AN ELECTRON ENERGY ANALYSER WITH A RETARDING POTENTIAL FILTER AND A NEW DIFFERENTIATING SYSTEM

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The design and construction of a simple, high resolution energy analyser with a new differentiating system is described in detail. An einzel lens developed by Brack was used for producing retarding potential. A small beam current of 10^{-15} A was measured by the use of a scintillator and a photomultiplier. In order to differentiate energy distribution curves and to improve the signal to noise ratio an A. C. modulation circuit with a phase-sensitive detector was used. The energy profile of a 26 keV electron beam and the characteristic energy loss spectrum of aluminium were measured to examine the characteristics of the analyser. The energy resolution obtained at 26 keV was 0.5 eV. This resolution was limited not by the resolving power of the electron filter but by the primary beam energy spread.

1. Introduction

Since the characteristic energy losses of electrons in metals was interpreted in terms of the collective oscillation of electrons by Bohm and Pines[1)2)] in 1952, many attempts to find the energy loss spectra of various metals have been carried out by the use of high resolution energy analysers[3)]. The investigations were mainly carried out by the Möllenstedt type analyser[4)-6)]. In this analyser, electron energies are analysed by deflecting electrons with an electrostatic cylindrical lens of very strong chromatic aberration. The resolution of this type analyser is usually about 1 eV for the accelerating voltage 20-50 kV. Dietrich raised the resolution to 0.25 eV for 35 kV by the use of a barium oxide cathode[7)]. Leonhard[8)] and Watanabe[9)] improved the analyser to record the variation of energy loss with the scattering angle on a photographic plate in a short time. This is a great merit of Möllenstedt type analyser. However, the construction of this analyser is rather difficult, because high accuracy is required for the mechanical construction of the filter lens.

As the electron energy analyser of a second type there is the retarding field analyser[10)-14)]. In this analyser, the high negative voltage is applied to the Faraday cage to collect the electrons with energies greater than the potential of the collector. This causes a disadvantage that the detector is also at the high voltage. This disadvantage is removed in the retarding field analyser of another type[15)-20)]. This has an electrostatic einzel lens, which consists of three concentric apertures, with the outer two electrodes connected to earth potential and the middle to high negative voltage. The detector is then at earth potential. In these retarding

field analysers, the variations of the high voltage have little effect on the measurement of energy losses, because the retarding and cathode potentials are supplied by the same high voltage supply. This is a great merit of this type analyser. The retarding potential measurement, however, requires for about 5-30 minutes. Therefore, it is very important to keep the beam current constant for a long time.

For the purpose of studying the characteristic energy losses of electrons by this type analyser, it is convenient to differentiate the energy distribution curve which is recorded on the output recorder, because the curve represents the integral distribution of electron energy losses. A method for obtaining differential spectra electrically was initiated by Leder and Simpson[21]. The principle is as follows. If a small alternating voltage of frequency f_m is superimposed on the retarding voltage, the beam current passing through the filter is modulated with the frequency f_m. A component of the beam current with the frequency f_m is proportional to the slope of the integral curve as illustrated in Fig. 1. Therefore, if only the f_m component of the current is amplified and recorded, the differential spectra is obtained.

As the electron energy analyser of a third type there is the deflection analyser[10)22)23)], which has the homogeneous magnetic field or the electrostatic field. The resolving power of this type analyser is generally poor, but Fert *et al.* has developed a magnetic deflection analyser of relatively high resolution (1/20000)[24]. A research group in Western Australia has obtained useful results in investigations of characteristic energy losses at low primary beam voltages 700-2000 eV by the electrostatic deflection analyser[25)-28)]. They investigated very clean surfaces of various metals and compounds by evaporating them inside the analyser. The electrons emitted from the cathode were scattered by the evaporated layers through 90° and detected. This caused a disadvantage that the variation of energy loss with the scattering angle could not be measured. A few investigators devised to combine the deflection analyser with the retarding field analyser to obtain high resolution[29)-32)]. In these analysers, however, either the specimen or the beam current detector was at high voltages.

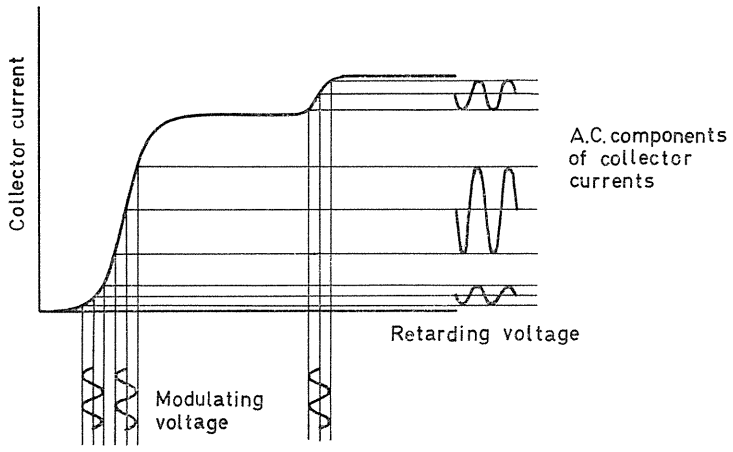

FIG. 1. A schematic illustration of the electrical differentiation.

TABLE 1. Typical analysers

		Resolution (eV)	Primary energy (keV)	
Ruthemann	1941～48	～1	2～8	Magnetic deflection
Möllenstedt	1949	～1	35	Chromatic lens
Watanabe	1954	1	22	Chromatic lens
Blackstock *et al.*	1955	2.9	100	Electrostatic deflection
Haberstroh	1956	～2	20～50	Retarding field
Fert *et al.*	1957	～1.5	30	Magnetic deflection
Dietrich	1958	0.25	35	Chromatic lens
Powell *et al.*	1958	1～2	0.7～2	Electrostatic deflection
Leder and Simpson	1958	0.6	15	Retarding field and A. C. modulation method
Horstmann and Meyer	1960	0.5	15～50	Retarding field
Kunz	1962	0.5	10～50	Retarding field and electrostatic deflection
Brack	1962	～0.5	40	Retarding field filter lens
Boersch and Miessner	1962	0.3	20～50	Retarding field filter lens
Klemperer and Shepherd	1963	2.2	10	Magnetic deflection
Boersch *et al.*	1964	0.017	20～50	Wien filter and a monochromator
Hartl	1964	0.012	30～50	Electrostatic deflection and a monochromator
Ichinokawa	1965	0.5	80	Cylindrical magnetic lens
Denbigh and Grigson	1965	1.3	40	Retarding field filter lens and A. C. modulation method

Recently, Ichinokawa has developed a new analyser with two cylindrical magnetic lenses[33]. It has high resolution of 0.5 eV at 80 kV and can be used at higher voltages. A disadvantage of this analyser is that it is difficult to stabilize the accelerating voltage and lens currents for 10^{-6} order.

In the high resolution analyser, the energy filter has high resolving power, but the overall resolution of the apparatus is limited by energy spread of the primary electron beam. In order to minimize the energy spread, monochromators have been developed[34)-39)]. An electron beam of about 0.05 eV half-width is obtained by the use of monochromator. Typical analysers ever developed are listed in Table 1.

In our laboratory, an analyser of simple mechanism and of high resolving power was constructed in order to study the characteristic energy losses of electrons in solids. It has a retarding field filter lens as an electron filter and has a new A. C. modulation circuit with a phase-sensitive detector*. It was designed to have simple mechanism, to be constructed inexpensively and to be easy to handle. The design and construction of the analyser is described and some examples of the energy analysis are given in this paper.

* Recently, Denbigh and Grigson[40)] and Bradbury[41)] have constructed the retarding field analyser with an einzel lens and an A. C. modulation system for the study of electron diffraction.

2. Apparatus

A schematic diagram of the apparatus is shown in Fig. 2. The analyser consists of an illuminating system, a specimen chamber, beam deflectors, an energy filter and an electron detecting system.

A fine electron beam was produced by a gun and aperture stops. The electron gun was telefocal type with double Wehnelt cylinders. Self-biasing voltages were applied to the cylinders. Electrons from a hair-pin tungsten cathode are accelerated through an anode and pass through apertures S_1 and S_2. The angular aperture of the primary beam was reduced to 2.5×10^{-4} radians by collimation through two apertures of about 50 μ diameter which were separated by a drift space 200 mm. The accelerating voltage of 20-30 kV was supplied by a transformer-rectifier circuit for an ordinary electron microscope and stabilized by a voltage stabilizer of a magnetic amplifier type. Its stability was 1×10^{-3}.

The fine electron beam bombards a specimen held at earth potential. A specimen holder could be moved along the axis perpendicular to the electron optical axis and rotated around the axis. The specimen chamber had an air lock.

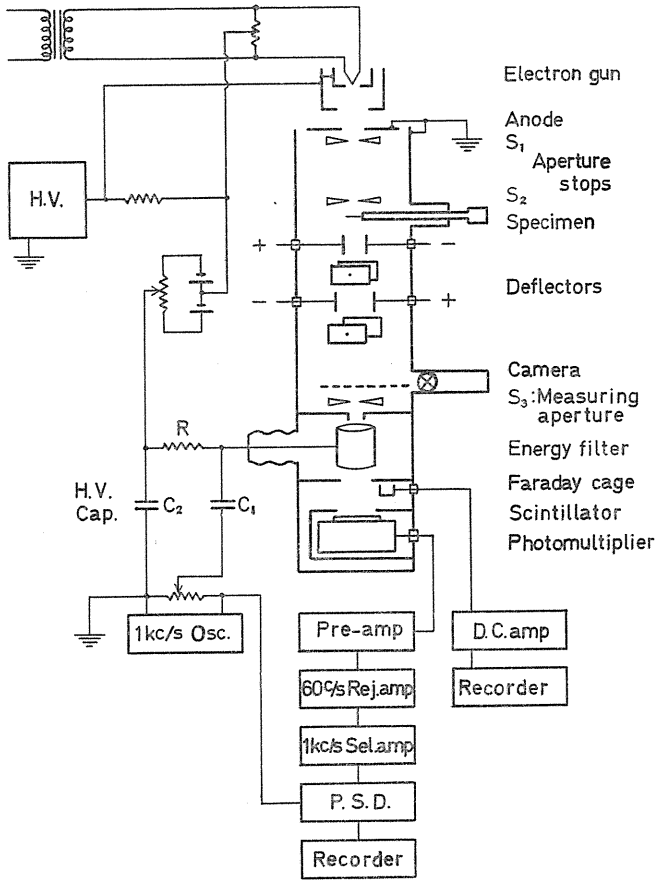

FIG. 2. A schematic diagram of the apparatus.

In order to analyse electrons scattered at any angles, two mutually perpendicular pairs of double condenser plates were arranged behind the specimen chamber. They were of the same type as Boersch and Miessner used[20]. Electrons scattered through any preset angles could be deflected and re-aligned with the electron optical axis by the deflectors. Deflection angles up to 0.1 radians could be measured by changing the deflector voltage. The diffraction pattern was observed on a fluorescent screen and recorded on a photographic plate, which could be inserted into the electron path in the front of the energy filter. Distance between the specimen and the plate was 266 mm. The camera box also had an air lock.

The measuring aperture S_3 selected an angular range of 1.2×10^{-4} radians only. A pencil of 50 μ diameter entered the energy filter. A retarding field filter lens of Brack type[19] was used as the energy filter. This lens has a saddle field that is flattened near the centre as much as possible. Only electrons with energies greater than the electrostatic potential of the saddle field can pass through the filter lens. They are reaccelerated to earth potential. The retarding potential was varied from -3 volts to $+80$ volts by the use of dry batteries and a continuous rotation potentiometer driven by a 2 rpm direct-current motor.

The electrons passing through the filter were detected by a scintillator and a photomultiplier which were placed in vacuum. The detector was on earth potential. A plastic scintillator film, 0.05 mm thick, was stuck on a window of a photomultiplier tube with silicone grease. The wave lengths of photons emitted from the scintillator lie from 4000 Å to 4500 Å. The photomultiplier tube of MS-9 S type, which was compact and had large sensitivity around 4000 Å, was set in an iron box which had a window. The stability of the high voltage for the photomultiplier was less than 5×10^{-5} and the stability of multiplication was 3×10^{-4}. A movable Faraday collector, which was connected with a dynamic condenser electrometer, could be inserted between the filter lens and the detector for the purpose of checking the multiplication of the detector.

An A. C. modulation circuit with a phase-sensitive detector was used in order to differentiate the energy loss curve. The available modulation frequency is limited by following conditions. The first condition is that the filtering voltage should be kept nearly constant until an electron emitted from the cathode has reached the electron filter. It requires that the modulation frequency is far smaller than 770 kc/s. The second condition is that electrons enough to give the information of the energy loss must be attained to the collector within a period that is much shorter than the period of the modulation frequency. It requires that the modulation frequency is lower than 10 kc/s. The third condition is that the modulation frequency must be as different as possible from the line frequency because the line field generates the greatest noise. Considering these conditions, 1 kc/s was used as the modulation frequency. The sinusoidal modulation signal, which was generated by a Wien bridge oscillator, was applied to the middle electrode of the filter lens through the high voltage capacitors C_1 and C_2. For the purpose of selecting the modulated component of the signal current the photomultiplier output was connected to a 1 kc/s tuned amplifier[42] and a phase-sensitive detector[43] after 60 cycle noises were rejected. The amplified signal was recorded by a chart recorder. These circuits had the maximum gain of 3×10^5.

The vacuum system consisted of an oil diffusion pump backed up by a rotary

pump and another rotary pump for forepumping. A liquid nitrogen trap was used. The vacuum pressure was normally about 5×10^{-6} mmHg.

3. Characteristics of the Analyser

1) Energy resolution

In order to measure the resolving power of the analyser the primary electron beam was analysed. The diameter of the beam entering the filter was varied by exchanging the measuring aperture. The observed integral distributions of the primary beam energies are shown in Fig. 3. This figure shows that the resolution increases with decreasing the measuring aperture. It is due to the fact that the saddle field is flat and homogeneous for a fine beam along the axis but is

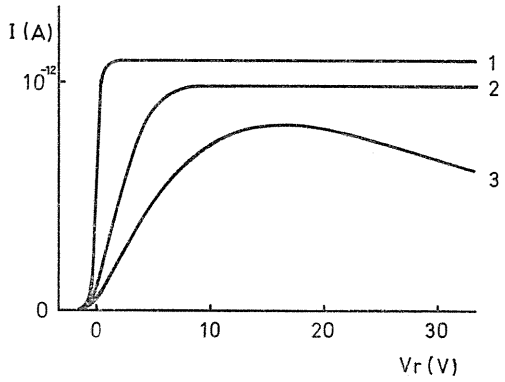

FIG. 3. Integral energy distribution curves, which were recorded using the different measuring apertures, of 30 keV electron beam. Diameters of the measuring apertures: 1 50 $\mu\phi$, 2 100 $\mu\phi$, 3 300 $\mu\phi$.

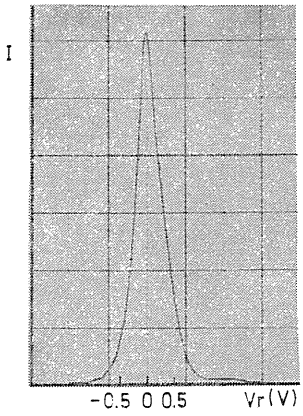

FIG. 4. An energy distribution curve of a 26 keV electron beam.

not so for a wide beam. In our analyser the measuring aperture of 50 μ diameter that gave the highest resolution was used. An example of the energy distribution curve of a 26 keV electron beam is shown in Fig. 4. This curve was obtained by the A. C. modulation method.

The energy distribution of primary electron beam is approximate Maxwellian. Some definitions of the energy bandwidth have been proposed by various authors[10]. For instance, Boersch defined the energy bandwidth of the integral Maxwellian distribution as the smallest of the energy interval which contained 63% of the primary beam current[11]. The bandwidth $\Delta E_{63\%}$ is given by

$$\Delta E_{63\%} = 2kT, \tag{1}$$

where k is the Boltzmann constant and T the absolute temperature of the cathode. The integral width ΔE_i[13)35)] of the energy distribution is defined by

$$\Delta E_i \cdot \mathrm{Max}[f(E)] = \int_0^\infty f(E)dE,$$

where $f(E)$ is the differential energy distribution function. Using the usual form of $f(E)$, we get

$$\Delta E_i = \mathrm{e}\, kT. \tag{2}$$

The half-width $\Delta E_{1/2}$ of the energy distribution is given by

$$\Delta E_{1/2} = 2.45\, kT. \tag{3}$$

From fhe distribution curve (Fig. 4), the bandwidths were measured to be $\Delta E_{63\%}=0.45\pm0.02$ eV, $\Delta E_i=0.61\pm0.02$ eV and $\Delta E_{1/2}=0.51\pm0.02$ eV. For the same temperature of the cathode (2400°K) the formulae (1), (2) and (3) give $\Delta E_{63\%}=$ 0.42 eV, $\Delta E_i=0.56$ eV and $\Delta E_{1/2}=0.51$ eV, respectively. The differences between the calculated bandwidth and the measured are less than 0.05 eV. This means that the resolving power of the filter itself is less than 0.05 eV.

In order to confirm this result, the integral energy distribution of the primary beam at lower cathode temperature (1700°K) was investigated by the ordinary retarding potential measurement. The integral curve obtained is shown in Fig. 5. From the curve $\Delta E_{63\%}=0.29\pm0.02$ eV, $\Delta E_i=0.36\pm0.02$ eV and $\Delta E_{1/2}=0.31\pm0.02$ eV were measured, and $\Delta E_{63\%}=0.29$ eV, $\Delta E_i=0.40$ eV and $\Delta E_{1/2}=0.36$ eV are calculated for $T=1700$°K from the formulae. The differences between the calculated bandwidth and the measured are also less than 0.05 eV*. These results mean that the resolving power of the filter itself is less than 0.05 eV. The energy resolution of the analyser, however, was about 0.5 eV owing to the energy spread of the primary electron beam.

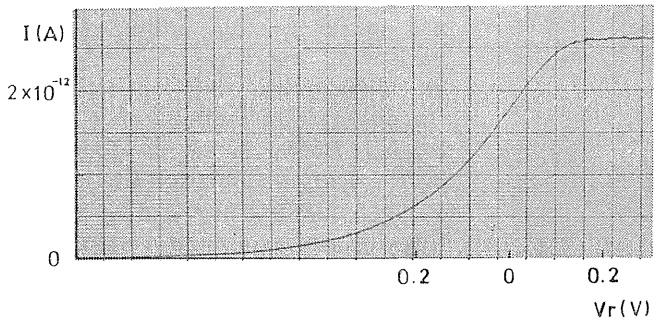

FIG. 5. A recording of the integral energy distribution curve of a 30 keV electron beam.

* Boersch had reported that the energy distribution in an electron beam was broadened with increasing current density[11]. The broadening was not significant in the present experiment because the current density was very small (1×10^{-9} A/mm^2).

2) Measurement of small beam current

It is desirable to make the beam current as small as possible, to avoid both changes in crystal structure of the specimen and deposition of impurities on the specimen surface due to the electron bombardment. The beam current, however, must not be so small as the fluctuation in the dark current of the photomultiplier exceeds the signal current. The photomultiplier used in the present experiment had a dark current of about 2×10^{-9} A and the fluctuation of about 5×10^{-10} A at 1000 V. This gives that the lower limit of the current is about 5×10^{-16} A without cooling the photomultiplier.

The fluctuation of the primary beam current was 2%. In general, the fluctuation of the beam current is caused by variations in the accelerating voltage, the heating current of the filament, the bias voltage and the pressure, the contamination of apertures and so on. Since a self-biased gun was used in the present experment, the variation in the primary beam current was less than about 10% of variations in the accelerating voltage and the heating current of the filament. Fluctuations of the high voltage and the heater current were less than 1×10^{-3} and 1×10^{-2}, respectively. Therefore, the instability of the beam current in this analyser was not due to these variations. It was supposed that the fluctuation of the primary beam current was caused mainly by the stray field. Reducing further the fluctuation of the beam current is very difficult. However, the noise in the measurement of the beam current can be reduced by using the A. C. modulation circuit.

3) Angular resolution

The angular resolution was 2.5×10^{-4} radians. It was the maximum divergence angle of the primary beam which was limited by the slit system.

Fluctuation of the high voltage had no effect on the angular resolution. The reason is as follows. The electron wave length is inversely preportional to the square root of the accelerating voltage V and the scattering angle θ proportional to the wave length, so that the variation of the scattering angle is given by

$$\left|\frac{\Delta\theta}{\theta}\right| = \frac{1}{2}\left|\frac{\Delta V}{V}\right| < 5\times10^{-4}.$$

Even for the maximum scattering angle that can be measured by the present apparatus (0.1 radians)

$$|\Delta\theta| < 5\times10^{-5} \text{ radians.}$$

This is less than the maximum divergence angle of the beam.

4. Energy Loss Spectrum of Aluminium

The energy losses of electrons transmitted through thin aluminium foils (200-1000 Å) were measured for the purpose of comparing with other experimental results. An example of the spectra at small angle scattering ($<2.5\times10^{-4}$ radians) is shown in Fig. 6. The primary electrons of 26 keV were used. The specimen was prepared by vacuum evaporation. The first peak in the figure corresponds to the zero-loss peak. The second peak and the third peak are at 14.9 ± 0.1 eV and 29.8 ± 0.1 eV, respectively. These peaks are due to the plasma oscillation.

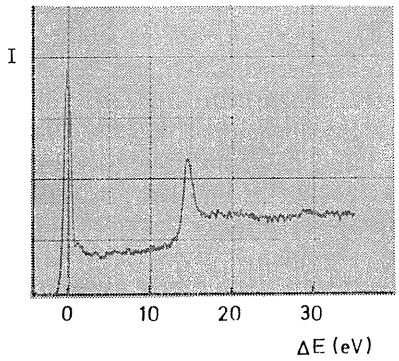

FIG. 6. An energy spectrum of 26 keV electrons in transmission through an aluminium foil.

The theoretical value of the plasma loss in aluminium is 15.8 eV and the best values obtained experimentally by various authors lie between 14.7 and 15.5 eV. The value obtained by the present experiment agrees well with these values.

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References

1) D. Pines and D. Bohm: Phys. Rev., **85** (1952), 338.
2) D. Bohm and D. Pines: Phys. Rev., **92** (1953), 609.
3) O. Klemperer and J. P. G. Shepherd: Advances in Phys., **12** (1963), 335.
4) G. Möllenstedt: Optik, **5** (1949), 499.
5) H. Watanabe: J. Phys. Soc. Japan, **9** (1954), 920.
6) L. Marton and L. B. Leder: Phys. Rev., **94** (1954), 203.
7) W. Dietrich: Z. Phys., **151** (1958), 519.
8) F. Leonhard: Z. Naturforsch., **9** (1954), 727.
9) H. Watanabe: J. Phys. Soc. Japan, **11** (1956), 112.
10) O. Klemperer: Reports on Progress in Physics, **28** (1965), 77.
11) H. Boersch: Z. Phys., **139** (1954), 115.
12) G. Haberstroh: Z. Phys., **145** (1956), 20.
13) M. Horstmann and G. Meyer: Z. Phys., **159** (1960), 563.
14) S. Kuwabara: J. Sci. Hiroshima Univ. **A-II 25** (1962), 407.
15) H. Boersch: Z. Phys., **134** (1953), 156.
16) G. Forst: Z. angew. Phys., **10** (1958), 546.
17) K. Keck and H. Deichsel: Optik, **17** (1960), 401.
18) J. A. Simpson and L. Marton: Rev. Sci. Instrum., **32** (1961), 802.
19) K. Brack: Z. Naturforsch. **17 a** (1962), 1066.
20) H. Boersch and H. Miessner: Z. Phys., **168** (1962), 298.
21) L. B. Leder and J. A. Simpson: Rev. Sci. Instrum., **29** (1958), 571.
22) E. Rudberg: Proc. Roy. Soc., **A 127** (1930), 111.
23) G. Ruthemann: Ann. Physik, **2** (1948), 113.
24) C. Fert and F. Pradal: CR Acad. Sci., **244** (1957), 54.

25) C. J. Powell, J. L. Robins and J. B. Swan: Phys. Rev., **110** (1958), 657.
26) C. J. Powell: J. nuclear Energy, **C 2** (1961), 57.
27) J. L. Robins: Proc. Phys. Soc., **79** (1962), 119.
28) P. E. Best: Proc. Phys. Soc., **80** (1962), 1308.
29) A. W. Blackstock, R. D. Birkhoff and M. Slater: Rev. Sci. Instrum., **26** (1955), 274.
30) C. Kunz: Z. Phys., **167** (1962), 53.
31) J. Lohff: Z. Phys., **171** (1963), 442.
32) O. Klemperer and J. P. G. Shepherd: Brit. J. Appl. Phys., **14** (1963), 85.
33) T. Ichinokawa: *Int. Conf. Electron Diffraction and Cryst. Defects*, Melbourne (1965).
34) H. Boersch, J. Geiger and H. Hellwig: Phys. Letters, **3** (1962), 64.
35) W. Legler: Z. Phys., **171** (1963), 424.
36) H. Boersch, J. Geiger and W. Stickel: Z. Phys., **180** (1964), 415.
37) W. A. M. Hartl: Phys. Letters, **13** (1964), 133.
38) W. A. M. Hartl: Z. Phys., **191** (1966), 487.
39) K. H. Gaukler: Z. Phys., **196** (1966), 85.
40) P. N. Denbigh and C. Grigson: J. Sci. Instrum., **42** (1965), 305.
41) G. R. Bradbury: *Int. Conf. Electron Diffraction and Cryst. Defects*, Melbourne (1965).
42) G. Valley and H. Wallman: *Vacuum Tube Amplifiers* (McGraw-Hill 1948), p. 398.
43) N. A. Schuster: Rev. Sci. Instrum., **22** (1951), 254.