N MICROSCOPIC TECHNIQUES FOR
THE STUDY OF METALS

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W. Bruce Zimmerman
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ELECTRON MICROSCOPIC TECHNIQUES FOR THE STUDY OF METALS

By

W. Bruce Zimmerman

AN ABSTRACT

A THESIS

Submitted to the College of Science and Arts of
Michigan State University of Agriculture and
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MASTER OF SCIENCE

Department of Physics

1957

Approved Harry M. Bendler

ELECTRON MICROSCOPIC TECHNIQUES FOR THE STUDY OF METALS

Abstract. An investigation is carried out as to the applicability and advantages of certain replica techniques for the study of metals by the electron microscope. The four methods investigated are the aluminum oxide, silver-carbon, direct carbon, and parlodion-carbon. They are judged in particular with respect to their resolution and their efficiency in producing good replicas. They are also used to illustrate that the mode of slip in very pure aluminum is homogeneous.

A subsequent study is performed with an aluminum-silicon alloy by using the parlodion-carbon replica technique. An etchant having the property of etching the alloy along preferred crystallographic planes is used to find approximately the crystallographic orientation of a grain of the polycrystalline alloy, as well as the orientation of the precipitates in the grain. The mode of slip in the supersaturated alloy is shown to be either homogeneous or inhomogeneous, the latter occurring only when the deformation is very great. The impervious nature of the aluminum oxide film is also illustrated.

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H. Bruce Zimmerman

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I. INTRODUCTION

It is now about eighty years ago that the great optician, Ernst Abbe, discovered the theoretical limit for the resolving power of the optical microscope. No objective, however perfect from the point of view of geometrical optics, could resolve details finer than about one-half the wavelength of light in the medium in which the object was imbedded.

There was little hope of overcoming this limit until the years 1924-1926 when three discoveries opened the way for the supermicroscope. In 1924, de Broglie put forward his bold hypothesis of matter waves. In 1925, Schroedinger discovered wave mechanics by combining de Broglie's ideas with Hamilton's Analogy of Dynamics and Optics. Finally, in 1926, H. Busch discovered the lens property of axially symmetric electric and magnetic fields, and laid the foundations of geometrical electron optics.

Work on the electron microscope began soon after these discoveries, and the first results with a transmission type electron microscope were published in 1932 by Knoll and Ruska.[1] This instrument has been developed at an almost incredible rate since that time, and has brought about an increase of resolving power of about 200 times. This is just as much as its predecessor, the optical microscope, did when compared to the naked eye. It enables the human eye, as Ashtbury[2] has stated, to look "into the underworld of the molecules themselves."

This instrument provides one of the more direct ways in which physical phenomena may be observed, e.g., it can be used to examine the behavior of postulated dislocations in metals through the appearance of slip bands or lines on the metal surface, and, in certain alloys, to observe the behavior of the precipitates which can only be poorly resolved, or not at all, with the light microscope. However, for electron microscopic examination of any bulk material by the transmission method, extremely thin replicas of the surface of the material are absolutely essential. The electron microscope has been developed to such an extent that a critical examination of the micrographs obtained with it shows that the amount of detail is no longer limited by the instrument itself, but by the replicas themselves. Thus a study of various techniques for the replication of metal surfaces was thought advisable. Although this investigation was by no means exhaustive, it did include the most promising of those methods now in use.

Slip lines in 99.99 per cent pure aluminum were used as a guide in comparing the degree of resolution of the various replica techniques. The micrographs obtained illustrated, at the same time, the mode and complicated nature of the slip process. Using what appeared to be the best of these methods, precipitation in an alloy of 1 per cent silicon in aluminum was studied, and a technique to allow the orientation of the precipitates in the crystal lattice was investigated.

II. EXPERIMENTAL APPARATUS

Electron microscope. An RCA transmission type electron microscope, model EMU-2C was used. It operates with an accelerating potential of 50,000 volts, and a resolution of 20 angstroms or better is attainable. To obtain optimum results and workability, some new equipment was added, namely:

(1) an externally adjustable condenser aperture to enable work at maximum beam intensity,

(2) an externally adjustable objective aperture to allow positioning of the objective aperture without breaking vacuum,

(3) an electrostatic pole piece compensator to counteract astigmatic effects caused by the pole pieces, and

(4) a diffraction unit for limited area diffraction work and quick centering of the microscope.

Evaporator. In order to make replicas suitable for use in the electron microscope, a chamber in which a near vacuum of at least 10^{-4} mm Hg can be maintained is very useful, and in this research was absolutely necessary. In this evacuated chamber, evaporation of certain materials is performed. The evaporator used in this research is illustrated in Figure 1. It consists essentially of a working space with terminals for mechanical and electrical connections that can be closed off from the surrounding atmosphere by a bell jar, a diffusion pump, a backing pump, an electrical system, and a valving system.

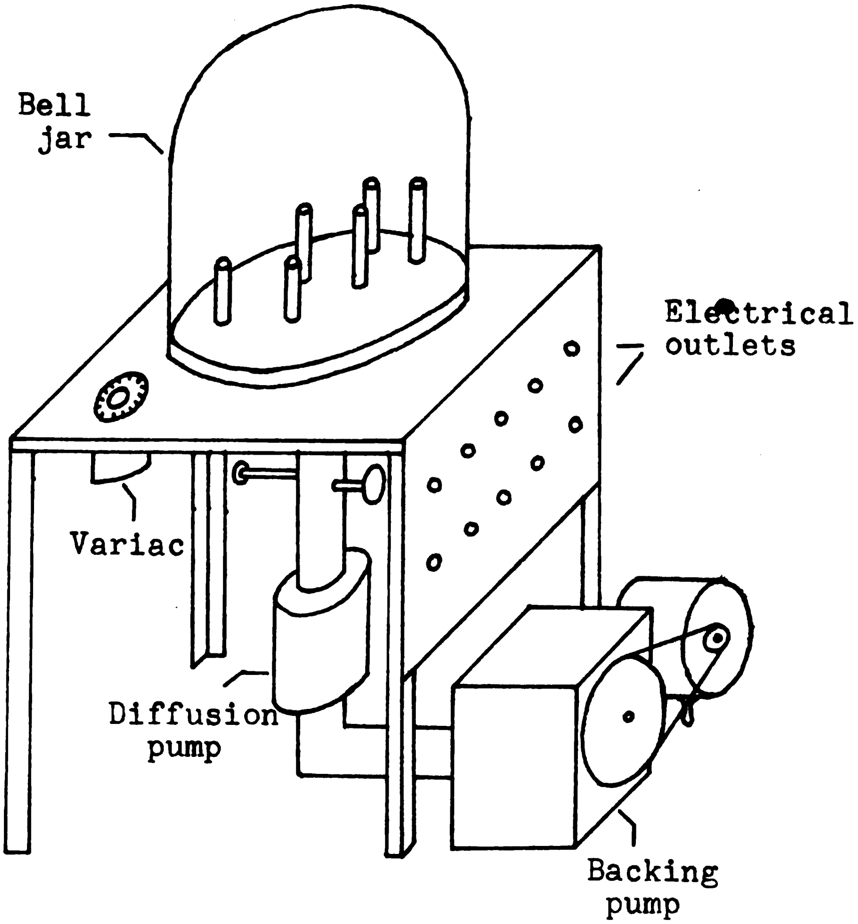

Figure 1. The evaporator

III. INVESTIGATION OF REPLICA TECHNIQUES

As stated in the introduction, single crystals of very pure aluminum were used as the specimen material. These crystals were grown without regard to orientation by the simple technique of melting a sample in air and then cooling it under a thermal gradient, as described by Heidenreich and Schockley[3] some years ago. The crystals were mechanically polished to a mirror finish, and the resultant deformed metal surface was removed by immersing in a chemical liquid, known commercially as Bright Dip, held at a temperature of about 90°C. The Bright Dip is a solution of 94 parts concentrated nitric acid and 6 parts concentrated phosphoric acid.

To produce the slip lines on the metal surface the crystals were deformed by twisting. These slip lines contain much fine detail which can only be seen with the electron microscope after a suitable replica has been prepared. Four replica methods were investigated, and compared as to their ease in making. They were:

(1) an aluminum oxide negative replica,
(2) a silver negative, carbon positive replica,
(3) a carbon negative replica, and
(4) a parlodion negative, carbon positive replica.

Aluminum oxide negative replica. This replica is formed after the technique described by Keller and Hunter[4] by anodizing the specimen in tartaric acid adjusted to a pH of 5.5 with ammonium hydroxide, the thickness of the replica being determined by the voltage. The aluminum oxide replica produced by the anodization is scored into one-eighth inch squares, and is removed by immersing the specimen in a saturated solution of mercuric chloride. The specimen is attacked by the mercuric chloride while the aluminum oxide replica is not, and thus the oxide replica squares can be made to float off the specimen surface. The replicas are moved to a cleaning bath of hydrochloric acid with a glass rod, are washed in a distilled water bath, and are then picked up on the specimen grids by sliding a grid underneath a replica and lifting upward. The replica clings to the grid and is dried by touching the edge of the grid with absorbent paper. It can now be used in the electron microscope.

If the specimen surface is called the positive surface, then the aluminum oxide replica of this surface will be a replica having a negative surface, i.e., hills on the specimen surface become valleys on the replica, and vice versa. Hence the name, negative replica.

Silver negative, carbon positive replica. This replica is formed in two stages. First a negative replica of the specimen surface is made by evaporating silver onto the surface. This is done in the evaporator as shown in Figure 2.

The specimen is held by a thread two or three inches above a tungsten basket filled with silver. A small steel rod is attached to the specimen so that during evaporation the specimen can be made to oscillate by means of a small electromagnet located to one side. The silver replica that forms can then be stripped mechanically from the specimen.

The second step is to evaporate carbon onto the silver negative replica until a dark blue color appears. The setup for this evaporation is illustrated in Figure 3. Two carbon rods are placed in holders in the evaporator about seven centimeters above the replica so that the pointed end of one presses against the blunt end of the other. The point has a diameter of about 0.8 millimeter, and it is pressed against

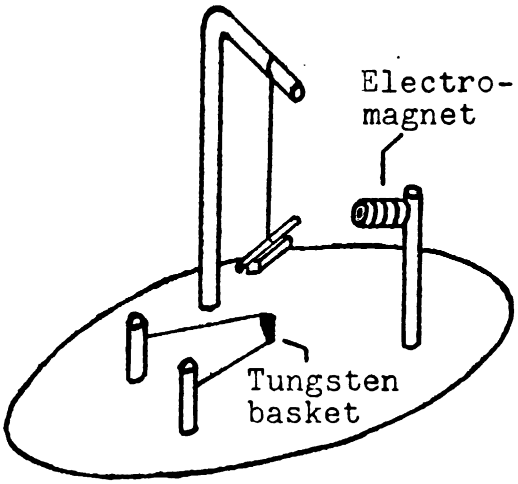

Figure 2. Setup for the preparation of the silver replica in the evaporator.

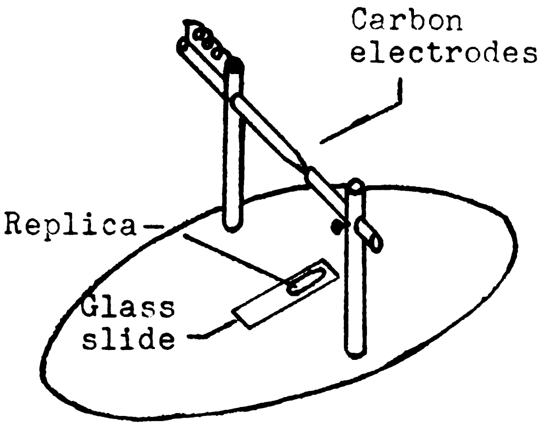

Figure 3. Setup for the preparation of the carbon replica in the evaporator

the blunt ended electrode by a spring with a force of 600 grams. After evacuation, a large current (about 50 amperes) is passed through the carbon electrodes and evaporation quickly takes place at the carbon point.

The resulting two layer replica is then cut into one-eighth inch squares which are placed on the surface of concentrated nitric acid with the silver side down. The acid dissolves the silver leaving the positive carbon replicas floating on the surface. These replicas are moved to several wash baths of water with a glass rod after which they are picked up on specimen grids.

Carbon negative replica. This replica is formed simply by evaporating the carbon directly onto the specimen surface until a lemon yellow color appears. After cutting the carbon film into squares, it can often be removed by slowly sliding the specimen into a water bath at an angle to the water surface with the replica side facing up. The carbon replicas tend to separate from the specimen and float on the water surface, ready to be picked up on specimen grids. Unfortunately, the carbon replica often adheres to the metal surface so tightly that it cannot be removed by this method. Then the only recourse is to a dilute acid which will etch away the aluminum specimen slowly, since any relatively violent action near the carbon replica will cause it to break apart. The carbon replica squares separate from the specimen, are moved to several wash baths of water with a glass rod, and are picked up on specimen grids.

Parlodion negative, carbon positive replica. This replica is also formed by a two stage process. Parlodion (4 or 5 per cent nitrocellulose in amyl acetate) is flowed onto the specimen surface and allowed to dry. A jet of steam is then allowed to play on the parlodion layer for a few seconds. The water molecules diffuse through the parlodion and form a very thin layer of moisture between the parlodion and specimen surface. Consequently, the parlodion is easily lifted from the specimen surface with tweezers, and a negative replica is obtained. It is taped to a glass slide and placed in the evaporator about three inches beneath the carbon point. A 15 second period of evaporation provides a carbon replica about 100 angstroms thick.

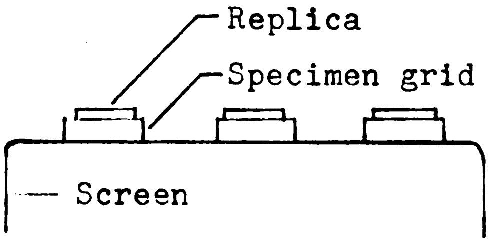

Figure 4. Replicas and specimen grids on a fine metal screen to facilitate the dissolving of the parlodion negative replica.

A fine metal screen is shaped as shown in Figure 4, wet with amyl acetate, and specimen grids placed on its surface. The two layer replica is cut into one-eighth inch squares, and they are placed on the grids with the parlodion against the grid. Since the grids are wet, the replica squares are held firmly in place. The screen is placed in a glass dish which is then filled with amyl acetate to the level of the screen. Within 15 to 30 minutes the parlodion dissolves completely leaving a positive carbon replica. Lifting the screen out, the replicas are dried by applying absorbent paper to the bottom of the screen.

Since the carbon films are so thin, they break apart easily. A carbon replica which is too thick will fall apart while the parlodion is being dissolved. If it is too thin it will often stay together during the solvation of the parlodion, but will break up on drying. This occurs when the amyl acetate film, which is stretched across the grid openings, breaks as the liquid is drawn off by the absorbing material. This action can be minimized by placing the screen with the grids and replicas, after the parlodion has been dissolved, in a solvent of amyl acetate with a low surface tension, such as methanol, and then drying.

Shadowing. A replica which is made in any of the ways described above is often so transparent to the electron beam in the microscope that contrast is almost non-existent. To improve contrast, a material with a high scattering coefficient is evaporated onto the replica at an angle of about 25° to 45°, the angle depending on the texture of the specimen surface that was replicated. Thus raised portions of the replica cast shadows, and a high degree of contrast is obtained.

In this work an oxide of tungsten was used with much success. A tungsten filament is shaped into a V, and then annealed in a vacuum by simply passing current through it. The filament is heated in this way to a bright red two or three times until, upon cooling, it appears shiny. It is then heated in air to a dull red so that a layer of tungsten trioxide forms on the surface. When the filament appears lemon yellow in color the heating is terminated, and it can now be used for shadowing.

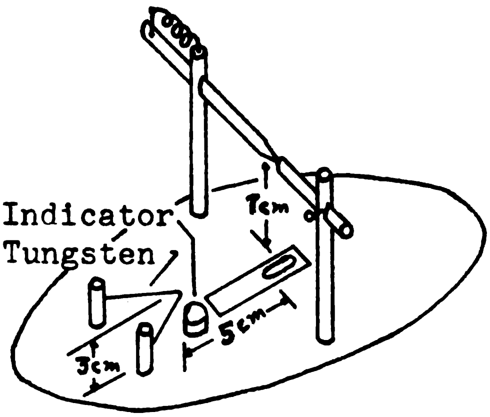

Figure 5. Setup for preshadowing and carbon evaporation.

The shadowing is carried out in the evaporator in a vacuum of about 10^{-4} mm Hg by heating the filament. The yellow of the tungsten trioxide changes to a dark color, indicating that perhaps tungsten dioxide has formed, and then evaporates quickly. Experience soon indicates the amount of shadowing to use, and, as a guide, the interference rings that spread out from the filament as the oxide evaporates can be used by placing a smooth surfaced metal near the filament. The setup is illustrated in Figure 5.

Although shadowing means another pump-down to vacuum, this can be bypassed whenever a negative intermediate replica is used by preshadowing it, i.e., by shadowing the negative replica first, and then immediately evaporating the carbon. The shadowing agent has always been found to adhere to the carbon replica and not to the negative intermediate replica.

Conclusions. Typical micrographs obtained from replicas made as described above are shown in Figures 10-14. The aluminum oxide replica was found to be the easiest to prepare, and often did not require shadowing. But Figure 10 shows that it gives a resolution inferior to that of the parlodion-carbon and direct carbon replicas. In addition, the specimen was ruined during the removal of the replica, and the replicas tended to be dirty.

The silver negative, carbon positive replica has the advantages of ease in stripping and not damaging the specimen surface, but did not lead to results comparable to the other techniques, as can be seen from Figure 11. A factor which may not have been adequately controlled was the heating of the specimen during the evaporation of the silver. This heating can be quite severe, and in some cases, cause the silver to assume a structure completely masking the structure of the replicated surface.

An example of a micrograph obtained from a carbon negative replica appears in Figure 12. This replica appeared to be capable of the greatest resolution of detail, but offered some difficulty in stripping. This method also often spoiled the specimen surface for further immediate replication, but destruction was not severe and a light repolishing would restore a smooth surface.

The parlodion negative, carbon positive replica technique was found to give a high yield of usable replicas and to preserve the specimen surface. Its resolution is comparable to the direct carbon technique, see Figure 13, and only at very high magnifications could considerable background structure be found. An additional advantage of this method is the ability of the parlodion to replicate faithfully relatively deep pits in the specimen surface, as will be seen in Chapter V. For most purposes this technique seemed to offer the obtaining of good results more consistently than the others, and was therefore used in the subsequent investigation.

It must be noted that, in general, the preparation of a carbon replica is ticklish and often time consuming. However, the carbon replica has the necessary properties of being unaffected by and transparent to the electron beam. In addition, it has the desirable characteristics of remaining unbroken for a great length of time once prepared, being unaffected by acids, and being able to hold extracted particles for diffraction work as well as photography.

IV. SLIP PHENOMENA IN ALUMINUM

An important element in the description of the process of plastic deformation in metals resides in the concepts of the theory of dislocations. One of the more direct ways of observing the behavior of the postulated dislocations is through the appearance of slip bands or lines at the metal surface. These disturbances represent, according to current theory, the emergence of successive dislocations that have propagated through a metal crystal relative to another, i.e., in plastic flow or deformation.

Slip in metals appears to take place according to two different modes. Following Wilsdorf[5], these modes are described as either homogeneous, i.e., slip on successive atomic planes, or inhomogeneous, i.e., slip on a single atomic plane. These two modes of slip are illustrated in Figure 6.

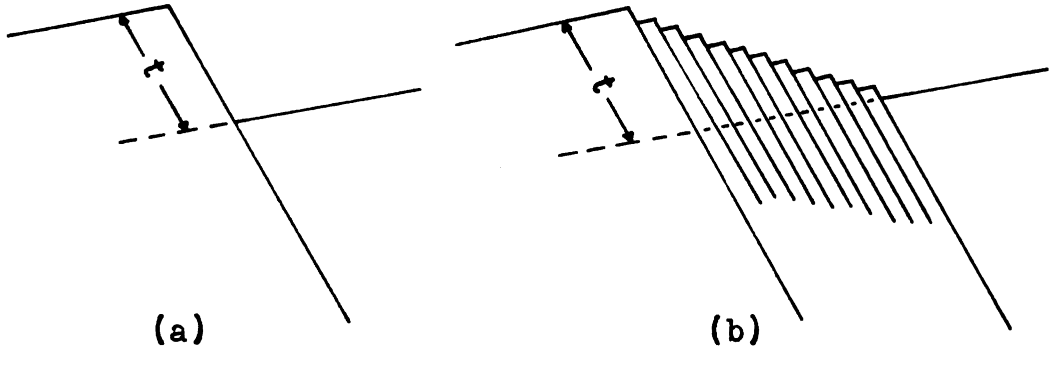

Figure 6. Two modes of slip: (a) Inhomogeneous slip of a distance t on one plane; (b) Homogeneous slip on a number of neighboring planes, with a total slip distance of t.

The slip surface is often an important crystal plane of dense packing. In very pure aluminum, the slip surfaces are the $\{111\}$ family of planes, although at high temperatures slip also occurs on $\{100\}$ and other planes.

The micrographs used in comparing the various replicas in the previous chapter also serve to illustrate the slip phenomena that takes place when very pure aluminum is deformed at room temperature. In the micrographs of Figures 10 and 11, only a faint trace of homogeneous slip can be seen. But the micrographs of Figures 12-14 clearly show that the slip is homogeneous.

That the dynamics of the slip process can become quite complicated is shown very clearly in Figures 13 and 14. In the former, a checkerboard effect is shown, formed by the intersection of two different slip systems, i.e., the intersection of two different members of the $\{111\}$ family on which slip has occurred. The latter indicates a slip band which appears to have essentially reflected itself with respect to a median plane. This is illustrated schematically in Figure 7.

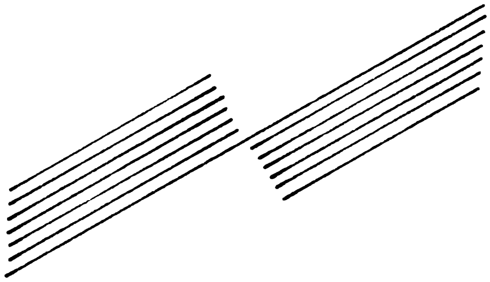

Figure 7. Schematic illustration of the slip band shown in the micrograph of Figure 14.

It may be noted that most of the micrographs show a pronounced network of lines, or an "orange peel" structure. To discover whether these lines correspond to ridges or valleys on the original specimen, the following type of reasoning must be performed. Consider the micrograph in Figure 20. A particle which is not actually replicated, such as a dust or an extracted particle, is first picked out to discover the direction in which projections on the replica cast shadows. Since the micrograph being considered was made from a preshadowed parlodion-carbon replica, the shadowing operation was performed on the parlodion negative replica. The extracted silicon particles are seen to cast white shadows toward the lower left hand corner of the micrograph. However, the network of lines being considered cast white shadows toward the upper right hand corner of the micrograph, i.e., in the opposite direction from that of the extracted particle shadows. Thus, since the silicon particles are projections, the network of lines must be valleys on the parlodion replica. This is illustrated schematically in Figure 8. But the network of lines was actually replicated from the original specimen. Therefore the network of lines correspond to ridges on the original specimen.

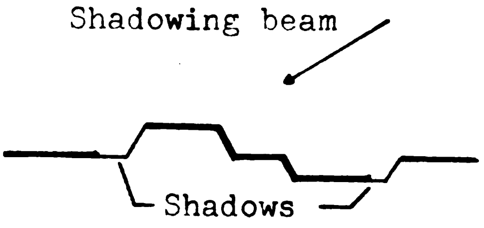

Figure 8. Schematic illustration of the shadowing of a projection and depression.

The "orange peel" structure was first thought to outline true mosaic blocks, or, in other words, to actually be a subgrain structure. But grain boundaries represent regions of higher energy than that of the grain, so it would be expected that these regions would be attacked by the chemical etch more vigorously. But this would make the "subgrain boundaries" appear as valleys on the specimen when replicated, and, as shown above, this is not the case. It is also noted that these ridges have no apparent effect on the slip lines, as they would if they were actually subgrain boundaries. Therefore, it would appear from these observations that these ridges are not subgrain boundaries.

Hirsch[6] has also presented a theoretical calculation of the density of dislocations that such "subgrain boundaries" would imply, and obtained results inconsistent with observation.

Very recently the conditions under which such ridges or "boundaries" are formed was examined in considerable detail, and the results indicate that they are a function of the etching characteristics of the etchant used to polish the surface.[6]

After the polishing of many specimens by the Bright Dip chemical polish, the Bright Dip deteriorates and forms etch pits on the specimen surface as well as polishing it. A typical micrograph of such an etch pit is shown in Figure 15.

V. STUDY OF PRECIPITATION PHENOMENA

As the specimen material, an alloy of 1 per cent silicon in aluminum was used. The phase diagram of the aluminum side of this alloy is shown in Figure 9.

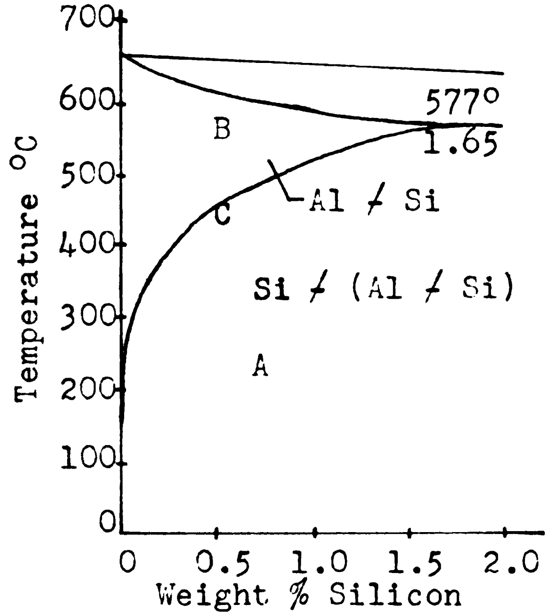

Figure 9. Phase diagram of the aluminum end for the aluminum-silicon alloy

Solution of the silicon in the solid aluminum is caused to take place by heating the alloy to a temperature in region B. During this solution heat treatment, the silicon atoms diffuse throughout the aluminum, and it is thought that small groups of these atoms continually form together and then disintegrate. Next the alloy is quenched by dropping it into water at room temperature. The small groups, or "knots" of silicon atoms are caught in the act of forming, and the lowered thermal agitation prevents their disintegration. At the same time many of the silicon atoms not in these groups are caught in the aluminum matrix, and a supersaturated solid solution is obtained. The alloy at this stage is quite soft and is almost as workable as pure aluminum. Hence it is at this point that the alloy is rolled to a desired thickness--about ¼ inch in this work.

The alloy is now aged by heating it to a temperature below that of curve C (anywhere within the range from 260°C to 500°C). The atoms in the knots become rearranged in a more regular pattern and form nuclei for the precipitate phase. As the aging is continued, general precipitation occurs throughout the matrix.

To investigate this precipitation visually, a magnification of about 3000 times is needed. Such a magnification with good resolution can only be obtained, at present, by the electron microscope.

A specimen about one inch long and one-quarter inch in cross section is cut from the original plate. A face of the specimen is mechanically polished by rubbing it on various grades of abrasive paper, moving from the coarser to the finer grades, and then lapping it on a polishing wheel using an abrasive of aluminum oxide. It is finally polished chemically by agitating it in Bright Dip at 90°C for 15 seconds. The polished face is replicated by the parlodion-carbon method, and shadowing was performed with tungsten dioxide at a 1:2 ratio. Figure 16 shows a typical micrograph obtained from such replicas. It is immediately seen that there are silicon precipitates of two types present: (1) pencil-shaped particles ranging in length from approximately one-half to one micron and in thickness from about one-eighth micron to one-quarter micron, and (2) thin plate-shaped particles with an average diameter of about one micron and a thickness of the order of 100 angstroms (see also Figure 24).

The orientation of the pencil-shaped particles is most interesting. Most of these particles are arranged in two directions which are at right angles to one another. However, by close scrutiny, a number of particles can be found lying in two directions different from the horizontal and vertical directions. These new directions make angles of 45° with the old ones. The different directions taken by the particles in this micrograph thus number four, and no more than this number of directions was ever found on other micrographs.

Unfortunately, a micrograph like that of Figure 16 does not enable us to tell on what crystallographic plane of the specimen we are looking. Therefore, a solution consisting of 9 parts concentrated hydrochloric acid, 3 parts concentrated nitric acid, and 5 parts distilled water was applied to the specimen surface after the Bright Dip treatment. This solution etches the aluminum on the $\{100\}$ planes at various places on the specimen surface. The resulting pits have faces which are $\{100\}$ planes and allow that grain in which a pit is located to be oriented crystallographically. Such pits are replicated nicely by the parlodion-carbon technique.

A steroscopic pair of micrographs of a pit are illustrated in Figure 17. (When first viewed the etch pit may appear to rise up at the viewer, but to obtain the true picture it should be made to fall away from the viewer.) Since the pit faces are $\{100\}$ planes, the plane surface around the pit can be said to be very near one of the family of $\{110\}$ planes.

It is known that at room temperature aluminum and its alloys slip on $\{111\}$ planes in $\langle 110\rangle$ directions. Since the dislocations which form in a slip process are regions of high stress concentration, they should aid nucleation. Thus we might suspect that nucleation should take place in these dislocations, and observe the silicon particles to have grown on $\{111\}$ planes in $\langle 110\rangle$ directions. If this is true, then the silicon particles on the $\{100\}$ faces of the etch pit in Figure 17 should form acute angles of about 55° with these faces, and those about the pit should form angles of about 35° or 90° with the $\{110\}$ plane of the surface. Viewing the pair of micrographs stereoscopically, we see that this appears to be the case.

How the pencil-like silicon particles orient themselves with respect to actual slip lines is shown in Figure 18. A specimen in which precipitation has occurred is quite difficult to deform manually, and the slip lines obtained were not pronounced enough to indicate whether the slip was homogeneous or inhomogeneous.

A specimen in which precipitation has taken place can again be caused to become supersaturated by reheating it to a temperature in region B of Figure 9. When such a sample is placed in an oven in which the temperature is held at 565°C for five minutes, quenched, polished, and replicated, a micrograph like that in Figure 19 is obtained. It shows that even in this short time much of the silicon has gone into solution. Figure 20 shows the state of the alloy when the solution treatment is continued for one hour. The small particles of silicon have completely disappeared, and some relatively large particles, which evidently do not go into solution, have formed. These clumps of large particles were found to be quite sparse and to form most often on grain boundaries.

If a specimen which has been solution heat treated to form a supersaturated solution is subsequently polished, strained by twisting, and replicated, then the micrograph of Figure 21 results. It shows that the slip in the supersaturated alloy is homogeneous, and also illustrates how well the parlodion-carbon replica technique resolves the fine elementary slip lines. However, if such a specimen is greatly deformed, slip which appears to be essentially inhomogeneous can result. This is illustrated by the stereoscopic pair of micrographs in Figure 22.

When a specimen in which precipitation has occurred is polished chemically, and then is solution heat treated for an hour before being replicated, a micrograph like that shown in Figure 23 is obtained. The micrograph shows that the solution treatment of the specimen has not affected the silicon precipitates on its surface. This indicates that if the aluminum oxide film has grown during the solution treatment, it has grown down into the specimen and not up from it. That is to say, oxygen molecules must have diffused through the oxide film to react with the aluminum underneath.

The stereoscopic pair of micrographs of Figure 24 illustrate interesting interference phenomena that occurs when the thin plate-shaped precipitates happen to be oriented to the electron beam in such a way that Bragg diffraction takes place. These interference lines appear as dark lines in the precipitates, and are known as extinction contours. They occur with a crystal of variable thickness or bent crystals. The fact that the contours are continuous indicates that the plate-shaped silicon precipitates are essentially single crystals. The extreme thinness of the plate-shaped silicon precipitates can also be best appreciated with this stereoscopic pair of micrographs. The extinction contour phenomena has been treated by the use of Brilliouin zones by Heidenreich[7].

VI. CONCLUSION

It has been shown that the parlodion negative, carbon positive replica technique can be used to replicate a microscopically rough surface and to also provide good resolution. Although it is doubtful that the micrographs obtained show a resolution any better than 50 angstroms, this quality of resolution is sufficient for a great deal of work. It is very advantageous to have a technique which can be depended upon to almost always give good usable replicas if a prescribed procedure is followed.

The modes of slip in very pure aluminum and in a supersaturated aluminun-silicon alloy were illustrated. The mode of slip in pure aluminum was strictly homogeneous, while that in the supersaturated alloy was either homogeneous or inhomogeneous, the latter occurring only under large strain.

By use of an etchant which attacks the metal being investigated along certain known crystallographic planes, it was shown how the orientation of a particular grain of the polycrystalline metal could be found. It was further shown that a quick qualitative orientation of the precipitates in an alloy could be made by the use of such etch pits. Such an investigation as this could be carried out on any metal for which there existed such an etchant.

In 1954 it was reported by Wilsdorf[8] that the precipitates in an alloy of aluminum and copper formed traces (i.e., rows of similarly oriented, closely spaced precipitates) which were interpreted as links in a dislocation network of the type postulated by Mott[9]. It was thought that perhaps similar traces could be found in an aluminum-silicon alloy. However, none were found.

VII. MICROGRAPHS

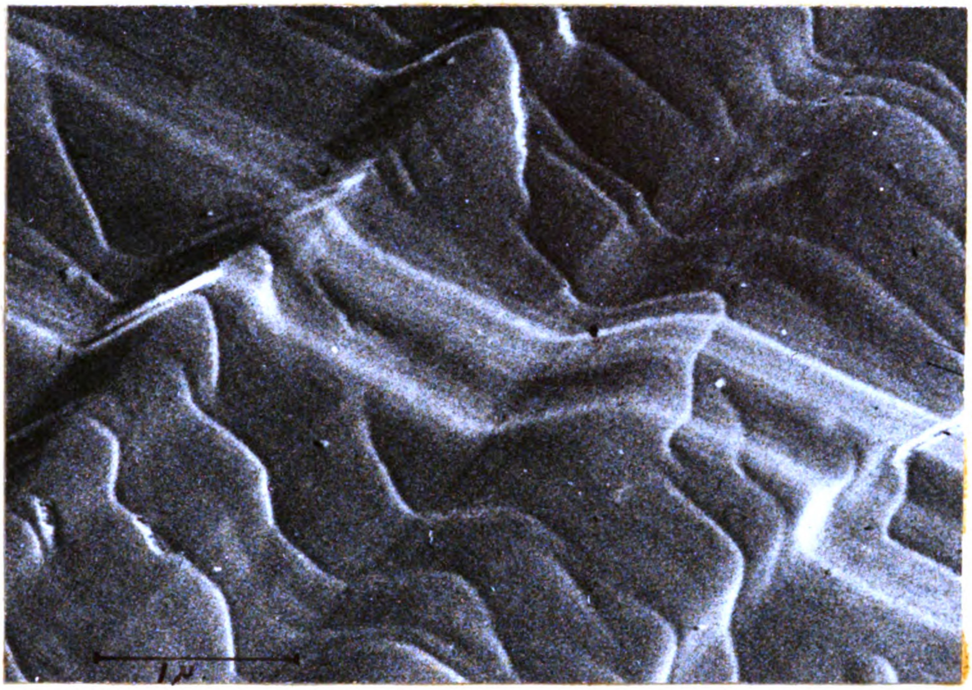

Figure 10. Micrograph of slip in aluminum photographed from an aluminum oxide replica. Magnification 27,000 X.

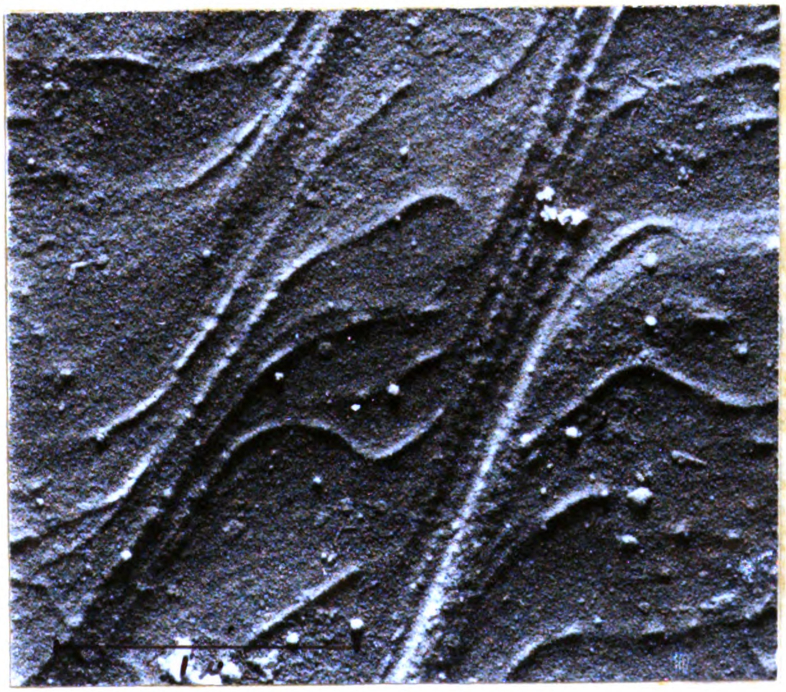

Figure 11. Micrograph of slip in aluminum photographed from a carbon replica made by the silver-carbon method. Magnification 40,000 X.

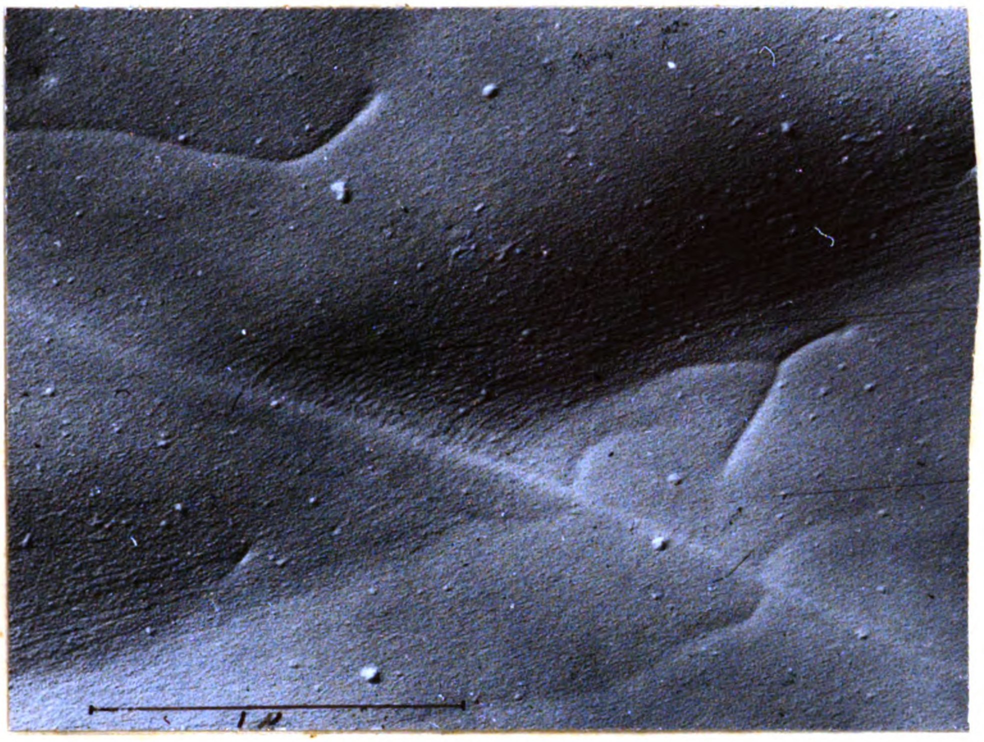

Figure 12. Micrograph of slip in aluminum photographed from a carbon replica made by the direct carbon method. Magnification 50,000 X.

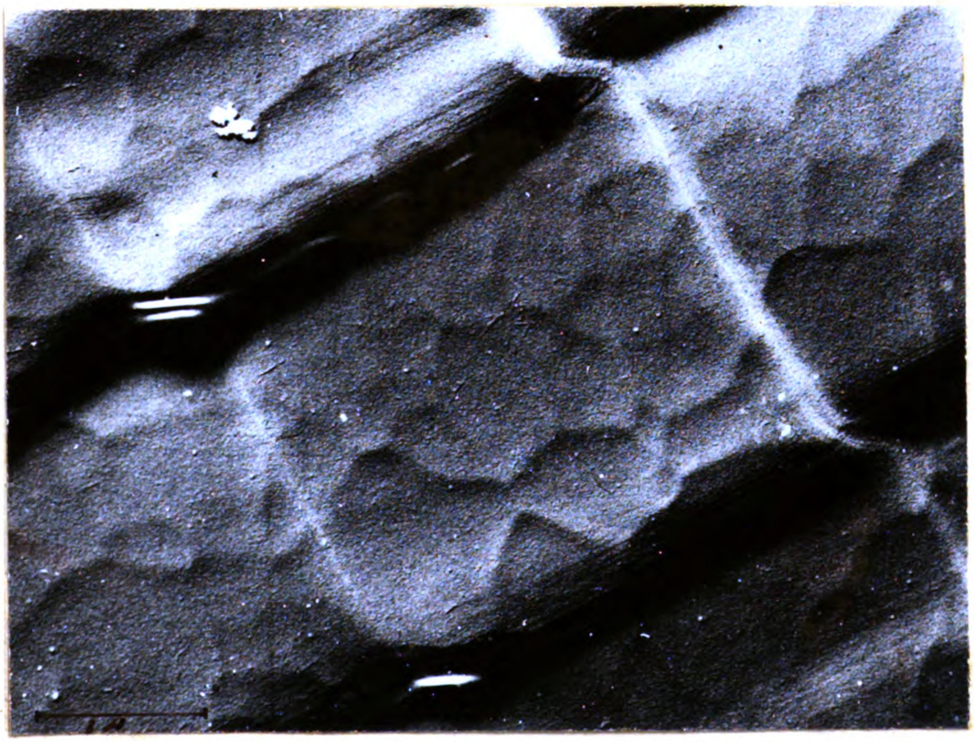

Figure 13. Micrograph of intersecting slip bands in aluminum photographed from a carbon replica made by the parlodion-carbon method. Magnification 23,000 X.

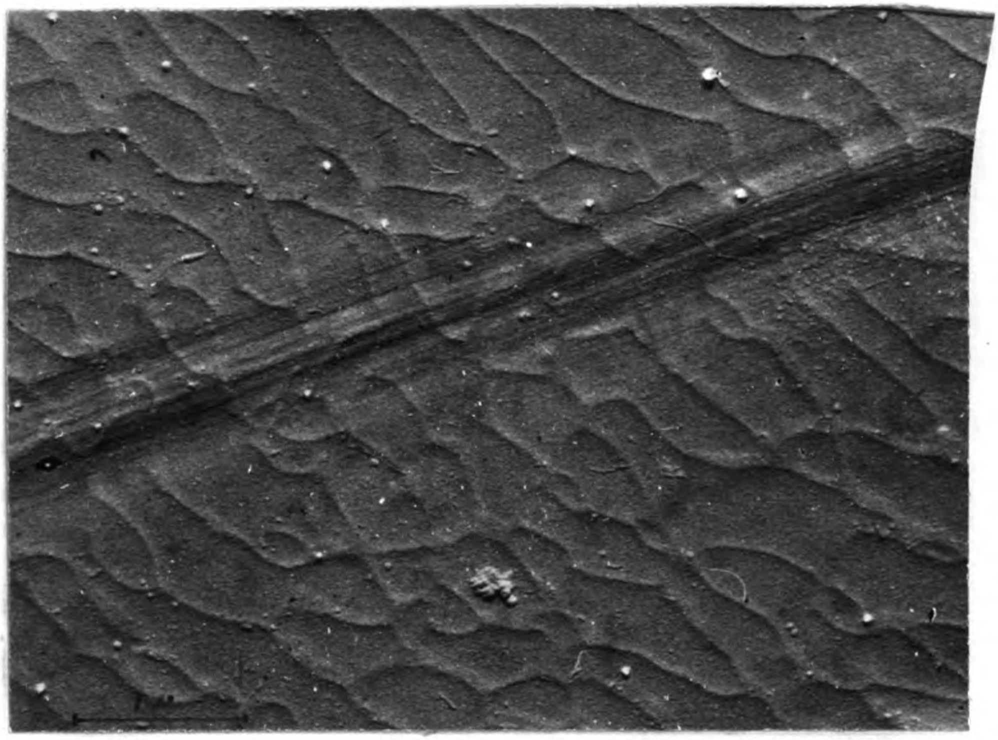

Figure 14. Micrograph of a slip band which appears to have essentially reflected itself with respect to a median plane. Photographed from a carbon replica made by the parlodion-carbon method. Magnification 23,000 X.

Figure 15. Micrograph of an etch pit made by deteriorated Bright Dip. Magnification 14,000 X.

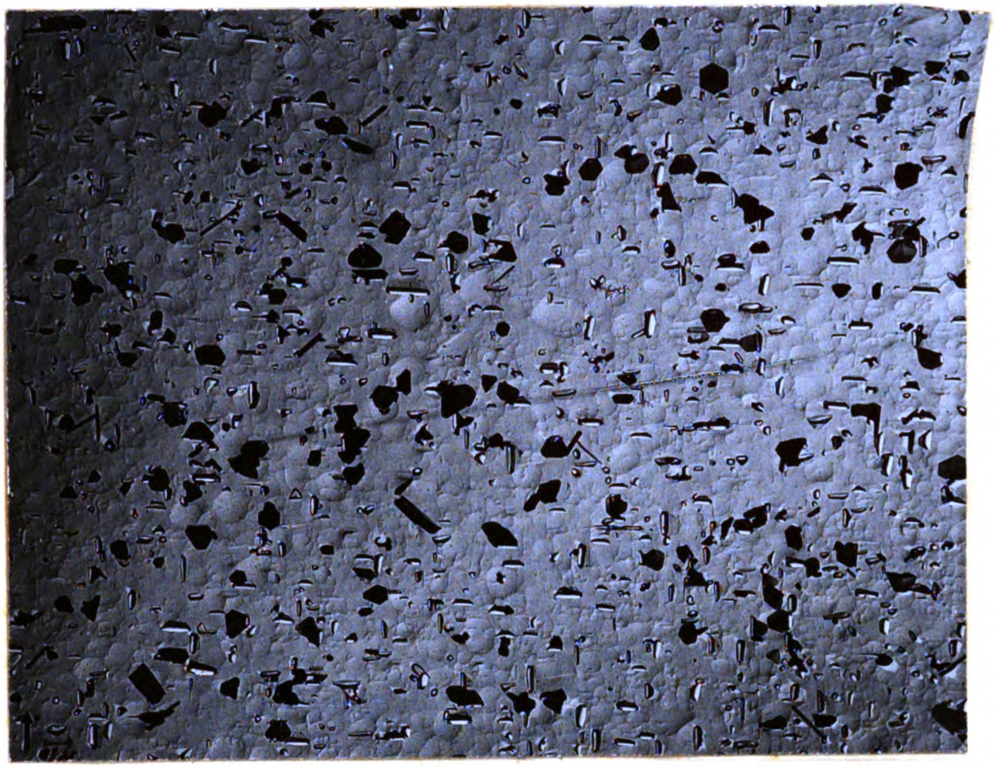

Figure 16. Micrograph of ordered precipitation in an aluminum-silicon alloy. Magnification 3600 X.

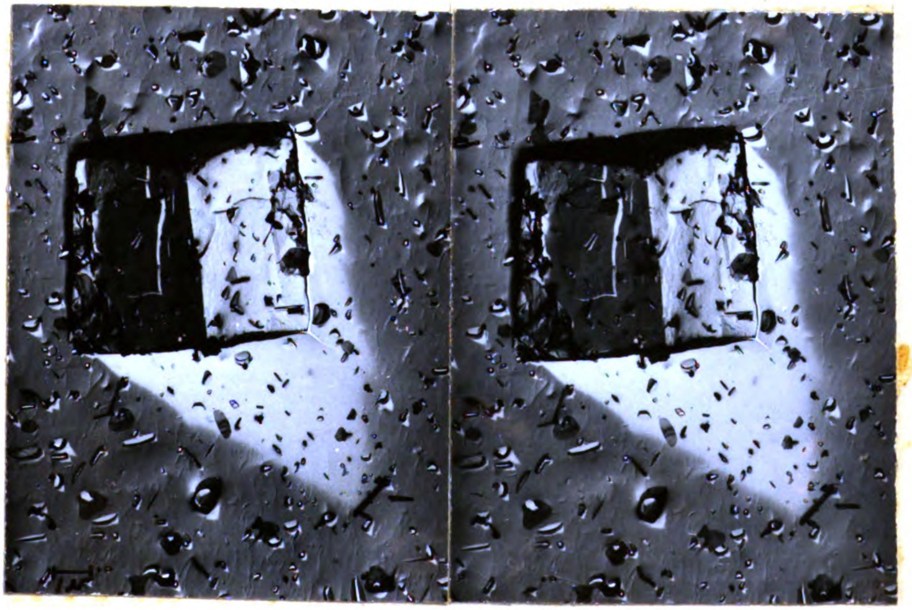

Figure 17. Stereoscopic micrographs of an etch pit with 100 faces from which the approximate orientation of the grain and precipitates can be found. Magnification 4500 X.

Figure 18. Micrograph illustrating the orientation of silicon precipitates with respect to slip lines. Magnification 4500 X.

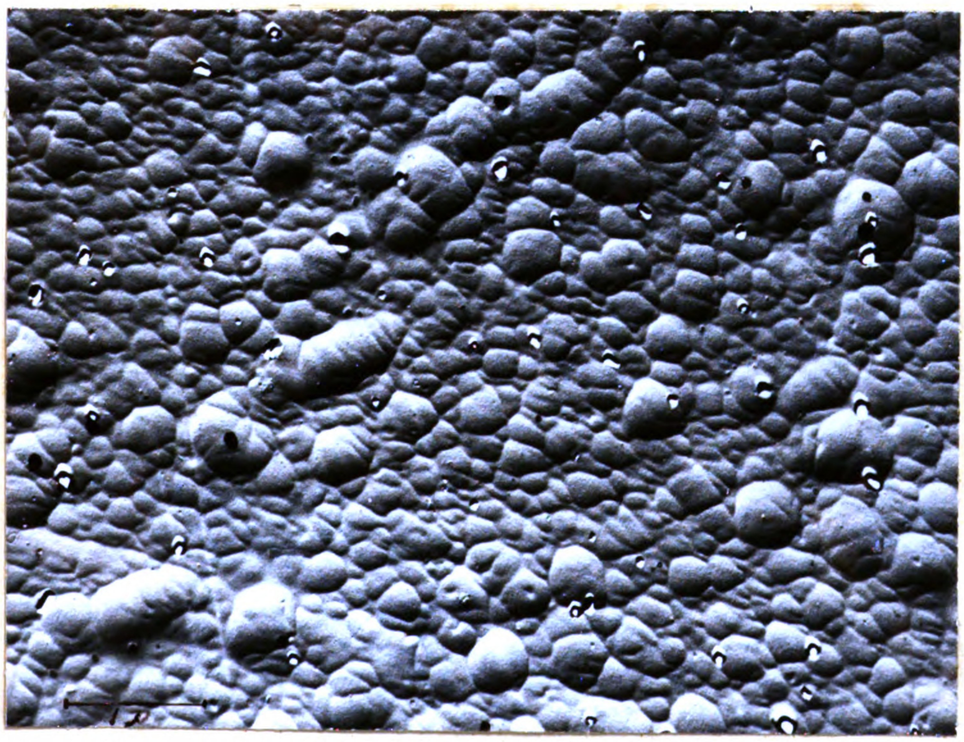

Figure 19. Micrograph of silicon precipitates going into solution. Magnification 19,000 X.

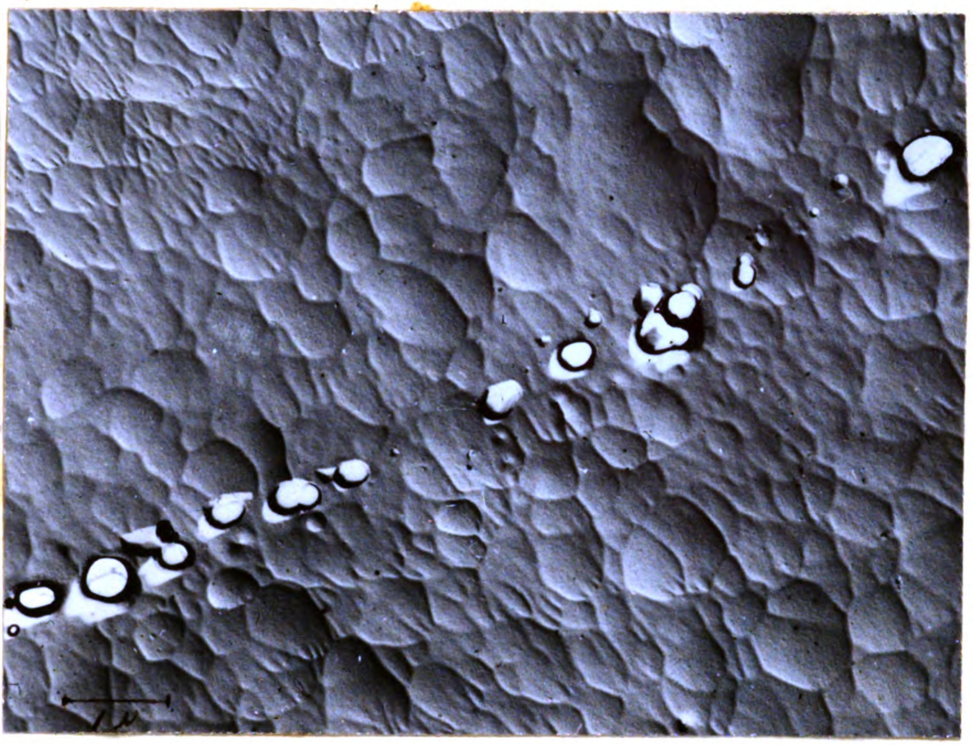

Figure 20. Micrograph of silicon particles which have not gone into solution in a supersaturated specimen. Magnification 14,000 X.

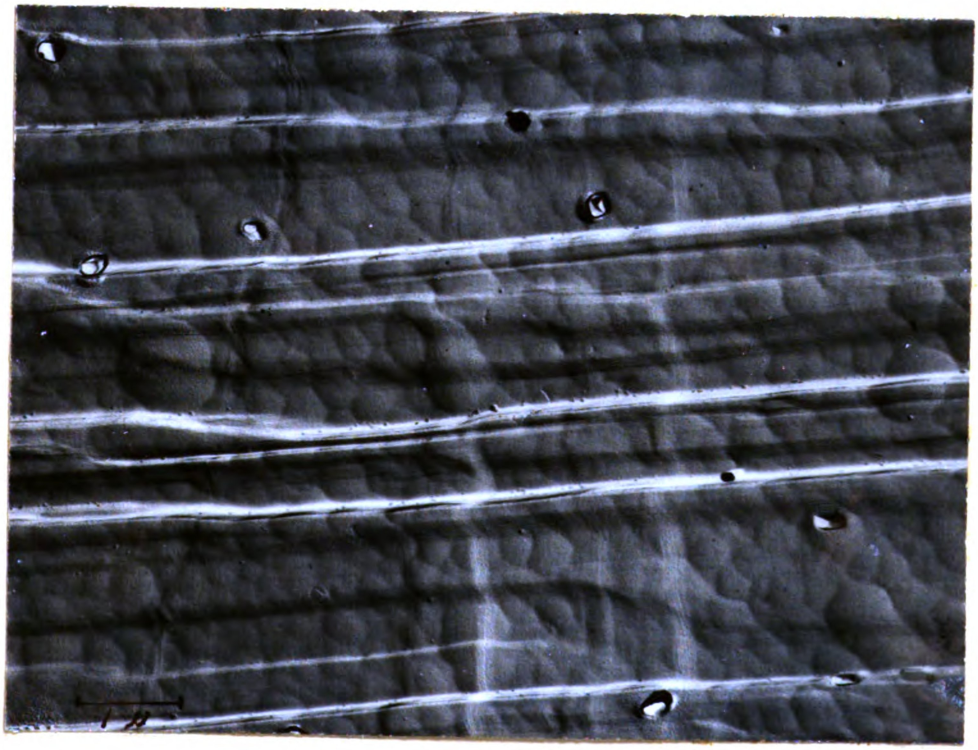

Figure 21. Micrograph illustrating homogeneous slip in a supersaturated specimen. Magnification 14,000 X.

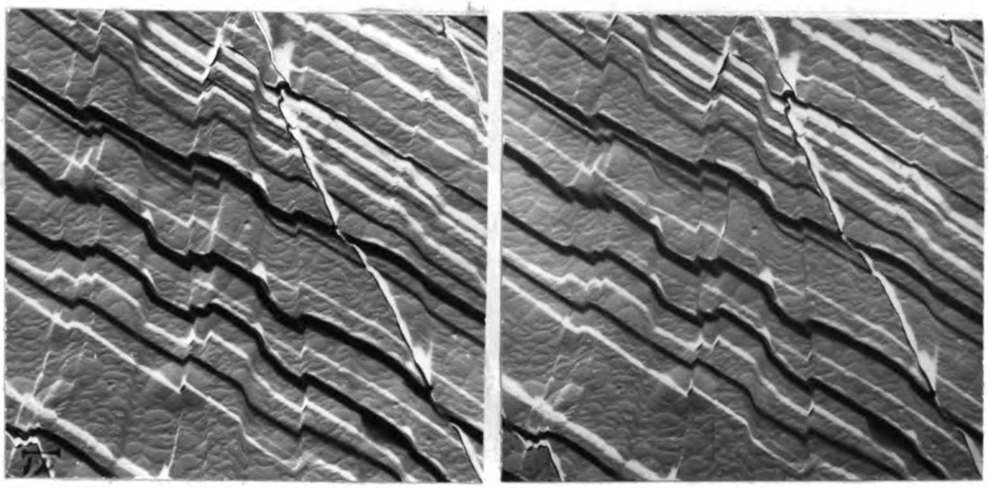

Figure 22. Stereoscopic micrographs illustrating inhomogeneous slip in a supersaturated specimen which has undergone heavy strain. Magnification 4000 X.

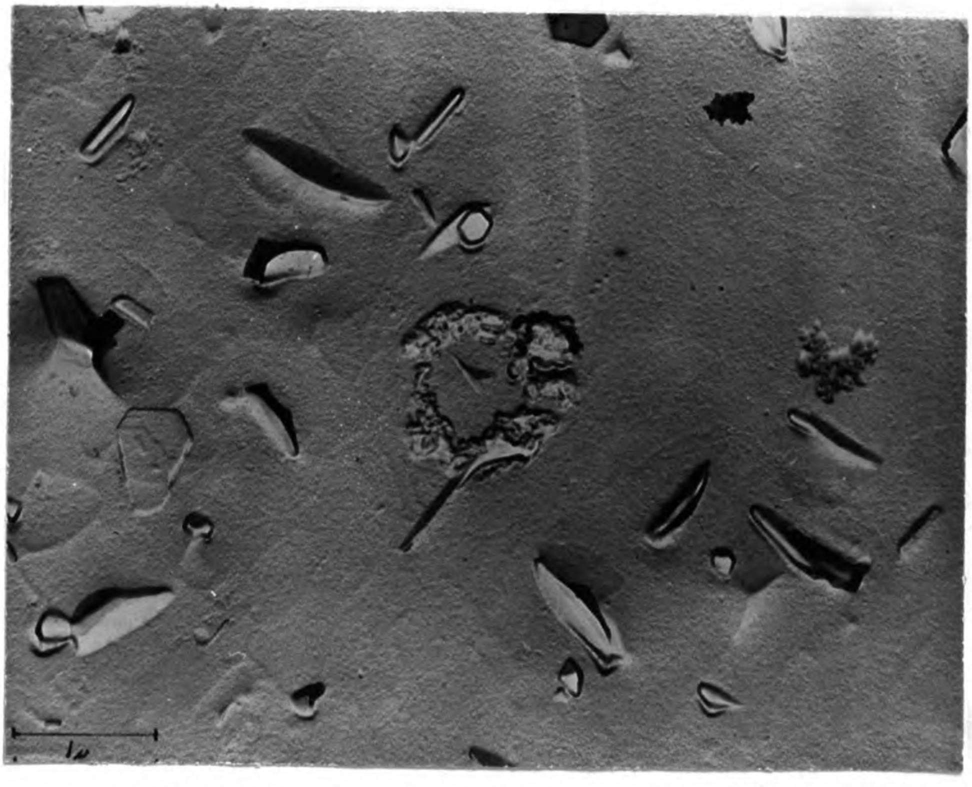

Figure 23. Micrograph of silicon precipitates caught in the aluminum oxide film of a specimen. Magnification 19,000 X.

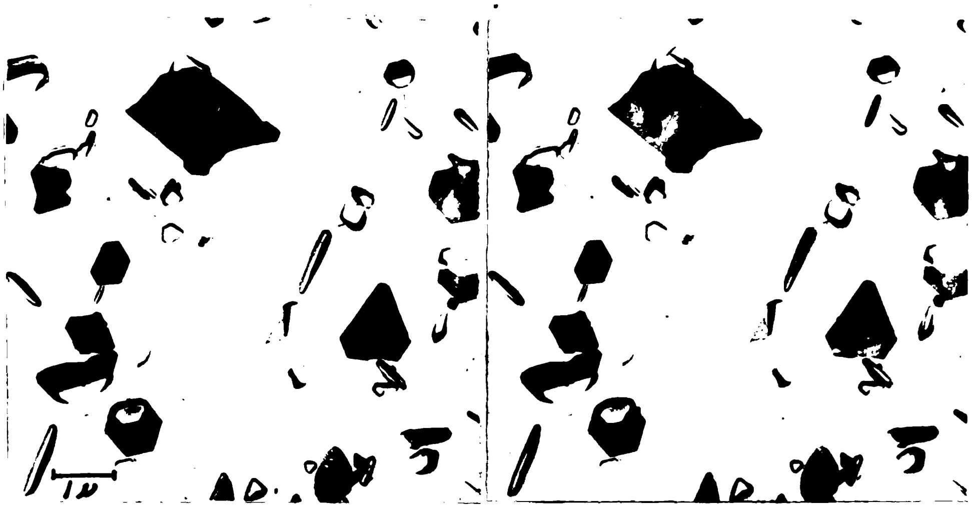

Figure 24. Stereoscopic micrographs showing extinction contours and the extreme thinness of the plate-shaped silicon precipitates. Magnification 8000 X.

VIII. BIBLIOGRAPHY

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