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# A Scanning Electron Diffraction System

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The paper describes a scanning electron diffraction system which has been built for the study of rapid changes in the crystal structure of thin-film specimens.

The system consists of a conventional single-lens diffraction camera, of equipment for sweeping the diffracted beams in two dimensions, and for obtaining displays of ring and beam intensity profiles.

The specimen is located close to the lens, which images the electron source to a fine spot on the viewing screen. Below the specimen is a system of magnetic deflecting coils which provide a controllable steady deflection in two dimensions of any part of the diffracted beams together with an alternating deflection. Thus any spot, or section of any ring, can be scanned across a fine pick-up slit. The fraction of the beam which traverses the slit is converted to light and amplified, without signal/noise degradation, by a photomultiplier. Thus the electron intensity profile of a spot in two dimensions, or the radial trace across an entire Debye-Scherrer pattern may be displayed directly. Simultaneously a second slit and a recording channel four orders less sensitive enables the profile of the primary beam also to be continuously displayed.

A brief discussion of the factors limiting the accuracy and sensitivity of the system is given.

The main advantages of such a system are: (1) increased accuracy of intensity measurements, 0.1% should be attainable with reasonably short scanning time, (2) electron manipulation of the intensity waveforms to remove the inverse high-power background, immediate integration of areas under profiles, ability to magnify any diffracted beam profile, (3) a very short time resolution of ring patterns, at present about 20 m secs with 2% accuracy. Thus rapid polymorphic and other structure transformations may be studied quantitatively as a function of time.

## Introduction

Since the intensities of currents of the order $10^{-12}$A can be directly displayed in the scanning electron microscope[1], it seemed clear that similar techniques could be used to measure diffracted electron beams and record them at a high rate. Moreover a preliminary study of solid state changes in alkali halides taking as little as 100 sec[2] had shown the need for such an instrument. A feasibility study showed that, for 2% accuracy on the peaks of rings, complete diffraction profiles could be recorded in 0.5 second. This is in contrast to the system of Bagdykyants[3] where recording times are given as 5 minutes. In fact read-out times of 50 milliseconds are obtained, which makes this system attractive for quantitative studies of high speed changes in the solid state.

## General description

Fig. 1 shows the principles of the system. The diffracted beams are focussed to the plane of two slits and any beam can be aimed into either slit by bending the rays in two dimensions with the aid of suitable magnetic fields. A direct component of field can bring any part of the diffraction pattern to the region of the slits, and an alternating com-

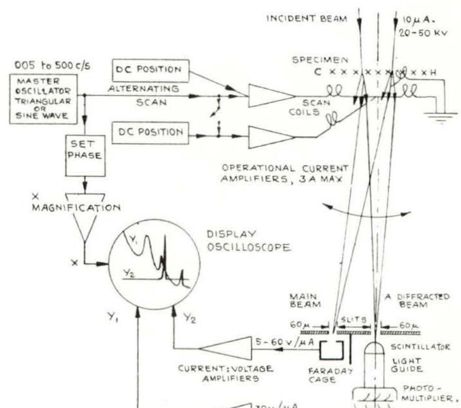



Fig. 1. Schematic diagram of scanning electron diffraction system.

ponent of field then scans it about the slit. Or entire ring patterns may be scanned past the slits. Through the slits electrons enter recording systems whose sensitivities differ by 4 orders, thus one system handles the primary beam, whilst the more sensitive system measures the diffracted beams. The profiles of primary and diffracted beams are then displayed on a twin-trace cathode ray tube, which has its time-base driven in synchronism with the scans.

**Some electrical details**

Magnetic deflection was chosen since the electron beams are not defocussed by the scan fields, and because it is not difficult to build accurate amplifiers to handle the large drive currents needed for deflecting high voltage beams.

The drive circuits for the scan were specially developed operational current amplifiers[4] such that both direct and alternating fields could be set up. Triangular sweep wave forms enable a direct check to be made of sweep linearity, since with inductive load only a linear waveform will give accurate registering of the pattern for both directions of travel. Since wave sweep with a phase adjuster is also satisfactory.

The pick up circuits are like those of the scanning electron microscope[5]. These must be of wide bandwidth, >1 Mc/s.

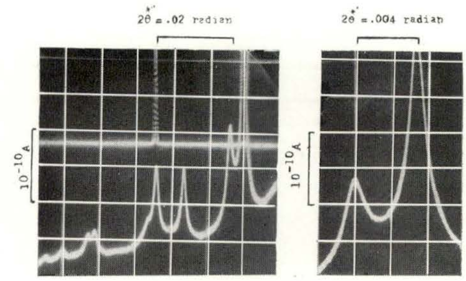
The display circuits permit magnification of the profiles in either dimension, and measurement of profile position.

Sweep, pick-up, and display circuits are direct coupled, thus any scan frequency from 0.005 to 50c/s can be handled.

**Results**

Fig. 2(a) shows typical patterns from a 300Å thick gold film, using 40 kV electrons. The incident beam intensity was $18\mu$A, the primary peak intensity was $6\mu$A, and current and angular deflection scales for the diffraction profiles are shown on the figure. The dashed effect is due to the use of a 150 kc/s beam switch to get the two traces. Fig. 2(b) shows the 111 and 200 ring profiles expanded. Noise is evidently comparable to trace thickness, giving a signal/noise ratio at the peaks of better than 50:1.

Rapid solid state changes have already been




(a) (b)

Fig. 2. Four sweeps on each trace, sweep time: 50 ms.

observed with the instrument. An example is amorphous to crystalline changes in silver films taking place in tens of seconds: for examination of changes as rapid as this a cine-camera must be used to record the traces.

**Limitations and developments of the system**

As with the scanning electron microscope, the ultimate speed of recording of the system is set by shot noise in the diffracted beams. The current in a 100 $\mu$ width slit at the peak of a strong reflexion is of order $10^{-9}$A, so that $10^4$ electrons arrive in 2 microseconds. A traverse across a pattern may take 500 slit-widths, so that for 1% accuracy on a peak, recording times of the whole sweep of 1 millisecond should be possible. Conversely a sweep rate of 10 c/s should permit intensity measurements of peaks to 0.1%. Present limitations on accuracy are due chiefly to 50 c/s stray field interference, and in recording speed to lack of bandwidth in amp-to-volt-amplifiers. 0.1% accuracy of position is a question of sufficient negative feedback in sweep amplifiers; cathode ray tubes resolving 2000 lines/face will ensure sufficient accuracy of display.

Once the diffraction information is in electrical form at a high enough signal level, a variety of manipulations is possible. (1) Fourier analysis of waveforms by a wave-analyser; the terms will be related to those of the Fourier synthesis of the crystals. (2) Inverse-6th power background could be removed, for example by imposing a triangular sweep waveform on the correct number of photomultiplier dynodes. (3) The area under any selected ring or spot profile may be integrated electrically.

**References**

1 Smith and Oatley: Brit. J. Appl. Phys. **6** (1955) 391.
2 Grigson: Dissertation, Cambridge University (1955).
3 Bagdykyants and Alekseev: Bull. Acad. Sci. USSR. Phys. Series **23** (1958) 766.
4 Grigson: Design of 1 kw DC Amplifier, AWRE Report, Aldermaston, England, April (1961).
5 Everhart and Thornley: J. Sci. Inst. **37** (1960) 246.

## DISCUSSION

D. A. SWICK: How do you calibrate the angle of scattering?

C. W. B. GRIGSON: Essentially by knowing the wavelength and using a standard specimen. That the scale came out at S.D.$=0.01$ radian total deflection was fortuitous; but it would be easy to provide preset adjustments to ensure this for any particular wavelength.

F. FUJIMOTO: What kind of scintillator substance did you use?

C. W. B. GRIGSON: Naton 136, a plastic scintillator based on polystyrene and made by Nash and Thomson, Scubitan, England. Pulse height is 60% of that anthracene, decay time is $1.6\times10^{-9}$sec.

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# Design and Characteristics of Parallel Incidence Electron Diffraction Apparatus

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The design and construction of an electron diffraction apparatus is described in which the sample is irradiated with a parallel beam of electrons, and the diffraction pattern is projected past a rotating sector onto a photographic plate by means of two large aperture magnetic lenses. The desirable feature of this design is that the focus condition is independent of sample position. Hence, this apparatus is particularly suitable for the study of low density samples and for recording reflection patterns at low angles of incidence.

It is demonstrated that scanning of the pattern by control of the projection lens current, coupled with electronic recording, permits the possible direct recording of a Fourier transform of the diffraction pattern. An additional advantage of electronic detection is the possibility of discriminating between diffraction due to the ambient gas and that due to a very low density injected sample.

## Introduction

Our objective is to construct an apparatus which is particularly suitable for structure determinations of low-density gaseous samples. We are primarily interested in vapors of metal halides and metal oxides which would effuse from a crucible containing the solid maintained at a high temperature, up to 2000°K. We also wish to extend the application of this apparatus to materials of very low stability, such as $O_2F_2$, which decomposes at ambient temperatures, so that the sample must be maintained at such a low temperature that the vapor pressure available is quite low. We were led to a set of specifications which includes special features as