Construction of 500 kV Electron Microscope

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A 500kV electron microscope was newly built in 1963 by close cooperation of Kyoto University and Shimadzu Seisakusho Co., as a development of the previous 300kV one made by them.

The high voltage generator is a pressure Cockcroft-Walton system driven with 720 cps input, and its output (variable up to 500kV) is supplied through an insulating cable to the 10 stage accelerator in a pressure tank on the microscope column. A double loop feed back system is employed and the output fluctuation is kept within as small as $\pm 5\times 10^{-6}$ per minute through continuous operations in the range of 100-500kV.

Such a high stability combined with the shorter wave length of electrons qualifies that the attainable resolving power of this electron microscope has to be higher than that of the conventional one.

The practical resolution so far obtained with thin crystalline specimen is 10Å. Although this is even the best value ever reported for the high voltage electron microscopy, it is expected that this instrument would be capable of revealing atomic arrays by the central illumination method.

1. INTRODUCTION

The reason why the present authors built the 300 kV electron microscope in 1957 was not only to construct a practical high voltage microscope for applied studies but to explore the possibilities which would be realized by elevating the accelerating voltage. The results of some investigations made with this instrument were reported previously[1].

Through these investigations, efforts were directed to inform quantitatively the effects of electrons upon various specimens when the accelerating voltage was varied. This was the first time in this field that such experimental data were made available. In one of these studies we proved quantitatively that the damage of the specimen due to the inelastic collision of electron was reduced with the increase of accelerating voltage. In the case of polymer crystals, the dose of electron, necessary to decompose the regular arrangement of molecules, was nearly linearly proportional to the accelerating voltage up to 300 kV[2]. This means that the damage at the same dose of incident electron becomes less as the electron energy is increased. Accordingly we can conclude that the inelastic cross section is the dominant factor concerning this effect.

Another important merit of the high voltage electron microscopy is the high transmission for the specimens with large mass thicknesses. On this subject there have been many theoretical predictions, but very few experimental data have been available about the controversial "transmissive power" of electrons so far as the

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electron microscopy was concerned[3]. The terms such as "apparent absorption" or "effective absorption", for example, make it obscure how to measure the transparency. Some quantitative measurements of transmissive power were carried out by us with the 300 kV electron microscope[1,4], but the results were not so consistent with the relation mentioned above[2], except for the effective absorption coefficient measured at the region of "anomalous transmission" where the inelastic cross section plays the main role. Apparent absorption controlling the transparency of specimen, includes both elastic and inelastic scatterings, which cause the decrease in number of electrons by the deflection from the image forming beam, and it also includes the loss of energy by inelastic collision, i.e. the "true absorption". Besides there remains a very important factor, i.e. the resolution or definition, though this is seldom considered as a measure of transparency. It would be advisable here to mention followings: an opaque glass can transmit light waves, however no one calls it transparent.

Besides this complexity, another controversial argument on the effect of high voltage electrons has arisen from the relativistic point of view[5]. It is evident that the relativistic nature of high velocity electrons is supposed to behave as the limiting barrier against not only the increase of the transmissive power but also the decrease of the damage upon the specimen. Concerning this point, no quantitative measurement has been made. The 300 kV electron microscope is not sufficient to slove this problem, since the relativististic effect is so slight in this region of electron velocity that the extrapolation of the measured value is to lead erroneous estimation.

Those are the main reasons why the present authors struggle again with the construction of high voltage electron microscope: first, to clarify the effect of increased accelerating voltage discussed above, and secondly, to construct a more useful microscope for the applied studies of electron-sensitive organic substances as well as electron-dense materials. At present there are two electron microscopes with operating voltage over 500 kV which were constructed after our 300 kV electron microscope. The one is the famous Toulouse 1 MeV microscope built by Du Pouy[6] and the other is the 600 kV instrument made by Popov in Russia[7]. They claimed that some living bacteria survived under the bombardments of high energy electrons[8,9], however they offered no data of quantitative measurements concerning, for example, the reduction rate of inelastic cross section. This is the first reason why the present authors needed for a higher voltage microscope than 300 kV. As to the second reason which is interconnected with the first one, considerations must be extended to the efficiency of the instrument. Although, in principle, 1 MeV or more is favorable for the analysis of the effect of varied electron velocities, an almost similar effect would be also attainable at several hundred kilovolts, since the increment of the effects becomes less as the velocity of electron approaches to that of light (β is 0.86 for 500 kV and 0.94 for 1000 kV respectively). As for the organic specimens, the theoretical estimation predicts that the inelastic cross section of electrons will approximate its minimum at 500 kV. The efficiency was evaluated also taking into acount the expences and toil as well as the space for housing. And finally the 500 kV was chosen as the maximum operating voltage for the new microscope.

Although the set up of this instrument in Shimadzu's factory was accomplished as early as 1963[10], it had many imperfections especially in the accelerating column. Eliminating these weak points by trial, a long test run has been continued up to date. The performance has so far reached a satisfactory stage, although a further great improvement is still expected. The following description is limited to refer to the main features of the new 500 kV microscope and the present status of progress at the end of 1964. Since the features differing from those of the 300 kV microscope are concentrated into the high tension part, the illustrations of the main column and evacuating system will be made only briefly.

The results of the applied studies obtained with this microscope will appear in separate papers in near future.

2. 500KV GENERATOR

The three step-up transformers system of cascade type was proved to be reliable and convenient for the 300 kV microscope[11]. It showed the utmost advantages for the low frequency of the current driven by the high voltage generator. For the first plan of the new 500 kV microscope, a five stage cascade type was considered. However this plan was discarded because of the difficulty of stabilization and the large space for

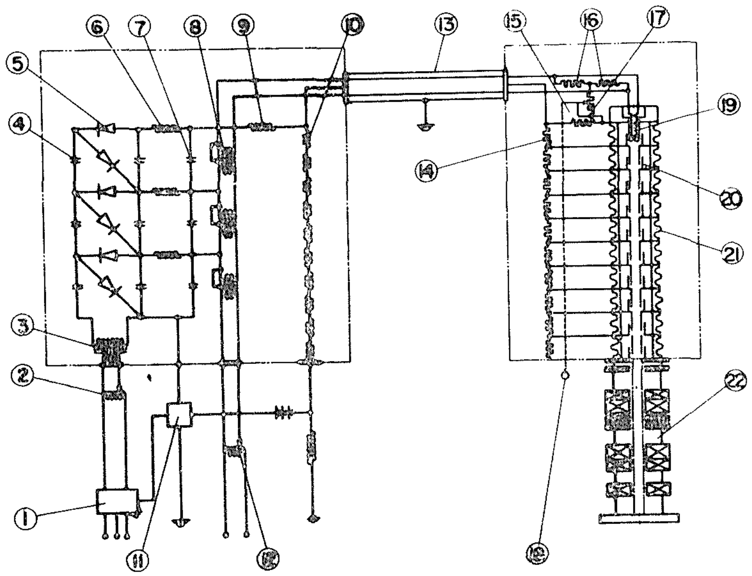

Fig. 1. Simplified diagram of 500 kV generator.
①: Frequency convertor, ②: High voltage controller (motor-driven auto-transformer), ③: Step-up transformer, ④: Capacitors, ⑤: Rectifiers, ⑥: Smoothing resistors, ⑦: Smoothing condensers, ⑧: Insulated transformer for filament power supply, ⑨: Break down resistor, ⑩: Potentiometer for detecting fluctuation, ⑪: Feed back regulator, ⑫: Filament power supply, ⑬: High voltage feeding cable, ⑭: Series resistor stack, ⑮: Break down resistor, ⑯: Balancing resistors, ⑰: Bias resistors, ⑱: Bias controller, ⑲: Electron gun, ⑳: Accelerating electrodes, ㉑: Insulators for accelerating electrodes, ㉒: Microscope column.

installation. Then a Cockcroft-Walton system was employed as the 500 kV generator and the principle to separate the generator and accelerator to be isolated from each other was succeeded from the experience of previous 300 kV[11] electron microcope."

The whole circuit of the new 500 kV high voltage generator is illustrated schematically in Fig. 1. Its main parts consist of a frequency convertor, a voltage multiplying rectifier system and a feed back system.

The 500 kV Cockcroft-Walton generator consists of 3 stage of double voltage rectifiers, and the input voltage from the main transformer is regulated continuously from 0 up to 84 kV by a motor-driven auto-transformer which controls the voltage supplied to the main transformer. The frequency of the current drived in the voltage multiplying circuit was chosen as 720 cps., since this range of cycles was proved to be most adequate for the selenium solid rectifier used in this circuit. The selenium rectifier is considered to be more reliable than the silicon rectifier which is poor in the self-recovering ability after an accidental break down by overvoltage.

The 720 cps input is supplied from a frequency converter which consists of a induction motor and a dynamo in direct coupling. This convertor is very stable for continuous operation at any range of its output current.

A double loop feed back system is furnished to stabilize the high voltage d.c. output. The fluctuation to be compensated is detected by a voltage divider acting as a potentiometric circuit, and fed back through an electronic and a magnetic regulaters. The electronic one is respondent to the rapid change of the high voltage and feeds back the compensating potential to the main circuit at a point where the time constant is supposed to be negligible small. And the magnetic regulater responds to the slower and greater change of high voltage and regulates the output of the converter. The stabilizer circuit was so designed as to work at several fixed voltages, i.e. 100, 200, 300, 400 and 500 kV.

The main part of this high voltage generator, including the rectifier system and the high resistors for potentiometric circuit and the three cascade transformers for the filament current, is set in a pressure tank filled with Freon gas of 1.6 atm. to prevent the surface leakages and discharges of the parts. The independent installation of high voltage generator from the microscope column benefits for the use of larger spacious tank in which the space among the electric components is wide enough to insert the sufficient shields for the elimination of induction effect from a.c. division. The gas pressure of 1.6 atom is high enough to insulate the whole system because of such large dimensions of its parts. This relatively low gas pressure makes it possible to reduce the thickness of the tank wall and is very convenient for the exhausting and refilling of gas.

As the result of such special precautions as described above, the stability of this high voltage generator realized is surprisingly high. Fig. 2 is an example of charts recording the voltage fluctuation of this generator. The measurement was carried out by detecting the voltage deviation of the output from the potentiomeric circuit. The load applied to the generator for this measurement was 100μA, i.e. the current through the high resistors of the voltage divider attached to the accelerating column,

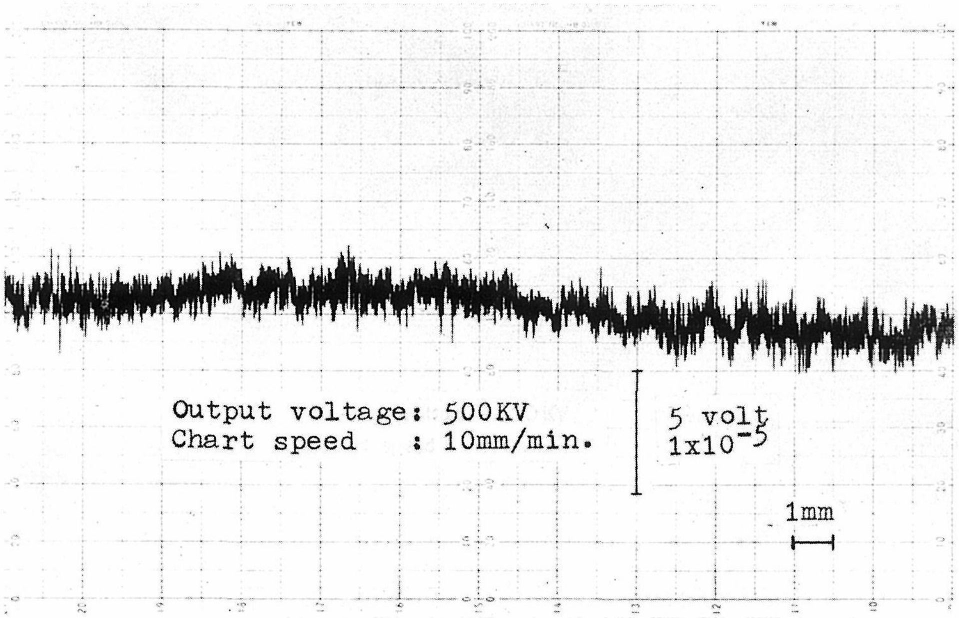

Fig. 2. An example of chart record which shows the stability of the output of high voltage generator.

And the recorder was connected to the detecting terminal with a cathode follower circuit. From many records as shown in Fig. 2, the fluctuation of high voltage was proved to be as small as $\pm 5 \cdot 10^{-6}$/min. at 500 kV. The ripple of high voltage measured separately with a specially deviced detector is also in the same range of magnitude. Similar values were also observed at 100, 200, 300 and 400 kV respectively, except for the slight increase of the ripple at lower voltages than 400 kV.

Such a high stability thus attained is the best ever reported for the generators operated over 200 kV[11,12], and is comparable to that of the best value for the 50-100 kV source for conventional electron microscope[13]. It will be worthwhile to draw attention to the fact that the stabilities of the accelerating voltage is limited also by the fluctuations in the initial velocities of the thermal electrons emitted from the gun and it would be substantially difficult to improve the practical stability of any 100kV microscope to the order less than 10^{-5} (cf. Chap. 10).

3. CABLE AND CABLE HEADS FOR 500KV SUPPLY

The 500 kV potential and power for the filament are supplied to the electron gun and accelerator system through an insulating cable. The cable is covered with synthetic rubber and is 14cm in diameter and 10m in length except two cable heads attached to both ends.* By the preliminary tests, this cable was proved to resist to over voltages up to 600 kV. It stood against the d.c. loading of 700 kV for the short time as well as the impulse loading at the same voltage.

* Specially prepared for this microscope by Dainichi Densen Co. Ltd..

Each end of this cable is molded with synthetic resin in a form of solid rod, and 5 or 10 corona rings made of aluminium are also fixed in the mold with appropriate spacings. By connecting each corona ring to the corresponding stage of high resistors of the potentiometric circuit in the generator tank or those of the voltage dividing circuit in the accelerator tank, the electric field along each cable head is distributed evenly. During the long test run for around one year there has been no trouble which arose from the cable itself or the cable heads. Since the electric circuit requires that the cable heads ought to be erected, the cable was led from the bottom of each pressure tank and the generator tank was raised 2m high by a supporting frame.

Two pressure tanks connected together with this cable make this 500 kV electron microscope perfectly shock-proof and eliminate any discharges from or along the outer surfaces of every high voltage parts. The perfect shock-proof is a very advnatageous feature of the new 500 kV instrument. In addition to this, the isolated generator-accelerator system gives a valuable convenience to the operational procedures for adjustments and repairs of each section. On the way of improvements, the components of the accelerator column has been replaced and reformed frequently. These operations have been carried out easily by opening the accelerator tank only. The occasion to open the generator tank has been quite seldom after the preliminary adjustments.

4. ELECTRON GUN AND ACCELERATOR

In the new circuit the potential for each stage of the accelerator is supplied by a voltage divider consisting of a series of high resistors.

The prototype of the 500 kV accelerator for this microscope was 5 stage tube made of 5 porcelain insulators and 5 sets of electrodes. In the early stage of test run, this was proved to be unreliable for a continuous operation. The electric field per each set of electrodes was so strong that small discharges were apt to occur. When an intense discharge happened to make the corresponding section of the high voltage divider short circuited, the remaining sections of the divider had to be loaded with over voltage which was able to induce the successive discharges between the remaining electrodes.

This tendency is only one demerit of the present high voltage circuit compared with that of the 300 kV one where the potential for each stage of the accelerator is supplied from the independent unit generator. To overcome this weakness, the number of stages for acceleration was increased. After many basic experiments on the growth process of electric discharge, a new accelerator tube was developed and has been improved to satisfy the severe demands for the removal of any detectable discharge which was to influence the stability of final accelerating voltage.

The present gun and accelerator system consist of 10 stages of electrodes and 10 glass insulating cylinders (8cm high in each). The total height up to the corona cap of the gun is 110cm. The cross section of this system is illustrated in the upper part of Fig. 3. The electric circuit combined with this system is shown in the right half of Fig. 1. For the simplicity of illustration, the cable head appears as being unified with the voltage divider in Fig. 3, however actually they are not unified in one column but are connected together. Three columns, i.e. accelerator tube, voltage divider and cable head are installed in a pressure tank placed upon the microscope body, and are located around the center of the tank keeping a necessary distance for insulation with one another and also from the inner surfaces of the tank.

The gas pressure in this tank is similar to that of the generator tank, i.e., 2 kg/cm^2. This relatively low pressure was chosen to make the weight of tank light enough to be mounted on the microscope. If any independent supporting frame were employed, it would introduce a vibration to the gun-accelerator system. Such vibration should be incoherent to the inherent vibration of the microscope body. Fig. 4 is the photograph of the whole system of the new microscope and it shows the large tank mounted directly on the microscope column.

The increase of the gas pressure is not so beneficial in the reduction of dimension of the tank, since the total height of accelerator tube is determined mostly by the surface distance of each glass insulator necessary for the elimination of minute discharges in vacuum. The higher pressure tank is rather suitable for the accelerator (or generator) producing intense electron beam where the minute discharges are permitted because the high stability is not required.

The electrodes in the present accelerating tube are made of stainless steel and are of similar shape which is determined not only on the basis of an electron optical design as a static lens but with the geometrical consideration for the electric field off axis. Since the power of the unit lens composed of such electrodes is too weak to converge very fast electrons at the later stages of acceleration, some modifications are applied to the top electrode facing to the gun. The diameter of the hole of this electrode is made smaller than those of others, and the potential difference applied between the gun and this top electrode is also small than that of following stage by adjusting the voltage divider. By this procedure a satisfactory convergence of electron beam is attained.

No magnetic shields are provided in the accelerator tube, since the deflection of beam by stray magnetic fields is estimated to be very small for the compactness of the construction. Comparing with the gigantic accelerator column of the Toulouse microscope, the height of this 500 kV accelerator is only one seventh.

The metal collars surrounding every electrode, as illustrated in Fig. 3, are provided to produce the even distribution of electric field along the inner surface of each insulator. They are notably effective to prevent the initiation and propagation of the minute discharges which are liable to occur through the very small gaps remaining between the glass insulators and metal plates supporting the electrodes. The discharge of this kind is supposed to be due to Malter effect[14]. All stages of this gun-accelerator column are fastened to each other by sandwiching O-ring vacuum seal. The degasing of the electrode is carried out by baking the parts in a vacuum furnace after machining and by ion bombardment after assembling.

The preliminary adjustment of the optical axis of the electrode system is carried out in the air by loading sections or whole system from 50 kV up to 200 kV. For the fine adjustment which is mainly necessary after filament exchange, cam mechanisms are furnished and they move the electrodes by the vacuum-sealed rods through the thick plates supporting the electrodes. These cam mechanisms are built in at the top of

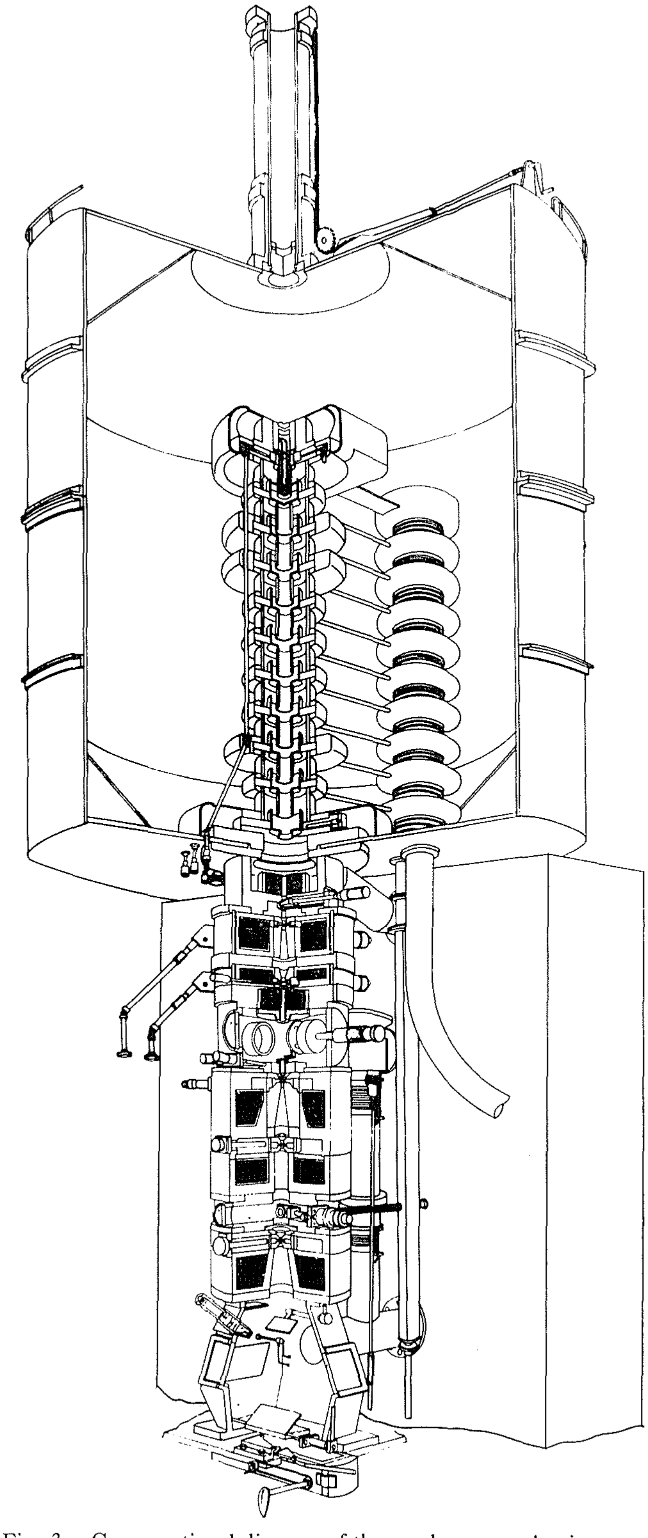

Fig. 3. Cross sectional diagram of the accelerator and microscope column.

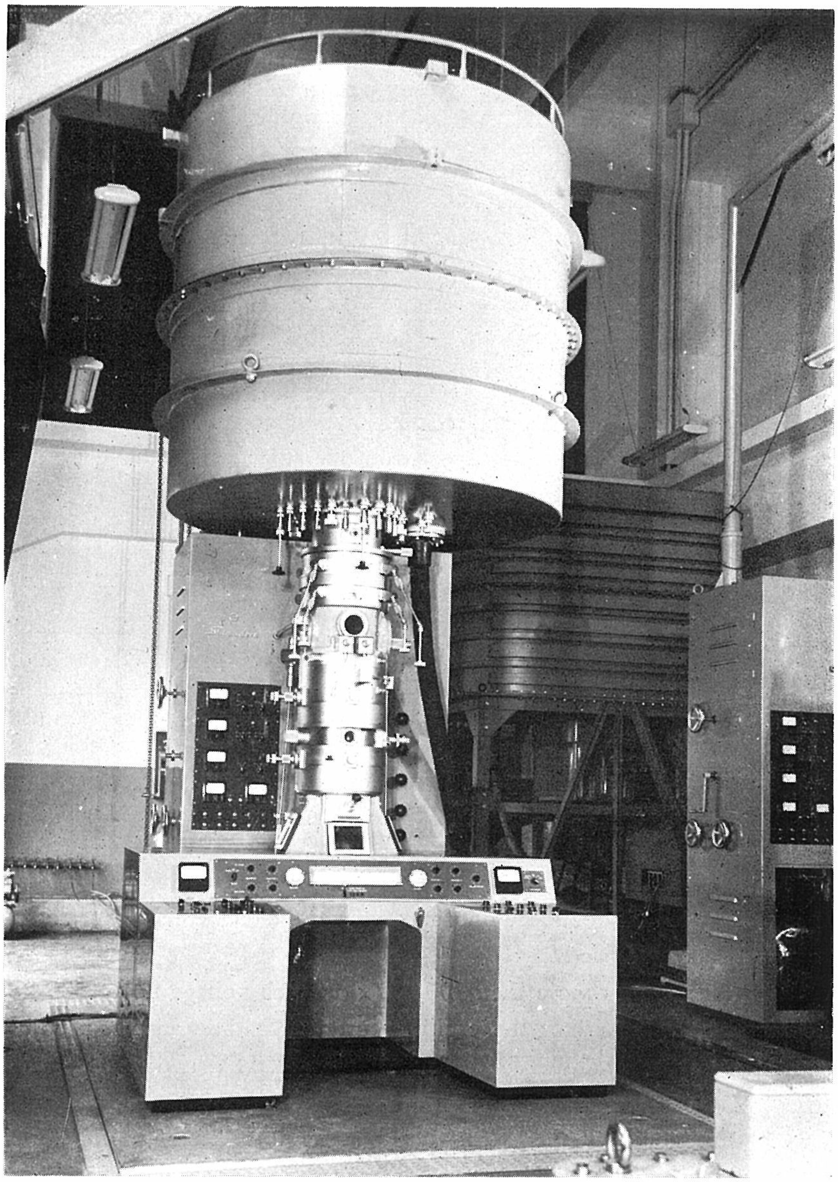

Fig. 4. Photograph of 500 kV electron microscope. The tank which appears in the right background is the high voltage generator and the regulator system installed in the supporting frame.

the gun as well as at the first and second accelerating stage, and controled from the outside of the tank by polymer (polyamide) shafts. Since the velocity of electrons traversing through these three stage is relatively small, their adjustment is effective enough to omit the fine adjustment of the following stages. The accelerator column as a whole also can be moved horizontally with the shafts through the bottom of the pressure tank. The electron gun is auto-biased and the resistance for bias potential can be varied by a long polymer shaft also led through the bottom of the tank. All of these adjusting shafts are surrounding the head of microscope column as shown in Fig. 4.

The filament exchange can be very easily performed by the aid of special device. By lowering the metal tube built in the top panel of the tank and pressing the tube end to the head of the gun, the filament assembly can be taken out without any leak of the gas filled in the tank.

Discharging of the residual high potentials after operation is practised by lifting a motor-driven rod which successively contacts the corona rings of the voltage divider. The sheath and the driving shaft of this groundug rod are illustrated under the voltage divider in Fig. 3.

A new gun-accelerator system is now on the running test. This column is assembled with 3 adjustable stages and 2 units of 5 stages of electrodes hermetically sealed with glass cylinders and it is expected to tolerate the operation at the voltage higher than 500 kV and to bring higher operational stability at 500 kV. Moreover another accelerating tube of 20 stages is now being prepared for the further development of this microscope to a much higher operational voltage.

5. MICROSCOPE COLUMN

The optical system of this microscope is quite similar to that of previous 300 kV microscope[11] except for the scale-up. As shown in Fig. 3 and Fig. 4, the system consists of double condensors, 2 sets of beam deflectors, an objective, an intermediate and a projector lens. The pole pieces of the latter two lenes are adjustable, removable and interchangeable.

As an example of the scale up, the ampere-turns of the objective lens of this microscope is 11,000 while that of 300 kV one is 5,600, and the diameters of the iron shields are 40 cm and 24 cm respectively.

Total magnification is more than $100,000\times$ at each voltage up to 500 kV, and by switching the range selector the exciting current for the projector is adjusted to the same lens power for 100 kV, 200 kV, 300 kV, 400 kV and 500 kV electrons, respectively.

To keep the stability of the exciting currents for these lenses, all of the electric circuits of the transisterized stabiliser, regulator and controller are stored in oil tanks to eliminate the effect of temperature change. By this precaution the drift of objective lens current is reduced as small as $2\cdot10^{-6}$/min.

For the fine adjustment of the optical axis, a wobbler circuit is availed, and by superimposing alternative current up to $\pm 2.5\%$ of the direct current flowing in each lens, the two condensor lenses and 3 imaging lenses can be aligned not only easily but precisely.

Two specimen chambers are provided as shown in Fig. 3. The one is for the ordinary microscopy and the other located between the intermediate and projector lenses is for the high resolution diffraction work.

The decrease of the diffracting angle of electrons at higher accelerating voltages requires longer camera lengths to obtain the diffraction pattern of a reasonable size than that of conventional electron microscope. By proper choice of the distances from lens to lens and lens to screen, the camera length for selected area diffraction at 500 kV is about 80cm in this microscope. Compared with that the length 35cm in the 300kV microscope, 80cm is long enough for the diffraction study even with 500 kV electrons. The camera length for this purpose can be extended, if necessary, to 120cm or more simply by raising the specimen position, that is, making the focal length of objective lens longer than 5mm which corresponds to 80cm camera length. Thus, there is no necessity of auxiliary intermediate or projector lens for this purpose. For extremely large magnification of the image far over 100,000×, an interchangeable pole picec of short focal length is available for the intermediate lens.

A new universal stage of goniometer type has been developed for this microscope. This stage enables the inclining movement of the specimen in any direction besides the conventional horizontal movement. The former two movements are motor-driven and controlled by two foot switches, while the latter (horizontal) movement is controlled manually. By this procedure the observer can tilt his specimen upto 12° in any desired direction. And that is a necessary technique for obtaining the maximum transparency of the point to be observed when the specimen is crystalline. The crystallographic analysis of farily thick specimen becomes possible by using this device with the high voltage electron microscope.

A special heating specimen holder has been developed to be mounted on the universal goniometer stage mentioned above.The conventional heating and cooling stages are also furnished.

6. VIEWING CHAMBER AND CAMERA

Since the lead glass plate of this 500kV microscope is 80 mm in thickness, it is difficult to apply the ordinary magnifying system such as in the 300 kV one.

One auxiliary fluorescent screen is provided in addition to the conventional screen so as to be swung into the position on the halfway from the projector to the conventional screen, as shown in Fig. 3. Thus the image on this auxiliary sceen has to be 4 times as bright as that on the lower one. A telescope with a large aperture objective is installed through the thick metal wall to magnify the image on the auxiliary screen up to 8 times as large as the image on the conventional screen at the bottom of the viewing chamber. The brightness of the magnified image is not less than that of the directly observed image on the lower screen.

The camera chamber is furnished with 24 photographic plates, so that either 24 exposures of 10cm × 7cm or 48 exposures of 5cm × 7cm are available. By means of the air-lock mechanism, these plates after exposure can be taken out without breaking the vacuum of the main column.

7. EVACUATING SYSTEM

The evacuating system of the new 500 kV microscope is quite similar to that of previous 300 kV one[11].

A schematic diagram of the whole pumping system is shown in Fig. 5. A flap-

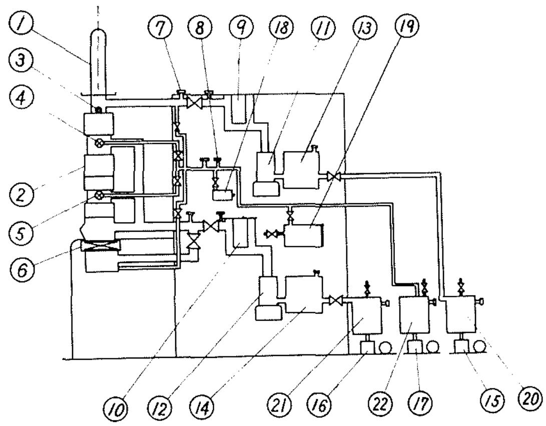

Fig. 5. Schematic diagram of the evacuating system. ①: Electron gun, ②: Microscope column, ③: Isolation vacuum flap-valve, ④: Air lock for specimen exchange, ⑤: Air lock for specimen exchange in case of high resolution diffraction, ⑥: Air lock for photographic plate exchange, ⑦, ⑧: Valves, ⑨, ⑩: Cold traps, ⑪, ⑫: Diffusion pumps, ⑬, ⑭: Vacuum reservers, ⑮, ⑯, ⑰: Boosters, ⑱: Dust filter, ⑲: Pre-evacuater for photographic plates, ⑳, ㉑, ㉒: Mechanical (rotary) pumps.

valve is located at the bottom of the electron gun so that it is separated from the lower half of the column (3 in Fig. 5). Each half has its own pumping system. Isolating the vacuum in the accelerator tube by this flap-valve, the high tension can be kept on all the time, even when the specimen or the photographic plates have to be exchanged. The use of this flap-valve is a characteristic feature of the evacuating system succeeded from the 300 kV microscope in which the convenience of this procedure has been proved.

Liquid nitrogen traps are provided on top of the individual diffusion pumps. Since they hold liquid nitrogen over 12 hours, continuous pumping for over night can be performed.

With these efficient traps, the vacuum in the accelerator tube is maintained as high as $2 \cdot 10^{-6}$ Torr. Such high vacuum is not required for the insulation between electrodes or along glass walls, but for the prevention of the electrode surfaces from the deposit of residual oil vapor which causes the building up of a dielectric membrane. This extremely thin membrane is considered to the main soure of Malter effect by which minute discharges leading occasional break down are initiated[14].

8. X-RAY SHIELDS

In this microscope the anode aperture, expected to be the most intense x-ray emitter, is omitted in the same manner as in the 300 kV microscope. The electrons of several hundreds kV energy produce strong white x-ray radiation far harder than the characteristic one excited by the electrons of lower energy than 100 kV. Accordingly there is little use of the employment of light metals, such as to reduce the hardness of x-ray. However, for the portions exposed to electron beam covers are employed to reduce the intensity of emitting x-ray.

The sections of the microscope column where the thickness of metal wall are too thin to prevent x-rays, i.e. the specimen and viewing chambers, evacuating tubes and the upper part of the first condenser, are covered with lead shields of 30-50mm in thickness. The lead glass plates used for the specimen chamber and the viewing chamber have sufficiently large thicknesses equivalent to 30-50 mm of lead.

Owing to these heavy shields the dose rate of stray x-rays measured at every point outside of the microscope is kept less than 1 mr./hr., even during the direct electron beam is hitting the fluorescent screen for the adjustment of the optical axis of the accelerator. As illustrated in Fig. 3, between the telescope and the auxiliary screen two mirrors are arranged so as to eliminate the direct incidence of x-rays to the ordinary glass lenses of telescope though the x-rays would be very weak when the magnification of image on the screen is large. For these precaution, at the general condition of operation including diffraction work with any procedure, the observer is perfectly protected from the x-rays emitted in the microscope.

9. ROOM FOR INSTALLATION

This microscope is relatively small in size as a 500 kV one, yet it is 488 cm high to the top of the air-lock device on the accelerator tank. Then the height of the room to house this microscope has to be larger than 6 m taking the operational requirement into account. An example of the installation of this microscope is illustrated in Fig. 6. The use of rubber dampers to eliminate mechanical vibrations is more advisable for this microscope which is taller than 300 kV. one.

Since the pressure tanks and the insulating cable are employed it is not necessary to keep the atmosphere of the room to be in low humidity, while the constancy of the room temperature is favorable to reduce the drifts of temperature in the stabilizers, reguraters and controllers.

10. PERFORMANCE OF THE 500KV ELECTRON MICROSCOPE

Obviously, the major merits of the ultra high voltage electron microscopy are attributed not only to the increased transmission of the electron beam through considerably thick specimens but also to the reduction of the irradiation damage to these specimens under heavy electron bombardments. These facts were actually pointed out for the first time by Kobayashi, one of the present authors, on the basis of rigid experimental results obtained with the previous 300 kV electron microscope as already reported in other papers[1,2,4]. However, these two major merits have been so much appreciated ever since that another significant advantage of increased accelerating

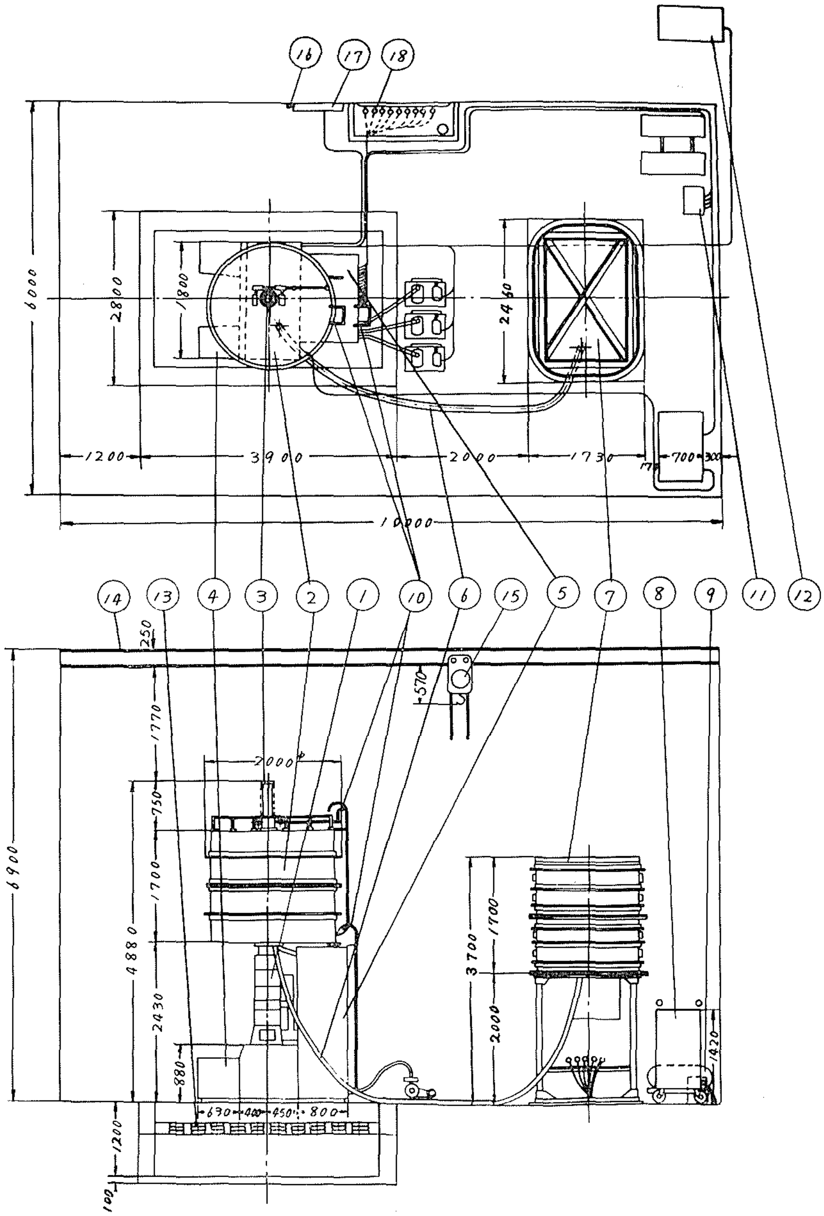

Fig. 6. Layout diagram for the installation of the 500 kV electron microscope. ①: Microscope column, ②: Pressure tank for the accelerator, ③: Air lock for filament exchange, ④: Controller desk, ⑤: Housing for the evacuating system, ⑥: Feeding cable for 500 kV, ⑦: Pressure tank for high voltage generator, ⑧: Stabilizer for lens current, ⑨: Air compressor for pneumatic valves, ⑩: Ladders, ⑪: Power supply for evacuating system, ⑫: Frequency convertor, ⑬: Rubber damper, ⑭: Guide rail, ⑮: Hoist, ⑯: Grounding, ⑰: Main electric power supply, ⑱: Water supply and sink.

voltage, that is the reduction in wave length, is apt to be too much underestimated up to date.

In the leading principle with which several preceeding high voltage electron microscopes were constructed as trial manufactures, rather poor attainable resolution has been taken for granted by the reason as follows: it was supposed that serious chromatic abberation should be caused not only by the instability of the accelerating voltage due to the technical difficulty but also by the multiple inelastic scattering of electrons, particularly for the transmission microscopy of thick specimens, and this defect would not be eliminated to a satisfactory extent.

According to the diffraction theory of image formation of the microscope, the resolution is supposed to be increased simply as the wave length of incident beam is decreased, when the geometrical and chromatic aberrations of imaging lenses are negligible. However, this conclusion cannot really be reached when it is taken into account that the effective diameter of the objective aperture can always be increased large enough to pass the first order reflections to contribute to the image formation. With an extremely thin specimen, it would be plausible that the inelastic scattering very seldom takes place in the case of high energy electrons. When it is assumed that such an extremely thin specimen is composed of a regular lattice structure, the sharpness of the electron image must be governed only by the chromatic aberration due to the fluctuations of electron energy which are *a priori* included in the incident beam. In this case, the effect of the spherical aberration can be negligibly small if focal plane is appropriately shifted from Gaussian focal plane, because these first order reflections are supposed to propagate in a very sharp annular zone. Since it is resaonable to assume that the image is blurred when Δf, the amount of defocusing due to the chromatic aberration, exceeds a quater of the wave length λ expressed in terms of the wave front aberration (Rayleigh limit), the following relationship can be obtained:

$$\Delta f = C_{chr} \frac{\Delta U}{U} \leq \frac{d^2}{\lambda},$$

where C_{chr}, $\Delta U/U$, and d are the coefficient of the chromatic abberation, the fluctuation of the accelerating potential and the spacing of the lattice plane to be resolved in the final electron image, respectively.

With a definite value of C_{chr}, it is easily deduced that even a poor stability is admissible for the resolution of the same order only if the wave length is short enough with increased accelerating voltage. In another words, a better resolution will be obtained with an increased accelerating voltage if the stability is kept constant. Since ΔU can not be reduced to less than 1 eV owing to Maxwell distribution in the initial velocity of the emitted thermal electrons[15], the ultimate resolution will reach 5 Å for 50 kV electron, 3 Å for 100 kV electron and even 0.8 Å for 500 kV electron, where $C_{chr}=$ 0.5 cm and the specimen is illuminated normally by the incident electron beam. As a matter of fact, the practical value of ΔU is much larger than this ideal limit. However, in the case of the present 500 kV electron microscope, the stability of the accelerating voltage turned out to be less than 1 part of 10^{-5} which is the best stability ever attained as far as the high voltage electron microscopes have been concerned. On the basis of the above equation, the practically attainable resolution with the present microscope is estimated to be in an order of 2 Å. In this case, it is not necessary to follow the oblique illumination proposed by Dowell[16] but the normal illumination is sufficient enough for the 2.35 Å spacing of (111) plane of gold crystal to be resolved. It has to be pointed out that the resolution of this order is realized only by the tilted illumination when a conventional microscope is applied[16,17].

With the previous 300 kV electron microscope, it turned out that the 12.5 Å spacing of the (001) plane of Cu-phthalocyanine crystal was scarcely resolved in practice. In the case of the new microscope, however, the image of the similar lattice was easily resolved with the sharpness of much better quality at the maximum accelerating voltage of 500 kV. Moreover, lattice images of 9.8 Å spacing as well as 12.5 Å one in rather thick crystals of Cu-phthalocyanine were clearly resolved as shown in Figs. 7 and 8. On

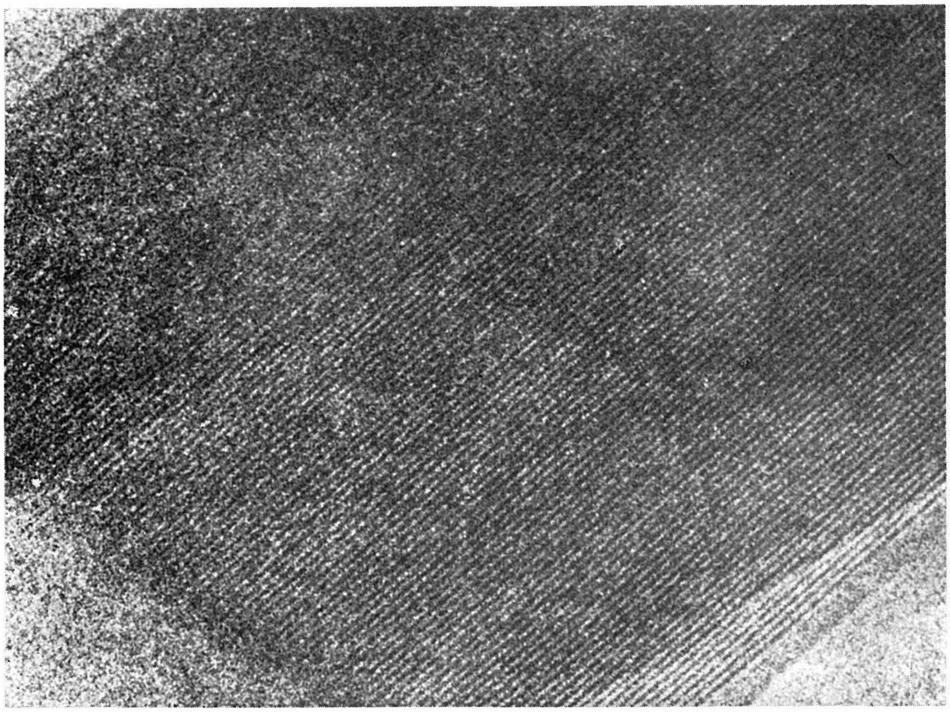

Fig. 7. Lattice image of Cu-phthalocyanine crystal, taken with the 500 kV electron microscope. Accelerating voltage: 500 kV, Lattice spacing: 12.5 Å.

the basis of these results, it has to be emphasized that the resolution of lattice images can be facilitated only by the ultra high voltage electron microscopy with much improved stability of the power supply.

The main obstacle, which impedes this 500 kV microscope from realizing such a high resolution as 2 Å, is the low sensitivity of the available photographic plates to 500 kV electrons. The long exposure makes it difficult to keep the specimen under electron bombardment without any horizontal shift exceeding 1 Å which is large enough

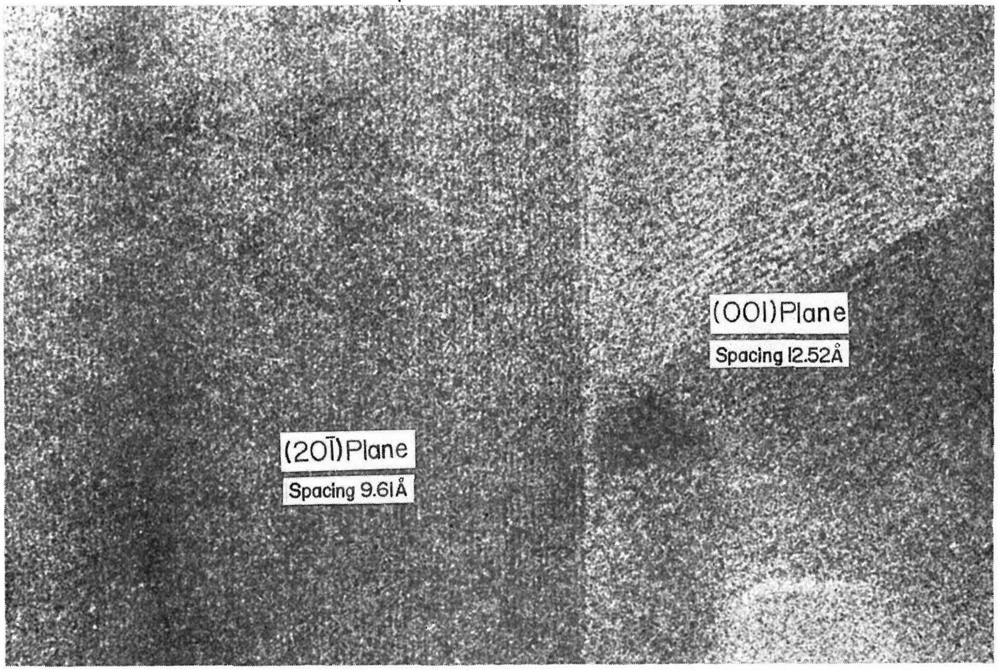

Fig. 8. Lattice image of Cu-phthalocyanine crystal, taken with 500 kV electron microscope. Accelerating voltage: 300 kV, Lattice spacing: 9.6 Å and 12.5 Å.

to blur out the image. At 500 kV some very minute discharges probably due to Malter effect are detected by an oscilloscope[14]. However the stability of the system watched with a sensitive detector does not seem to be affected by this kind of discharge, for the duration of the discharge is of the order of 10^{-5} second which is negligibly short compared with the time constant of the generator.

Concluding this paper it would be worth while to refer to the major field where this 500 kV electron microscope is expected to be the most powerful. Assuming the inelastically scattered electrons are suffered from a mean energy loss of 25eV, that means, when the main component of the specimen is carbon and the thickness of the specimen is rather large as in the order of the "Bremsdicke"[18], the least value of $\Delta U/U$ is estimated to be $5\cdot10^{-5}$ for 500 kV electrons while $2.5\cdot10^{-5}$ for 100 kV electrons. In another words, if the specimen is rather thick, the spacings less than 15 Å are not resolved by 100 kV electron microscope, while the resolution of 500 kV one can reach as high as 4 Å. This has been proved by the photograph shown in Fig. 7, in which the 12.5 Å spacings are clearly resolved by this 500 kV electron microscope even when many layers are superposed. In addition to this effect, it must be emphasized that the "Bremsdicke" itself increases with the elevation of the accelerating voltage.

The high resolution microscopy for thick specimens (0.1-1 μ range) should be the main distinctive feature of this 500 kV electron microscope.

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