

JAERI - M
82-157

RESEARCH PROPOSAL FOR ADVANCED DIFFRACTOMETER
(US-JAPAN COLLABORATIVE RESEARCH
IN NEUTRON SCATTERING)

November 1982

Masashi IIZUMI and Satoru FUNAHASHI

日本原子力研究所
Japan Atomic Energy Research Institute

JAERI-Mレポートは、日本原子力研究所が不定期に公刊している研究報告書です。
入手の問い合わせは、日本原子力研究所技術情報部情報資料課（〒319-11茨城県那珂郡東海村）あて、お申しこしください。なお、このほかに財団法人原子力弘済会資料センター（〒319-11茨城県那珂郡東海村日本原子力研究所内）で複写による実費頒布をおこなっております。

JAERI-M reports are issued irregularly.

Inquiries about availability of the reports should be addressed to Information Section, Division of Technical Information, Japan Atomic Energy Research Institute, Tokai-mura, Naka-gun, Ibaraki-ken 319-11, Japan.

©Japan Atomic Energy Research Institute, 1982

編集兼発行 日本原子力研究所
印 刷 (株)高野高速印刷

RESEARCH PROPOSAL FOR ADVANCED DIFFRACTOMETER
(US-JAPAN COLLABORATIVE RESEARCH IN NEUTRON SCATTERING)

Masashi IIZUMI and Satoru FUNAHASHI

Division of Physics,
Tokai Research Establishment, JAERI

(Received October 14, 1982)

This paper is the proposal submitted to the US-Japan meeting (1980) for the collaborative research in neutron scattering, in which it is proposed to carry out various kinds of new measurements by using the wide angle, curved linear position-sensitive counter. The time-division measurement of the diffraction patterns and the single-crystal diffractometry utilizing the inclination geometry are the principal research area proposed.

Keywords: Neutron Diffraction, Time-division Measurement, Inclination Diffraction Geometry, Modulation Measurement, Texture Measurement

新型中性子回折装置による研究の提案

(中性子散乱日米協力のために)

日本原子力研究所東海研究所物理部

飯泉 仁・船橋 達

(1982年10月14日受理)

これは中性子散乱日米協力計画を協議するため1980年に開催されたハワイ会議に原研の提案として提出された計画案である。曲線型一次元位置検出型の広角カウンターを用いて、種々の新しい測定を行うことを提案しているが、特に中性子回折パターンの時分割測定及び傾角配置による単結晶回折が主要な提案の内容である。

PREFACE

This paper was presented at the Technical Meeting on U.S.-Japan Non-Energy Collaborative Research in Neutron Scattering held in Honolulu, Hawaii from June 30 to July 2, 1980. It was the first formal proposal from JAERI for the collaborative research.

Prior to this proposal it had been proposed by the Oak Ridge scientists to build the high-resolution powder diffractometer with the curved linear position-sensitive counter.

The main purpose of the proposal was to widen the scope of the research to be made and accordingly to modify the concept of the instrument to be constructed by describing what kind of research we, JAERI group, are interested in.

The principal modifications proposed in the present paper were

(1) to adopt the inclination-geometry of the diffractometer to be used for the single-crystal diffractometry and

(2) to facilitate the time-analysis capability in order to make the instrument to be used for the time-division measurement of the whole diffraction pattern.

This proposal was agreed upon basically in the Meeting and the design of the instrument has been carried out along the proposal. The main differences between the proposed concept and the one actually under design are

(1) the beam hole at HFIR to which the instrument is to be installed has been changed from the vertical beam hole originally proposed to one of the horizontal beam holes (HB-4A) where more intense beam is available, which is necessary in carrying out the time-division measurements and

(2) some of the proposed research such as the measurement of the texture of polycrystalline materials have been given up because they require a very flexible and versatile arrangement of the diffractometer. The inclination geometry has been realized in the design as the flat-cone arrangement

which is less powerful than the equi-inclination geometry but more suitable for the counter and shielding design since the scattered neutrons always hit the counter perpendicularly.

Although the paper was not published at that time, it is now considered to be worth while to publish it as a document of the US-Japan Collaborative Research which is now to be realized as it has been contemplated. It should be remarked that some of the schematic figures in the present paper was drawn for the vertical arrangement of the instrument and hence completely different from the arrangement actually under design.

On this occasion the authors would like to express their thanks to Y. Hamaguchi for his encouragement and to the US scientists in Oak Ridge for their friendly cooperation.

Contents

I.	Introduction	1
II.	Research Programs	3
	A. General	3
	B. Time-Division Measurements	4
	a. Once-Through Measurements	5
	b. Repetitive Measurements	8
	C. Field Modulation Method	9
	D. Quasi-Static Measurements	11
	E. Crystal Rotation Camera	12
	F. Texture Measurements	15
	a. Stationary Measurements	16
	b. Time-dependent Measurements	18
III.	Requirements of the Machine	19
	A. General	19
	B. Beam Intensity	19
	C. Time-Division Measurements	21
	D. Three Circle Goniometer	23
	E. Counter Positioning	23
	F. Cryostats and Furnaces	27
	G. Graphic Display	27

目 次

I. 序 論	1
II. 研究計画	3
A. 一般論	3
B. 時分割測定	4
a. 単発現象の測定	5
b. くりかえし現象の測定	8
C. 場の変調による測定	9
D. 準静的な測定	11
E. 結晶回転カメラ法	12
F. 集合組織の測定	15
a. 定常状態での測定	16
b. 時間変化の測定	18
III. 装置の必要条件	19
A. 一般論	19
B. ビーム強度	19
C. 時分割測定	21
D. 三サークルゴニオメーター	23
E. カウンターの配置駆動	23
F. クライオスタットと電気炉	27
G. グラフィックディスプレイ	27

I. INTRODUCTION

In the ORNL part of the U. S.-Japan cooperation on neutron scattering it has been proposed to construct a powder diffractometer with a linear position-sensitive counter. Based on this concept of the instrument we, JAERI scientists, have proposed to modify it to an advanced diffractometer with the following additional functions.

- (1) Time-division data collection
- (2) Single crystal diffractometry
- (3) Texture measurements.

In this memo we are going to describe the details of the research programs using this advanced diffractometer. It will be emphasized which kind of new research field will be made practicable by the introduction of the advanced functions to the basic concept of the instrument.

The key function of the proposed instrument is the time-division data collection. This is especially valuable in the alloys and other materials research to which our main research efforts are going to be concentrated. Moreover the method is expected to introduce a new technique which is useful in the fundamental solid state physics.

It must be reminded that in general a new facility gives rise to a development of quite new, unforeseen research fields, if it is really innovative. We expect that the following statements are just a partial anticipation of the future problems to be investigated by using the proposed instrument.

In the latter half of this memo we are going to describe the required performance of the new instrument in order to realize the additional functions mentioned earlier. In this description we assume the basic concept of the instrument proposed by the ORNL side and describe only additional requirements.

II. RESEARCH PROGRAMS

A. GENERAL

There are three essential ingredients which make it possible to develop new experimental techniques and accordingly to carry out new research programs. They are (1) intense neutron beam of HFIR, (2) efficient data collection capability of linear counter and (3) versatile data processing ability of a computer with large memory capacity and with graphic display function. The combination of (1) and (2) may reduce the minimum counting time required to obtain a diffraction pattern to a few seconds. The greatly improved efficiency can be utilized to carry out the time-division measurements, by which we observe the dynamic behavior of a condensed matter through the change occurring as a function of real time. This is particularly interesting in order to investigate the behavior of a system which is far from the equilibrium, provided the time evolution takes place rather slowly. The research program of this category will be described in Sec. B.

As an extension of the time-division measurements it is also possible to develop a novel technique which we tentatively call the field modulation method. This will be explained in Sec. C.

The efficient data collecting capability can also be utilized for other purposes. This is just to obtain a large number of diffraction patterns as functions of some parameters. The parameters can be temperature, pressure,

stresses, applied fields and so on. Then we can investigate a details of the quasi-static change of the structures and of the short or long range orders in solids and liquids especially in the vicinity of phase transitions (Sec.D Quasi-static Measurements).

The parameter can be the orientation angles of a sample. If the sample is a single crystal a map of scattered intensity distribution on a certain plane in a reciprocal lattice will be obtained after the reconstruction of the pattern by computer and graphic display device (Sec E. Crystal Rotation camera). If the sample is a polycrystalline sheet with preferred orientation a texture pattern is obtained (Sec.F. Texture Measurements).

B. TIME-DIVISION MEASUREMENTS

In the time-division measurements our interest is to observe the transient behavior of the system after introducing a stepwise change of temperature, pressure, fields etc. in a system. If the change occurs irreversibly the transient is observable as a once-through process. In this case we can not observe very rapid change because of the lack of scattered intensity. If the change is reversible with the change of state we can accumulate the scattered intensity for a number of repetitive cycles. Then a fairly rapid change is observable in this case. Our estimate of the observable time scale is one tenth of a minute in the once-through measurements and ten milliseconds in a repe-

titive measurement.

Anyway the real-time observation is restricted to fairly slowly varying dynamics in comparison with the dynamics observed by spectroscopic methods. A lot of interesting time-dependent processes taking place in condensed matters are beyond scope of this method. Nevertheless there are many important problem areas which are suitable to the method. The diffusion-controlled processes in solids are typical example of importance. Some kinds of chemical and biological processes are also interesting. Moreover we may expect that the introduction of the method actuates development of unforeseen problems. In the followings we concentrate on the investigation of the unmixing process of supercooled binary solid solutions and see how the method can be used to disclose the important aspects of the problem.

a. Once-Through Process

When an irreversible approach to an equilibrium is accomplished by a diffusion process in a solid at low temperature, it is observable as a once-through process. The time evolution of the diffraction pattern should be measured following through the irreversible change of the sample. The unmixing of supercooled solid solutions is a typical process of this type to which our primary interest is directed.

The unmixing proceeds either through the "nucleation and growth" or the spinodal decomposition according to

the supercooled condition. When the solution is supercooled to the metastable region of the phase diagram, the nucleation and growth takes place, while the spinodal decomposition occurs when the solution is brought to the unstable region of the phase diagram.

The spinodal decomposition is characterized by the continuous and coherent unmixing taking place simultaneously in the whole lattice. It gives rise to a modulated structure consisting of a two-phase mixture typically with a 10 - 200 Å size. The time constant of the decomposition is roughly $10^{12} |D|^{1/2}$ sec, where D is a negative diffusion coefficient. For the most of binary alloys of interest we can choose the temperature range in which the time constant has a proper value for the time evolution to be measured by the time-division measurements.

Spinodal decomposition of various kinds of alloys has been extensively studied by the X-ray small angle scattering. But there still remain several important problems which are only resolved by a detailed and careful measurements of the time-dependence by the neutron diffraction. They are, for example, (1) clustering in the course of quenching to a low temperature under variable rate of quenching speed, (2) time evolution of the decomposition process as a function of aging temperature and also (3) the time-dependence of the reversion process. The neutron is particularly useful in getting a clear contrast of the scattering by solute from that by the solvent in very important systems such as Fe-Cr, Cu-Ti and Al-Mg.

Although the decomposition has been studied by the small-angle neutron scattering (SANS), the large-angle diffraction is more informative than SANS. The modulated structure gives rise to side bands in the vicinity of fundamental Bragg reflections. The detailed study of the side bands ^{in a single crystal sample} reveals the structure of lattice modulation as well as the compositional modulation. Since the SANS can see only the latter, the complementary use of SANS and the large-angle neutron diffraction may be very effective to determine the structure, though the time-dependent SANS measurements have not been realized yet.

The rapid time evolution of the aging process has been observed by the intermittent aging method in which the quenched sample is heated to the aging temperature for a certain short period of time and is then quenched and the measurement is carried out at low temperature. This cycle is repeated and aging time is assumed to be additive. The validity of this intermittent measurements has not been established. Observation of real time evolution is naturally superior to the intermittent measurements.

Another interesting technique is the time-dependent observation of the decomposition under repetitive pulse heating of the sample kept at the low-temperature isothermal condition. One can adjust the time scale of the evolution with a suitable choice of average power.

The nucleation and growth process is another interesting phenomena. Early stage of the process is especially interesting. We haven't had a direct means of observing

thermal fluctuation which gives rise to the formation and disappearance of the cluster, or embryo. This stage may perhaps be beyond scope of the present time-division method, by which we can only observe the growth of the nucleated precipitates. The in situ observation of the coarsening of the precipitates under heat treatment is a good example of the once-through time-division diffraction measurements.

b. Repetitive Measurements

If the initialization of the time-dependent measurement can be done easily and rapidly, the measurement can be carried out repetitively to obtain a good statistics of data. Then the observable time scale can be reduced to one tenth or one hundredth of a second. The lower limit of the time scale is determined either by the inhomogeneity of the condition (say, temperature) in the sample or by the uncertainty in the flight time of neutrons.

There are a wide variety of problems to be investigated by this method. The particular problem we are interested in is the reversion process in supercooled solid solutions. When a decomposed solid solution is heated to a temperature which is still in a miscibility gap, it sometimes restores the homogeneous state before decomposition. With the decrease of temperature we can again observe the decomposition. This decomposition and reversion process are repetitive with the cyclic change of temperature of the solid solution. Then we can investigate

the time dependence of the structure change occurring in the decomposition and reversion.

C. FIELD MODULATION METHOD

As a special case of the repetitive time-division measurements let's consider the time change of diffraction patterns after a change of applied field is introduced to the sample. Suppose we apply magnetic field to a magnetic material. The simplest procedure is the one in which we remove abruptly the field and see the response of the magnetic part of the diffracted neutrons. Essentially the same but more accurate and more informative measurements of the response can be done by the field modulation method proposed in the followings.

In this method we apply periodic magnetic field to a sample. Any change of diffraction pattern induced by the modulation field is detected by the Fourier analysis of the time-dependent diffraction data collected by the repetitive time-division method. Information obtained by this method may be diverse according to the magnetic state and magnetic property of the sample.

As a simple case let's consider to apply magnetic field $H = H_0 + H_1 \cos \omega t$ ($H_1 \leq H_0$) perpendicular to the scattering vector. The magnetic scattering length is expressed as $b_m = b_m^0 + b_m^1 \cos \omega t$ so long as the response is linear. Here b_m^0 is the magnetic scattering length in the absence of the oscillating field. Then the scattering

includes two periodic components, i. e. $2b_m^0 \cdot b_m^1 \cos \omega t$ and $(1/2)b_m^1{}^2 \cos 2\omega t$. It should be noted that the $\cos \omega t$ term is enhanced by b_m^0 even though b_m^1 is not large in general.

Merits of the field modulation method are that the magnetic scattering is separated from the nuclear part and that the long-time instrumental drift does not affect the result. Therefore high statistics experiments can be carried out. For example, very weak ferromagnetic scattering of polycrystalline sample may be detected by this method. Paramagnetic scattering will be measured in the presence of intense nuclear scattering.

Another merit of this technique is that ω can be changed in order to study dynamic behavior of spins. For example, in the vicinity of the freezing temperature of the spin glasses, dynamic behavior of spins will be drastically reflected to the ω -dependence feature.

Critical phenomena around the second order phase transitions are also interesting objects of this method. Critical slowing down near the Curie temperature of ferromagnetic materials will be directly observed by applying on-off modulation as well as sinusoidal one. Dynamic behavior of spins near the second order transitions caused by magnetic field will be also observed by this method.

Various modifications of this method may be possible. If we apply the static field H_0 in the scattering plane leaving H_1 in the perpendicular direction, the $\cos \omega t$ term will disappear and the enhancement due to cross term with

b_m^0 will be lost. In this case, however, we shall be able to apply high magnetic field enough to saturate magnetization. Such configuration will be effective for ordered magnetic materials in general.

The field modulation method will possibly be applied to other magnetic systems, such as antiferromagnets, spin reorientation materials, metamagnetic materials etc.

This method can as well be applied to the dielectric materials by the use of modulated electric field.

D. QUASI-STATIC MEASUREMENTS

The use of the linear counter gives rise to a tremendous increase of the amount of diffraction data we can obtain within a reasonable machine time. This makes it possible to observe detailed changes of diffraction patterns as functions of state variables such as temperature, pressure, stresses and fields. Suppose we change the condition of sample fairly rapidly keeping the sample always at the equilibrium. Then we can "snap-shot" the diffraction patterns in the course of quasi-static change.

This technique is valuable to determine the equilibrium phase diagram in various materials. It is also useful to observe details of any kind of structural changes, whether they occur abruptly or gradually.

The research programs we are especially interested in are (1) phase changes in alkali-metal graphite intercalates as a function of temperature or vapor pressure

of alkali-metals, (2) change of structures of liquids at supercooled state and those of binary liquids with a miscibility gap, (3) change of clustering in one-phase solid solutions and (4) change of structures of amorphous substances, for example, crystallization from amorphous state.

E. CRYSTAL ROTATION CAMERA

One of disadvantages of the neutron diffraction method in comparison with the X-ray or electron diffraction methods has been the difficulty of taking diffraction photographs. If we can get a photograph indicating the distribution of diffracted neutrons in the reciprocal space, it would be very powerful in various kinds of experiments. Especially the search for superlattice reflections or diffuse scattering in the reciprocal space becomes very efficient. In the conventional neutron diffraction method the search is usually limited to selected lines with high symmetry in the reciprocal space. There has, therefore, been a possibility to overlook superlattice reflections and diffuse scattering outside the search. If the scattering is incommensurate, as it is in the problem areas of recent interest, the possibility of overlooking increases. In these cases the photographic search is very valuable.

By the ingenious use of the linear counter together with the intense monochromatic beam we can obtain a map of scattered intensity on the screen of the graphic

display device very easily and very rapidly. This is essentially equivalent to the X-ray or electron diffraction photographs.

The "photograph" of the scattered neutron distribution on a zone is simply obtained by mounting the sample crystal with the zone axis perpendicular to the scattering plane defined by the monochromatic beam and linear counter and by rotating the crystal around the zone axis continuously or step-wisely. ^(Fig.1a) The map is constructed from a series of diffraction patterns measured for successive angles of rotation. Perspective plotting of the scattered intensity on the zero-level plane on the reciprocal space is shown on the screen by an appropriate transformation of the coordinates.

In order to observe the scattering not on the zero level of the reciprocal space, one may use an Eulerian cradle in order to bring upper level reciprocal points to the zero level scattering plane. However, in this geometry every point on the linear counter "sees" different level of the reciprocal space. In order to take the full advantage of the linear counter it is more suitable to arrange the scattering geometry in such a way that every point on the counter "sees" the same level of the reciprocal lattice at the same time. This can only be realized by translating the counter from the zero scattering plane (Fig.1b). In order to get rid of the central blind area which appears inevitably on each upper level of the reciprocal lattice, we can utilize the equi-inclination technique usually

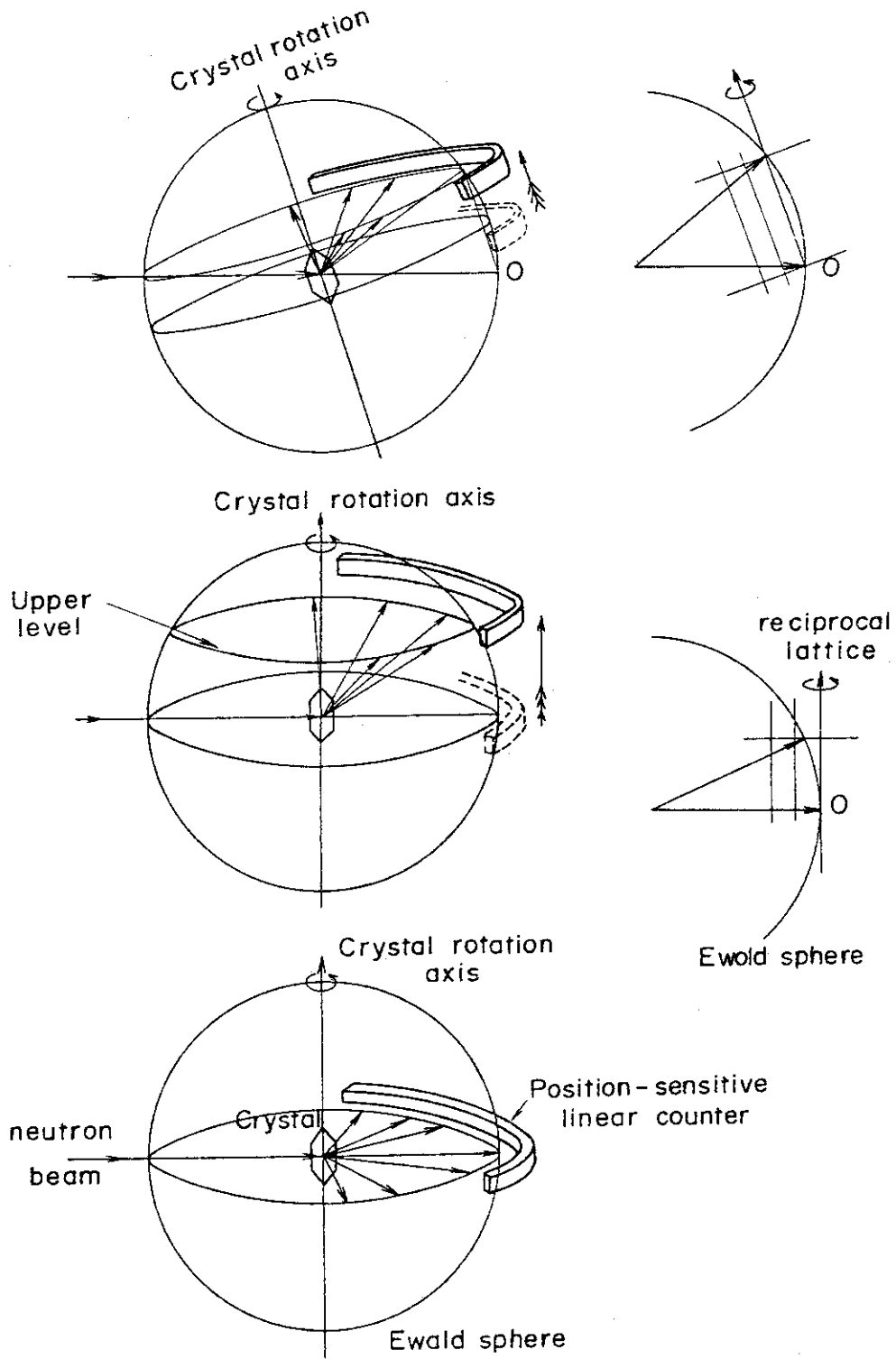


Fig. 1 Crystal rotation camera

adopted in the Weissenberg camera of X-ray diffraction. For this purpose it is necessary to translate and incline the counter together with the same amount of inclination of the rotation axis of the crystal (Fig.1c).

The crystal rotation camera has a broad application including not only the crystal structure determination but also the diffuse scattering and superlattice measurements associated with various kinds of phase transitions. It should also be emphasized that this method is very useful in the study of low-dimensional systems.

The structural phase transitions in the graphite intercalates, the structural phase transitions involving incommensurate structures, the diffuse scattering in low-dimensional magnetic materials and the modulated structures in alloys are examples of the problems we intend to study by this method.

F. TEXTURE MEASUREMENTS

Most of the engineering materials are random polycrystalline aggregates which develop preferred orientation, or textures, upon solidification, plastic deformation, recrystallization, phase transformations and so on. Since the properties of the materials are affected by the texture and they are somewhat controllable by the manufacturing process, the X-ray diffraction has been routinely used to determine the pole figures of the texture in industry. Nevertheless the X-ray diffraction method has serious

disadvantages and some times it is difficult to obtain accurate textures. This is due to the high absorption and consequent limited penetration of X-rays. The important subsurface texture in thick metallic samples can only be obtained by employing destructive machining or etching to them.

Since, in contrast, thermal neutrons have a high penetrating power for most materials, the neutron diffraction method is highly superior to the X-ray method. Therefore the neutron diffraction method has been increasingly applied to the texture measurements. Recently JAERI group started a research program in which the change of textures of metals with temperature, rate of deformation etc. is the object of investigation.

a. Stationary Measurements

In the conventional measurement of textures a pole figure is obtained by measuring diffraction intensity as a function of orientation angles of a sample with the counter being fixed at a certain Bragg angle of principal reflection (Fig. 2a). The time required to obtain a pole figure is greatly reduced if the intensity along a certain Debye-Scherrer ring is obtained simultaneously by the use of the linear counter. This can be done by arranging the plane of the curved linear counter perpendicular to the incident neutron beam and translating its position to a suitable scattering angle (Fig. 2b).

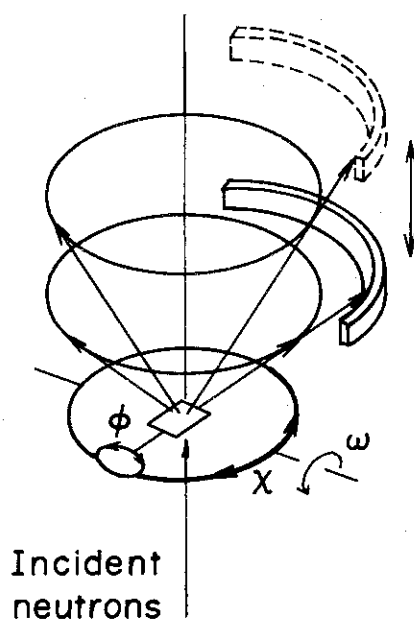
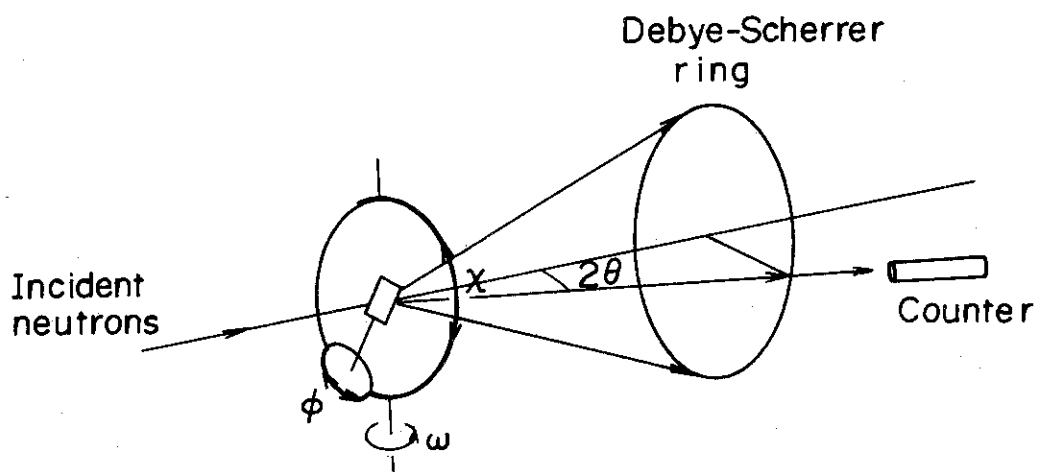


Fig. 2 Texture Camera

The pole figure is obtained by changing ω and χ stepwisely in the transmission measurement and by changing χ and ϕ in the reflection measurement. We can do almost without χ movement by the use of the linear counter. Then the pole figure is obtained by scanning only one angle. Combined with the high intensity a single figure may be plotted within several minutes.

b. Time-dependent Measurements

It is very interesting to observe in situ the change of the texture pattern with the change of sample conditions (temperature and stresses). This is undeveloped but very promising field of research. In the in situ measurements of the texture change we will be able to limit the part of the pole figure to be investigated. Then the desired part is obtained within a minute or so and the dynamic change of the texture with deformation is observable. This is quite informative and useful in determining the condition of the deformation in order to obtain the required distribution of the polycrystalline aggregates.

III. REQUIREMENTS OF THE MACHINE

A. GENERAL

The basic concept of the instrument is the diffractometer with a position sensitive linear counter as proposed originally by the ORNL scientists. Most of the research programs described so far constitute quite new types of problems which require versatile additional functions of the instrument. The feasibility of the research entirely depends on the technical realization of the requirements. Most of them are not so difficult to accomplish. However, the beam intensity (Sec. B) and the counter positioning (Sec. E) are likely to become serious problems.

B. BEAM INTENSITY

Most of the research programs are such advanced ones that can only be undertaken by the use of high flux beam. In particular the feasibility of the once-through time-division measurements is crucially dependent on the beam intensity.

In order to observe the once-through process with the unit time-division of five seconds neutron flux at the sample is estimated at least to be 3×10^7 n/cm²·sec. This estimation is based on the fact that the minimum counting time per step required to obtain a powder diffraction pattern is about 30 seconds when we use our most intense beam at JRR-2 which is estimated to be 5×10^6 n/

cm²·sec at the sample position for 40 meV neutrons (bent PG monochromator with 30 min collimation).

Bragg reflections from a single crystal sample are naturally easier to observe with lower flux. However, the side bands related to a modulated structure given rise to as a consequence of the spinodal decomposition are fairly weak especially at the earliest stage of decomposition. Our primary interest in the time-division measurements lies in observing the time evolution of the side bands from its earliest stage to the final formation of GP zones. This can only be observed by the use of single crystals together with the intense beam ($2-5 \times 10^7$ n/cm²·sec).

Realization of such an intense beam at the proposed instrument site is likely to be very difficult. The 90° reflection by the monochromator and the long distance from the monochromator to the sample position give rise to a very good resolution of both the wavelength and angular divergence but accordingly to a poor intensity.

An elaborate design of the monochromator and beam path might make up somewhat for the lack of intensity. Nevertheless the required beam intensity is likely to be attainable only by the use of monochromatized beam with the horizontal reflection to a medium Bragg angle. Thus the ad hoc usage of the linear counter on an existing instruments should be considered for the specific measurements which require very intense beam.

C. TIME-DIVISION MEASUREMENTS

In order to undertake the time-division measurements some circuits should be added to the detector electronics of the linear counter. The block diagram of the circuits is indicated in Fig.3. The time-division can be realized simply by adding to the add-one circuit a register indicating the starting address of the memory blocks in which the angular data from the linear counter should be accommodated. The starting address on the register is changed step by step every time it receive a signal from the time-interval unit.

In the time division and field modulation measurements the data collection should be done synchronously with the change of external conditions applied to the sample. This may be ensured by using the trigger signal from the same time-interval generator to control the external conditions.

The control of data accumulation in the quasi-static measurements can also be carried out by using the same register. The case of temperature change is illustrated in the figure. The electromotive force of the junction is AD-converted and is compared with the standard voltage. Every time the emf exceeds the voltage the starting address is changed to that of the next memory block and also the standard voltage is changed by a predetermined increment.

The type of the linear counter and the detector electronics should be chosen by considering the requirements of the time-division measurements. We think the multi-wire counter together with the wire-to-amplifier

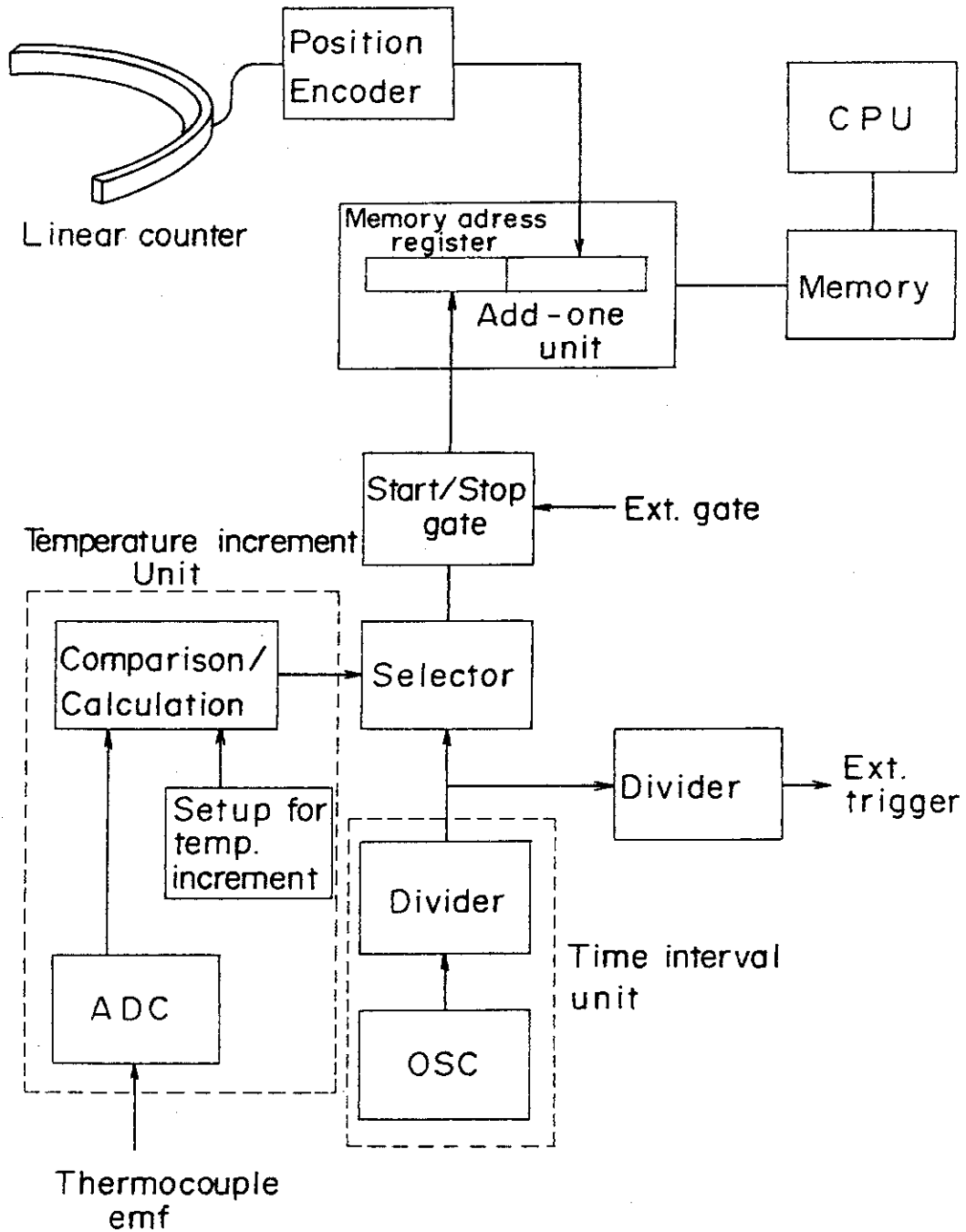


Fig. 3 Block diagram for the time-division measurements

electronics is superior to the counter with resistive wire with the rise-time analyzing electronics.

The size of the memory to be used for the data acquisition in the computer should be very large, say 1 Mb.

D. THREE CIRCLE GONIOMETER.

The single crystals and texture measurements proposed in Part II require a three circle goniometer at the sample position. Unusual arrangement of the proposed instrument requires a special type of three circle goniometer which has a horizontal ω axis (Fig.4). The χ -circle and goniometer head should be large enough to install cryostats, furnaces and other equipments.

E. COUNTER POSITIONING

In powder diffraction measurements the counter position is fixed or it may only be enough to rotate it within the vertical scattering plane just to cover the wider scattering angles. On the other hand ^{the} crystal rotation camera and the texture measurements proposed in Part II require very flexible positioning of the counter. The counter is translated and sometimes inclined from the vertical scattering plane.

For this purpose a new type of cradle should be devised. Fig. 5 illustrates schematically one of the designs which are to accomplish the required flexible

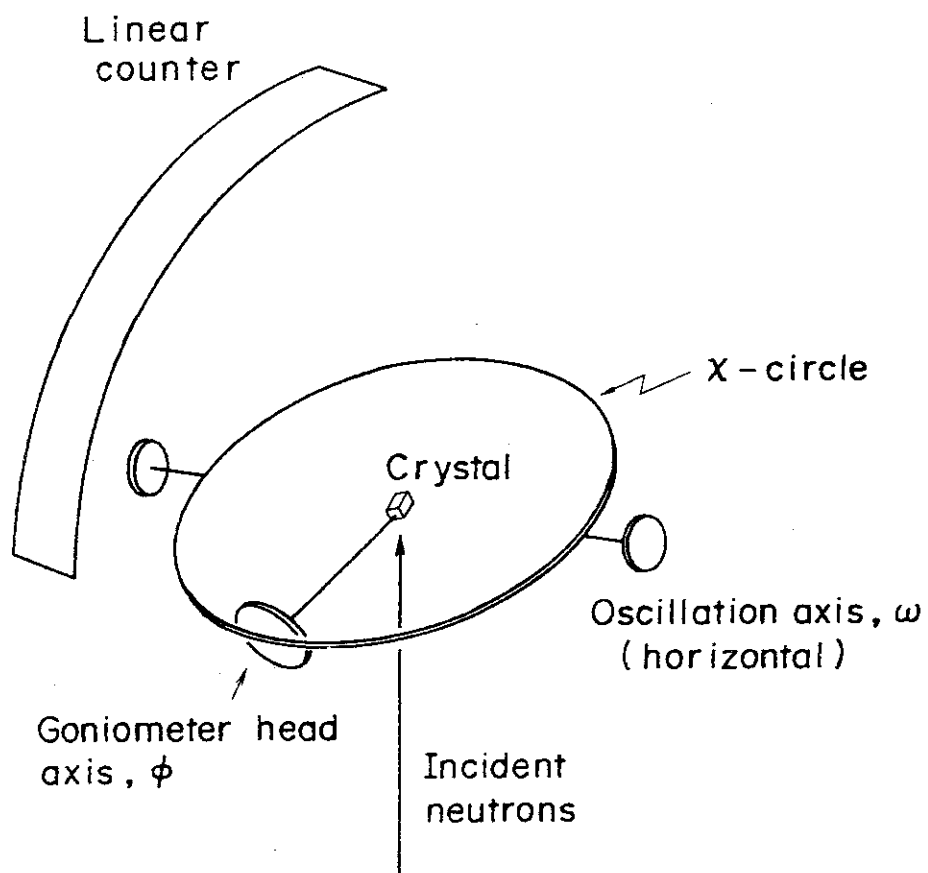


Fig. 4 Three circle goniometer with horizontal oscillation axis.

positioning. It consists of three moving parts: a pair of beam (B) on which the circle frame (A) carrying the counter translates and the counter carrier (C). Both A and B are able to rotate around horizontal axes. A typical arrangement for the rotation crystal camera at equi-inclination geometry is shown in Fig.5b in which the counter takes almost vertical position and displaced and inclined by the required amounts to measure scattering on the upper level of the reciprocal lattice. In the texture geometry shown in Fig.5c the counter takes the horizontal geometry and translates upward or downward for the counter plane to coincide with the Debye-Scherrer rings of the sample. If we restrict the geometry only to these two cases we can do without the rotation of the circle frame (A) against the beam (B); the circular face can be fixed to the position perpendicular to B. However the redundant freedom may not be useless.

The cradle for these purposes may be unusually huge-scale. However, the precision of the positioning is not required to be so strict as it is for the moving parts of usual spectrometers, because the finite vertical aperture of the linear counter covers the inaccuracy of the positioning.

It should be noticed that with this flexible positioning of the counter the sample is not always situated at the center of curvature of the linear counter and therefore diffracted neutrons do not always hit the front face of the counter perpendicularly. Therefore the counter

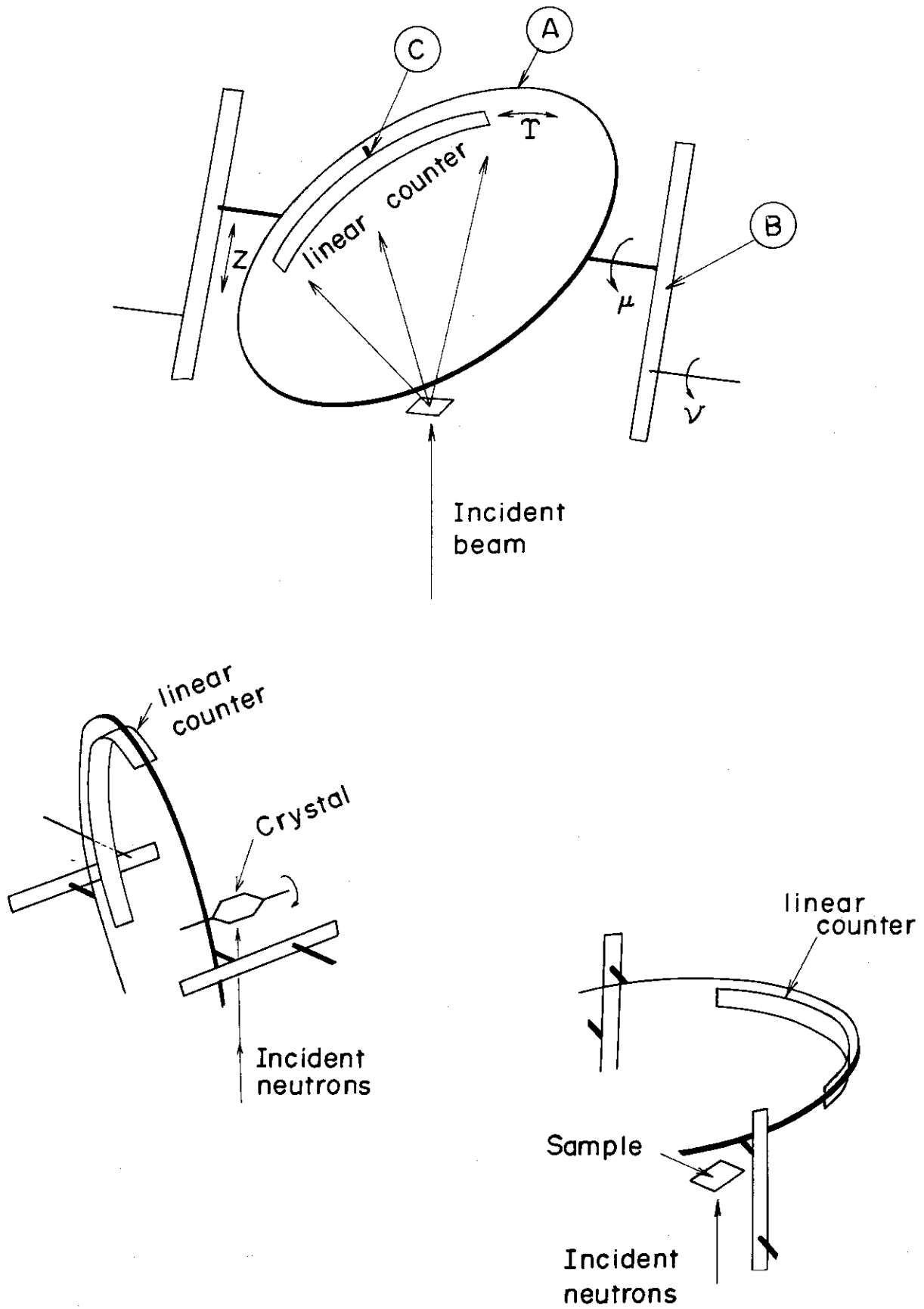


Fig. 5 Cradle for the counter positioning

shieldings should have a wide aperture which may cause the background troubles. The restricted aperture of the counter and shieldings sets limit to the translation range of the part C on the part B. In this respect the incident neutrons with shorter wavelength are suitable for obtaining the rotation "photograph" of the upper level diffraction. In the texture measurements it is convenient to adjust the wavelength of incident neutrons in such a way that the Bragg angle takes nearly 90° . The resolution effect of the texture measurements may be fairly complicated. Almost the same degree of collimation of both the horizontal and vertical angular divergences is desirable.

F. CRYOSTATS AND FURNACES

Auxiliary equipments such as cryostats and furnaces of standard specification should be furnished. The unusual geometry of the instrument requires proper equipments which can not be shared with other existing instruments. In order to mount on the three circle goniometer we recommend the cryostats with a closed cycle refrigerator. A special cryostat/furnace for the time-division measurements of the spinodal decomposition will be designed and prepared by JAERI.

G. GRAPHIC DISPLAY

In most of the proposed research programs we must

acquire and process a huge amount of data. An efficient and versatile graphic display device with well organized software will greatly help to carry out the measurements and data-processing. Devices of the similar function have already been developed in ORNL for the X-rays and neutron small-angle scattering measurements.