V. *Effects of Strain on the Crystalline Structure of Lead.*

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[Plates 2–6.]

The effects of strain upon the crystalline structure of metals and the subsequent effects of annealing at low temperatures have already been studied by Ewing and Rosenhain,* and the work described in the following pages is in many ways a continuation of theirs. A variety of lead was obtained in which the crystalline structure was on a particularly large scale. This furnished a metal specially well adapted for experiments on the influence of strain on crystalline structure, and the author, on the suggestion of Professor Ewing, was successful in obtaining single crystals of sufficient size to furnish a test piece possessing a uniform orientation throughout the part under test. As the effects of a strain could then be studied in individual crystals, the problem was greatly simplified.

The material used was a pure lead, commercially known as "chemical lead," its chief use being for purposes such as the lining of acid chambers. It is obtained by treating ordinary furnace lead in Pattinson crystallisation pots, and by this process practically all other metals which were originally alloyed with it are removed. The first sample obtained had been cut from a casting, and was about 6 inches by 4 inches in area by $1\frac{1}{2}$ inches deep. One of the large faces had been in contact with the air during solidification and the other with the bottom of the mould. The former showed in a very beautiful manner, and without any treatment, the crystalline structure of the material. The crystalline grains were clearly lined out by the slight differences of level at which they had formed, the boundaries showing up as fine lines. These grains varied from about $\frac{1}{2}$ sq. inch to 4 sq. inches in area and were of quite irregular shape. Many of them exhibited distinctive markings on their surfaces, these generally taking the form of slight ridges. In many cases one fairly

* 'Phil. Trans.,' A, vol. 193, 1899, pp. 353–377, and 'Phil. Trans.,' A, vol, 195, 1900, pp. 279–301.

large ridge ran down the centre of the crystal, and from this others branched away at right angles, giving the appearance of a sort of skeleton.

In order to see how the growth of the crystals had proceeded throughout the casting, the rough sides were smoothed with an ordinary wood plane, and the whole casting was subjected to a prolonged etching in dilute nitric acid. The effect of this treatment upon the upper surface was to bring out in a very striking manner the orientation of the different crystals. The surface of each crystal became covered with a number of geometrical pits, these pits being similar and similarly situated over its area, though varying in shape and position from one crystal to another. The crystals accordingly showed either bright or dark, as the light striking the sides of the pits was reflected or not into the eye. As the specimen was revolved the crystals flashed out one after another from almost black to a brilliant white. Fig. 1 (Plate 2) is a photograph of this surface in its natural size, and the different shades of the various crystals can be clearly seen. The appearances observed after etching are precisely similar in kind to those which are already well known as occurring when ordinary metallic surfaces are subjected to microscopic examination; but here they are exhibited on an exceptionally large scale.

The first effect of etching upon the sides of the specimen was to reveal a number of minute crystals all over the side surfaces, but as the action proceeded these disappeared, and the large crystalline structure of the lump could be seen. The crystals upon the upper surface of the casting were found to only descend to a depth of about $\frac{1}{4}$ inch, and a second system had grown vertically upwards from the bottom of the mould to meet them. The sections of the latter parallel to the upper surface of the casting were generally of rather smaller area than those upon the upper surface.

As will be seen in fig. 1, some of the crystals do not reflect the light quite equally from all parts of their surface, but seem to be shaded. The shaded appearance is only visible in certain illuminations, the orientation of the crystal appearing quite uniform in all others. When examined under the microscope, it was found that the edges of the etched pits upon them were not quite parallel in the various parts. Fig. 2 shows part of such a crystal magnified 12 diameters, and it can be seen that the edges of the pits between the two dark bands running across the centre are not quite parallel to those on the portions on either side; the difference is, however, slight. The effect is probably caused by a slight disturbance whilst the crystal was forming, one part being moved relatively to another when the metal between them was still molten. Such a disturbance might be brought about by external causes, such as a shaking of the mould, or by stresses set up as the metal cooled. The difference of orientation is still further brought out by the fact that the acid has acted more strongly where the two portions join than upon the surrounding area; this etching out has produced furrows, which appear as the dark bands in fig. 2. Such an action is always seen between two patches of different orientation, and

where the difference is great, as it is between two crystals, a furrow which can be easily seen without any magnification is produced. It may be accounted for by electrolytic differences between the two portions producing a more violent action where they meet.

The large size of the crystals in this casting suggested that it would be possible to obtain a fairly large sized specimen of lead having a uniform orientation throughout, so that the effects of strain upon such a specimen might be studied. To do this an attempt was made to obtain a casting in which individual crystals went right through from top to bottom, as they could then be separated one from another with a minimum amount of cutting. It was found to be very difficult to do this by direct casting, as crystallisation always seemed to take place from both top and bottom, thus producing a casting such as has been described above. The following method was therefore resorted to :—

The lead was melted in a flat sheet-iron dish over a small gas furnace, an iron plate being placed between the flame and the dish to equalise the temperature. The gas was then turned low and the surface of the metal was allowed to solidify, one corner, however, being kept molten by removing the crust with a hot iron spike. Owing to the contraction of the lead upon solidifying the growth of the various crystalline grains could be observed, a slight difference of level being formed between the solid and molten portions. The growth was, perhaps, best seen in those crystals which formed with two cubic axes parallel to the surface. Fig. 3 illustrates diagrammatically the manner of this growth. One or more parallel arms, such as are seen in the figure, shot out from the place where crystallisation started and gradually spread outwards into the molten metal. At certain intervals along these came others at right angles to them, then a third set from these parallel to the first, and so on, numerous successive sub-branches growing at right angles and gradually filling in the intervening spaces. The growth of an individual crystal proceeded in this manner until arrested by meeting either the sides of the dish or another crystal. The outlines of the branches remained even after the whole casting was cool, forming the ridges which have been already mentioned.

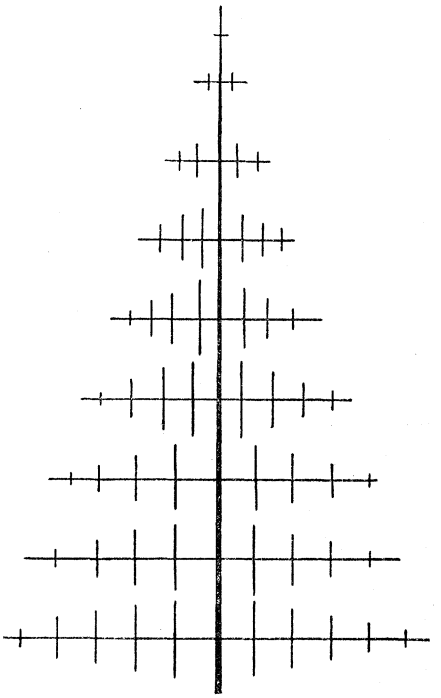

Fig. 3.

When a sufficiently strong crust had formed, the dish was lifted up and the still molten metal was poured out through the free corner. When cool, it was found that a certain portion round the edges of the dish had solidified to the full depth of the

metal, but that in the centre there was simply the upper crust, about $\frac{1}{8}$ inch to $\frac{1}{4}$ inch in thickness, and this could be easily cut away from the surrounding portions. The crystals forming the crust fell away from one another to a certain extent when unsupported by the metal beneath. This was due to the well-known fact that the metallic impurities form a eutectic with part of the lead and collect in the crystalline boundaries. This eutectic has a lower freezing-point than the pure lead, and hence remains molten after the main mass has solidified. The boundaries are, therefore, lines of weakness, and the crystals tend to fall apart when the supporting metal below is poured away.

The upper surface of the crust which had been in contact with the air was, apart from the skeletal markings, fairly plane, but the lower surface was covered with numerous small spiky projections, these being regular in shape and position over each individual crystal, but varying from one crystal to another. Fig. 4 (Plate 2) shows such a surface natural size, and the various crystalline grains can be easily distinguished by their different textures. It will be seen that in some cases certain spikes are raised rather above the others, forming ridges which run in different directions across the crystals. These correspond to those upon the upper surface which have been mentioned above. A portion of the same surface at the junction of three crystals is shown magnified to 10 diameters in fig. 5, and the different shape and orientation of the spikes upon each crystal can be clearly seen. These spikes have more or less curved surfaces, but generally roughly assume the form of octahedra. Some of them exhibit a somewhat wavy surface, as if the metal had solidified in successive layers. Such markings are probably due to a certain unsteadiness when the molten metal is poured away, the latter flowing two or three times over the solidified surface before finally leaving it.

The various crystals were now separated one from another with a fretsaw. The rough sides were smoothed by cutting away the spiky projections with a sharp knife, and the crystals were then etched with dilute nitric acid.

The etching consisted of two operations; the crystals were first placed in a 20 per cent. solution of nitric acid for about half an hour. This removed any roughness from the surface and produced a fairly smooth plane specimen. They were next immersed in a 5 per cent. solution, in order to produce the geometrical pits by means of which most of the following phenomena have been studied. In order to obtain a good development of such pits it was found necessary to prolong the etching for from one to two days; if particularly large pits were required the process was even longer. It was found in general that the stronger the acid solution the smaller were the pits, and *vice versâ*. With a 5 per cent. solution a beautiful system of contiguous pits covering the entire surface of the specimen could be produced which was easily visible at a magnification of 20 diameters or less.

The shape of these etched pits has been found to be that of a portion of a negative cubo-octahedron. Such a figure is shown in fig. 6 (*a*) and (*d*); in (*a*) one of the

cubic faces is parallel to the plane of the paper, and in (*d*) an octahedral face is nearly parallel to the same plane. It possesses faces which are alternately squares and hexagons, and it will be seen that in the following photographs the faces of the pits always consist of all or part of one of these figures. Fig. 6 (*b*), (*e*), are drawings of the pits which (*a*) and (*d*) respectively would make if made to penetrate a certain distance into the plane of the paper, and may be compared to the etched pits seen in the photographs. In some cases the octahedral faces were very strongly developed,

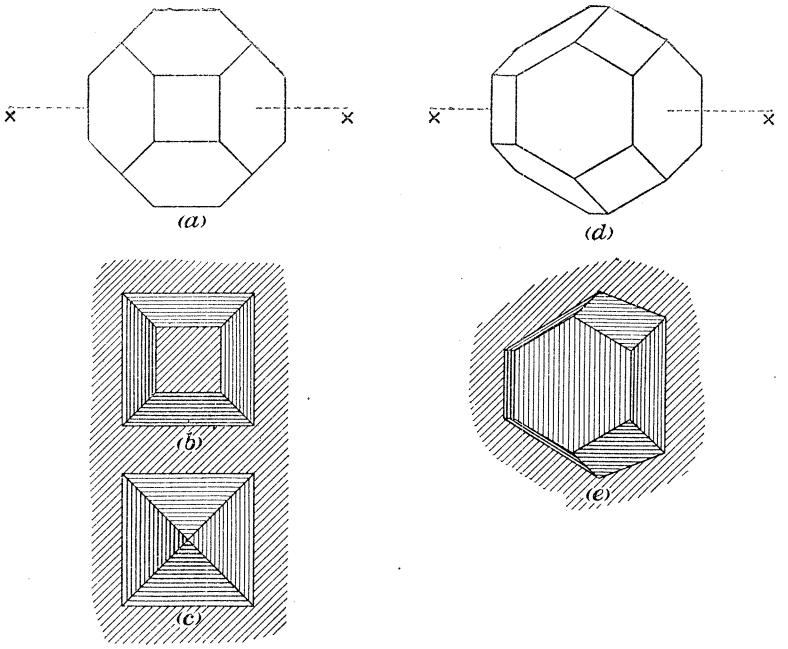

Fig. 6.

and the cubical faces were practically non-existent. The figure then developed into the regular octahedron. Fig. 6 (*c*) shows the type of pit we should expect in such a case when a cubic face is parallel to the surface, and may be compared with the pits seen in figs. 9 and 14. The relation of these pits to the directions of the skeletal crystal formation was found by scratching lines to show these directions upon the surface before etching, taking care to make the scratches deep enough not to be eaten away. It was then seen that where a square face of the pits was parallel to the surface of the specimen, the sides of the square made angles of 45° with the direction of the ridges. That is to say, the sides of the cube from which the cubo-octahedron was formed were parallel to these directions. Hence the pits are correctly placed as regards the crystalline axes, if we assume that the ridges are parallel to the cubic axes of the crystal. From the manner in which these ridges are formed by the crystal growing by successive branches at right angles to one another, as is illustrated in fig. 3, such an assumption seems not improbable. (See Plates 3 and 4.)

When examined under the microscope, the pits at first sight appear to be projections from the surface, but by means of the focussing screw it is readily shown that they are not projections, but true pits.

A similar appearance is also seen in the micro-photographs and is apt to be rather misleading as to the nature of these geometrical figures, but in all cases they were found to be, as stated above, pits descending into the surface.

The single crystals, when separated one from another, possess all the usual plastic properties of an ordinary sample of lead composed of numerous small crystals united together. They show no signs of sudden cleavage or parting and may be bent double without breaking, and may be hammered out into any shape.

The first observations made on the effects of strain on single crystals were in connection with the formation of slip-lines. The specimens were cut to about 2 inches to 3 inches long by $\frac{1}{2}$ inch wide by $\frac{1}{8}$ inch thick, and were generally taken from a single crystal; if, however, a sufficiently large specimen could not be obtained from a single crystal, the piece was cut so that the central portion, where the effects of strain were studied, consisted of a crystal extending right across the specimen from side to side, as well as through from front to back. After the specimens were cut out they were carefully etched until a complete system of geometrical pits had formed upon their surfaces; they were then washed until all the acid was removed, and were quickly dried. The surface remained bright for a sufficient time to enable observations to be made and photographs to be taken, but it gradually became tarnished if left exposed to the air.

The specimens were strained in tension in the small machine described by Ewing and Rosenhain. This could be fixed to the stage of the microscope, so that a portion of the specimen was kept under observation during the whole process. As the stress was applied the specimen gradually elongated, this elongation being due to the numerous small slips along the gliding planes of the crystal. These small slips are first visible under the microscope as either bright or dark lines upon the surface, according to the illumination of the specimen, just as Ewing and Rosenhain first observed them; but when the amount of strain becomes large the actual steps formed can be clearly seen. A point of chief interest in the experiments was the relation of the slip-lines to the etched pits upon the surface, that is to say, to the crystalline axes. Figs. 7 and 8 are photographs taken of surfaces after straining in tension. In fig. 7, which is at a magnification of 45 diameters, the etched pits had been very slowly produced and are of large size; they are not quite contiguous, but portions of the original surface remain between them. It will be seen that the slip-lines on one face of a pit are parallel to one edge of that face. Again, the illumination is such that a partially formed hexagonal face shows bright, and it will be seen that slip has occurred along planes parallel to this face, so that the small steps also appear bright. In fig. 8 (Plate 3) the pits were of considerably smaller size, and a higher power (100 diameters) was necessary to show them clearly. As before, the

slip-lines follow the sides of the pits in such a manner that the plane of slip is parallel to a hexagonal face; in fig. 9 this face and the slip-lines show up bright. Owing to the difference of level in the surface due to the pits, all of it could not be brought into focus together—this accounts for the somewhat blurred patches in the photographs. From these and other observations it may be concluded that lead tends to slip along planes perpendicular to the octahedral axes of the crystals, and there would, therefore, be at least four possible directions in which slip could occur. Four systems of slip-lines have already been noted in strained lead by EWING and ROSENHAIN.

When a single crystal is strained in tension, the slip along the gliding planes does not take place to an equal extent right across the specimen. The surface, originally fairly plane, becomes slightly undulated as the stress is applied, the undulations running diagonally across the specimen at an angle of about 45° to the direction of pull. The general character of the slip-lines can be best examined upon a plane unetched surface not broken up by pits. Such a surface exists on the upper side of the crystal, namely, the side in contact with the air when cast as described above and not afterwards etched. If we examine the slip-lines formed upon it when strained in tension, we find that the undulations are caused by the slip-lines being larger and more numerous along certain areas. When the specimen is re-etched after straining, the undulations are still visible as diagonal bands, which appear either slightly brighter or darker than the surrounding material, according to the illumination used.

Fig. 9 shows part of such an etched surface at a magnification of 45 diameters. A band of pits, more brightly illuminated than the rest, runs across the centre of the figure, and similar, though less distinct, bands can also be traced running parallel to it. The following points can be noticed: (1) that there is no distinct boundary between the light and dark portions, the change from one to the other being gradual; (2) the bands do not exhibit the same illumination; (3) the sides of the etched pits upon the bright parts are not quite parallel to those upon the darker ones.

Such slight differences of orientation occurring after a single crystal has been strained are evidently not due to a re-crystallisation. Professor EWING has suggested to the author that it is quite possible to account for them if we consider that the strain is not homogeneous. It has been stated above that the number of slip-lines was not the same all over the strained crystal, but that certain parts showed more signs of slip than others. From this we would gather that when once slip occurs in a certain part it tends to go on there rather than in other parts of the specimen. Hence these parts get more or less distorted from their original shape, while other parts of the same crystal contiguous to them either have not changed at all, or have done so in a less degree. It may readily occur that the contiguous portions, which were originally in parallel orientation, become relatively inclined through the distortion of the material between—the material between behaving as a

strained wedge—and hence differences in orientation such as are seen in fig. 9 may arise within a single crystal.

In cutting out the single crystals from the casting, a certain amount of local strain was given at the edges by the action of the saw, and it was found, upon etching, that where such strain had occurred the specimen no longer exhibited a uniform orientation, but that numerous small areas of different orientations had appeared. Such a breaking of the crystals has already been mentioned as appearing upon the sides of the casting shown in fig. 1, where it had been sawn from the surrounding metal. Some specimens also had been accidentally strained by bending, and in these cases a similar breaking-up was visible over the strained area when the specimen was re-etched. Further experiments were therefore made to obtain more definite results as to the production of comparatively small crystals by strain.

A specimen was carefully cut with a sharp knife from the centre of a single crystal in such a manner that as little strain as possible was given. After etching it was found that the orientation was uniform all through. It was now bent nearly double between the fingers, straightened again, and re-etched. Great changes were now visible over the strained area, the orientation no longer being uniform, but broken up into numerous small areas, each with a different orientation. The greatest change had occurred on what had been the concave side when the specimen was bent, that is to say, where the metal had been subjected to compression. Figs. 10 and 11 show the compression and tension sides respectively of a specimen originally uniformly oriented throughout (that is to say, originally a single crystal) after it had been strained in the manner described above and then re-etched. It will be seen that on the compression side a large area in the centre has been split up into numerous small patches of different orientations, the variety of shades within this area showing in a striking manner the extent of the change. On the tension side two or three isolated patches have appeared possessing new orientations, but the amount of change there has been far less than on the compression side. It is also noticeable that the ends which were not subjected to any strain have not in any way altered. When we look more closely into the newly oriented patches, it is seen that a great number of straight-line boundaries exist between them, and this fact is still more striking when we use a higher magnification. It is then seen that in some cases the boundaries are quite irregular and have been more or less eaten out into channels during the etching process, but that in others the boundaries are straight and sharp, one orientation changing quite abruptly into the other with no such channel between them. In the latter cases it was also apparent that there was some distinct geometrical relation existing between the pits on either side of the boundary, an edge of the pits on either side being always parallel to the boundary between. In many cases there were two parts joining in a straight line, but surrounded by an irregular boundary. Such is the case in the newly oriented patches seen in fig. 11. Each of the patches consists of two such parts, and the straight line between them

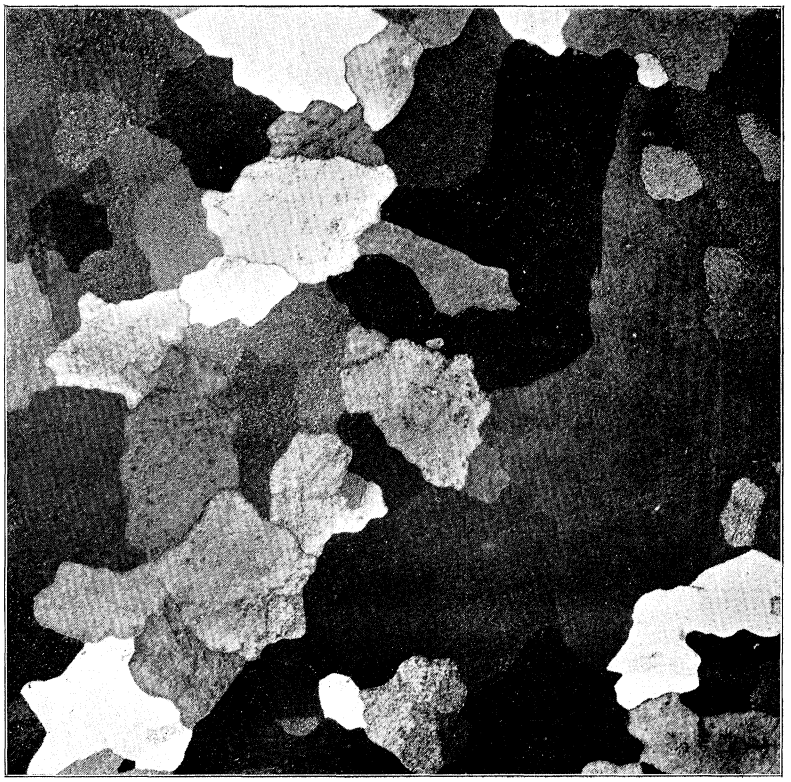

Fig. 1. Cast lead, etched; no magnification.

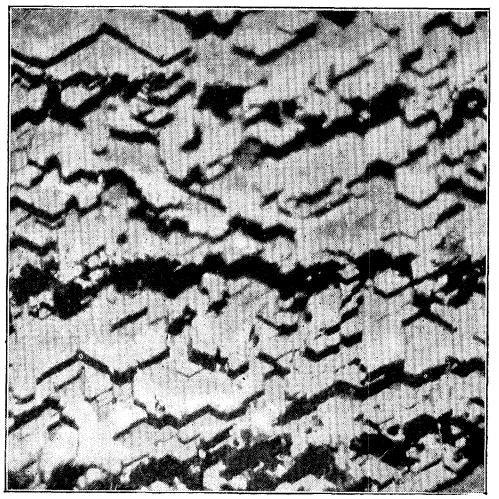

Fig. 2. Etched cast lead × 12.

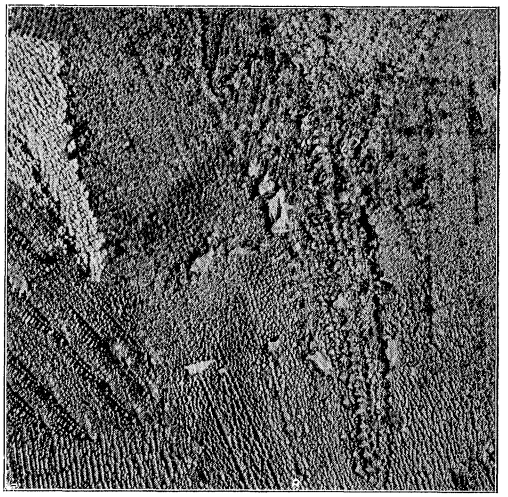

Fig. 4. Under side of casting; no magnification.

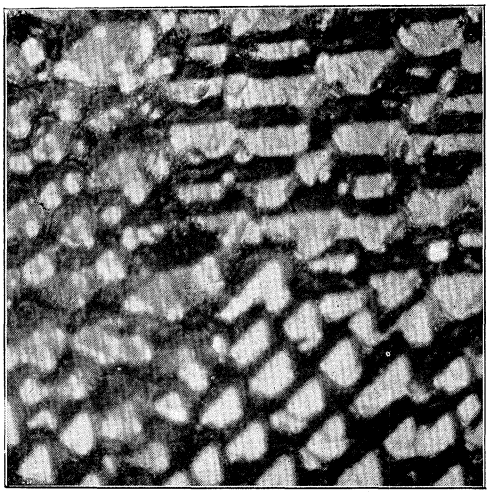

Fig. 5. Part of fig. 4 × 10.

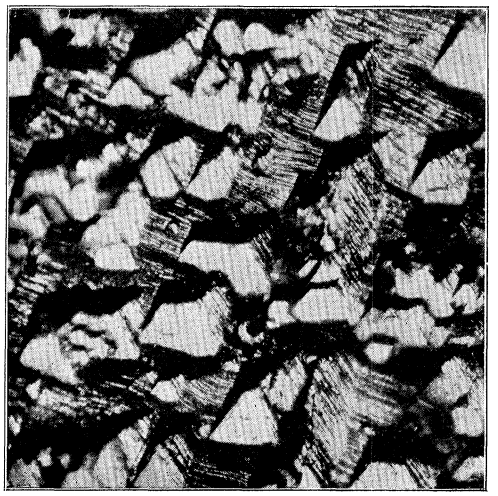

Fig. 7. Slip-lines × 45.

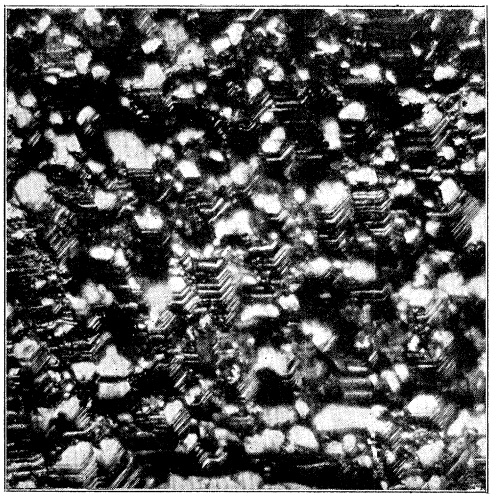

Fig. 8. Slip-lines × 100.

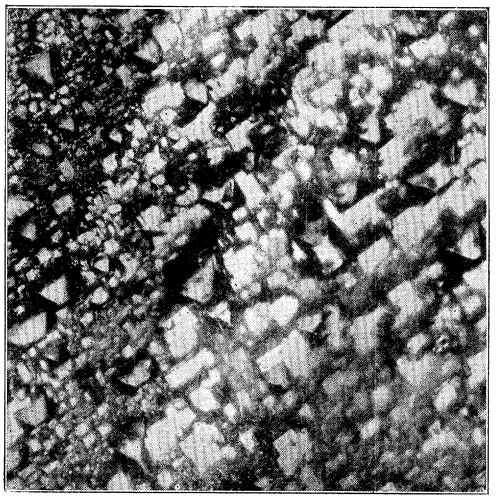

Fig. 9. Part of a single crystal after straining in tension and re-etching × 45.

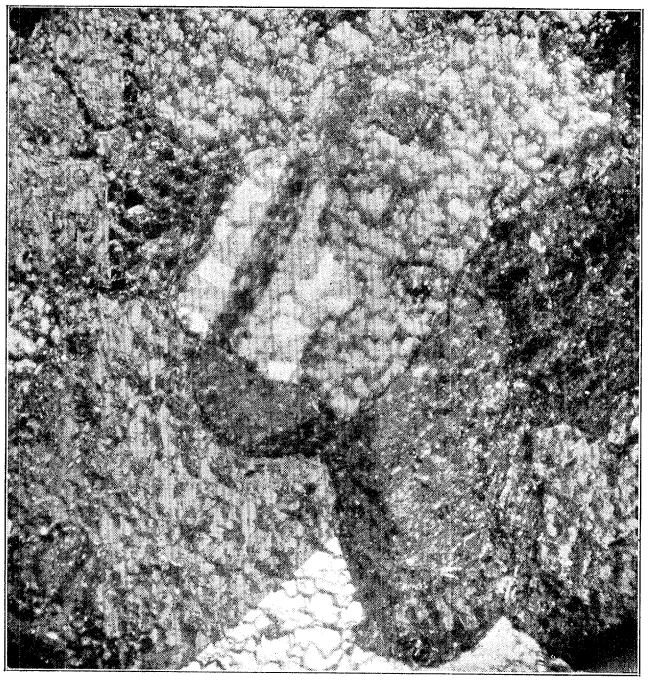

Fig. 12. Twins in strained crystal × 45.

Fig. 10. Strained crystal, re-etched × 6.

Fig. 11. Other face of same crystal × 6.

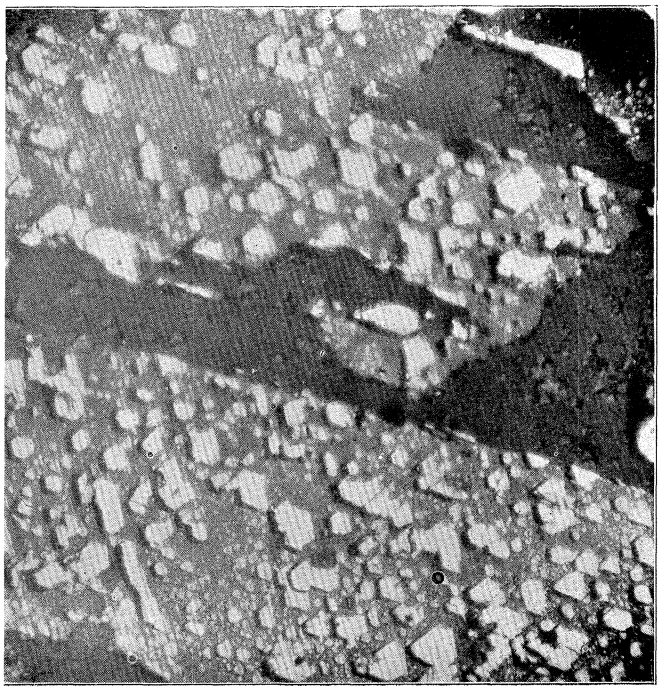

Fig. 13. Twins × 45.

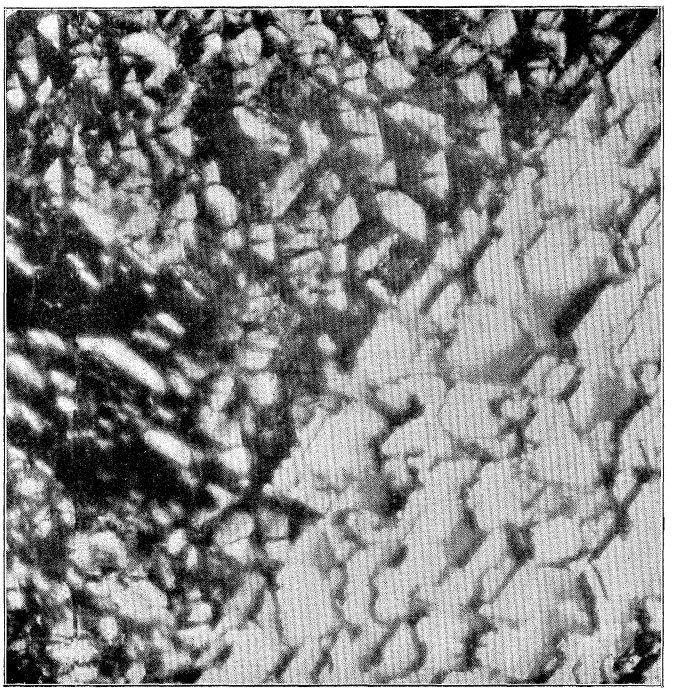

Fig. 14. Twins × 45.

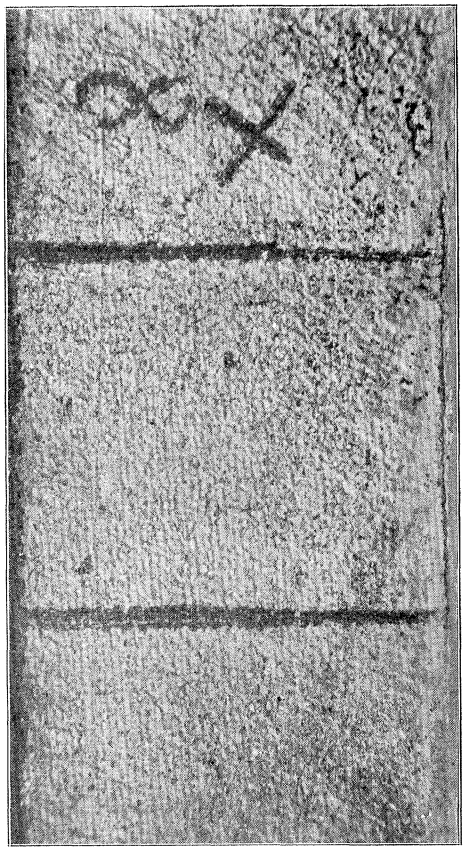

Fig. 15. A single crystal after straining in tension and re-etching × 5.

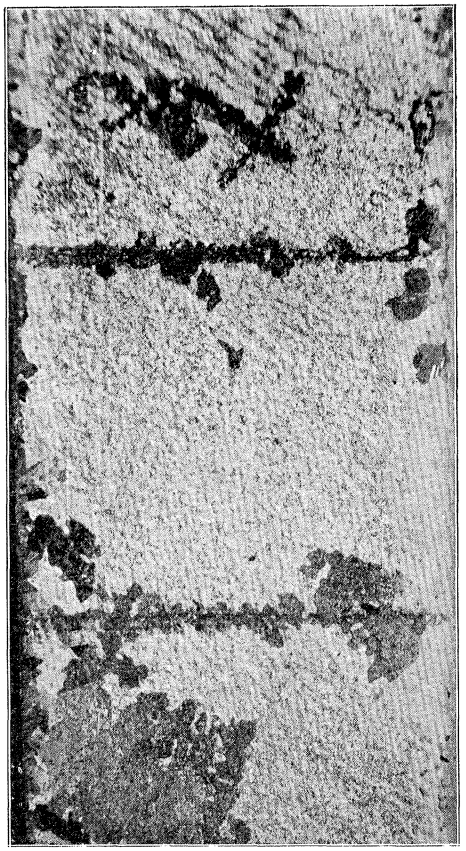

Fig. 16. Same after cooking 5 minutes at 60° C.

Fig. 17. Same after 10 minutes at 60° C.

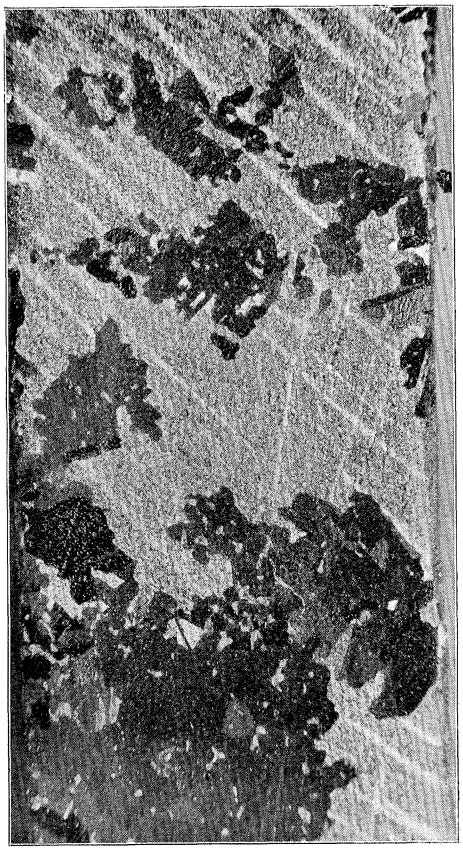

Fig. 18. Same after 20 minutes at 60° C.

Fig. 20. A single crystal after straining in tension and re-etching × 5.

Fig. 22. A single crystal after straining in tension and re-etching × 4.

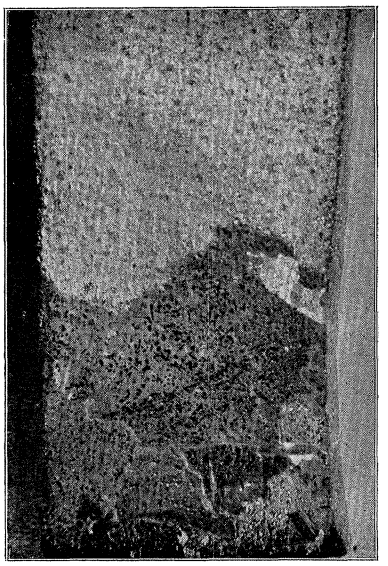

Fig. 23. Same after 3 weeks at atmos. temp.

Fig. 19. Same after 40 minutes at 60° C.

Fig. 21. Same after cooking for 20 minutes at 100° C. and re-etching.

Fig. 24. A single crystal after straining in tension, cooking 2 hours at 100° C., and re-etching.

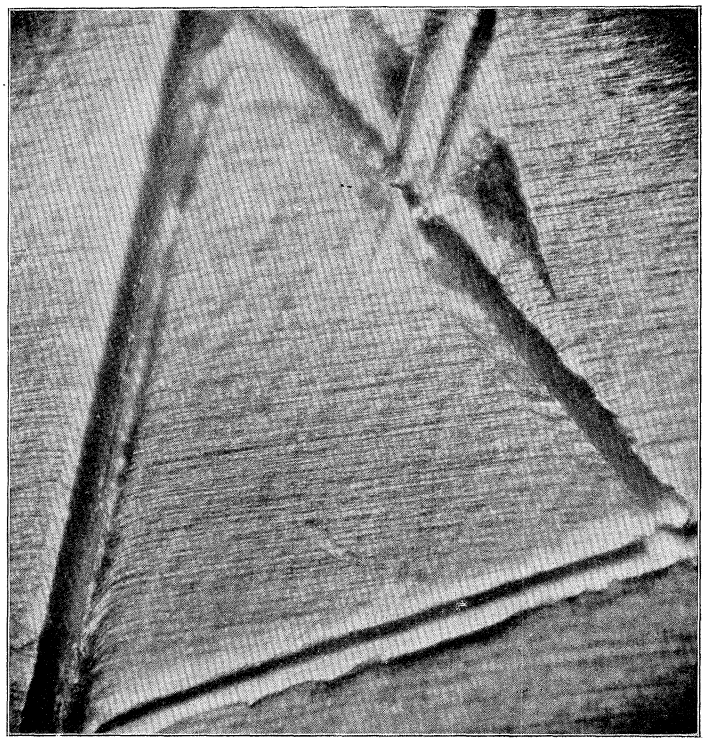

Fig. 25. Slip-lines × 45.

Fig. 26. Same surface after etching.

Fig. 27. Slip-lines × 45.

Fig. 28. Same surface after again straining.

can be contrasted with the curved irregular outline surrounding them. Similar cases can also be seen in fig. 10, though surrounded by new orientations and not, as in fig. 11, by the original one. In other cases there would be two or more sets of parallel straight-line boundaries, the two orientations occurring alternately; and in others a patch showing one orientation would be surrounded on more than one side by straight lines, these being always parallel to an edge of the pits on either side. Figs. 12 and 13 illustrate these cases; all are taken at a magnification of 45 diameters. In fig. 12 we see two portions at the bottom, one bright and one dark, joining in a straight line, but surrounded by an irregular boundary; while in the centre we have a crystal with a band with parallel sides running across it, the parts on either side of the band being in similar orientation. In fig. 13 (Plate 4) we have a patch including various portions of a different orientation, though similar to one another. Some of the boundaries in this case appear irregular, but a rather higher magnification shows that these apparently irregular boundaries are made up of numerous short straight lines. This photograph is also interesting as the etching had not been carried so far as in most of the other cases and the etched pits are not contiguous. It hence gives some idea as to the manner in which they are formed.

Such straight boundaries are a strong indication that the portions on either side are connected by a twin relation, and the fact is further brought out when we examine the geometrical relation of the pits on either side. It has been said above that these pits, which are portions of a cubo-octahedron, are placed in a correct direction to the crystalline axes, and are in fact representations in miniature of what the external form of the crystal would probably be if allowed to assume its proper shape. Take the case shown in fig. 14: we have two main orientations in the picture, one having pits with a large hexagonal face nearly parallel to, and the other having pits with a cubic face parallel to, the surface. In the latter case, the cubic faces of the pits have, for the most part, been etched away, and the pits are practically octahedra. If we draw the complete form of the latter, we shall obtain the figure shown in fig. 6*a*, the dotted line being at right angles to the boundary between the two parts in fig. 14. Now, if we give this figure a turn of 180° about the octahedral axis marked xx, that is to say, bring it into a position in twin orientation to its original one, it will appear as shown in fig. 6*d*. Then, on comparing the etched pits on either side of the boundary in fig. 14 with 6*a* and 6*d*, the twin relation which exists between them is seen. It was found in all cases that the pits on either side of a straight boundary agreed with figures drawn in such a way. The method is of course merely a rough one, and no accurate results could be obtained from it, but it should be quite possible with a suitable goniometer to actually measure the crystallographic angles of the pits on either side. Such measurement of etched pits in lead have already been made by Professor MIERS, but the work is rather beyond the scope of the present paper.

The tension side of a specimen such as that described above was of course sub-

jected to a certain amount of compression when the specimen was re-straightened after being bent. A trial was, therefore, made to see if any splitting up of the orientation could be produced by tension alone. A crystal was strained in tension in the small machine mentioned on p. 230, the stress being applied until local contraction and fracture occurred. The two halves were then re-etched and examined, and it was found that the greater part of the specimen was unchanged, but near the fracture, where, due to the local drawing out and transverse contraction, the strain must have been specially severe, a splitting up had occurred similar to that in the bent specimen described above. That is to say, in place of the original uniform orientation covering its whole surface, numerous patches with various orientations appeared.

EWING and ROSENHAIN have already drawn attention to the progressive growth of crystals which occurs after a specimen of lead or other metal has been severely strained, especially when the specimen is moderately warmed. On the suggestion of Professor EWING, the author next tried the effect of cooking these large strained crystals, that is to say, subjecting them to prolonged exposure to moderate temperatures. The first specimen tried was one similar to that shown in figs. 10 and 11. It had been strained by bending between the fingers and then re-straightening. After etching it was found that a certain area in the centre had re-crystallised, the specimen showing a similar appearance to figs. 10 and 11. It was then cooked for some hours at a temperature of about 100° C. and then re-etched. After this treatment the area re-crystallised was far greater than before. This was especially striking upon the tension side, where, as in fig. 11, only a few isolated patches had existed before cooking; afterwards, however, a large area was found to have changed, the patches with new orientations being quite contiguous, though of rather larger individual size than upon the compression side. That such a growth of new orientations was in some way due to the straining of the specimen was clearly proved by roasting an unstrained specimen. In this no change was produced, but after straining and again cooking the specimen was found upon re-etching to have to a great extent re-crystallised. It was also found that it was not necessary for the specimen to show any signs of re-crystallisation before annealing, but that so long as a certain amount of strain had been given, whether upon re-etching after this strain the orientation showed any signs of alteration or not, yet after cooking at 100° C. the re-crystallisation either continued or was started. Specimens were strained both by bending and tension, and in both cases a further re-crystallisation occurred after heating. It has been mentioned above that, when a specimen had been strained in tension, only a small area near the fracture showed any signs of alteration when re-etched, but it was found that if such a strained specimen was afterwards heated, the whole orientation could be changed. In studying this effect it was indeed found to be far more convenient to strain the specimens in tension, as the amount of strain to which they were subjected could be more easily regulated.

The method of carrying out these experiments was as follows:—A specimen about 2 inches long and $\frac{1}{2}$ inch broad by $\frac{3}{16}$ inch thick, was cut from a single crystal and given a certain amount of tensional strain in the small machine mentioned on p. 230, re-etched and photographed. It was then cooked for a short time by exposure to a certain temperature in a small asbestos-lined wooden oven, heated by an electric incandescent light. After this cooking the specimen was re-etched, first in a 20 per cent. solution of nitric acid to remove all former pits, and then in a 5 per cent. solution to produce a new set. A second photograph was then taken, and further processes of cooking, re-etching, and photographing were gone through until no further changes were visible. The photographs were taken with a 4-inch Ross lens, fitted to the front of the camera, diffused daylight being used as an illuminant. The specimen was kept immersed in a weak solution of nitric acid whilst being photographed, in order to preserve a clean surface. Care was taken to get the illumination the same throughout the series, in order that the same patches of new orientation could be recognised in the different photographs. In all the following illustrations (figs. 18 to 32) the direction of the pull was parallel to the long edges of the specimen, that is to say from top to bottom of the page.

Fig. 15 shows a specimen after straining in tension and re-etching, magnified to five diameters. The two lines seen running across had been scratched with a sharp steel point, and were originally 1 centim. apart, the straining being carried on until they were 1·5 centim. apart; the other marks were for the sake of identification. The only change visible before annealing was a small amount of re-crystallisation along the scratches, where the material was, of course, subjected to fairly severe local strain, and in two places between the lines, where it will be seen the new orientations appear as two small dark patches. Fig. 16 shows the same specimen at the same magnification after cooking for 5 minutes at 60° C. and re-etching. It will be seen that great changes have taken place; the former patches of new orientation have greatly extended, and others have appeared in various parts of the specimen. The greatest change is at the bottom left-hand corner, where a large area has re-crystallised. Figs. 17, 18, and 19 show the same specimen magnified five diameters after further successive cookings of 5 minutes', 10 minutes', and 20 minutes' duration respectively, at 60° C. The re-crystallisation has continued until practically the whole of the original orientation is altered. In fig. 18 (Plate 5), the scratches have been almost entirely etched away, and with them the small local changes which they had produced. The stage at fig. 19 seems to be a final one, and further cooking produced no further changes. In cases such as this, where the material had been subjected tc a uniform strain throughout, the patches of new orientation go right through the specimen, both sides showing a very similar pattern. In this specimen the final formation consisted for the most part of a few large differently oriented areas penetrating right through, each to a large extent split up by twin orientations in bands and patches, as can be seen in fig. 19. It will be noticed that the area of the

specimen gets gradually less in each photograph; this was due to the etching, which had to be fairly severe in order to entirely eat away the old pits and exhibit the new structure, and which was sufficient, when repeated several times, to produce a considerable change in the size of the specimen.

If we examine this series it will be seen that when once a patch with a new orientation is formed, and is surrounded by other new ones, it remains practically unaltered when subjected to further annealing. There is a slight change of outline in some cases, but this can be accounted for if we consider that the boundaries need not be at right angles to the surface, and will, therefore, change slightly as the etching solution eats down to successive depths. The structural change is entirely confined to the original orientation, which is gradually split up into numerous different ones, and when once these are formed they persist.

The slight differences of orientation which appear after straining, when the specimen is first etched, appear to some extent upon these photographs as bright bands across the original orientation. They are, however, far more clearly illustrated in the next series. Fig. 20 shows the crystal after straining in tension and re-etching magnified 5 diameters. It exhibited no signs of re-crystallisation before annealing, but the "strain bands" (as they may be called) were particularly well developed and can be clearly seen in the photograph. Fig. 21 shows the same specimen after annealing for 20 minutes at 100° C. It is evident that the strain bands have no influence on the re-crystallisation, but are swallowed up in an exactly similar manner as the original orientation. A remarkably fine development of etched pits was obtained on this specimen, and these are clearly visible even under the low magnification at which the photographs were taken.

The next experiment was to see whether any change took place in a strained crystal of lead at atmospheric temperatures. The specimen was strained in tension, and is shown (magnified to 4 diameters) in fig. 22 after this straining and re-etching. The stress had been applied until local contraction had commenced, and the strain thus produced was sufficient to cause a certain amount of re-crystallisation to be visible immediately upon re-etching. The band of patches of new orientations can be seen running diagonally across the specimen at one end. The photograph reproduced in fig. 23 was taken from the same specimen after three weeks. The specimen was kept in a small glass jar, and was simply subjected to the slightly varying temperature of the room. In fig. 23 it will be seen that re-crystallisation has continued until nearly the whole of one end has changed, the band widening out in both directions so as to fill in the right-hand bottom corner and extend further upwards.

It is clear from this series that, although a further re-crystallisation occurs at ordinary atmospheric temperature, yet it is much slower than when the temperature is slightly increased, as had been the case in the former series. It has been found in all cases that the higher the temperature at which the strained piece is kept the

quicker does re-crystallisation proceed. In some specimens which had been only slightly strained no visible change was seen until a fairly high temperature was used. One specimen was strained in tension and annealed for 20 hours at 60° C. without any visible change taking place. When, however, it was annealed for two hours at 100° C., the re-crystallisation seen in fig. 24 occurred. It is interesting to notice the large size of the patches showing new orientations in this photograph.

An important question now presented itself for solution by experiment. Is the re-crystallisation which is apparent immediately after etching in a severely-strained crystal a direct and instantaneous effect of the strain, or is it a growth which occurs during the interval of time that has elapsed between the straining and the examination? The experiments of EWING and ROSENHAIN showed that a slow progress of growth goes on at atmospheric temperature in ordinary lead after severe straining, which may result in the formation of comparatively large crystals in a severely crushed specimen after the lapse of several days or weeks, and the experiments just described show a similar slow change. In the present instance we are concerned with individual crystals in a structure of much coarser grain than was dealt with in their experiments, and with the comparatively short interval of time (some five minutes at least) which was required to prepare the specimen for examination by etching after the strain had been applied. At first it was not suspected that the re-arrangement of crystals seen after straining was other than an immediate effect of the strain, but the author has now satisfied himself that this is not the case. The re-arrangement does not occur in the act of straining like the re-arrangement (by twinning) which one can produce on straining a crystal of calcite. It occurs after the strain has taken place, during the time that elapses before the crystal is etched for re-examination, and though it requires only a short interval of time for its development, it is to be classed with the progressive growth demonstrated by EWING and ROSENHAIN and confirmed by the experiments already described in this paper.

That this was the case was first suspected from observations of the character of the re-arranged crystals. If these had been produced by successive twinning actions forming a direct result of the strain, as in the twinning of calcite, we should have expected to find the straight-line boundaries characteristic of twins, not only between the patches having new orientation, but also between these and the unchanged portion of crystal in which its original orientation was preserved. Now, although there are numerous straight-line boundaries between the patches of new orientation, demonstrating the twin relation of these patches to one another, it is remarkable that twin boundaries are not to be found between the unchanged portion of the crystal and any of the re-crystallised portions. Twins to the original orientation of the crystal would undoubtedly be formed if the action was the direct result of straining, such as has been described above, but in all cases the boundaries between any new patches and the original structure were found to be irregular in outline, and to be eaten out into channels by the etching solution; and no traceable geometrical

relation existed between the etched pits on either side of them. All these characteristics pointed to their not being twin boundaries.

One experiment directly bearing on this point consisted in examining the slip-lines which were formed as the process of straining went on. If the action was one of successive twinning, when one twin had formed the slip-lines should, as the strain proceeded, form in a new direction over its surface, and hence, as more and more strain was given, numerous systems of slip-lines running in various directions should appear, so grouped as to exhibit the twin character of the crystals produced by the earlier part of the straining. Experiments were, therefore, made to see whether the slip-lines which were formed during the application of the strain gave any such indication of a change of orientation in any part of the crystal, and thus to find out whether re-crystallisation was a direct and immediate result of the strain, or happened after the strain had been given.

As was mentioned on p. 231, a plane unetched surface, such as that obtained from the surface of the casting in contact with the air during solidification, should be used in studying the direction of the slip-lines. A specimen was, therefore, cut from an unetched single crystal, and the spiky projections were cut away from the under side. The specimen was strained by bending nearly double and re-straightening. It was known from previous experiments that after such a strain the lead always gave evidence of re-crystallisation, but, on examining the specimen under the microscope, it was found that the slip-lines extended in parallel systems all over the surface of the crystal. An area was marked by scratching upon the surface with a steel point, and was photographed. This is shown in fig. 25 (Plate 6), and although the scratches have produced a certain amount of local displacement of the slip-lines, it is obvious that these all run in directions which are uniform over the whole strained area. The surface was now etched, and the same area was again photographed (fig. 26). Numerous patches of different orientation are seen to have appeared which have no apparent relation with the slip-lines in fig. 25. It is clear, therefore, that these patches have been developed subsequent to the strain, and not in the process of straining.

In the next experiment the specimen was strained first of all in a similar manner to the above, that is to say, by bending. This developed simple uniform marking by slip-lines. The specimen was then allowed to rest for about five minutes, and was not etched. A further strain was then applied by tension. When this was applied, it was seen that upon the outlying parts the slip-lines ran in directions uniform with those already formed, but upon the middle area, which had already been severely strained, they ran in numerous directions which were parallel over certain small patches, but bore no apparent relation to the original direction. It was clear from this that over the part which was severely strained to begin with, the interval of rest had caused crystals to form differing from the original orientation, their existence being manifested by the new directions which the slip-lines assumed when

further strain was applied. Figs. 27 and 28 are photographs taken (at a magnification of 45 diameters) of a marked area on the specimen after the first and second straining respectively.

The final experiment consisted in straining a single crystal in tension until fracture occurred, and examining the slip lines. It was found that even in this case, although the surface was to some extent broken up by wavy bands, such as are described on p. 231, yet the general direction of the slip lines was constant all over the crystal, right up to the fracture. In other words, the rearrangement in structure of which the previous experiment had given evidence does not occur during the application of a strain, even when that is continued up to the limit of fracture.

From these experiments we would gather that the formation of the patches of new orientation always takes place *after* the stress has been removed and is not directly the result of a general revolution of some of the crystalline elements in the process of straining. From former experiments we have learned that in parts of the crystal where the strain has been severe the patches of new orientation appear almost immediately after the stress is removed and gradually extend from these into the remainder of the strained portion. There appears, therefore, to be no broad distinction between the change which is visible (on re-etching) almost directly after straining and that which takes place after a certain lapse of time. Such differences as are found depend on the amount of strain to which the material is subjected and the temperature at which it is kept; severe straining and a high temperature both tend to increase the subsequent rate of change of structure.

This re-crystallisation which has been shown to go on in an individual crystal must be distinguished in one important particular from that which was observed by EWING and ROSENHAIN to go on in strained specimens of lead composed of numerous crystals united together by a thin film of eutectic formed of part of the lead united with the metallic impurities. In the present case the specimens were composed of practically pure lead and the action was one of a splitting up of the originally uniformly oriented crystal into numerous differently oriented parts, the action proceeding without the aid of any eutectic. In ordinary lead EWING and ROSENHAIN found that certain crystals gradually increased in size by swallowing up their neighbours, and they have suggested that this was due to a "solution and diffusion of the pure metal constituting the crystals into the fusible and mobile eutectic forming the intercrystalline cement." It is interesting to note in this connection that in the case of a single crystal strained so as to show newly oriented parts, such parts show no inclination to grow into *one another*. When once the whole of the specimen becomes newly oriented, further cooking produces no further change. This, so far as it goes, may be regarded as in agreement with the theory of diffusion through the eutectic, as there would in the case of an originally uniformly oriented crystal be no eutectic between the newly oriented parts, and hence no such growth would be possible.

With regard to the formation of twin crystals we may, however, apply a similar

explanation in both cases. To quote from EWING and ROSENHAIN'S paper: "When a metal solidifies from the liquid state it does so by the formation of skeleton crystals starting from a great number of centres, and the arms of these skeletons continue to grow until arrested by meeting with other growths. From these arms other arms again shoot out, and so on until the entire metal is solidified; but each crystalline element as it settles into place on any of these arms must assume the proper orientation to enable it to fit in, and in the process of filling space by means of such a system of many meeting and interlacing arms the formation of a twin would be almost impossible. But when the metal crystallises after severe strain it does so by the growth of skeleton arms that must often start from a cleavage plane of an actual solid crystal, and probably the new elements deposited upon such a plane would find it as easy to assume the twin orientation as the normal."

In the present case it is exceedingly probable that practically all the patches of new orientation start from a cleavage plane, and hence the formation of twin crystals would be exceedingly common, as in fact it is.

In conclusion, the author would like to express his thanks to Professor EWING for the great help and many suggestions which he has given. The research has been carried out, under his direction, in the Engineering Laboratory at Cambridge.

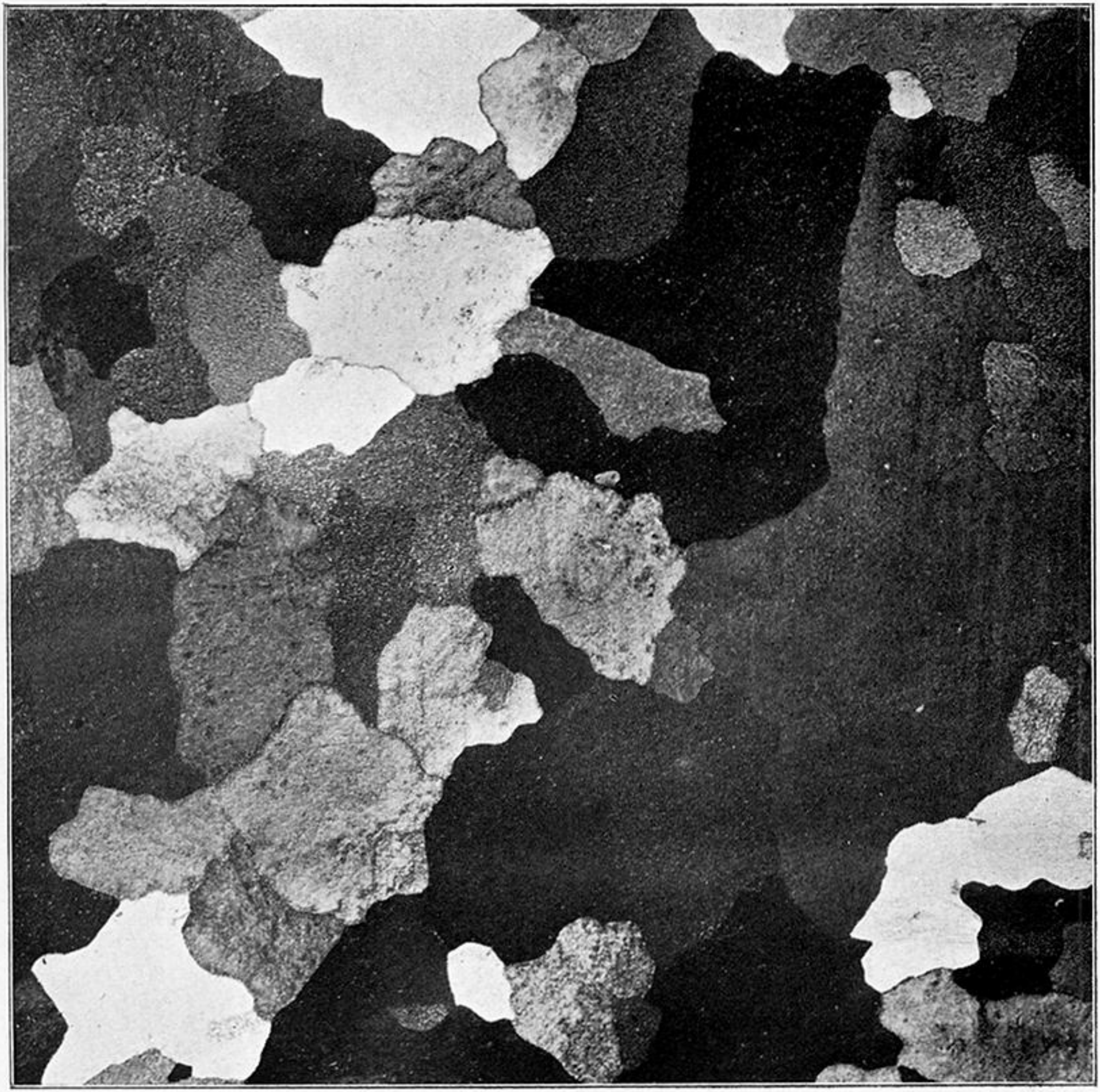

Fig. 1. Cast lead, etched ; no magnification.

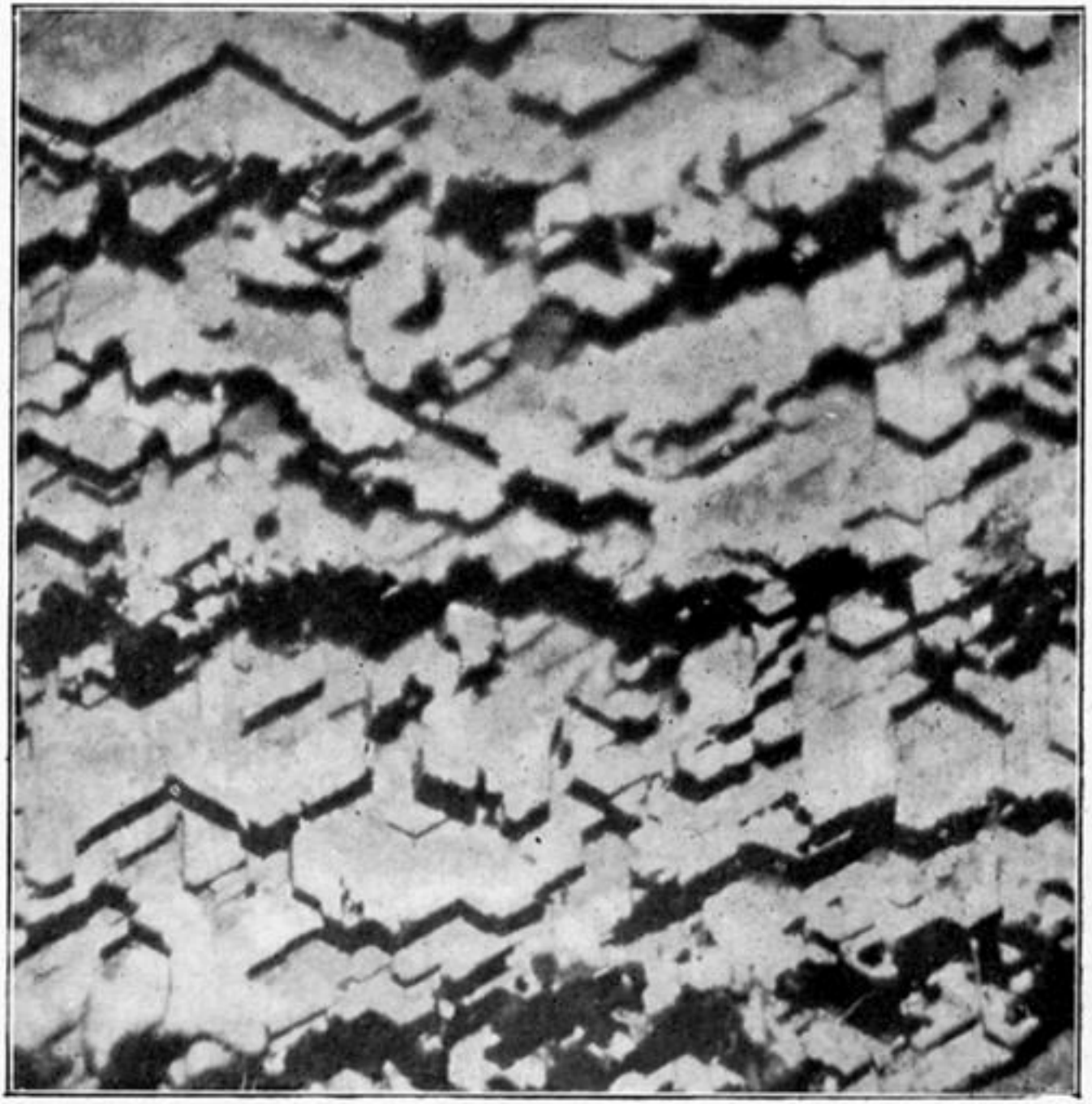

Fig. 2. Etched cast lead × 12.

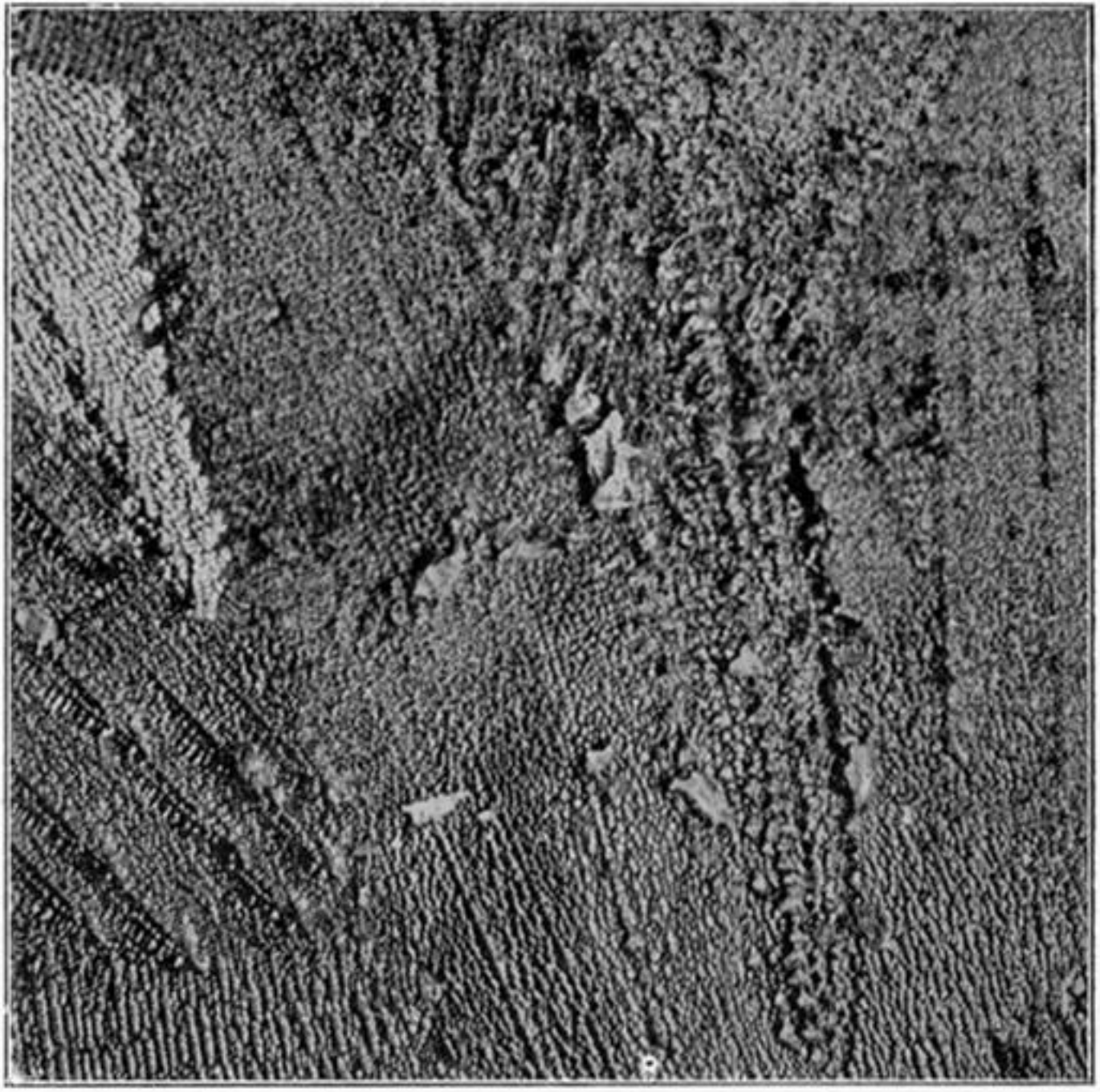

Fig. 4. Under side of casting; no magnification.

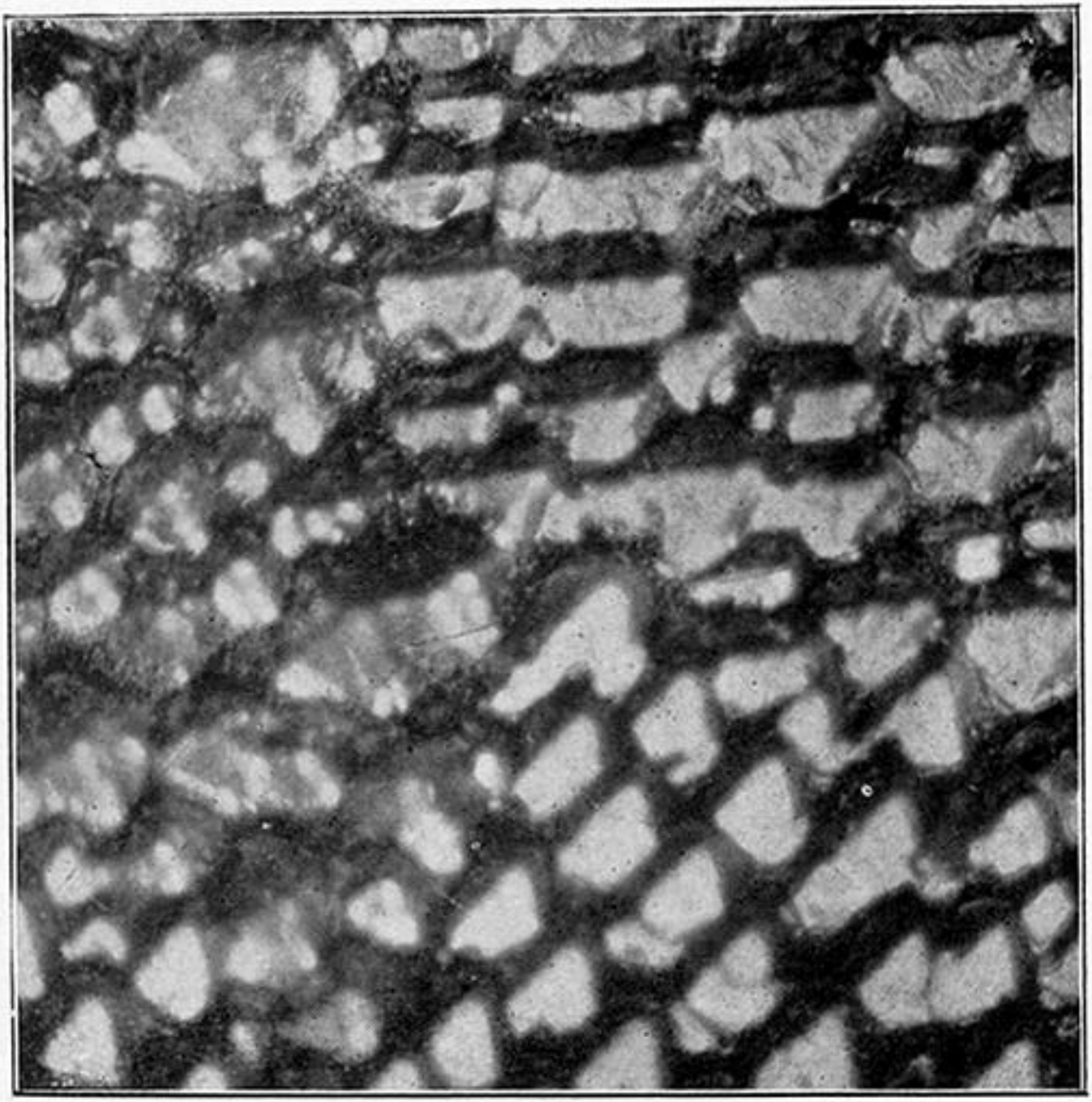

Fig. 5. Part of fig. 4 × 10.

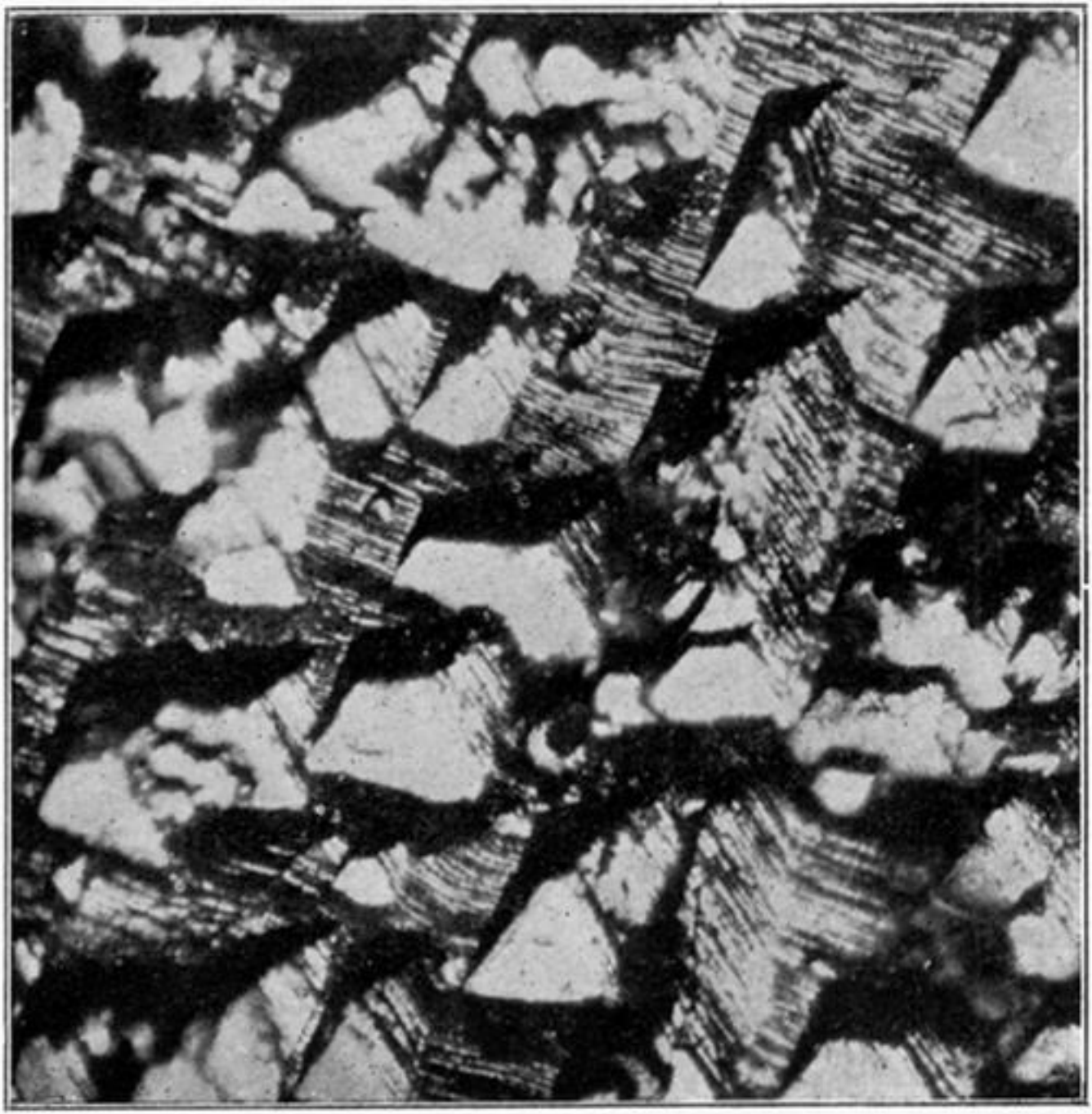

Fig. 7. Slip-lines × 45.

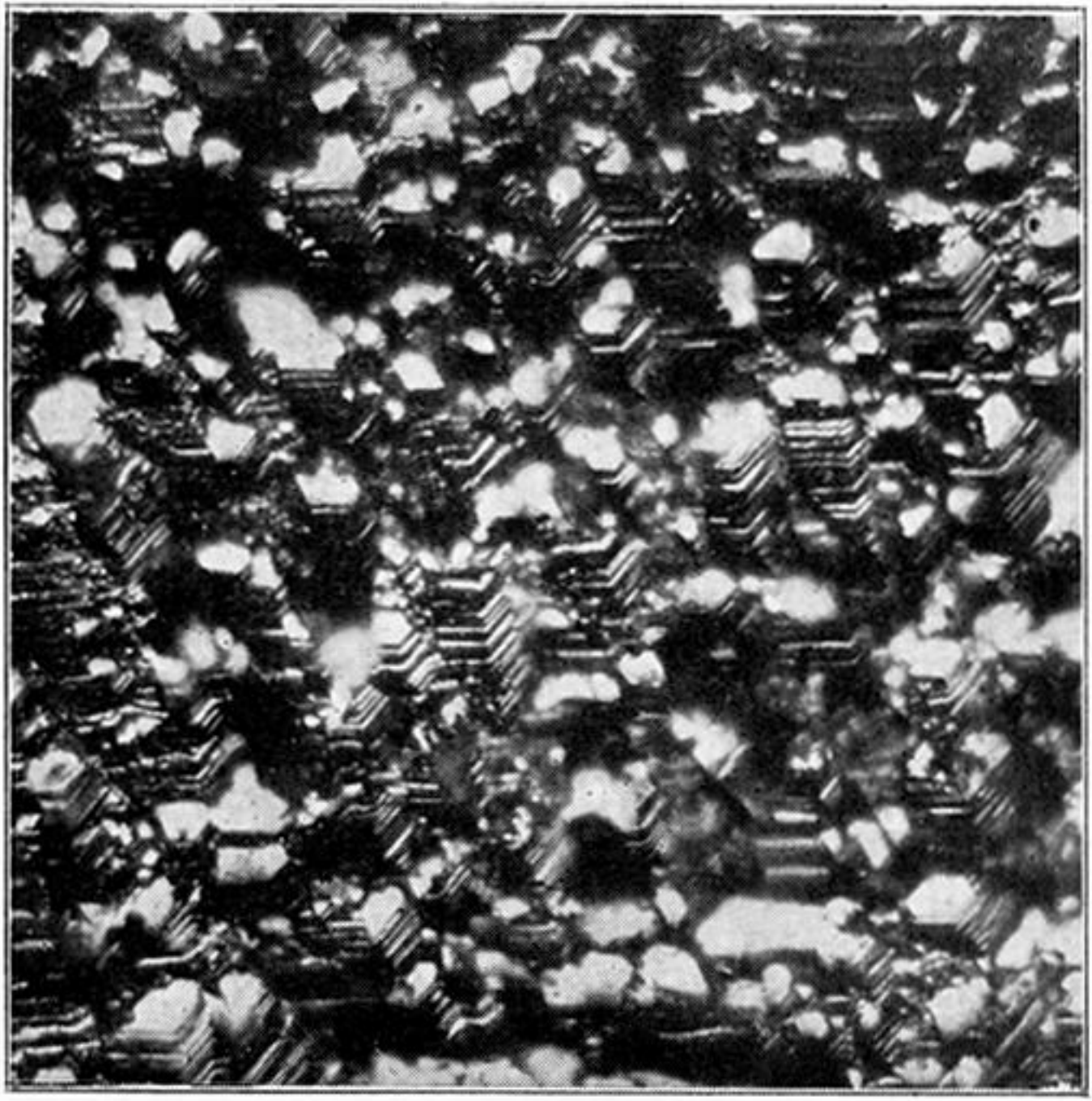

Fig. 8. Slip-lines × 100.

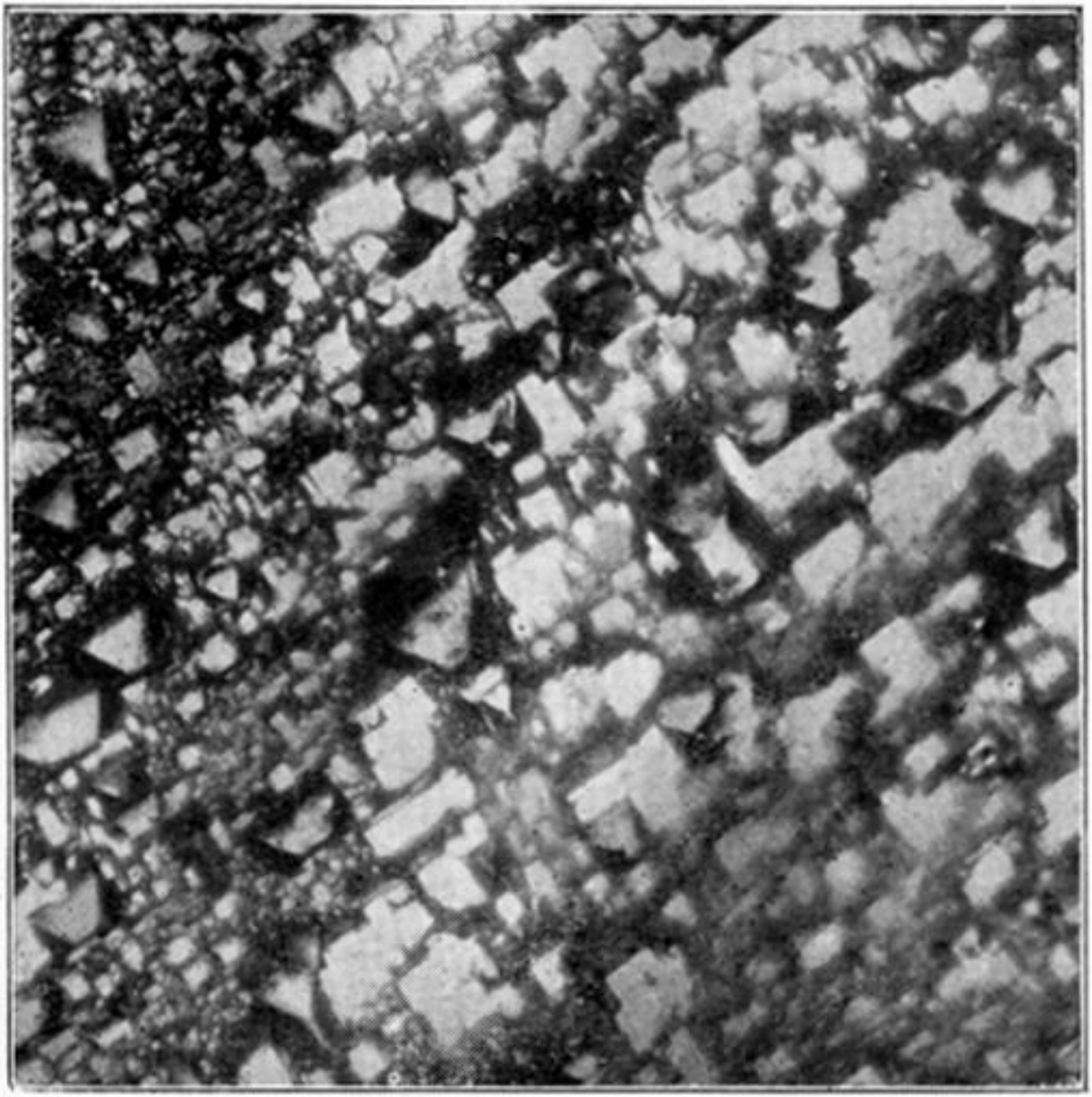

Fig. 9. Part of a single crystal after straining in tension and re-etching × 45.

Fig. 10. Strained crystal, re-etched × 6.

Fig. 11. Other face of same crystal × 6.

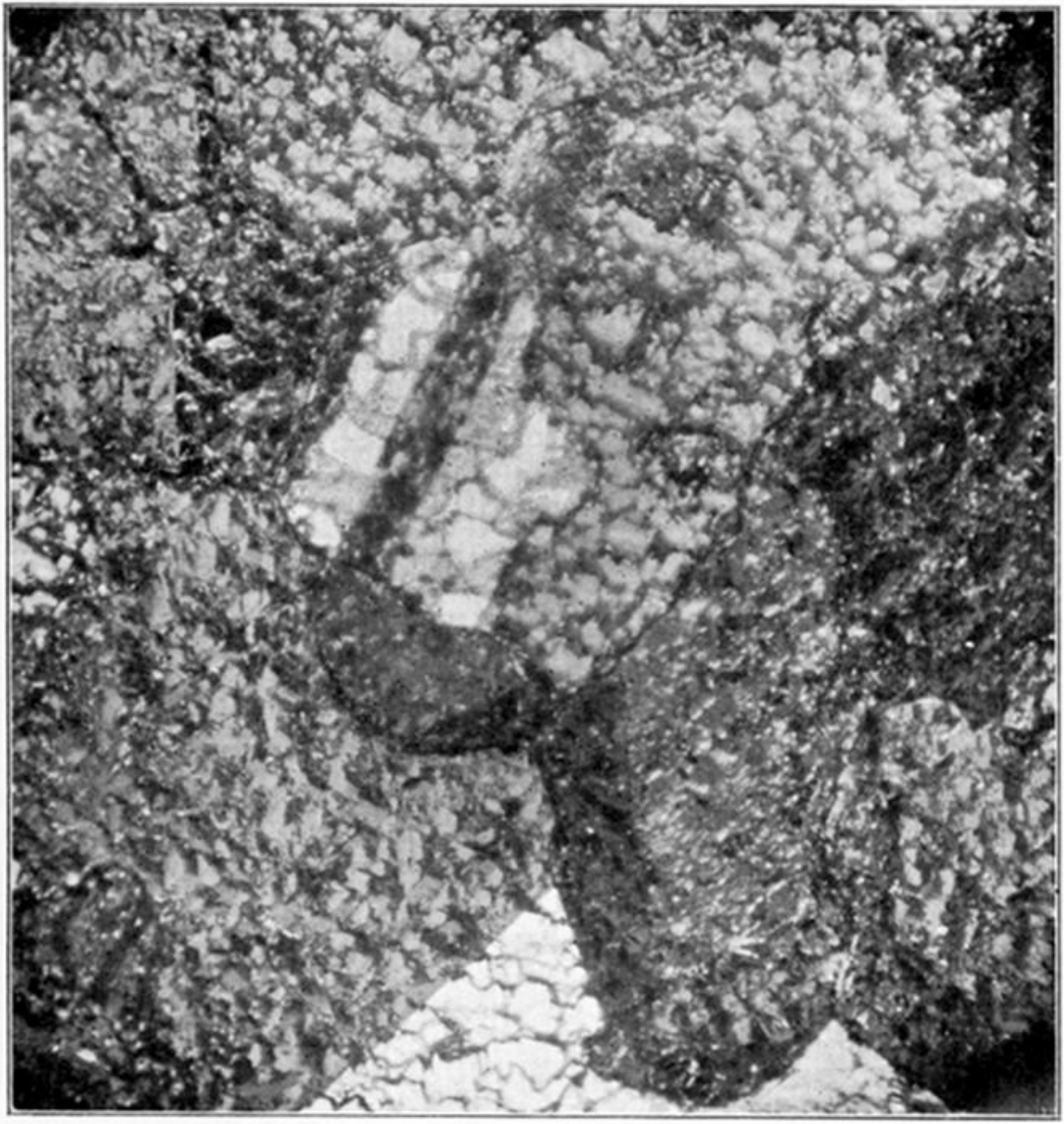

Fig. 12. Twins in strained crystal × 45.

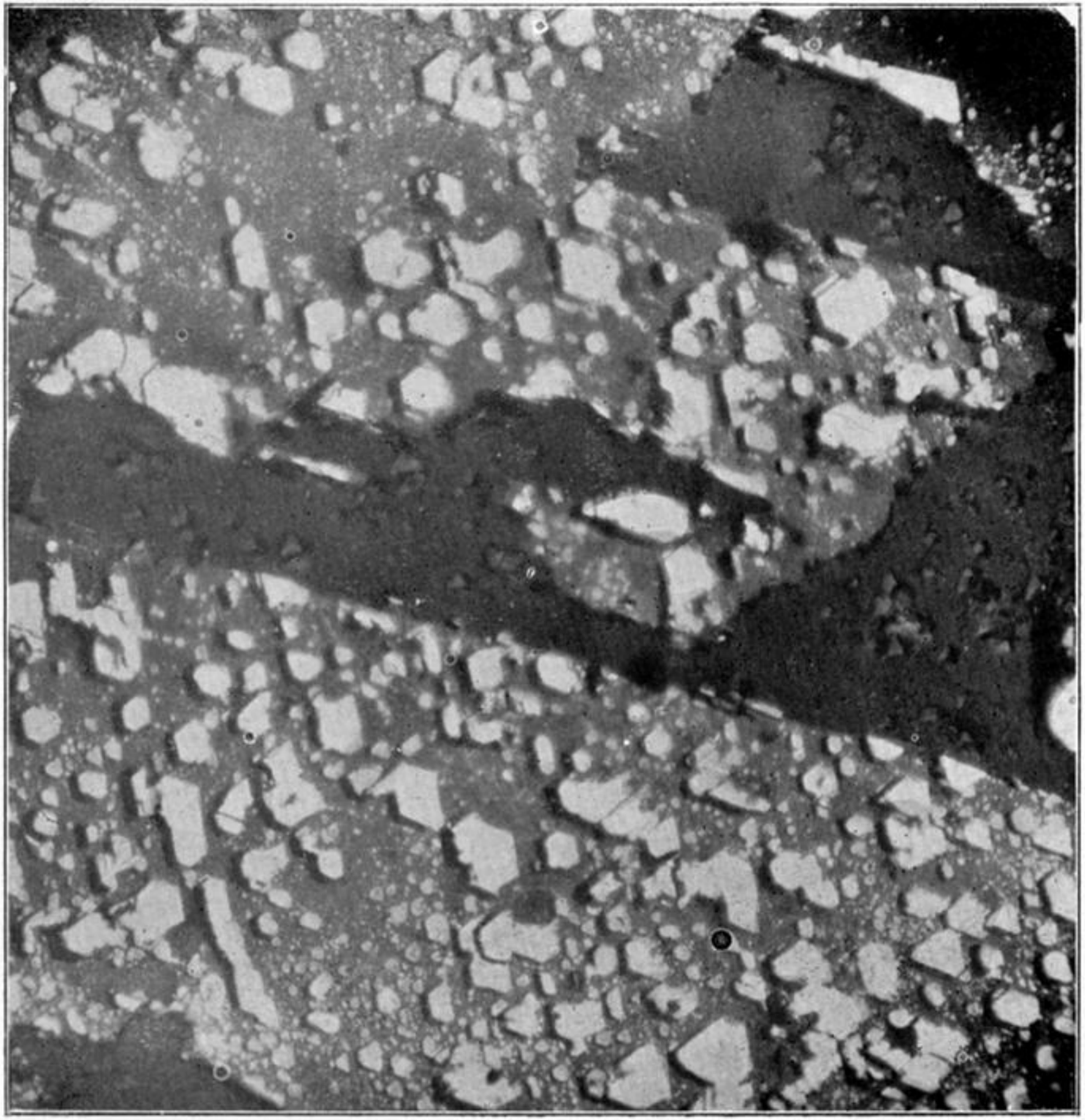

Fig. 13. Twins × 45.

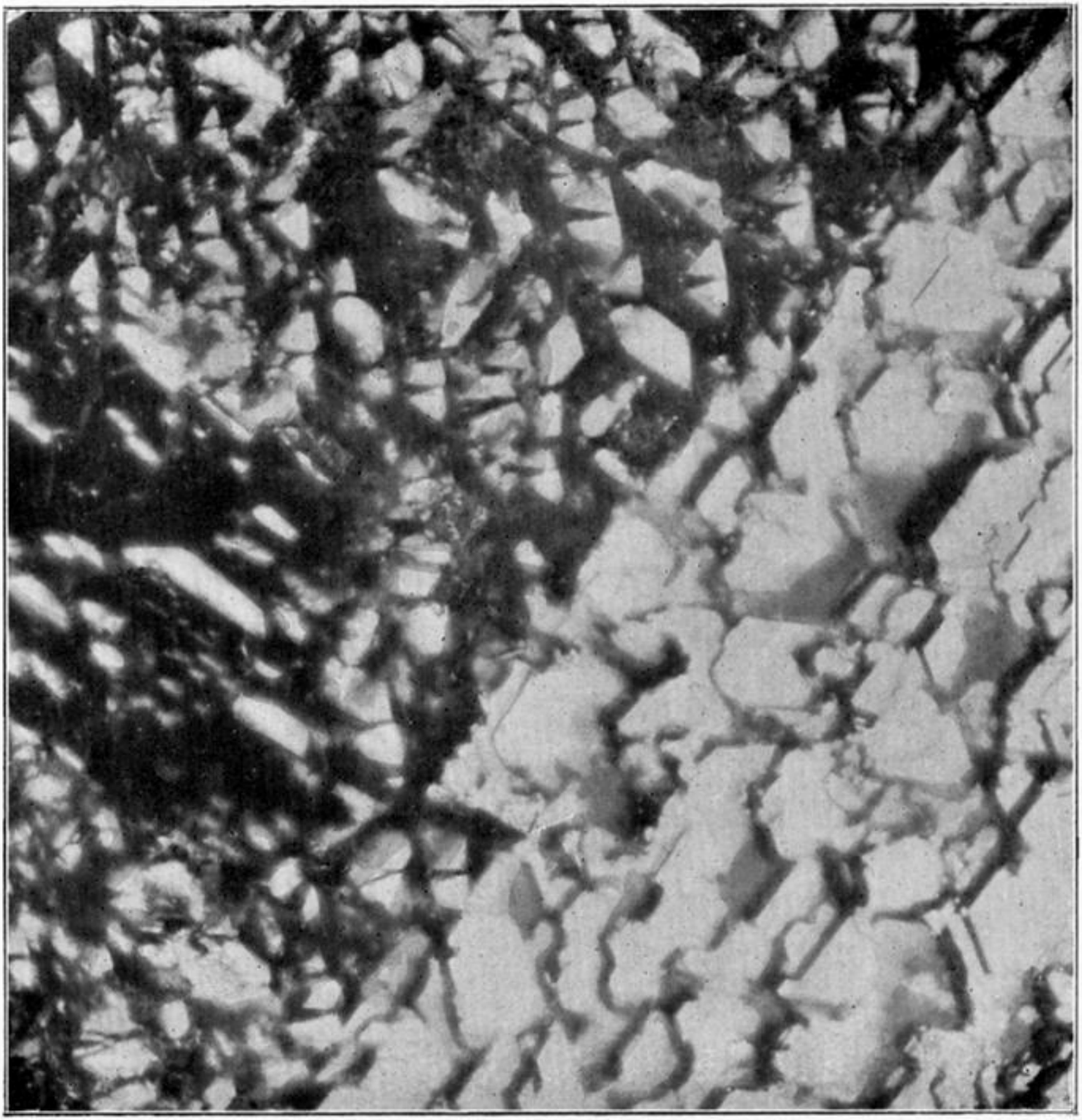

Fig. 14. Twins × 45.

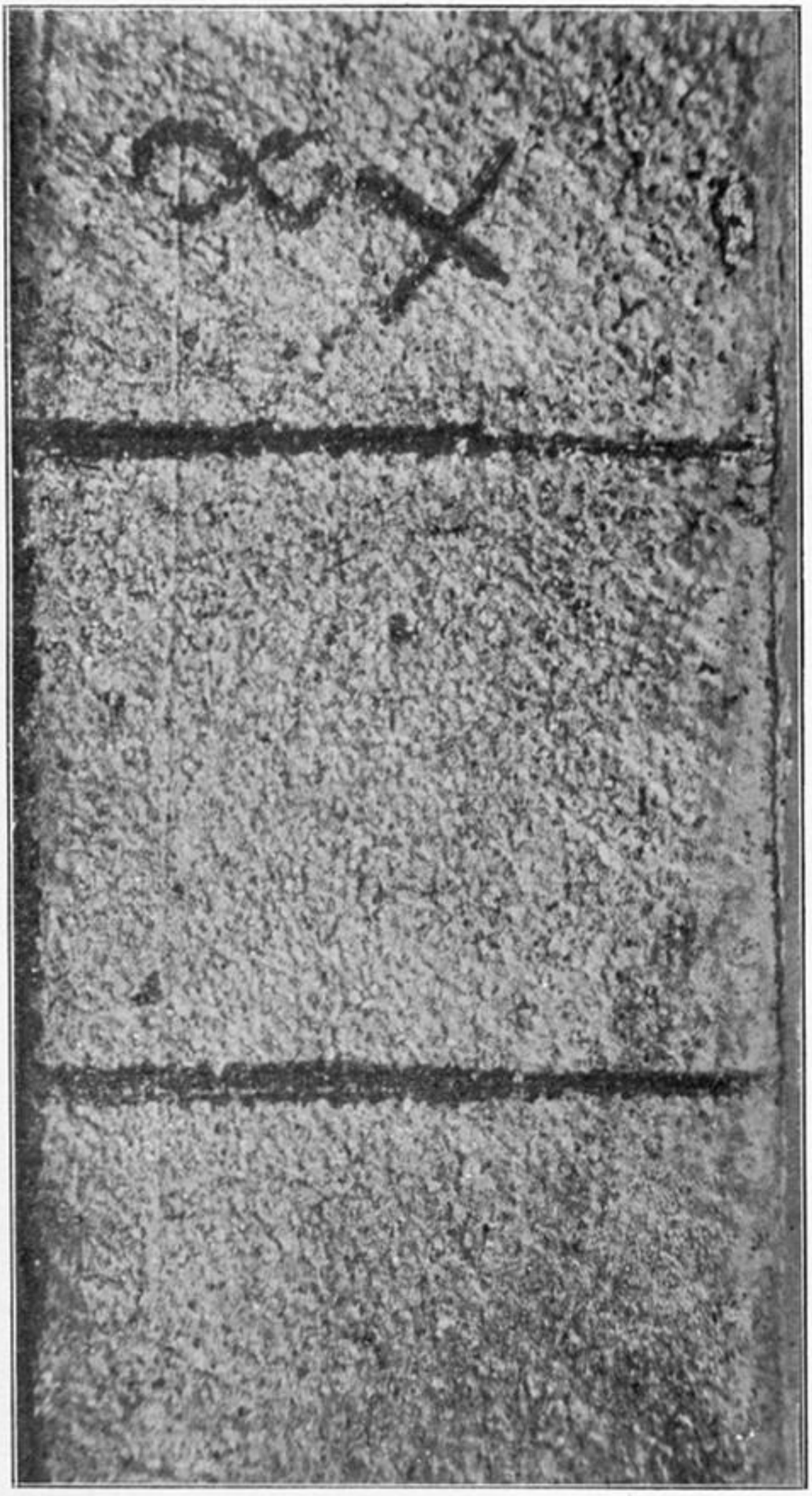

Fig. 15. A single crystal after straining in tension and re-etching × 5.

Fig. 16. Same after cooking 5 minutes at 60° C.

Fig. 17. Same after 10 minutes at 60° C.

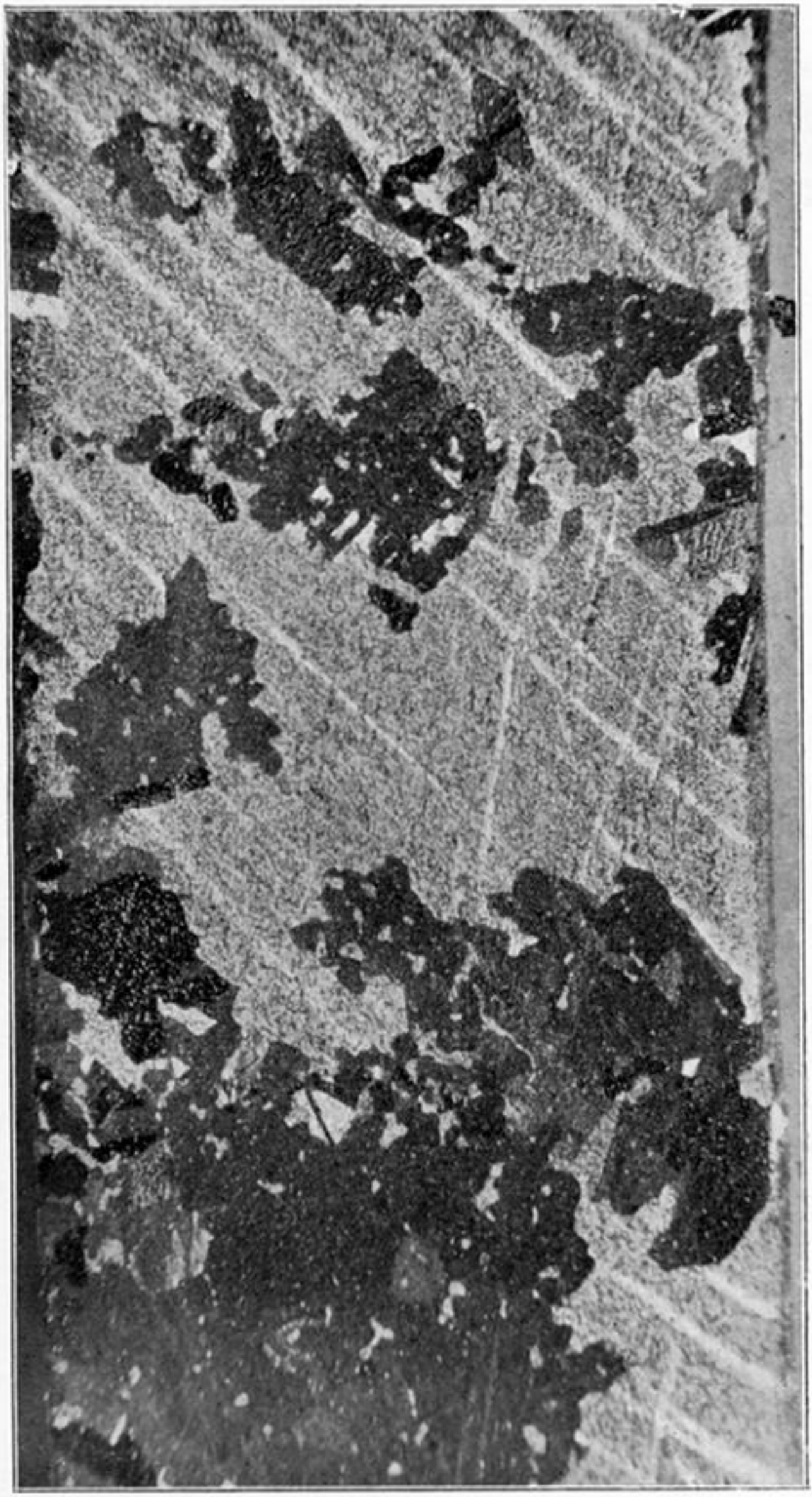

Fig. 18. Same after 20 minutes at 60° C.

Fig. 19. Same after 40 minutes at 60° C.

Fig. 20. A single crystal after straining in tension and re-etching × 5.

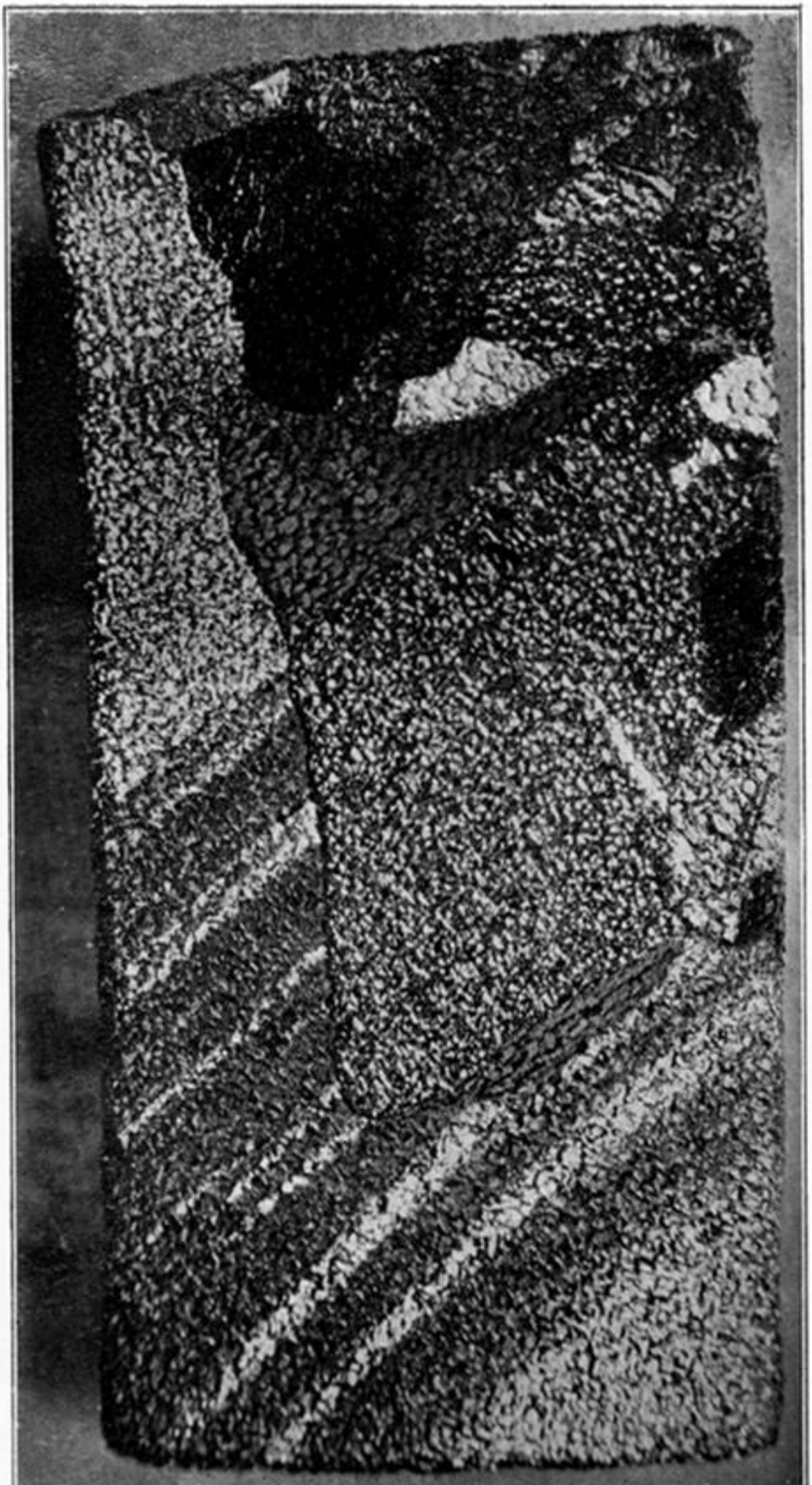

Fig. 21. Same after cooking for 20 minutes at 100° C. and re-etching.

Fig. 22. A single crystal after straining in tension and re-etching × 4.

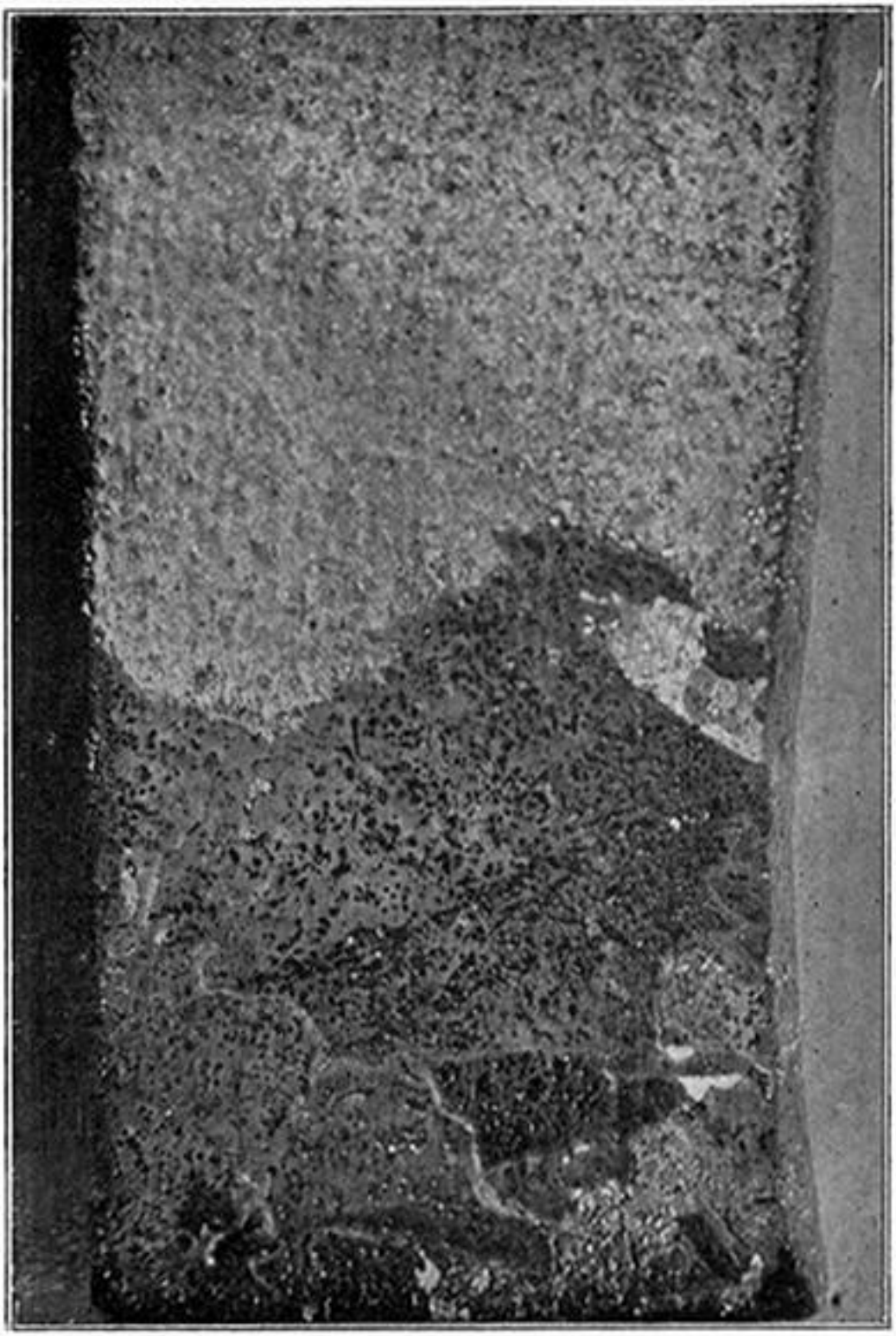

Fig. 23. Same after 3 weeks at atmos. temp.

Fig. 24. A single crystal after straining in tension, cooking 2 hours at 100° C., and re-etching.

Fig. 25. Slip-lines × 45.

Fig. 26. Same surface after etching.

Fig. 27. Slip-lines × 45.

Fig. 28. Same surface after again straining.