digits, each with BCD output digits.

6. Micro-computer module

The Intercept Jr. 6950 made by Intersil, U.S.A.[4,5] is chosen as a controller of the X-ray diffractometer. A block diagram of the module is shown in Fig. 7. This module possesses the IM6100 CPU LSI. All memory and I/O devices are connected to the IM6100 bus. The twelve bit bus carries time-multiplexed address and data from memory and I/O devices, which are, therefore, tri-state devices. In addition to the twelve bit bus, select lines providing timing information to strobe data on and off are needed. The monitor ROM and 256 × 3 RAM are mapped to select the proper devices during memory I/O. The Intercept Jr. 6950 uses a 12 switch keyboard, through which the program input into RAM is possible. Each key is connected to the corresponding DX line, and the accumulator can be loaded with the content of the switch register. Two display registers, each with four decimal (BCD) digits, are provided.

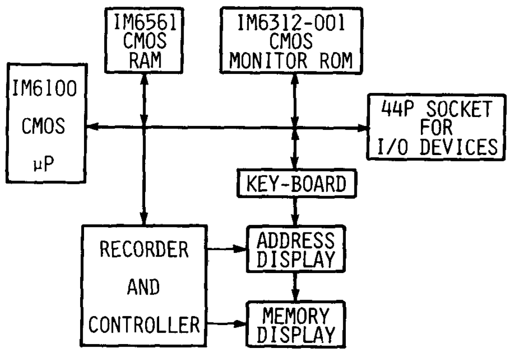


Fig. 7. Block diagram of the Intercept Jr. 6950 micro-computer board.

7. Paper tape reader and puncher

As a paper tape reader, the Ricoh PTCR-35C is used[6]. Punched holes are read by phototransistors, and maximum reading speed is 2000 frames/min. Clutch magnet driver signals from an external control unit are required to be supplied as pulses of 10-12 ms in width at intervals longer than 30 ms.

As a tape puncher the Ricoh TCP-25 is used[7]. Maximum punching speed is 1500 frames/min. Signals to the clutch magnet are required to have a width of 15 ms, which are supplied at intervals longer than 40 ms.

8. TSS computing facility

Calculation of orientation matrix and production of an input paper tape of angle data are performed using TSS of ACOS Series 77 NEAC System 900 (Tohoku University Computer Center (TUCC)). For the orientation matrix determination, program SRO-A is prepared and stored in a TSS file. Details of this program are given in Appendix Al. TSS programs prepared for measurements and analyses of the SRO diffuse scattering from the disordered fcc alloys will be described in part II of this series.

III. Micro-Computer System

1. Outline of the system

The diagram of the measurement control system is shown in Fig. 8. I/O devices, about which we will mention below, are connected to the IM6100 bus of the micro-computer.

Random Access Memory (RAM) Unit ([2] in Fig. 8) serves as a memory to register temporary data and a software stack for subroutine return addresses. Programs needed for the system control are stored in a P/ROM [3].

In addition to the 12 switch keyboard equipped on the Intercept Jr. 6950, switch registers [4] are provided as Program Selector, Orienter Remote Controller, Pause and X-ray Down Switch, and Ending Switch. Program Selector consists of the switches which correspond, respective-

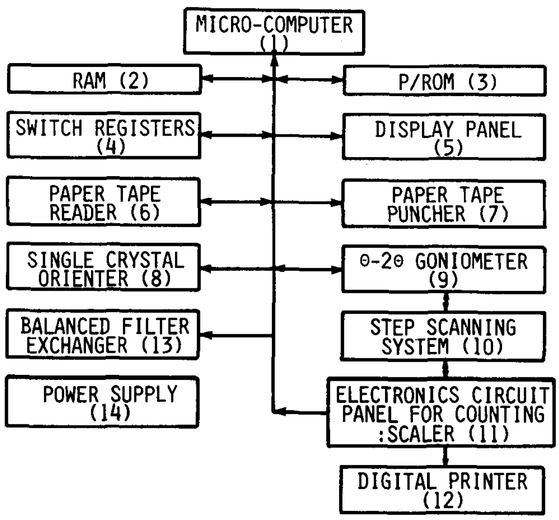


Fig. 8. Block diagram of the micro-computer control system. Power supply is composed of DC27V, DC12V, DC6V, DC5V, DC-5V sources, which are stabilized, except 27V.

ly, to the main routine program, the program for manual drive of χ and ϕ axes, the program to reset the routines to the initial state, and the program for θ-2θ step scanning. Orienter Remote Controller is available, when the stepping motors are to be manually driven, and it contains four switches corresponding to high-speed-clockwise, high-speed-counter-clockwise, low-speed-clockwise and low-speed-counter-clockwise operations. Pause Switch is used to interrupt the main routine for X-ray intensity measurement, and Reset Switch makes the routine to restart successively. When there is a trouble with X-ray generator, the Pause Switch becomes active automatically. Ending Switch is prepared for stopping the main routine completely.

Setting Angle Display Panel [5] has 15 seven-segment LEDs, and indicates three angle values to 2 places of decimals in degree. These LEDs can also indicate the parameters of the measurement conditions by switching the modes.

Paper Tape Reader [6] is used for reading the angle data punched with the aid of TSS. Paper Tape Puncher [7] is used to punch out the observed intensity data.

θ-2θ Goniometer [9] is equipped with a Goniometer Driver [10], which is the Rigaku Step Scan Mechanism reconstructed for the present purpose, and the driver can also partially control the Electronic Circuit Panel for diffractometry composed of Scaler [11] and Timer, Ratemeter, Recorder and Digital Printer [12].

Ross balanced filters[3)] are exchanged by turns by Electromagnetic Shutter [13].

2. Memory units, switch registers and display panel

As a RAM, Jr. RAM Module 6951-M1KX12 is used. Twelve IM6518 CMOS RAM chips are used to implement the 1024 (words) × 12 (bits) array for this board. The RAM on this board may be made nonvolatile by using

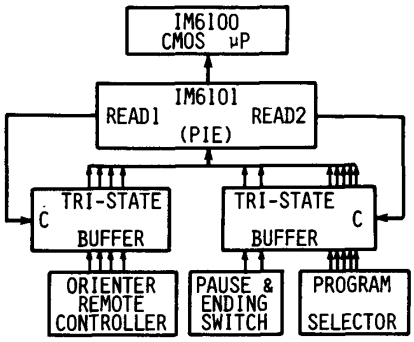


Fig. 9. Block diagram of the switch registers (Program Selector, Pause & Ending Switches and Orienter Remote Controller).

two "AA" type penlite cells in the chips provided. As the ROM, Jr. P/ROM Module 6952-P2KX12 is used, on which we put the IM5624, 512 × 4, tri-state output avalanche induced migration programmable bipolar P/ROMs to provide more than 1800 words for storing programs.

Fig. 9 illustrates a diagram of the switch registers. Each switch is connected to the DX line through a tri-state inverting buffer, which consists of MC14069 CMOS inverter and SN74LS365 TTL non-inverting buffer, and is gated by the active low pulse from the read output of the IM6101 parallel interface elements (PIE). The inputs to the buffers are pulled up to VCC via 50-200 kohm registances. When a switch is active, the input is shorted to the ground, and the DX line becomes at high level. The accumulator is loaded with the data put on the DX line, when the CPU executes an Input/Output Transfer (IOT) instruction.

Display Panel has 15 decimal (BCD) digits of LED, see Fig. 10. They are driven by a BCD-to-7 segment decoder/driver (MC14513) with the latch enable input shorted to 0 V, and 15 transistors (2SC372) that enable successive digits in turn. The transistors, when turned on by the outputs of the 4-line to 16-line decoder (MC14514), connect the LED common cathode to a low voltage. The drivers source current to individual segments, lighting these up for the time that the bit keeps that digit selected. The CPU executes an IOT instruction for display and loads three 4-bit latches (MC14076) with 12 bit data (DX 0-11) divided into three parts. Data on 4 lines of the DX 0-3 are used for inputs to base-lines of LED select transistors to illuminate the LEDs in turn. Data on the DX 4-7 and DX 8-11 contain the information of the measurement conditions (slit system, counting time, etc.) and setting angles (χ, ϕ and 2θ) in degree, respectively. The two sets of latched data are selected by a data multiplexer, for which the output disable inputs of the two MC14076 are used. The inputs are controlled by a manual switch.

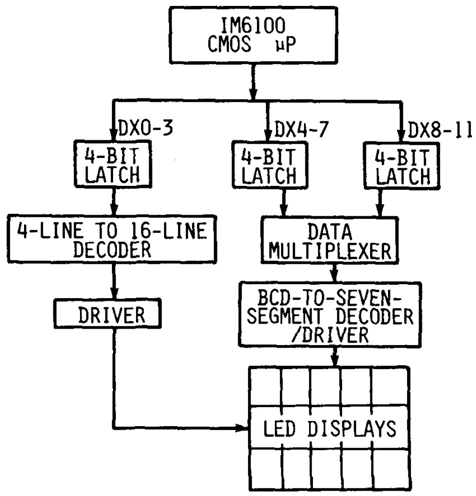
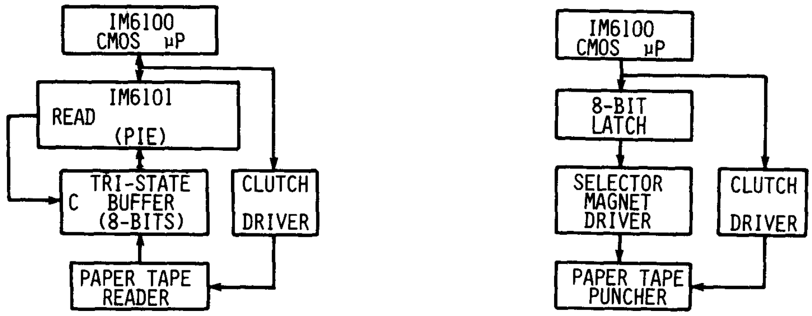


Fig. 10. Block diagram of the LED display for indicating the angle values.



(a) (b)

Fig. 11. Block diagrams of the paper tape reader (a) and puncher (b) drivers. The clutch drivers share a unit in Fig. 12.

3. Drivers of tape reader and puncher

Block diagram of a Paper Tape Reader Driver is shown in Fig. 11(a). 8 outputs from the reader are active low signals of 12 V, and are loaded into the DX bus through tri-state inverting buffers, which are gated by the strobe read signal from the IM6101 (PIE). That is, when inputs of tri-state buffer go low, the accumulator is loaded with the lower 8 bit data. The reader clutch is switched on by an n-p-n darlinton transistor array of 2SC234 and 2N3055, which is turned on by an active low pulse of 12 ms. This pulse is produced by widening the output pulse from the CPU with a pulse width transformer (MC14011-MC14069), after the IM6100 fetches an IOT instruction for the clutch magnet drive.

Fig. 11(b) shows a block diagram of a Puncher Driver. The CPU loads a 8 bit latch, for which MC14076 CMOS 4-bit D-type latches are used, with digits each from the accumulator. Outputs from the latch are connected to the inputs to the Selector Magnet Driver, which contains MC14049 CMOS inverting buffer-SN7404 TTL inverter-2SC234 transistor arrays. Clutch Driver is the same as that of the Paper Tape Reader driving circuit mentioned above.

4. Drivers of single crystal orienter and θ-2θ goniometer

Fig. 12 shows a block diagram of a drive system of the Single Crystal Orienter. As a part of the interface, the SPD4201 driver unit by Oriental Motor is used. Inputs to the driver unit are a TTL signal exchanging the rotation directions and a pulse of 50 ms as a drive pulse. The signal can be replaced by a switch of a high impedance state and 0 V level, as which a reed relay is used in the present sys-

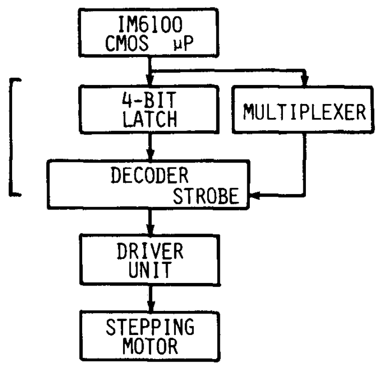


Fig. 12. Block diagram of the step motor driver. This interface shares a unit indicated as C with the clutch drivers of the paper tape reader and puncher.

tem. The pulse is the same as that switching the clutch magnet in the tape reader and puncher.

Rigaku θ-2θ Goniometer (SG-7) and Electronic Circuit for detecting X-rays are combined to do θ-2θ step scanning by Step Scan Mechanism. For the present purpose, the mechanism is reconstructed and connected to the micro-computer system. Fig. 13 shows a block diagram of the X-ray detecting system. On θ-2θ motion starting by a signal which switches on the power supply of the θ-2θ motor, the goniometer generates pulses at intervals of 0.04° (2θ), which are loaded into the DX bus through tri-state buffer and counted to be compared with the calculated angle to be scanned through. When the counted pulse number reaches the calculated one, a motor stop pulse is generated. By the pulse, the goniometer motor stops to detect the X-ray intensity. X-ray count end signal from Scaler/Timer (in fixed count method or fixed time method) is latched, and then the scaler outputs (24 bits 12V active low signal) are loaded into the DX bus through tri-state buffer as mentioned above. The digital printer shares the scaler outputs.

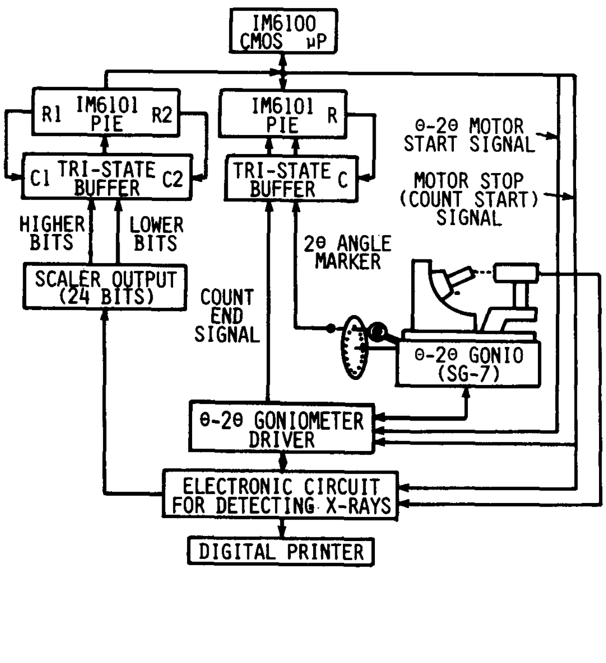


Fig. 13.
Block diagram of the driver of the θ-2θ goniometer and electronic circuit for detecting X-rays.

5. Balanced filter exchanging mechanism

Block diagram is shown in Fig. 14. Two strobe write signals from IM6101 switch the output level of an R-S latch, which is used to energize a reed relay in a circuit of an electromagnetic shutter with Ni and Co foils as balanced filters for Cu radiation.

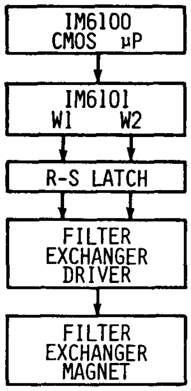


Fig. 14. Block diagram of the electromagnetic shutter driver for exchanging Ross balanced filters.

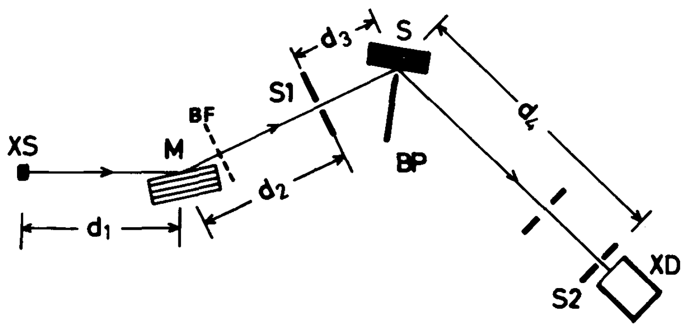


Fig. 15. Illustration of the optical system. XS:X-ray source, M:monochromator, BF:balanced filters, S1,S2:slits, S:sample, XD:X-ray detector. In the present case, $d_1=d_2=110$mm, $d_3=60$mm, $d_4=200$mm, S1=0.5(h)×0.8(v)mm^2, and S2=2(h)×1(v)mm^2.

IV. Performance

1. Resolution of the optical system

Fig. 15 schematically illustrates the optical system. In this system, divergence of the incident beam depends on the mosaic spread of the monochromator crystal in the horizontal plane (plane of the figure) and on the heights of the X-ray focal spot and the slit S1 perpendicular to the plane. The mosaic spread varies depending on the position of the crystal surface and is 0.2° at the optimum setting. The height of the focal spot is 0.53 mm and the optimum slit height is considered to be comparable to the spot size, since the effective divergence is given by a convolution of the angles subtended at the position of the slit by the spot length and vice versa. Divergence of the scattered beam is dependent on the beam cross-section at the sample position and the size of the receiving slit. When slit S1 of 0.5(horizontal) × 0.8(vertical) mm^2 is used, the beam cross-section is 1 mm high and 0.6 mm wide. If the size of the receiving slit is 2(h) × 1(v) mm^2, divergence in the plane of incidence is 0.2° and 0.6° for the incident and scattered beams, respectively, and divergence perpendicular to the plane is 0.5°.

2. Noise and parasitic scattering

Natural noise in the detecting system is reduced to 0.04 cps by adjusting PHA.

Main origin of parasitic scattering is considered to be air scattering from the volume near the sample. Correction for the air scattering can be made using a beam trap (or a baffle plate) in place of the sample and applying a geometrical factor which depends on 2θ. This factor in the present system was experimentally determined by a setting shown in Fig. 16, and the result is given in Fig. 17. At 2θ below about 15° this correction is not reliable. The baffle plate is designed to blow the sample with He gas and this method may be effective at the lower angle of 2θ.

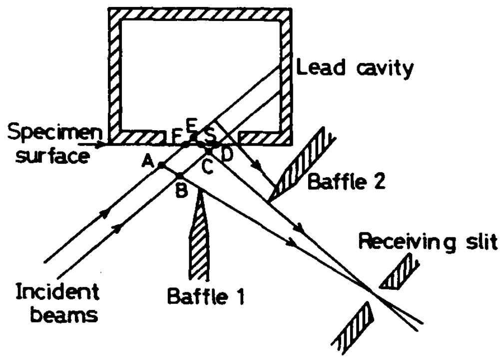


Fig. 16.
Air scattering measurement device. Detected intensity is from a volume ABCE, which nearly equals ABDF in the case of SRO diffuse scattering measurement.

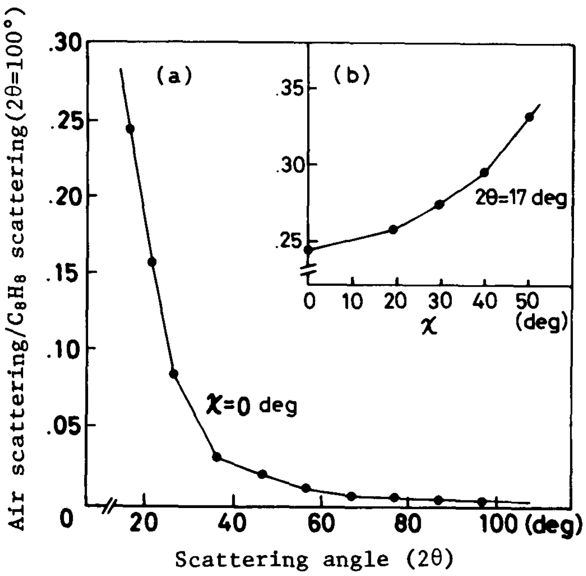


Fig. 17.
Air scatteiring from the volume near the sample depending on 2θ and χ angles, in the case that the baffle plate is used.

3. Intensity and stability of incident beam

Incident beam intensity can not be measured directly with present day X-ray counting equipment, where dead time corrections become appreciable above 10^4-10^6 cps. In the multi-foil technique, an extrapolation over 5 to 6 orders of magnitude would be required, and therefore the technique is erroneous. As used widely in diffuse X-ray scattering experiments, a technique of measuring the intensity scattered from polystyrene substituted for the sample is used in the present work. The scattered intensity at $2\theta=100°$ is 4000-8000 counts/sec/sr, which corresponds to 20-40 cps in the case of 1×1 mm^2 slit size of S2 in Fig. 15. The SRO diffuse scattering is comparable to the polystyrene scattering intensity, and requires 100 sec of counting time to hold the statistical error within ± 2 % for one measurement point.

V. Measurement Procedure

1. Sample setting

In the case of the short-range order diffuse scattering measurements, a sample crystal of a disc shape ($\geq$10 mm in diameter) is usually used. The sample must be placed in a way that the crystal surface (not the crystal lattice plane) is normal to the ϕ axis. By this, the crystal surface can be maintained symmetrical with respect to the incident and diffracted beams, so as to have an absorption factor independent of θ-2θ angle.

Precise knowledge of the lattice parameter and the wavelength of X-rays is needed in the present method. Knowing approximate index of the surface normal of the sample helps manual searching of the required reflections. A program SRO-A has been prepared as a TSS file of ACOS Series 77 NEAC System 900 in Tohoku University Computer Center which includes the following processes;

[1] Preliminary angle calculation (χ, 2θ) for peak hunting, and

[2] orientation matrix (UB) determination.

Process [1] is for obtaining values of χ and 2θ angles for the reflections to be searched manually. In hunting the reflection, χ and 2θ circles of the diffractometer are, at first, set at the values calculated roughly in the Process [1], and ϕ motion is made till the reflection is sensed. At this stage, no receiving slit is placed in the front of a detector, and χ and ϕ motors are manually drived. With a receiving slit in position, then, correct angle values for the reflection peak are found with the use of the ratemeter and recorder.

2. Determination of orientation matrix

Angle values of χ, ϕ and 2θ for three independent reflection peaks can be used for determining the orientation matrix (UB) by Process [2] in SRO-A, which is explained in Appendix A1.

3. On-line process of diffuse intensity measurement

A flow-chart of the intensity measurement process is shown in Fig. 18 and in greater detail in Fig. A2 in Appendix A2. The program counter (PC) in the CPU is set to a starting address, and the program is run. The switch registers of Program Selector are read by an IOT instruction, at first ((2) in Fig. 18). When χ or ϕ Manual Drive Program Switch is on, the motor can be drived by the Orienter Remote Controller. If a Main Process Switch for balanced-filter method or

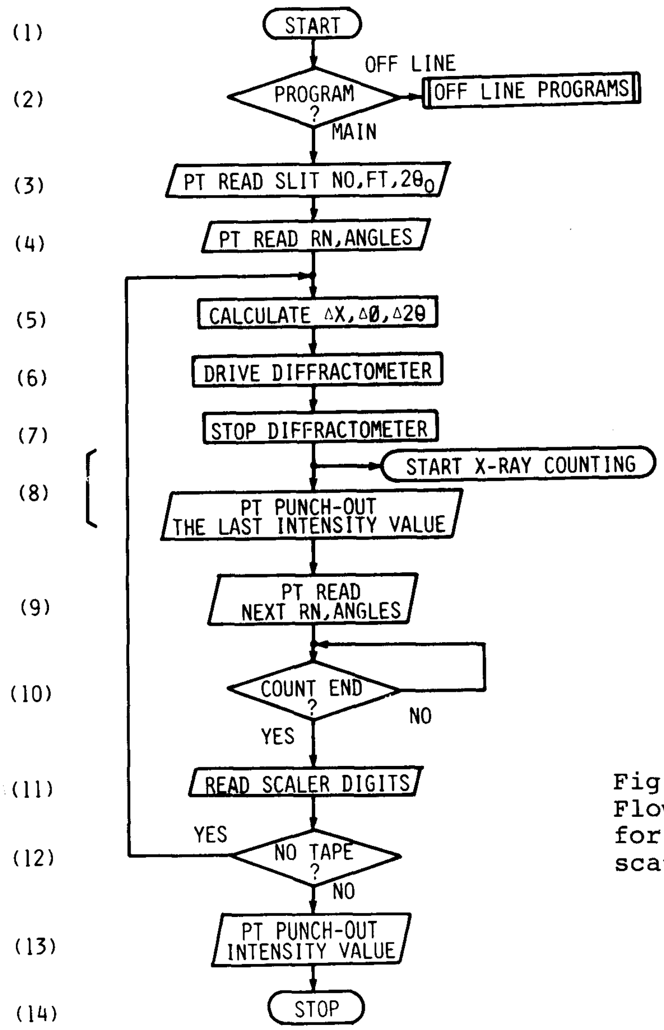


Fig. 18.
Flow-chart of a software for measuring the diffuse scattering.

non-filter method is on, a paper tape, on which parameters of the initial condition of measurement (fixed time or count, reference number of the slit system, initial setting angle of 2θ etc.) have been punched, starts to be read (3), and the information is displayed on the LED Display Panel. Then, the routine pauses till a reset switch is pushed on. At this stage, it is possible to check the setting parameters of the instrument. With the switch on, a paper-tape containing angle values of χ, ϕ and 2θ corresponding to a datum point to be investigated and a reference number of the point is read (4). Angle difference between this point and the last one are calculated (5). χ, ϕ and 2θ are driven one after another. After the 2θ motor is stopped (7), counting of X-ray quanta starts, and simultaneously an intensity value measured at the last step is punched out on a paper-tape, if it is not the first step (8). Then, the angle values of the next point are read with the paper-tape reader (9). At this stage, the CPU waits a count end signal from the Scaler/Timer (10). If the signal becomes active, scaler digits are read by an IOT instruction and stored in the RAM (11). The process of (5-12) in Fig. 18 is repeated. In the balanced-filter method, the process (8) is carried out in turn for Ni and Co filters without driving χ, ϕ and 2θ circles.

4. Input and output data of measurement

There are two kinds of data on the input paper tapes. One of them is the information of the initial setting condition of the instrument, and it is possible to record 2θ value of 2 figures in degree at a starting point and any seven figures on the micro-computer RAM through the paper tape input. The seven figures are useful for recording fixed time or fixed count value, slit system number, etc.. The other is the setting angles (χ, ϕ and 2θ) corresponding to the datum point. In the tape, 5 figures representing a reference number of the point and three sets of 5 figures representing χ, ϕ and 2θ values in degree are punched for one step of measurement. Angle values are calculated down to two places of decimals, and multiplied by 100 to be treated as integers. As mentioned above, for χ, ϕ and 2θ, one step represents an angular displacement of 0.01°, 0.05° and 0.04°, respectively, and therefore, integral values to be punched must be a multiple of 1, 5 and 4 respectively. The format of the data punched on the tape is given in Appendix A3.

Measured intensity data are punched out on a new tape with a reference number, which can identify the data in a TSS file of reflection index list. Intensity values are of 6 figures. The format for the input to a TSS file is given in Appendix A3.

5. Data processing

In the present system, no monitoring device is attached. Stability check of the incident beam intensity is made by measuring the identical reflection point as a standard for every score of steps. Observed intensities are normalized with respect to the standard point intensity. This is the first step of data processing in TSS. Successive process of calculation is divided into the following four steps;

[1] Index identification comparing the reference numbers with an index list in TSS file,

[2] air scattering correction and normalization with the polystyrene scattering intensity,

[3] separation of the SRO intensity and the atomic displacement modulation intensity by Borie-Sparks method[1,2]

[4] Fourier transformation of the SRO intensity and the atomic displacement modulation intensity to obtain the Warren-Cowley α parameters and the atomic displacement γ, δ and ε parameters.

TSS program for these analyses is prepared, and will be described in part II of this series.

V. Concluding Remarks

The system described in the present paper is installed in the room of X-ray diffraction laboratory of RIISOM and has been shown to be used successfully for the diffuse scattering intensity measurements of Cu-based disordered alloys. Extension of the measurements to the study of short-range order in crystals subjected to high pressure and high temperature is scheduled along with the improvement in data acquisition rate.

Acknowledgement

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Appendix

A1. Definition of crystal orientation on the Single Crystal Orienter

As mentioned in V. 1 and 2, SRO-A is prepared for angle calculation of χ, 2θ circles for preliminary peak hunting, and for orientation matrix determination. The former program is based on the following equation; Let [u,v,w] and h_1, h_2, h_3 be the indices of the surface normal vector and those of a reflection peak to be searched, respectively. χ angle is calculated as

$$\cos\chi = \frac{uh_1+vh_2+wh_3}{\sqrt{(u^2+v^2+w^2)(h_1^2+h_2^2+h_3^2)}} \qquad \text{(A1)}$$

2θ is calculated with a Bragg's law.

On the diffractometer the crystal is rotated about three axes (θ or ω, χ and ϕ), and the detector is rotated about one axis (2θ). A right-hand p_1 p_2 p_3 coordinate system is defined relative to the diffractometer as shown in Fig. A1(a). When all the circles are at

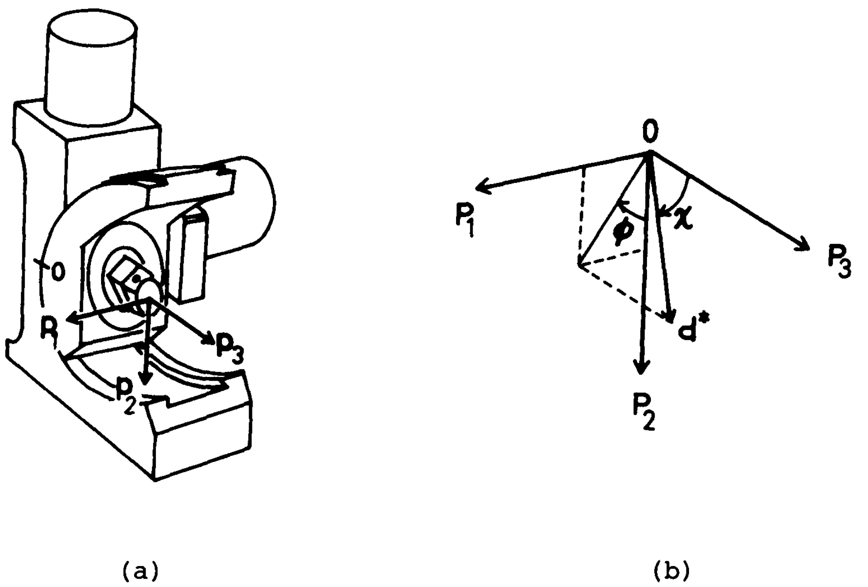

Fig. A1. (a) Quarter-circle orienter at χ=0. (b) A relation between a datum point and χ, ϕ angles to satisfy a Bragg's law.

their zero setting, a normal to a lattice plane has an orientation expressed by this coordinate system. This lattice plane can be set to a position for a Bragg reflection by turning the crystal on the ϕ axis until the normal falls in the plane of the χ arc. The χ circle is then rotated to bring the normal into the plane of incidence, and the normal is set according to Bragg's law by adjusting ω to θ_{Bragg} and 2θ to $2\theta_{Bragg}$.

Reflection from the lattice plane is represented in reciprocal space by a vector d* along the normal. The length of d* is equal to $2\sin\theta_{Bragg}/\lambda$, when the radius of the Ewald sphere is taken as $1/\lambda$ in reciprocal space. The p_1,p_2,p_3 components of the vector d* can be defined from the setting angles for the Bragg reflection:

$$p_1 = |d^*| \sin\chi \sin\phi,$$
$$p_2 = |d^*| \sin\chi \cos\phi, \quad \text{(A2)}$$
$$p_3 = |d^*| \cos\chi.$$

Refer to Fig. A1(b). Let a vector Q_j be represented by (q_1,q_2,q_3) in a coordinate system relative to the crystal lattice. Its length is taken as $|d^*|$. The coordinates (p_1,p_2,p_3) and (q_1,q_2,q_3) are connected by

$$\begin{pmatrix} p_{1j} \\ p_{2j} \\ p_{3j} \end{pmatrix} = \begin{pmatrix} a_{11} & a_{12} & a_{13} \\ a_{21} & a_{22} & a_{23} \\ a_{31} & a_{32} & a_{33} \end{pmatrix} \begin{pmatrix} q_{1j} \\ q_{2j} \\ q_{3j} \end{pmatrix} \quad \text{(A3a)}$$

or

$$(p_{ij}) = (a_{ij})(q_{ij}) = UB\ (q_{ij}), \quad \text{(A3b)}$$

where UB is the orientation matrix. If three independent sets of (q_{1j},q_{2j},q_{3j}) (j=1-3) and the corresponding (p_{1j},p_{2j},p_{3j}) are known, the elements of UB matrix can be calculated as

$$UB = (p_{ij})(q_{ij})^{-1}, \quad \text{(A4)}$$

where (p_{ij}) and $(q_{ij})^{-1}$ are 3×3 matrices. (p_{ij}) is known from the observed values of χ, ϕ and 2θ by equation (A2). The UB matrix resulted from an execution of the program [2] of SRO-A is stored in a TSS file UBMX.

SRO-A has a supplementary function calculating an angle by means of the UB matrix for any datum point. From equation (A2), χ, ϕ and 2θ are obtained as follows;

$$\chi = |\cos^{-1}(p_3/|d^*|)|,$$

$$\phi = \tan^{-1}(p_1/p_2),$$

where χ is in between the limits of 0° to 90°. Since ϕ must be represented by a value in the range from 0° to 360°, ϕ is rewitten as

$$\phi = \begin{cases} \phi' + 360t & \text{(degree) for } p_2 > 0, \\ \phi' + 180 & \text{(degree) for } p_2 \leq 0, \end{cases}$$

where

$$\phi' = \mathrm{Tan}^{-1}(p_1/p_2),$$

$$t = \begin{cases} 0 & \text{for } \phi' \geq 0, \\ 1 & \text{for } \phi' < 0. \end{cases}$$

A2. A detailed flow-chart of measurement process

See Fig. A2.

A3. Format of the tape

[1] Initial setting parameter tape

This tape is produced by a puncher in TSS and read in advance of the X-ray intensity measurement. The format is as follows;

a	b	c	d	e	f	a
Feeder	No 1	SP	No 2	2θ	No 3	Feeder

(a) Feeder consists of an array of codes except 20_{16}-$3F_{16}$,

(b) Number 1 is of 3 figures, (for example, as the nearest peak index),

(c) Space or Null code of JIS character,

(d) Number 2 is of one figure, (for example, as type of slit system),

(e) 2θ value of 2 figures in degree as a starting point of the goniometer,

(f) Number 3 is of 3 figures, (for example, as the fixed time in second or fixed count).

FLOW CHART OF DIFFUSE SCATTERING MEASUREMENT PROGRAM

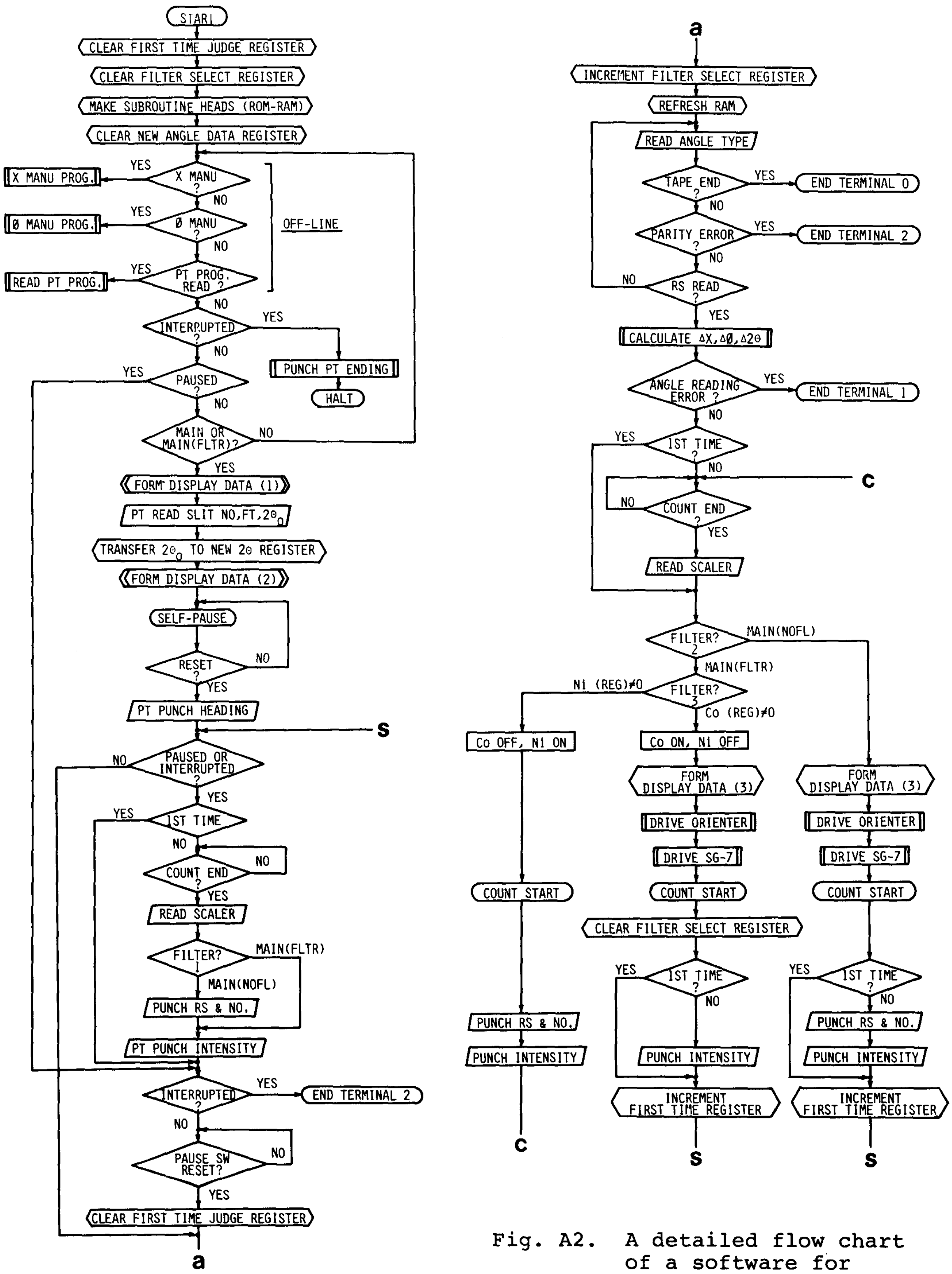

Fig. A2. A detailed flow chart of a software for measuring the diffuse scattering.

[2] Angle data tape

This tape is produced in TSS and read in the measurement process. The format is as follows;

a	b	c	d	e	f	b	c		g
Feeder	RN	χ	ϕ	2θ	RS	RN	χ	.....	$
	├── Record 1				──┤	├── Record 2	──┤		

(a) Feeder consists of an array of codes except 20_{16}-$3F_{16}$,

(b) Reference Number (RN) is of 5 figures punched with the 8-bit JIS character codes,

(c) χ, (d) ϕ and (e) 2θ angle values are of 5 figures,

(f) Record Separator (RS) consists of codes except 20_{16}-$3F_{16}$,

(g) $ code of JIS character implies the end of records.

[3] Output data tape

This paper tape is produced by the Paper Tape Puncher in the micro-computer control system, and the data are read and processed in TSS. There are two kinds of format for the measurement with the balanced filters and that without the filters.

Case 1 : Non-filter method

a	b	c	d	c	e	c	f	g	h	c	f	g	h	
Feeder	SI	RS	No2	RS	No3	RS	RN	SP	Int.	RS	RN	SP	Int.	....

Case 2 : Balanced-filter method

a	b	c	d	c	e	c	f	g	h	g	i	c	f	
Feeder	SI	RS	No2	RS	No3	RS	RN	SP	Int1	SP	Int2	RS	RN	....

(a) Feeder is an array of Null code,

(b) SI ($0F_{16}$) code of the 8-bit JIS character,

(c) Record Separator consists of CR($0D_{16}$), DEL($7F_{16}$)×6, LF($0A_{16}$) and DEL×5.

(d) Number 2 is of one figure, which is identical to No.2 in the input tape.

(e) Number 3 is of 3 figures, which is identical to No.3 in the input tape,

(f) Reference Number of the datum point,

(g) SP code of the JIS character (20_{16}),

(h) Intensity value (counted with Co filter in Case 2) is of 6 figures,

(i) Intensity value counted with Ni filter in Case 2 is of 6 figures.

Ending of these paper tapes is as follows;

	a	b	c	d	e	f	g	h	
....	CR	DEL × 6	LF	DEL×5	$$$EOF	DC3	CR	LF	....

(a) CR ($0D_{16}$) of the JIS character,

(b) DEL ($7F_{16}$) × 6,

(c) LF ($0A_{16}$),

(d) DEL × 5,

(e) $ (24_{16}) × 3, E (45_{16}), O ($4F_{16}$) and F (46_{16}),

(f) DC3 (13_{16}),

(g) CR,

(h) LF.