March 10, 1970 KARL-JOSEF HANSSEN 3,500,043
METHOD FOR HIGH CONTRAST IMAGING OF PHASE OR AMPLITUDE OBJECTS
IN A CORPUSCULAR RAY DEVICE, SUCH AS AN ELECTRON MICROSCOPE
Filed May 10, 1967 3 Sheets-Sheet 1

Fig.1

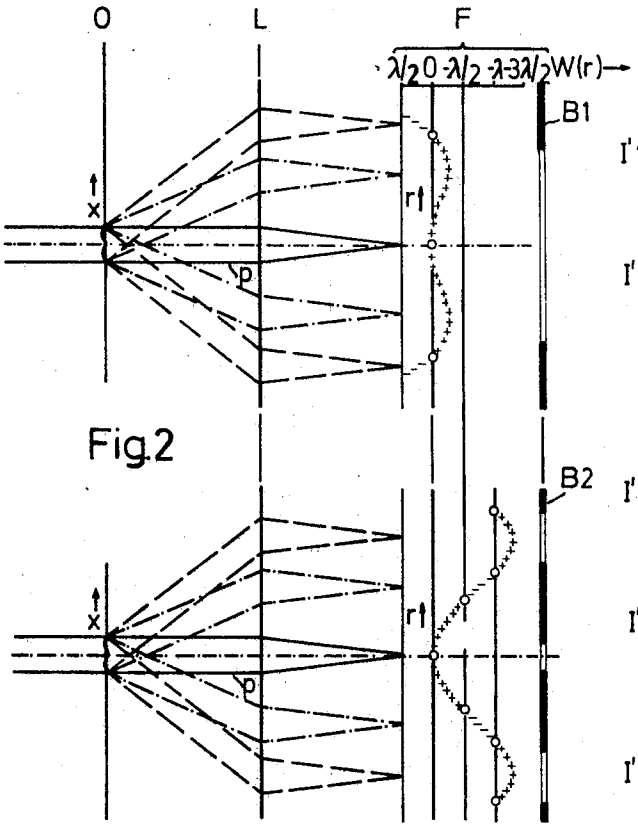


Fig.2

Fig.4

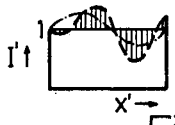


Fig.5

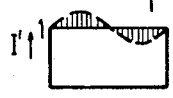


Fig.8

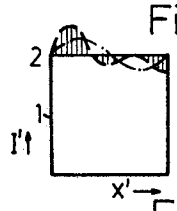


Fig.7

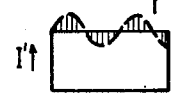
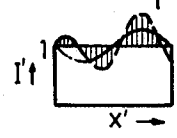


Fig.6



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Fig. 9

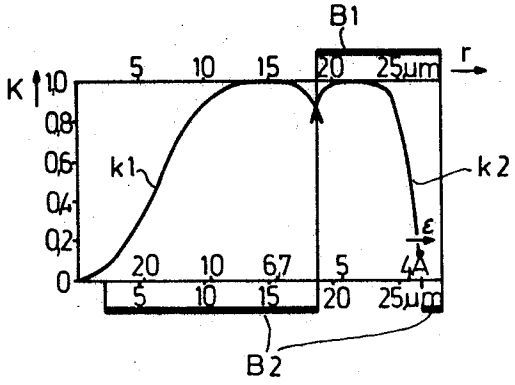
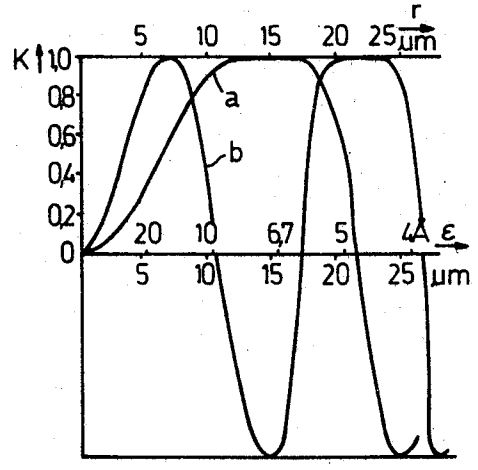


Fig. 3



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Fig. 10

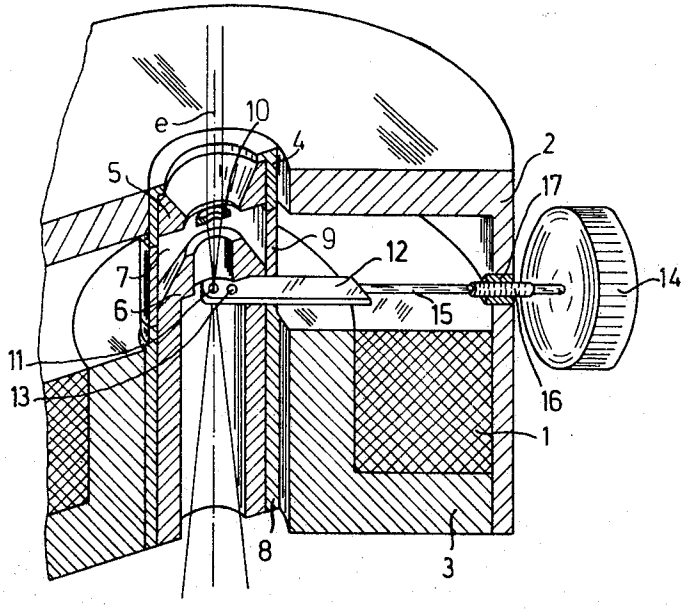
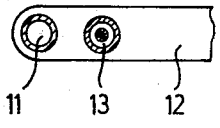


Fig. 11



United States Patent Office 3,500,043 Patented Mar. 10, 1970

3,500,043

METHOD FOR HIGH CONTRAST IMAGING OF PHASE OR AMPLITUDE OBJECTS IN A CORPUSCULAR RAY DEVICE, SUCH AS AN ELECTRON MICROSCOPE

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Filed May 10, 1967, Ser. No. 637,425
Claims priority, application Germany, May 13, 1966, H 59,395
Int. Cl. H01j 37/26
U.S. Cl. 250—49.5 6 Claims

ABSTRACT OF THE DISCLOSURE

A method for high contrast imaging of phase or amplitude objects in a corpuscular ray device, particularly in an electron microscope. The corpuscular ray device has an objective lens capable of producing wave aberration. Correcting diaphragms are successively situated in the corpuscular ray path. Each correcting diaphragm has, next to each other, regions or zones which are alternately permeable and non-permeable to the corpuscular rays. These zones of the correcting diaphragms are arranged in such a way that only those space frequencies of the object reproduced with positive contrast or only those reproduced with negative contrast reach the image plane. At a first value of excitation of the objective lens a first correcting diaphragm is introduced into the corpuscular ray path such that only those space frequencies are allowed to reach the image plane which at the first excitation value of the objective lens are reproduced with only one character of contrast. After photographically reproducing that first image in the image plane, this first correcting diaphragm is displaced out of the corpuscular ray path and replaced by a second correcting diaphragm which is used with a second value of excitation of the objective lens. This produces a wave aberration resulting in transmission of those space frequencies that are represented with the same contrast character during use of said second diaphragm as those transmitted during use of said first diaphragm but comprising, at the second excitation value of the objective lens, at least some of the space frequencies which were suppressed by the non-permeable zones of the first diaphragm, so that the image derived by way of the first correcting diaphragm is at least partially completed by way of that derived with the second correcting diaphragm.

My invention relates to a method for high-contrast imaging of phase or amplitude objects in a corpuscular ray device, particularly an electron microscope, having an objective lens which will produce a wave aberration and while using in the corpuscular ray path a correcting diaphragm which has arranged one next to the other zones which are alternately permeable and non-permeable to the corpuscular rays, to produce in the image plane images of space object frequencies which are of the same contrast character, namely either only of positive contrast or only of negative contrast.

It is known to carry out optical corpuscular ray investigations, such as, for example, investigations with an electron microscope, of very thin objects with high resolution at small ray apertures while utilizing phase contrast effects. The object details which are characterized by the distribution of the potential which is present in the object influence primarily the phase of the waves which travel through the device. For the theoretical handling of the actions which take place, it is of advantage to visualize the object, according to Fourier, as being made up of a sinusoidal phase pattern of the most widely different space frequencies and to consider the imaging of only predetermined space frequencies. Several space frequencies are respectively transmitted to predetermined locations in the plane of the exit pupil of the objective lens, customarily the plane of the aperture boundary.

At a weak sinusoidal phase pattern, the waves are deflected in a pair of directions which are situated symmetrically to the optical axis. The deflected waves have with respect to the primary wave a phase shift $\lambda/4$ (λ=corpuscular wave length).

For high contrast imaging of the particular space frequencies, the phase shifting must be reversed by the imaging system or must be completed in a suitable manner.

It is already known with optical systems to eliminate such phase shifting by arranging in the back focal plane of the lens a phase turning foil in such a way that the primary rays do not undergo any phase turning while, on the other hand, the phase turning foil provides for all of the deflected rays a phase turning of $\lambda/4$. This phase turning foil does not, however, take into consideration the image error of the objective lens and is therefore effective only to a very limited degree. Thus, it has been demonstrated that the objective lens of an electron microscope, for example, will in any event provide a phase influencing of the deflected rays. These relationships are covered by the concept of wave aberration. Thus, these considerations deal with a phase turning suffered by the rays during passage thereof through the objective lens, or, in other words, these considerations deal with the deflection of the wave front of every imaging point from a spherical configuration, considered at the plane of the exit pupil of the lens. As has been demonstrated by investigations, the wave aberration $W(r)$ is not a lens constant, but depends not only the radial distance r of the considered point of the back focal plane of the lens from the primary ray but also on the particular objective excitation and is represented by the relationship

$$W(r)=-Ar^4+Br^2+\text{terms of higher powers}$$

In the latter equation A is a first constant which is proportional to the aperture error constant of the objective lens and B is a second constant which is proportional to the defocusing.

For these reasons it is not possible to eliminate defective phase shifting during imaging only by use of a foil which turns the phase over the entire cross section of the beam through a value $\lambda/4$ (or a suitable multiple thereof).

It is a primary object of my invention to provide a method which will make it possible to image all of the interesting structure of a given object.

In particular, it is an object of my invention to provide a method which will achieve imaging while eliminating the undesirable effects of wave aberration. Furthermore, it is an object of my invention to provide a method which will produce imaging which is of a contrast which is of only one character, namely either only positive contrast or only negative contrast.

Furthermore, it is on object of my invention to provide a method which will make it possible to derive from the images achieved with my invention photographs which can singly have all of the desired information thereon or which can be composed of separate photographic sections which can be superimposed upon each other to give a complete picture of the structure of a given object.

My invention is illustrated by way of example in the accompanying drawings which form part of this application and in which:

FIGS. 1 and 2 are respectively diagrammatic representations of the manner in which the corpuscular rays reach the image plane and respectively coact with differ-

ent diaphragms under different operating conditions to perform the first and second steps of the method of my invention;

FIG. 3 is a graph illustrating the relationship between phase contrast transmission and aperture and wave length;

FIGS. 4–8 are graphs showing the relationship between image intensity and an image coordinate;

FIG. 9 is a graphic illustration of the relationship between the phase contrast transmission factor in relationship to other factors, similar to the graph of FIG. 3;

FIG. 10 is a schematic fragmentary sectional elevation showing in perspective part of a corpuscular ray device used with interchangeable diaphragms according to my invention; and

FIG. 11 is a fragmentary top plan view of an assembly of a plurality of different diaphragms according to my invention.

FIGS. 1 and 2 graphically illustrate the relationship between the dependence of wave aberration of a given location at the rear focal plane F and the excitation of the objective lens.

In FIGS. 1 and 2, the object is to be considered as situated at the object plane O, while the objective lens plane L is shown to the right of the object plane O. The primary corpuscular ray p is shown extending along the central, principal axis of the schematically illustrated structure, and surrounding the primary corpuscular ray p are the deflected rays shown in FIGS. 1 and 2 for two space frequencies, these deflected rays being annularly distributed about the primary rays with an astigmatism-free objective lens. The distance between the locations which are considered in the back focal plane F from the primary ray p is represented at r while the object coordinate x is indicated at the left of FIGS. 1 and 2.

Each of FIGS. 1 and 2 illustrates the behavior of wave abberation $W(r)$ in the back focal plane F at a predetermined value of excitation of the objective lens. In the case of FIG. 1, the situation provided under conditions of weak underfocusing is illustrated. As is apparent from the illustrated behavior of wave aberration $W(r)$ in dependence upon the radial distance r from the primary ray p, there is a relatively large central region of positive phase contrast which is surrounded by an outer annular region of negative phase contrast which forms an extension of the central region of positive phase contrast.

Under conditions of intense underfocusing, as illustrated in FIG. 2, the central region of positive phase contrast becomes substantially smaller. This smaller central region of positive phase contrast is immediately surrounded by an annular region of negative phase contrast which forms an extension of the region of positive phase contrast and which is itself surrounded by an annular region of positive phase contrast which in turn forms an outer extension of the intermediate region of negative phase contrast. To the last-mentioned outer region of positive phase contrast there is then an adjoining outer region of negative phase contrast, and so on, so that there are successive annular regions of alternately opposite phase contrast situated at progressively greater radial distances from the primary ray p.

It is known that with phase objects, there will be imaging with positive contrast, in the above indicated sense, only of those space frequencies whose transmission occurs through a location having a radial distance r from the primary ray in the back focal plane and a wave aberration $W(r)$ between $-n\cdot\lambda$ and $-(n+\frac{1}{2})\cdot\lambda$, where $n=0, \pm1, \pm2, \pm3 \ldots$ and λ is again the wave length of the corpuscular rays. The corresponding value for amplitude objects is $-(n-\frac{1}{4})\cdot\lambda$ and $-(n+\frac{1}{4})\cdot\lambda$.

FIG. 3 shows the relationship between the phase contrast transmission factor K on the one hand and the aperture coordinate r as well as the wave length ϵ of the space frequencies on the other hand, at a predetermined aperture error and a predetermined wave length λ. Thus, referring to FIG. 3, the curve a illustrates weak underfocusing according to FIG. 1 while the curve b illustrates the more intense underfocusing according to the behavior of wave aberration as illustrated in FIG. 2. In both curves the regions of positive and negative phase contrast alternate. It is desirable to make either the regions of positive transmission factor as large as possible or the regions having the negative phase contrast transmission factor as large as possible, so that as many space frequencies as possible are transmitted either with only positive contrast or with only negative contrast. If the imaging is mixed, which is to say if there are portions of positive contrast and negative contrast mixed together, then in an arrangement as shown in FIGS. 1 and 2 where the different space frequencies produce at the given wave aberration images which are in part of positive phase contrast and in part of negative phase contrast, it is difficult to derive from the structural image definite conclusions with respect to the structure of the object.

It can be demonstrated that the maximum positive phase contrast will be encountered at a wave aberration of $-(n+\frac{1}{4})\cdot\lambda$, while the maximum negative phase contrast will be encountered at a wave abberation of

$$-(n+\tfrac{3}{4})\cdot\lambda$$

It is however not possible to achieve solely by a suitable choice of the value of the excitation of the objective lens a sufficiently great region of only positive phase contrast or only negative phase contrast.

As a result of these considerations, it is expedient to use a correcting diaphragm which has zones which are respectively permeable and non-permeable to the electron rays. These zones are arranged so that a field of the diaphragm which is permeable to the electron rays is located next to a zone of the diaphragm which is non-permeable to the electron rays, and thus the permeable and non-permeable zones of the diaphragm alternate with each other. "The size and arrangement of the alternating zones of the diaphragm which are permeable and non-permeable to the electron rays are such that only space frequencies of the objects which are imaged with exclusively positive contrast or with exclusively negative contrast are transmitted to the image plane." Such a diaphragm is schematically shown in FIG. 1, and also such diaphragm is schematically shown in FIG. 2. As may be seen from FIG. 1, the correcting diaphragm B1 prevents transmission of those space frequencies which are imaged with negative phase contrast. Thus, for this purpose the correcting diaphragm B1 has a ring-shaped, annular zone which is impermeable to the corpuscular rays while the central circular zone of the diaphragm B1 is permeable to the corpuscular rays.

In the case of FIG. 2, it will be seen that the correcting diaphragm B2, used with more intense underfocusing, with the resulting more complicated behavior of wave aberration, has a more complicated construction than the diaphragm B1. Thus, it will be seen that the diaphragm B2 has a pair of annular areas which are not permeable to the corpuscular rays, and of course these latter areas alternate with a central circular area and an annular area or field both of which are permeable to the corpuscular rays. It will be noted that both diaphragms have in common the feature being provided with a central region which is permeable to the primary ray p.

As contrasted with the above-discussed use of a foil for constant phase turning, correcting diaphragms as described above and shown in FIGS. 1 and 2 have the advantage of taking into consideration the influence of the phase of the different waves which depends upon the distance from the primary ray or principal axis resulting from wave aberration. This favorable result is however achieved only at the cost of suppressing a portion of the wave frequency region. As a result there is a loss of information with respect to the structure of the object.

With my invention, however, it is possible by utilizing

correcting diaphragms of the above type to image all of the interesting structure of a given object. The solution to the problem is achieved with the method of my invention by first situating in the path of the corpuscular rays a first correcting diaphragm the construction of which is such that at a first value of excitation of the objective lens only space frequencies of a single contrast character, namely either of positive contrast only or of negative contrast only, will be imaged in the image plane at the particular value of excitation of the objective lens. This first image in the image plane is then photographically reproduced and then this first correcting diaphragm is displaced out of the corpuscular ray path and is replaced by a second correcting diaphragm which is situated in the path of the corpuscular rays and which has such a construction that at a given second value of excitation of the objective lens only space frequencies which are imaged exclusively at positive contrast or exclusively at negative contrast will be transmitted to the image plane, and the second excitation value of the objective lens is selected in such a way with respect to the first excitation value as to obtain wave aberrations which, when the second correcting diaphragm is used, will provide at the image plane images of those space frequencies which were suppressed by the first correcting diaphragm. Of course, the phase contrast which is transmitted by the second correcting diaphragm will be the same as the phase contrast which is transmitted by the first correcting diaphragm.

In the event that it is not possible to image all of the interesting space frequencies by the successive use of only two different correcting diaphragms at the two different excitations of the objective lens which result in the different wave aberrations, then these steps can be repeated with additional correcting diaphragms and additional selections of excitation of the objective lens to provide preselected wave aberrations. Thus, a solution to the problem provided by this latter general case will include, after the imaging with the first and second correcting diaphragms at the different values of objective lens excitation, the use of further correcting diaphragms which are successively introduced into the path of the corpuscular rays and which have size and dimensions, with respect to their alternating zones which are permeable and non-permeable to the corpuscular rays, which at the selected values of objective lens excitation used for these additional correcting diaphragms will provide additional imaging either only at positive contrast or only at negative contrast of space frequencies in the image plane in such a way that at the excitation values of the objective lens which are selected to provide pre-determined wave aberrations, all of the space frequencies which were suppressed by the first correcting diaphragm will be imaged successively by the second and the following correcting diaphragms.

According to a preferred method of my invention, the correcting diaphragms are introduced into the path of the corpuscular rays at the region of the back focal plane of the objective lens, as is indicated in FIGS. 1 and 2.

Thus, in principle, my invention deals with the superpositioning of images of the same object achieved by way of corpuscular ray optics at different lens excitations while using different correcting diaphragms. This superpositioning can be provided as by photographically reproducing the successive images achieved by the different correcting diaphragms on the very same sheet of photographic material, which is to say on the same sheet of film or on the same photographic plate. However, it is also possible to provide different distinct pictures respectively resulting from the use of the different correcting diaphragms, and then these several pictures can be superimposed one upon the other to provide the complete picture.

The number of steps taken according to the method of my invention, which is to say the number of correcting diaphragms which is used, is limited by the background intensity which increases with the number of method steps. This factor can be appreciated from FIGS. 4–8 which illustrate the behavior of the image intensity I′ at values of 1 and 2. These diagrams of FIGS. 4–8 show the behavior of image intensity I′ for the space frequencies in FIGS. 1 and 2 over the image coordinate x'. Thus, FIGS. 4 and 5 illustrate the relationships of FIG. 1, FIGS. 6 and 7 illustrate the relationships of FIG. 2, while FIG. 8 illustrates the superpositioning of images achieved by using the diaphragms **B1** and **B2**.

In the case of FIGS. 4 and 6, the behavior of the intensity when the diaphragms **B1** and **B2** are respectively displaced out of the ray path is illustrated, while in the case of FIGS. 5 and 7 the behavior of the intensity is shown with the diaphragms of FIGS. 1 and 2 respectively situated in the path of corpuscular rays so that in the case of FIGS. 5 and 7 only those space frequencies are imaged which have positive contrast. It is apparent that the average image intensity in all cases is constant.

However, the case is different with the superpositioning according to FIG. 8, as provided with my invention. In FIG. 8 the average brightness is doubled, since in correspondence with the different wave aberrations the diaphragms **B1** and **B2** allowed different space frequencies to reach the image plane, but with both diaphragms the primary corpuscular ray passed through to the image plane. As a result, the number of exposures with different correcting diaphragms is limited.

FIG. 9 illustrates the behavior of the phase contrast transmitting factor K with the method of my invention in a diagram which corresponds to the illustration of FIG. 3. The diaphragms **B1** and **B2** are schematically represented in FIG. 9. As may be seen from FIG. 9, there is in total a broader region of positive contrast transmitting factor K achieved by the double exposure of the same film using both diaphragms. The use of the diaphragm **B1** results in an image of space frequencies having the wave length ϵ, which is illustrated in connection with the curve $k1$, while the curve $k2$ applies to the exposure while using the diaphragm **B2**.

Thus, when carrying out the method of my invention, there should be on hand a set of correcting diaphragms and information should be available to indicate to the operator the different values of lens excitation which should be definitely selected so that by simple manipulation of suitable knobs or switches it becomes possible to make the exposures one after the other with the different correcting diaphragms and at the different lens excitations which respectively are to be used with the different diaphragms.

FIG. 10 illustrates schematically an objective lens for an electron microscope used with a pair of correcting diaphragms carried by a diaphragm shifting structure which is also illustrated in FIG. 11.

The objective lens of FIG. 10 includes, as is known, a coil **1** through which current flows to achieve a magnetic flux, and there is an iron return flow path for the magnetic flux provided in the illustrated example by the components **2** and **3**. The magnetic flux path is closed through the annular component **4**, the pair of pole shoes **5** and **6**, which define the lens gap **7** between themselves, and the tubular component **8**. The flux is compelled to traverse the lens gap **7** by making the component **9** of a material which is magnetically inert, such as, for example, brass.

Beneath the object-carrying diaphragm **10** there is a correcting diaphragm **11** which is held in the corpuscular ray path by the diaphragm slide **12**, this one correcting diaphragm **11** being situated at least approximately at that location where the electron stream e becomes concentrated to a point by the action of the magnetic lens field in the gap **7**. As may be seen from FIG. 11, in the illustrated example the correcting diaphragm **11** is constructed in a manner similar to the correcting diaphragm **B1**, shown in the example of FIG. 1, from zones or regions which are alternately permeable and non-permeable to electrons.

The diaphragm slide **12** in the illustrated example carries the second correcting diaphragm **13** which, as is also apparent from FIG. 11, has the same construction as the diaphragm B2 of FIG. 2.

Both of the correcting diaphragms **11** and **13** can, by moving the slide **12**, in a direction transverse to the electron stream *e*, be displaced into the path of the rays. For this purpose the drive **14** which is shown in FIG. 10 may be used, this rotary drive including the rotary driving rod **15** whose left end, as viewed in FIG. 10, engages the right end of the slide **12**, this left end of the rotary driving rod **15** carrying the slide **12**, for example, while being turnable with respect thereto in such a way that the slide **12** is compelled to move axially with the rod **15** while the latter is capable of turning with respect to the slide **12**. For example, the left free end of the rod **15** may terminate in an outwardly directed collar or flange, and this left free end may be received in a T-slot formed at the right end of the slide **12**, so that while such a collar or flange can turn in such a slot with respect to the slide **12**, nevertheless the latter is compelled to move axially together with the rotary rod **15**.

This rotary driving rod **15** carries in the region of the component **2** an exteriorly threaded portion **16** which has its threads coacting with the interior threads of a sleeve **17** which is mounted within an opening of the component **2** and fixed to the latter. Drives of this general construction are known. The connection between the rod **15** and the slide **12** is such that the slide **12** will only move transversely to the corpuscular ray path without participating in the rotary movement of the rod **15**. To prevent rotary movement of the slide **12**, the components **9** and **6** are formed with slots whose configuration is such that the slide **12** can pass freely through these slots but cannot rotate with respect to the components **9** and **6**.

Of course, the slide **12** can have still more diaphragms, and, in fact, it can be provided with openings which do not carry any correcting diaphragms in the event that it is desired to carry out operations without correcting diaphragms.

Furthermore, there are other possible constructions which may be used for interchanging the correcting diaphragms.

I claim:

1. A method for high contrast imaging of phase or amplitude objects in a corpuscular ray device, such as an electron microscope, having an objective lens for focusing an image in an image plane which lens produces wave aberrations, said method comprising the steps of sequentially placing into the ray path a plurality of correcting diaphragms, one at a time, each having mutually adjacent zones permeable and non-permeable respectively to corpuscular rays and positioned so that only such space frequencies of the object reach the image plane which are imaged with only one character of contrast, namely either only with positive contrast or only with negative contrast, first exciting the objective lens at a first value of excitation while situating in the ray path a first one of said correcting diaphragms which allows only those space frequencies to reach the image plane that are imaged at only one contrast character at said first excitation value, photographically reproducing the image at said image plane so as to form a first picture thereof; then displacing the first correcting diaphragm away from the corpuscular ray path, and exciting said objective lens at least once more at another value of excitation and situating in the corpuscular ray path another one of said plurality of correcting diaphragms which at said other value of excitation of said objective lens, allows only those space frequencies to reach the image plane that are represented with the same contrast character as that transmitted through the diaphragm previously situated in the ray path, said other excitation value of said objective lens being selected to produce a wave aberration such that, with the other correcting diaphragm, at least some of those space frequencies are reproduced the imaging of which was suppressed by the non-permeable zones of the preceding correcting diaphragms, so that a picture derived by way of each next diaphragm will at least partially complete the picture derived by way of the first diaphragm.

2. In a method as recited in claim **1**, wherein each correcting diaphragm in introduced into the corpuscular ray path at the region of the back focal plane of the objective lens.

3. In a method as recited in claim **1**, the step of photographically reproducing the respective images produced in the image plane during use of each of said plurality of diaphragms on the same photographic material.

4. In a method as recited in claim **1**, wherein the images respectively produced in the image plane during use of said plurality of diaphragms are separately photographed on separate photographic sheets, and then superimposing the latter sheets one upon the other to derive from all of said sheets a photograph wherein the partial photograph derived by way of said first diaphragm is at least partially completed by the partial photograph derived by way of said second diaphragm and the respective partial photographs derived by way of the additional diaphragm.

5. In a method for high contrast imaging of phase or amplitude objects in a corpuscular ray device, such as an electron miscroscope, having an objective lens for focusing an image in an image plane which lens produces wave aberrations, and using in the ray path a correcting diaphragm with mutually adjacent zones, permeable and non-permeable, respectively, to corpuscular rays and positioned so that only such space frequencies of the object reach the image plane which are imaged with only one character of contrast, namely either only with positive contrast or only with negative contrast, the improvement comprising the steps of first exciting the objective lens at a first value of excitation, situating in the ray path a first correcting diaphragm which allows only those space frequencies to reach the image plane that are imaged at only one contrast character at said first excitation value, photographically reproducing the image at said image plane so as to form a first picture thereof; then displacing the first correcting diaphragm away from the corpuscular ray path, and exciting said objective lens at a second value of excitation and situating in the corpuscular ray path a second correcting diaphragm which at said second value of excitation of said objective lens, allows only those space frequencies to reach the image plane that are represented with the same contrast character as that transmitted through said first diaphragm, said second excitation value of said objective lens being selected to produce a wave aberration such that, with the second correcting diaphragm, at least some of those space frequencies are reproduced the imaging of which was suppressed by the non-permeable zones of said first correcting diaphragm, so that a picture derived by way of said second diaphragm will at least partially complete the first picture derived by way of said first diaphragm.

6. In a method as recited in claim **2**, the steps of successively introducing into the corpuscular ray path, after imaging with said first and second correcting diaphragms, additional correcting diaphragms at additional values of excitation of said objective lens with said additional correcting diaphragms, the additional diaphragms being such that, at said additional values of excitation of said objective lens, respectively, only those space frequencies are allowed to reach the image plane which are reproduced with the same character of contrast as those transmitted during use of said first and second diaphragms at the first and second values of excitation of the objective lens, respectively, said additional values of excitation of the objective lens being selected to produce a wave aber-

ration such that the space frequencies, which were suppressed by the non-permeable zones of said first and said second diaphragms are reproduced in succession by using the additional correcting diaphragms, so that the picture achieved by way of said additional diaphragms substantially completes that achieved by way of said first and second diaphragms.

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