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Beech Hill,
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# THE PREPARATION OF THIN METAL FOILS BY MICROTOMY: AN ASSESSMENT OF THE TECHNIQUE, AND ITS APPLICATION TO METALLURGICAL INVESTIGATIONS WITH THE ELECTRON MICROSCOPE

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HEAD OF LABORATORY

# ABSTRACT

The research was undertaken to investigate the use of ultra-microtomy as a method for preparing thin sections of metals and alloys for transmission electron metallography.

The work was divided into three parts:-

1) Comparison of thin films of the same sample prepared by microtomy and electropolishing.
2) Investigation of the effect of various cutting liquids on the structure of the thin sections.
3) Application of microtome techniques to the preparation of thin films from samples for which present electropolishing methods are inadequate.

1,2) In order to investigate these two topics, two alloys, aluminium-4% copper and aluminium-20% silver were used as these have already been studied extensively by using electropolishing to prepare the thin foils. It has been found that using 'Epikote' resin* as the cutting liquid, thin films were obtained which were almost as good as the electropolished foils. Other cutting liquids invariably caused the thin films to either curl up or break up on cutting.

3) Instances where electropolishing could not adequately be used include stress-corrosion and metallic layers on a metal substrate.

Thin films were obtained with the microtome of 70/30 brass stress-corroded in ammonia. The features present in these films were similar to those in electropolished thin foils of strained material, which were prepared by other workers in an investigation into stress-corrosion.

*Supplied by Shell Chemical Co. Limited.

Thin films have been successfully produced from nickel plating on brass, however, distortion of the structure required electron diffraction analysis to positively identify the phases present.

Galvanised iron and soft solders on iron were chosen for the examination of metallic layers deposited at high temperatures. Thin sections through the galvanised layer were obtained which showed that diffusion during the coating process had produced a very strong bond. However, the intermetallic phases of iron and zinc could not be positively identified by electron diffraction because the patterns were diffuse, mainly due to the strains introduced during cutting. Soft solders on iron presented difficulty in obtaining coherent sections of the interface because the bonding was not sufficiently strong. It was found that thin sections of the layer and the metal substrate together could best be obtained if the plane of the interface was perpendicular to the plane of cutting, in addition to the bonding at the interface being strong enough to withstand the stresses introduced during cutting.

The anodised oxide layer on aluminium has been examined previously by ultra-microtomy, and it was thought that copper oxide films could be similarly examined, however, this proved to be difficult as the film was not very adherent. However, by electro-plating a layer of copper on the oxide, it was possible to obtain thin sections of the oxide.

It was found that microtomed thin films were liable to recrystallisation in the electron microscope owing to the heavy deformation introduced during cutting.

# THE PREPARATION OF THIN METAL FOILS BY MICROTOMY: AN ASSESSMENT OF THE TECHNIQUE, AND ITS APPLICATION TO METALLURGICAL INVESTIGATIONS WITH THE ELECTRON MICROSCOPE

## 1. INTRODUCTION

Since 1956 metals and alloys have been extensively examined by direct transmission in the electron microscope, and a number of techniques are now available for the preparation of suitable thin foils.

Heidenreich(1) in 1948 appears to have been the first to use electropolishing to thin discs of aluminium and aluminium-copper, 3mm in diameter and 125µ thick, until suitable thin foils were produced at the centre of the specimen. The microscopes available however, at that time did not allow him to exploit this technique fully. Microtomy was used by Haanstra(2) to cut sections, about 100Å thick, of soft metals with a diamond knife. Other techniques include chemical etching(3,4), vacuum evaporation, ion bombardment(5,6), electrodeposition(7), deposition from solution(8), mechanical deformation(3), thinning molten drops by surface tension(9), cleavage techniques(10-14), distillation from the vapour(15), and spark-machining(16).

Most of these techniques are either rather specialized or can lead to preferential dissolution of one phase with respect to another. Another difficulty in the case of electro-polishing is to achieve uniform removal of metal from all parts of the specimen surfaces, and this requires a uniform current density. In the case of surface effects and thin metallic layers at the surface these difficulties tend to be accentuated because the chemical properties of the surface layer are often considerably different to the bulk of the material. Microtomy is not dependent on the different chemical properties and to assess its value for the study of surface layers, investigations were undertaken to examine stress corrosion of 70/30 brass, oxide on copper, nickel plating on brass, the galvanised layer on steel, and soft solders on soft iron.

It is known that the metallographic strudture of sections obtained by microtomy invariably shows some degree of distortion(17), so to evaluate its extent, age-hardened aluminium alloys were prepared by both microtomy and electropolishing techniques, and a comparison made of the structures obtained.

In microtomy, the sections are collected from the surface of a liquid bath behind the knife edge, and generally water is used but there are some instances where its use is prohibited e.g. in the case of easily oxidised metals. To find a suitable alternative to water, sectioning was carried out using other liquids.

## 2. EXAMINATION OF THE SURFACE LAYERS OF METALS

### 2.1. Stress-Corrosion of 70/30 brass

When many alloys are stressed in a corrosive medium, they fail at stresses well below those required for failure in a normal tensile test. The fracture path in brasses and bronzes may be either trans-granular or intergranular, depending on the stress and heat treatment of the specimen. Swann and Nutting(18) found that electropolishing of tensile strained specimens (giving a condition similar to stress-corrosion) showed preferential attack along stacking faults in copper - zinc and copper-aluminium alloys, whereas in copper-germanium and copper-silicon alloys, the matrix is attacked in preference to the stacking fault.

It was suggested, however, that this experiment may not in fact give the same results as normal stress-corrosion(18). Microtomy was thought to be capable of correlating these results with true stress-corrosion, as thin films can be prepared directly from the stress-corroded sample without recourse to further etching or electro-polishing.

Samples of 70/30 brass in sheet form, 0.05" thick and fully annealed were loaded to just below the yield point (15,000 p.s.i.) in an atmosphere of ammonia saturated with water vapour at 20°C, failure occurring in 12 to 20 hours. Specimens were obtained at three hourly intervals, and the less corroded ones were mounted directly in the microtome chuck and those which were heavily corroded mounted initially in cold setting plastic before placing in the microtome. Sections were cut from small areas $<$0.05mm square, of thickness 1000 to 2000Å, and collected in the usual way from the water surface behind the knife onto grids covered with a carbon support film.

Cracks were observed and figures 1 and 2 show stacking faults, DD, which are very unlikely to be produced as a result of the cutting mechanism because the plane of maximum shear stress is more or less normal to the thickness of the section(19). The stacking faults are parallel to the cracks and in figure 1 the crack appears to be a continuation of a stacking fault. These cracks are trans-granular and appear to be continuous rather than made up of a number of small separately nucleated cracks which have subsequently joined as has been observed in metals fractured by the application of stress only(20,21).

In the badly stress-corroded specimens, corrosion occurred at the grain boundaries and wide cracks invariably developed.

The results provide confirmation of the work of Swann and Nutting, showing that electropolishing of strained material produces characteristics similar to stress corrosion.

## 2.2. Thin metallic layers on a metal substrate

### Introduction

There are many instances when metals are used coated with a thin film of a different metal. Typical examples are galvanised iron, nickel plating, chromium plating, etc.. Using conventional thin film techniques, it is however, very difficult to study the interface between the two metals and it was decided to attempt to use microtome sectioning for preparing sections through the interface.

### 2.2.1. Metal deposited at room temperature

### Nickel Plating on Brass

The specimens were mounted in bakelite and sectioned across the interface between plating and brass. The type of section obtained depended on the cutting conditions, i.e. the cutting speed, the thickness of cut, the quality of the knife edge and the liquid meniscus behind the edge. Figure 3 shows some splitting at the interface A-A, due to the difference in strain produced in two materials of different mechanical properties. Figure 4 shows a case where the interface is coherent B - B, and although the brass portion (R) of the section is badly distorted, the nickel (N) is much less distorted at the interface.

### 2.2.2. Metal deposited at elevated temperatures

#### a) Zinc (galvanised) layer on steel

A galvanised coating on steel is formed at 450°C in a molten zinc bath by dipping the carefully cleaned steel into the liquid metal for about 15 seconds. As a result, intermetallic phases of iron and zinc are formed, and optical examination revealed a thin layer A - A, next to the steel (Figure 5) and adjacent to that, a thicker layer B - B showing no detailed structure. Next is a layer of columnar structure C - C, and at the outside, a layer D - D containing small crystals within a matrix, which is presumably zinc. The picture was not nearly so clear by electron microscopy, although Figure 6 shows an intermetallic phase B as well as zinc C, but the phases $\Gamma$, $\delta_1$, and $\zeta$ could not be positively identified by electron diffraction, although some rings on the patterns corresponded to reflections from $\Gamma$ and $\zeta$. Generally, the patterns were diffuse, due to the thickness of the sections and to the fact that the large number of reflections from these phases are very close together and so could not be resolved. Some of the broadening of the patterns may be due to strains, or the small crystal size produced by the formation of the $\delta_1$ phase, so an anneal at 300°C for ½ hour was given to a specimen, but the diffraction pattern from a cut section was no different as a result. This probably indicates that the diffuse patterns are due to the deformation introduced during cutting.

If the section was sufficiently thin, heating caused by an increase in the electron beam current, produced evaporation of the zinc, which then condensed in another area of the carbon support film on the grid. Figure 7 shows the small crystals (about 0.1µ in linear dimensions), some hexagonal shaped, which were formed.

### b) Solders on Iron

Soft solders containing 60% tin, 40% lead and 40% tin, 60% lead, were bonded to a finely ground surface of soft iron using a commercial flux called 'Fluxite' which had the effect of dissolving any oxides present. The specimens were mounted in bakelite and sectioned in the usual way, cutting in the plane normal to the iron-solder interface.

The sections obtained were thin enough for electron diffraction but distortion was evident, although not preventing the solder (S) being distinguished from the iron (I)(Figure 8). From the diffraction patterns, lead and β-tin were identified, as expected, but some sections showed hexagonal crystals (C) (Figure 9), and indexing on the hexagonal crystal system gave an 'a' value of 5.3Å. However, after checking against the known oxides of the metals present and chlorides of zinc and ammonium, no identification could be made, so it was postulated that the crystal originated from the flux particularly as it was only observed near the interface.

## 2.3. Oxide films on copper

Microtomy has proved very successful for examining the oxide layer on aluminium(22), and it was decided to study the application to another metal-oxide system in which the oxide was less adherent. The metal chosen for this purpose was copper and the oxide was produced by heating in air. A suitable film thickness was formed by heating at 300-350°C for 5 - 10 minutes, the thickness being estimated to be 0.1μ by its straw colour(23).

On subsequent microtome sectioning, it was found that as the oxide was so non-adherent, it was difficult to prepare sections showing both the metal and the oxide. To support the oxide, copper was plated onto it, having previously deposited Au/Pd or bent the specimen so that the plating could obtain electrical contact with the metal.

Figure 10, shows an oxide layer (O) 0.1 - 0.3μ thick sandwiched between the Cu plate (P) and Cu matrix (M), but only fragments are observed, indicating that fracturing of the oxide occurred on sectioning and many sections examined showed no oxide present at all.

## 3. COMPARISON OF MICROTOMY AND ELECTROPOLISHING IN THE PREPARATION OF AGE-HARDENED ALUMINIUM ALLOYS

Aluminium-4% copper and aluminium-20% silver are two alloys which age-harden, and as the precipitates are initially in the form of G.P. zones, with dimensions of up to 100Å, electron microscopy has to be used in order to observe them. The heat treatment applied to the Al-Cu consisted of homogenizing at 540°C followed by ageing at 160°C for 5 hours giving G.P.(2) zones or, more accurately, $\theta''$ precipitates, since they are coherent intermediate precipitates rather than zones. The Al-Ag was homogenized at 525°C, then aged at 160°C for 3 days to give principally G.P. zones with a few $\gamma'$ precipitates. It was also aged for 2 hours at 220°C to give more $\gamma'$ precipitates in addition to the G.P. zones. The specimens were in sheet form, the Al-Cu being 0.040" and Al-Ag 0.005" thick.

Thin foils of these specimens were prepared by the technique described by Nicholson, Thomas and Nutting(24).

The Al-Cu was reduced from 0.040" to 0.005" thickness by the scanning jet machine, developed by Kelly and Nutting(25), using 30% nitric acid and an applied potential of 80V. Final polishing was carried out in Lenoirs' solution (817ccs $H_3PO_4$, 134ccs $H_2SO_4$, 156grs $CrO_3$, 40ccs $H_2O$) at 70°C, using a horizontal flat Al cathode below the specimen, the 'window' method and a voltage of 10 - 12V. The Al-Ag thin foils were prepared by the same technique, without the initial thinning by the scanning jet machine. Any oxide on the thin foils was removed by immersing in phospho-chromic acid solution (350ccs $H_3PO_4$, 160grs $CrO_3$, 650ccs $H_2O$) for 10-15 minutes. For sectioning, the specimens were mounted in bakelite, which necessitates heating up to 150°C for a few minutes. It was considered that this short length of time at 150°C would not cause an appreciable change in the type or amount of precipitation.

Figure 11 shows θ" plates 500 -1000Å long in an electropolished sample and the diffraction pattern of a similar area has streaks in the ⟨100⟩ directions, as observed by Nicholson, Thomas and Nutting(26). The microtomed sections of this specimen did not show any similar structure (Figure 12), the reason being that these sections are much smaller in area than the thin areas in foils prepared by electropolishing. Thus the probability of finding an area of the correct orientation is considerably reduced in the former case.

The preparation of Al-Ag thin foils presented less difficulty as the specimens were thin enough (0.005" thick) for electropolishing without any initial thinning. Figure 13 illustrates the formation of spherical G.P. zones (Z) about 100Å diameter, in an electropolished foil, together with plate-like Y' precipitates, about 20Å wide and 1000-5000Å long, in a specimen aged for 3 days at 160°C. Figure 14 gives an enlarged view of the zones.

The electron micrographs of microtomed sections of a specimen given the same ageing treatment were not always similar. Figure 15 shows severe distortion, Figure 16 gives no indication of either G.P. zones or Y' precipitates while Figure 17 has straight plate-like precipitates and in addition small spots, distributed uniformly, are observed about 100Å in diameter, corresponding to the G.P. zones described by Nicholson et al(26). The section giving this structure was obtained using 'Epikote' resin in place of water in the bath, and as there was so little distortion, further cutting was carried out (see Section 4)to determine if this resin was generally better for cutting than water.

The Y' precipitates formed in Al-Ag on ageing for 2 hours at 220°C were very densely distributed (Figure 18).

In Figure 19, small crystallites (C) are observed within the matrix but the Y' precipitates are apparently unaffected by the heating. It is known that the temperature rise in the specimen during observation in the electron microscope may be several tens of degrees Centigrade, and these small grains are almost certainly the result of recrystallisation of the matrix in the microscope. In some cases the matrix appeared to recrystallise completely, Figure 20, to produce a grain size of about 1.5μ diameter, compared with about 1.5mm diameter for a similar specimen in the same aged condition before preparation and examination in the microscope.

## 4. THE EFFECT OF VARIOUS CUTTING LIQUIDS

The requirements of a liquid for use in a bath behind the knife edge during cutting are:-

a) a high surface tension,
b) chemical inertia to the specimen,
c) low vapour pressure at room temperature,
d) ability to be removed from the cut section without causing contamination.

The principal cutting liquid used is water, which fulfils most of these requirements. It has a high surface tension(73 dynes/cm.) enabling sections to float on its surface; it has a low rate of evaporation; it does not contaminate sections, i.e. it does not react with either the embedding materials or many metals, although some metals become oxidised.

For metals that oxidise fairly rapidly, xylene has been used as it is non-reactive, but it has the disadvantage of being volatile, therefore, the meniscus at the knife edge does not remain long enough for sections to be cut and collected without frequent replenishment. Since it was considered that cutting liquids must have a high surface tension, two liquids were tried, quinoline and furfural which had surface tensions, 45.0 and 43.5 dynes/cm. respectively. But cutting using both of them produced the same effect. The sections would ~~curled~~ up and sank or became suspended in the liquids.

The usefulness of a highly viscous liquid was examined and first results showed that it had excellent cutting properties. Figure 17 shows the sort of fine detailed structure that can be observed in a cut section. The liquid used was 'Epikote' resin which has a viscosity of 90/150 centipoise (c.p.) ( water has a viscosity of 1 c.p.), is colourless, and readily soluble in chloroform enabling the sections to be cleaned before examination in the electron microscope. A summary of the properties of these cutting liquids is given in Table I.

To examine the profile of sections, which would show the type of surface produced on cutting, sections were cut using water and 'Epikote' resin. A nick was made in the sample so that the section of the section could be subsequently cut parallel to the first cutting direction. The sections were mounted in methacrylate to give them support in the electron microscope, and as 'Epikote' softens methacrylate, water had to be used as the cutting liquid on re-sectioning. Figure 21 shows the profile of a section cut using the resin. This is of a uniform thickness, approximately 7500Å, which is somewhat greater than the usual 1000-2000Å normally considered as the optimum thickness for cut sections to be examined in the electron microscope. The surfaces of the original sections are both smooth and Figure 22 shows this at much higher magnification. Figures 23 and 24 are cross sections of sections cut using water, Figure 23 showing both surfaces to be rough and also a variation in thickness of the original section which is badly curled. These effects demonstrate the heavy deformation present in sections cut on water.

## TABLE I

## CUTTING LIQUIDS FOR ULTRAMICROTOMY

| Liquid | Surface tension (dynes/cm) | Viscosity (c.p.) | Comments |
|---|---|---|---|
| Quinoline | 45.0 | - | Dark brown liquid in which sections curled up and became suspended in liquid. |
| Furfural | 43.5 | 1.5 | Sections would not float on surface and became suspended in liquid. |
| Xylene | 30 | - | Evaporated quickly but useful for metals that oxidise easily. |
| Water | 73 | 1.0 | Sections float on surface readily but often sections break up on cutting. |
| Epikote Resin | ~50 | 90-150 | Colourless liquid which enables sections to be cut with less distortion than with water, also larger areas. Dissolves readily in chloroform. |

## DISCUSSION

### 1) Stress - Corrosion of 70/30 Brass

The results obtained were very similar to those found by Swann and Nutting(18) in that cracks were observed along the stacking faults. However, it is possible that during the present work both the faults and cracks were produced by the cutting process.

The stacking faults in the microtomed samples were always at some angle, usually 45° to the cutting direction. Some recent work on silver(27) has shown that stacking faults introduced during cutting, however, are normally at right angles to the cutting direction in the plane of maximum shear stress. In addition, many of the cracks along the stacking faults observed in the present research contained corrosion product which could only have been formed during stress corrosion and not in subsequent cutting. Thus, both the faults and cracks were probably formed during stress corrosion confirming the results of Swann and Nutting, and showing that electro-polishing strained samples of 70/30 brass has the same effect on the structure as normal stress corrosion.

### 2) Nickel Plating on Brass

In many cases the nickel layer remained firmly attached to the underlying brass in the microtomed slices. This effect could be the result of a strong bond between the deposited nickel layer and the brass or alternatively to the two layers having similar mechanical properties.

However, parting of the two metallic layers was occasionally observed which demonstrated how critical the cutting conditions have to be if undistorted flat sections are to be produced.

### 3) Galvanised layer on steel

It has been shown previously(28) that during the process of depositing the galvanised layer, three intermetallic phases ($\Gamma$, $\delta_1$, and $\zeta$) are formed between the iron and zinc and it was expected that these would be detected by electron diffraction studies of the microtomed samples. However, only one intermetallic phase could be found ($\Gamma$) and the electron diffraction patterns were generally rather diffuse.

The other phases ($\delta_1$, and $\zeta$) could probably not be identified because of the similarity of the structures and the relatively low accuracy of electron diffraction techniques (approximately ± 2%) compared with X-ray diffraction.

The diffuse patterns are probably due to either the very close spacing of the diffraction lines, or the extremely small grain size produced during the $\delta$ - $\delta_1$ transformation at 630°C or to the strained matrix produced during cutting.

### 4) Soft Solders on Iron

It was possible to prepare thin sections even though there was a tendency for the solder layer to break away from the iron. Electron diffraction patterns were obtained which could be identified as lead and $\beta$-tin showing the feasibility of this technique for examining the structures of thin layers of very soft metals on a relatively hard metal.

In addition, hexagons of an unknown phase were observed which were thought to originate from the flux.

### 5) Oxides on Copper

It was found to be extremely difficult to section through the oxide film on copper, and the problem was resolved by plating on top of the oxide prior to cutting. The distortion due to cutting was very large, and in most cases, the oxide film was lost. But there were occasions where it remained attached (Figure 10), although not held by the plating in the original position.

### 6) Ideal Conditions for the Cutting of the Interface of a Thin Metallic Layer on a Metal Substrate

These observations of cutting through the interface of layers on the surface of metals suggest that strong bonding between the surface layer and underlying metal is necessary for complete sections to be obtained. Also the plane of cutting must be normal to the plane of the interface, and the cutting direction parallel to the line of the interface. Since the shear stress produced at the knife edge is in a plane approximately normal to the rake face(19), the stress is probably less likely to cause fracture at the interface if the specimen passes the knife edge so that the interface and underlying metal pass at the same time. It is probable that if the interface were such that the metal was cut followed by the surface layer, i.e. the interface parallel to the plane of maximum shear, the stress would in many cases be sufficient to part the two layers. However, further experiments would be required to prove this point.

The extent of distortion of each material of a section was different (see Figures 3, 4, 6 and 8). This implies that the mode of deformation of each was different which would again probably lead to parting at the interface, suggesting that good intact foils would only be produced when the mechanical properties of the metal and surface layers are similar.

## 7) Comparison of Electropolishing with Microtomy as Techniques for Preparing Thin Foils of Age-hardened Aluminium Alloys

Age-hardened aluminium-copper and aluminium-silver alloys θrovided a good case for comparing the techniques of microtomy and electropolishing since they contained small zones and precipitates which have been extensively studied in thin foils prepared by electropolishing. These zones and precipitates are situated in a particular orientation with respect to the aluminium matrix. To observe the plate-like zones in aluminium-copper they must lie in a plane parallel or nearly parallel to the electron beam. Sections of aluminium-copper were examined but no zones were observed. This was possibly due either to the severe distortion during cutting which could mask the coherency strain fields around the $\theta''$ precipitates, or to the difficulty of obtaining a section with the plates in the correct orientation with respect to the electron beam.

Aluminium-silver was slightly different because although the spherical G.P. zones were coherent, the $\gamma'$ precipitates were not, although they were orientation dependent. To obtain a section showing these precipitates it was found that the plane of cutting must be normal to the plane of the plate-like precipitates, and the cutting direction must be in the plane of the precipitates. Figure 17 shows the structure when this condition is satisfied, and it will be seen that this corresponds very closely to the structure of a thin foil prepared by electropolishing. If however, the section is cut in the direction normal to that described above, no precipitates or zones were observed (Figure 16). The distorted network of precipitates in Figure 15 may be due to the fact that the cutting direction was not exactly in the plane of the plate-like precipitates.

## 8) The Function of the Cutting Liquid

The function of the cutting liquid has not been considered very extensively in the literature, although R. Phillips(17,22) does mention some of the most important chemical and physical properties required. He states that the liquid should have low viscosity, high surface tension, and a suitable density, presumably such that sufficient buoyancy is given to the section to enable it to float on the surface. It should also provide a horizontal meniscus at the knife edge and all the edge should be wetted. It should not evaporate so quickly that constant replenishment of the bath is necessary, but quickly enough for the liquid to evaporate from the section when it is on the grid, leaving it clean, dry and uncontaminated prior to examination in the electron microscope. The liquid may also provide a certain amount of lubrication.

Distilled water is most commonly used, as it satisfied most of the above requirements; it is not very satisfactory, however, for metals which readily oxidise. For these metals, xylene is recommended but it has the disadvantage that evaporation is rather rapid.

V. A. Phillips(27,29) used a wetting agent with the water, either a mixture of water-25% ethyl alcohol or water-5% acetone, but the reason for using these mixtures was not stated.

The use, however, of 'Epikote' resin as a cutting liquid led to some interesting observations. The sections obtained (Figure 17) were relatively free from distortion, and quite large in area (about 0.5mm square which is 5 to 10 times the normal size). The major difference in physical properties of this resin compared with water is its high viscosity (90-150c.p. compared with 1 c.p. for water). The high viscosity possibly helps to provide some form of physical support to the section, and this makes up for the deficiency in surface tension (50 dynes/cm, compared with 73 dynes/cm for water). Also, it is suggested that 'Epikote' may provide good lubrication at the knife edge, enabling the specimen to pass with a much smoother action than would be the case with a liquid having poor lubricating properties.

The two chemical reagents, quinoline and furfural, did not prove to be very suitable cutting liquids, mainly because of their fairly low surface tension combined with a low viscosity. Xylene also has a low surface tension, which is probably a factor partly accounting for the difficulties such as the lack of flat sections produced during its use as a cutting liquid.

It appears therefore, that the ideal cutting liquid must have either high surface tension or relatively high viscosity, or more ideally, a combination of both. The rate of evaporation must be low, and the liquid must not contaminate the section during any stage of the preparation. A certain amount of lubrication may be desirable but further work is needed to confirm this observation. The density of the liquid must be such that sufficient buoyancy is provided for the section to float on the surface. It was found that 'Epikote' resin most clearly approached the ideal liquid, as it satisfied the majority of these requirements, its main disadvantage being that it had to be removed from the section with chloroform before examination in the microscope.

## 9) Factors Determining the Preparation Technique

In deciding what method of preparation to use for the preparation of thin foils for examination in the electron microscope, a number of factors have to be considered. The form of the specimen is very important, and since microtomy does not require the specimen to be of any particular shape initially, as it is ground to a pyramidal tip, this technique has an advantage over electropolishing where the specimen must be either in the form of thin sheet(30) or discs (31). The grinding has been shown not to influence greatly the metallographic structure. This was deduced from the fact that diffraction of a section from a tip prepared by filing, showed that the material had recrystallised with a fine grain size, whereas spot splitting was very slight using grinding. A limitation is the fact that the area which can be examined is small (about 0.5mm square, at the maximum), but it is possible to obtain series of sections giving an idea of the variation of structure with depth. The hardness of the specimen obviously has an effect on its cutting properties, but materials of hardness up to 600 V.H.N. have been sectioned successfully. Of equal importance is the ductility, for it is quite clear that ductile materials can be cut far more readily than brittle materials. Due to the distortion no reliable information can be obtained on dislocations using microtomy.

## FURTHER WORK

1. The thin films prepared using 'Epikote' resin as the cutting liquid contained much less deformation than those obtained with the normal fluids. So the use of other liquids having similar properties should be investigated.

2. A more detailed study of the amount of deformation using 'Epikote' resin could be made by comparing with thin sections of the same sample using water as the cutting liquid.

3. In cutting metallic layers on a metal substrate a study could be made of the relationship between the mechanical properties of the two metals and their influence on the coherency between the metals when sectioned with the ultra-microtomes.

## CONCLUSIONS

1. Microtomy provides a suitable alternative method for preparing thin films of metallic specimens for electron microscopy, when electropolishing or etching techniques cannot be used, provided distortion is unimportant.

2. Interfaces of metallic layers on a metal substrate can be prepared by microtomy provided there is strong bonding between the two materials.

3. It is difficult to obtain sections of brittle oxide layers.

4. The direct comparison between electropolished and microtomed thin films showed that the metallographic structure was similar, but some distortion of microtomed sections was inevitable.

5. 'Epikote' resin was the best cutting liquid tried in the present work, as it enables larger and less deformed sections to be obtained compared with those produced using the other liquids including water.

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P. P. Dennis

I. S. Brammar

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# LIST OF ILLUSTRATIONS

| Figure No. | Plate No. | Specimen | Magnification |
|---|---|---|---|
| 1 | | Stress-corroded Brass | 40,000 |
| 2 | | " " " | 80,000 |
| 3 | 50683 | Nickel Plated Brass | 4,000 |
| 4 | 50730 | " " " | 4,000 |
| 5 | 119 | " " " | 2,250 |
| 6 | 50913 | " " " | 8,000 |
| 7 | 51285/6 | " " " | 20,000 |
| 8 | 51005 | 60% Sn, 40% Pb Solder on Iron | 12,000 |
| 9 | 51788/9 | 60% Sn, 40% Pb Solder on Iron | 20,000 |
| 10 | 50897 | Oxidised Copper | 48,000 |
| 11 | 54386/7 | Al-4%Cu Aged 5 hrs at 160°C | 120,000 |
| 12 | 29120 | " " " " | 40,000 |
| 13 | 29482 | Al-20%Ag Aged 3 days at 160°C | 60,000 |
| 14 | 54562 | " " " | 160,000 |
| 15 | 29181 | " " " | 32,000 |
| 16 | 54876 | " " " | 56,000 |
| 17 | 54871 | " " " | 40,000 |
| 18 | 54551 | " " " | 40,000 |
| 19 | 54862 | " " " | 56,000 |
| 20 | 54878 | " " " | 12,000 |
| 21 | 55057 | " " " | 4,000 |
| 22 | 55066 | " " " | 60,000 |
| 23 | 55072 | " " " | 32,000 |
| 24 | 55068 | " " " | 32,000 |

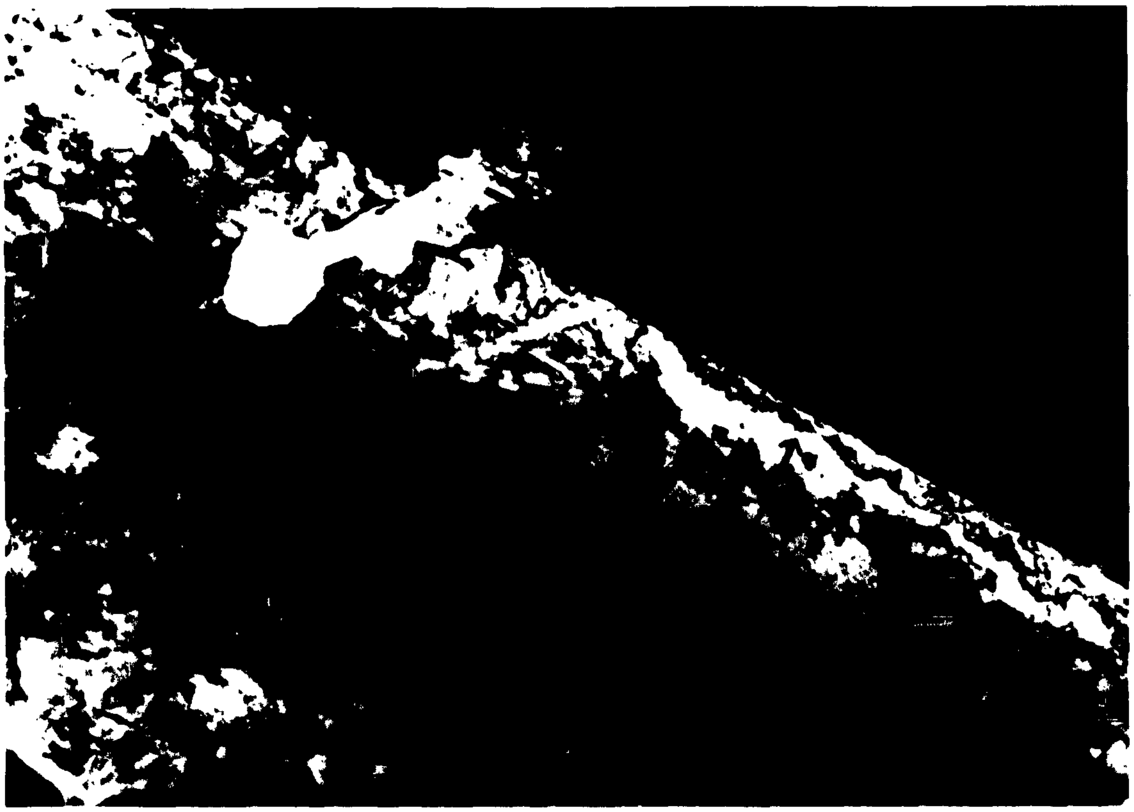
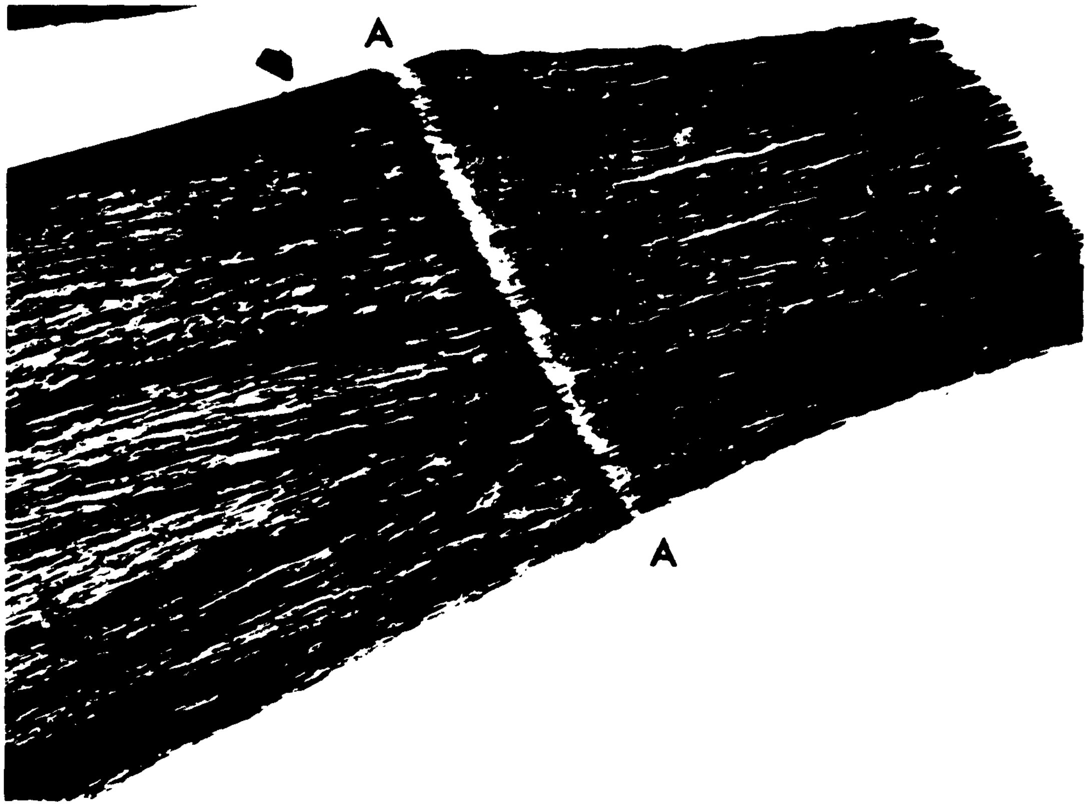


FIGURE 1

FIGURE 2



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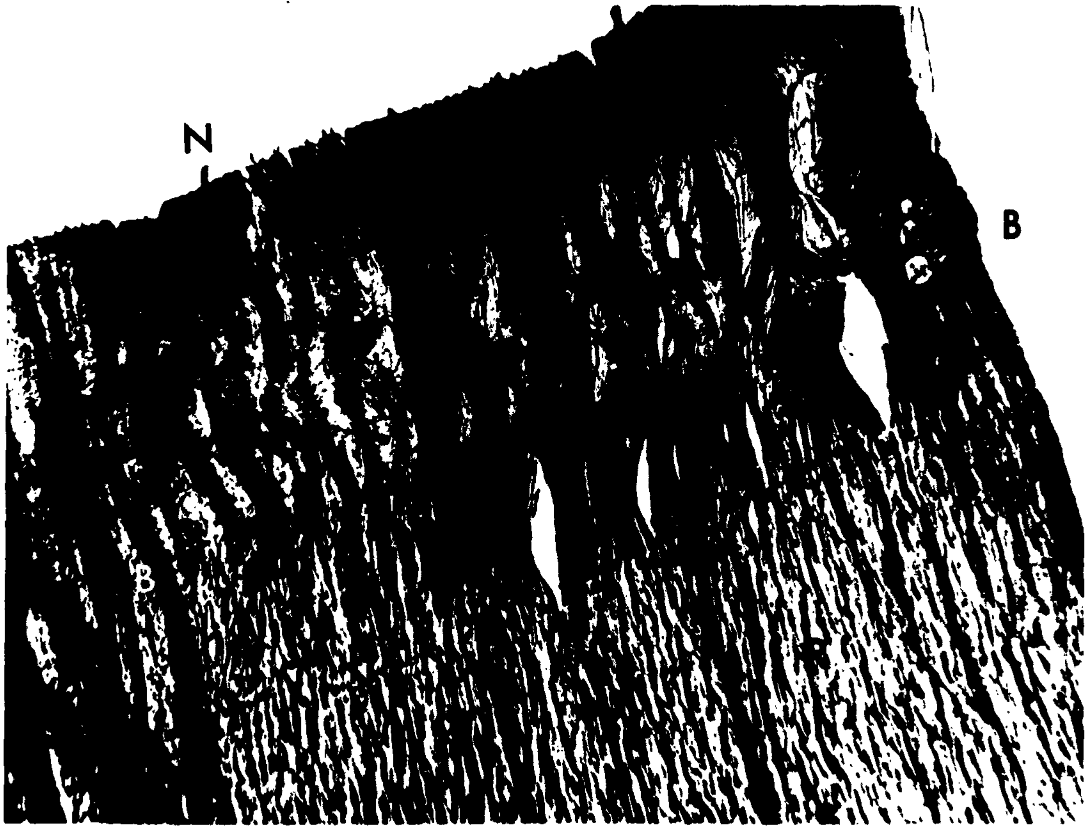

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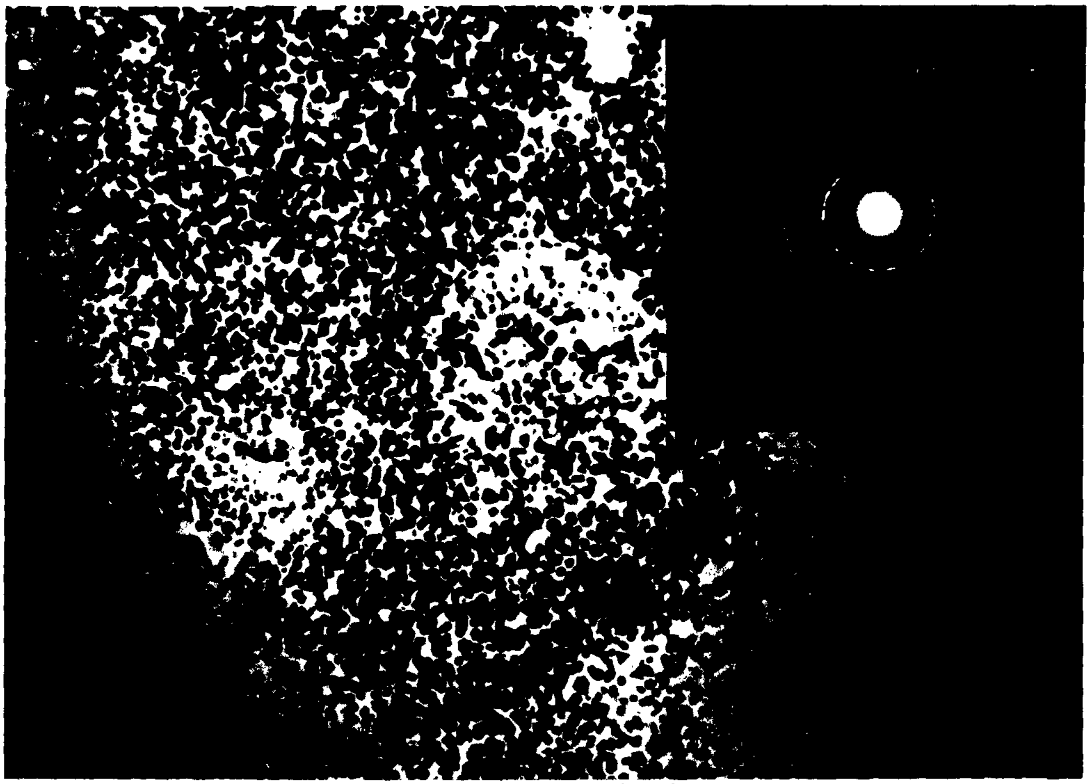
Figure 5 Plate No. 119 X2,250

Figure 6 Plate No. 50913 X8,000



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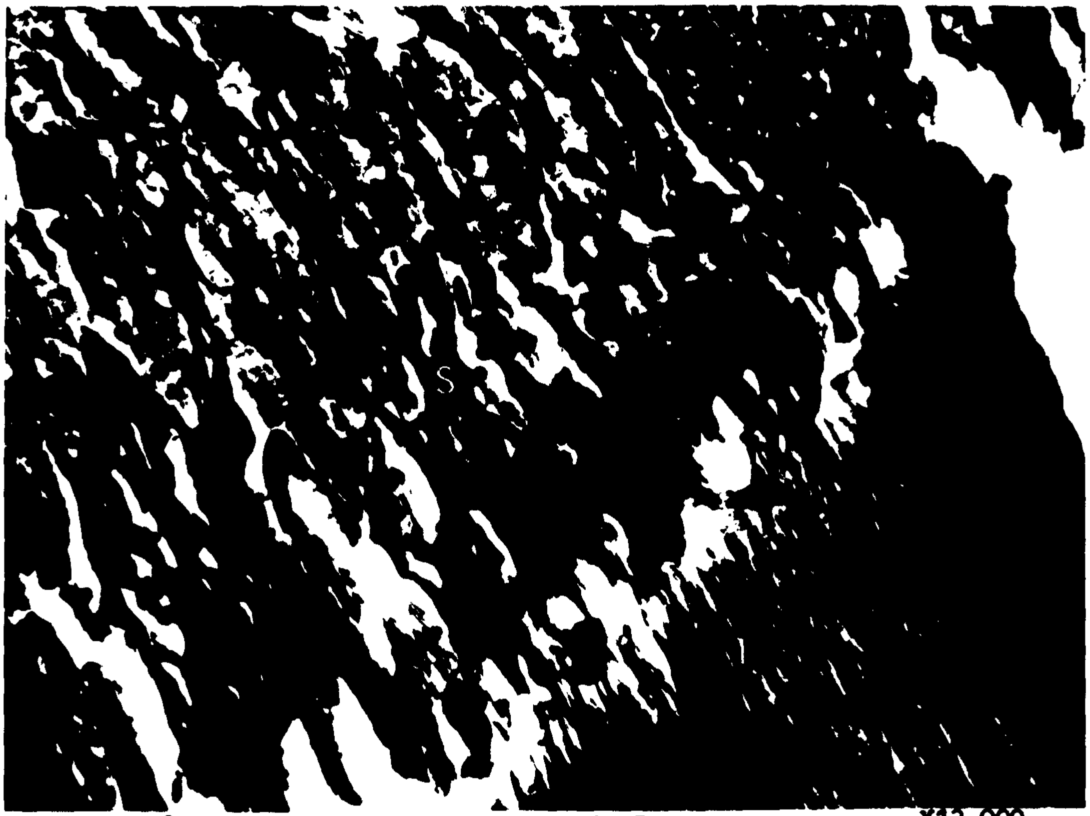
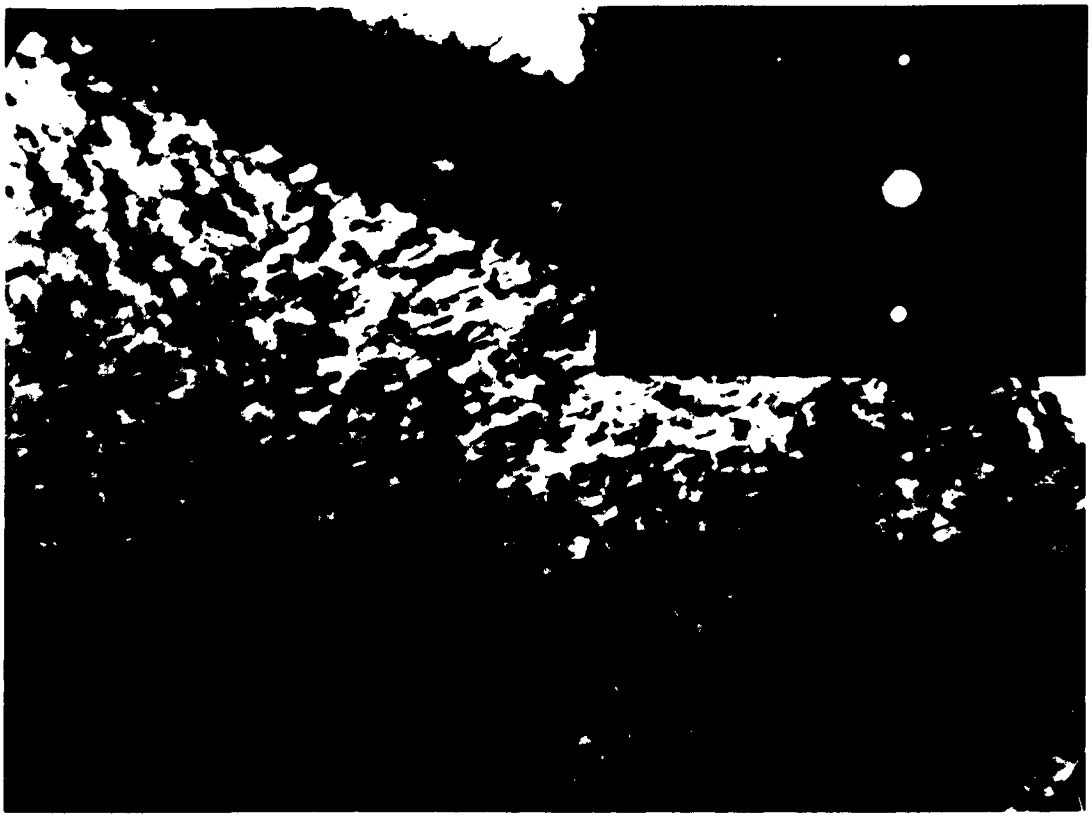


Figure 8 Plate No. 51005 X12,000

Figure 9 Plate No. 51788/9 X20,000

Figure 10 Plate No. 50897 X8,000



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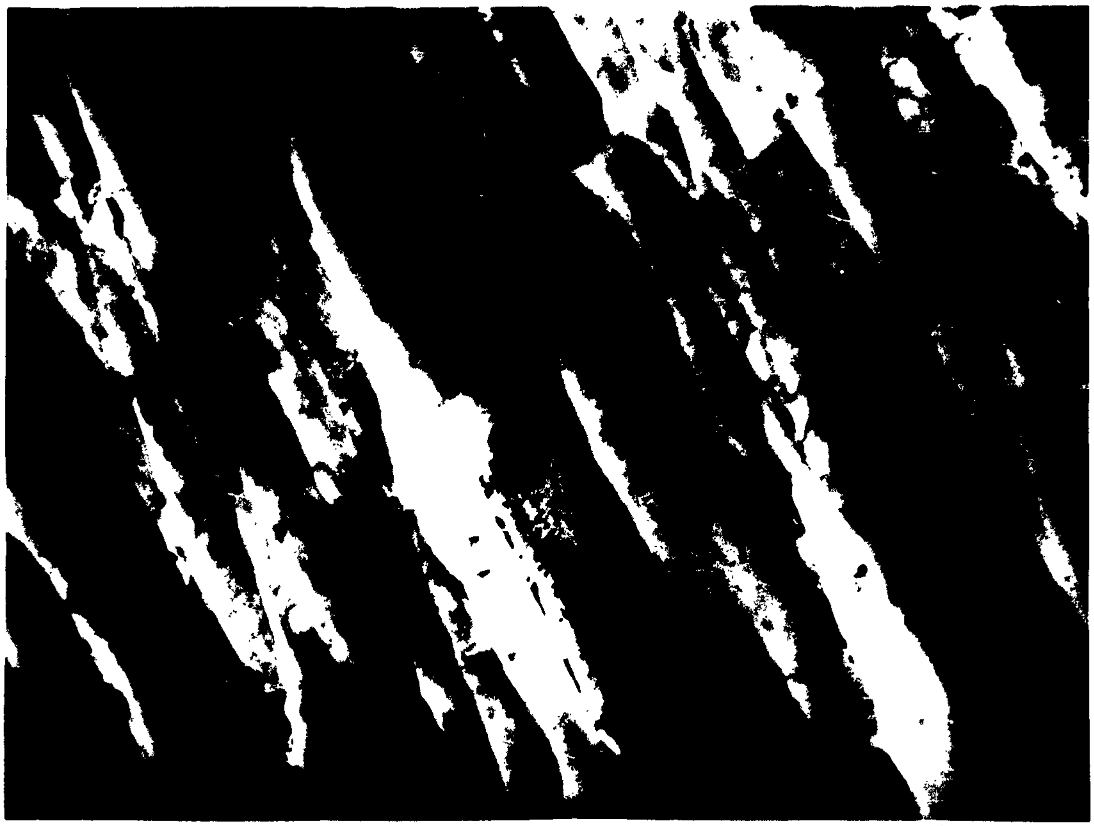

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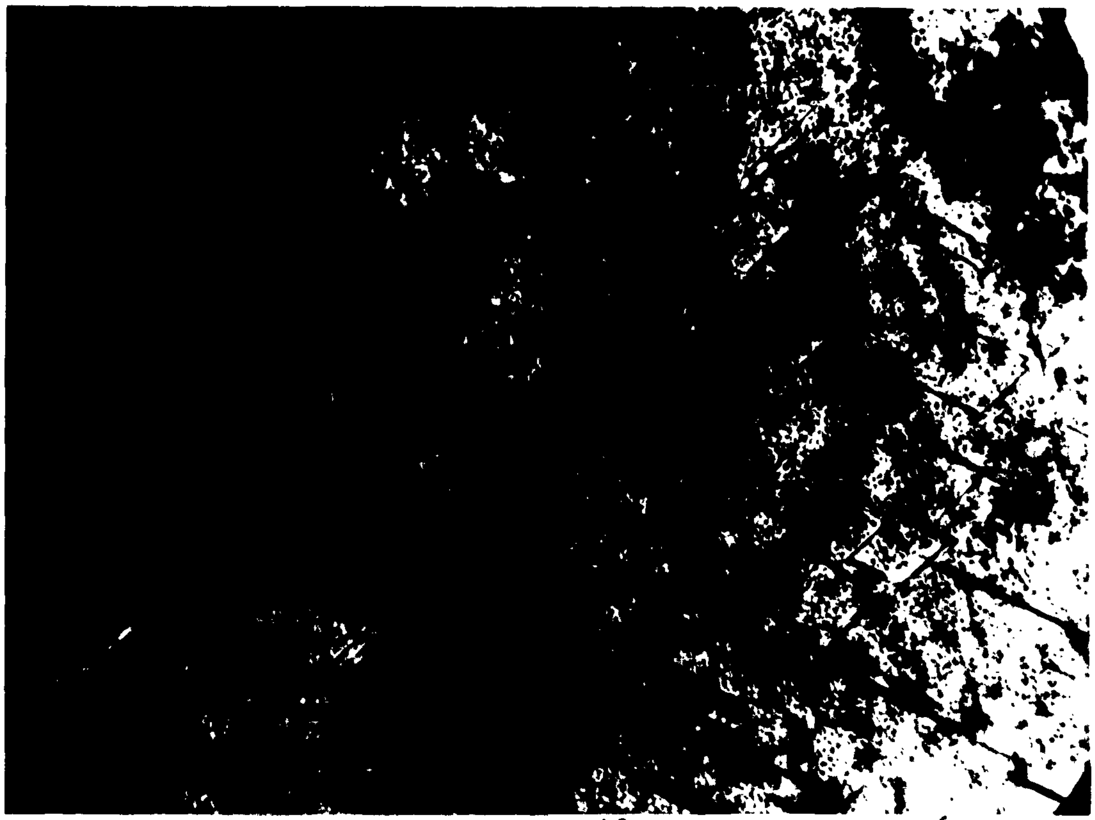

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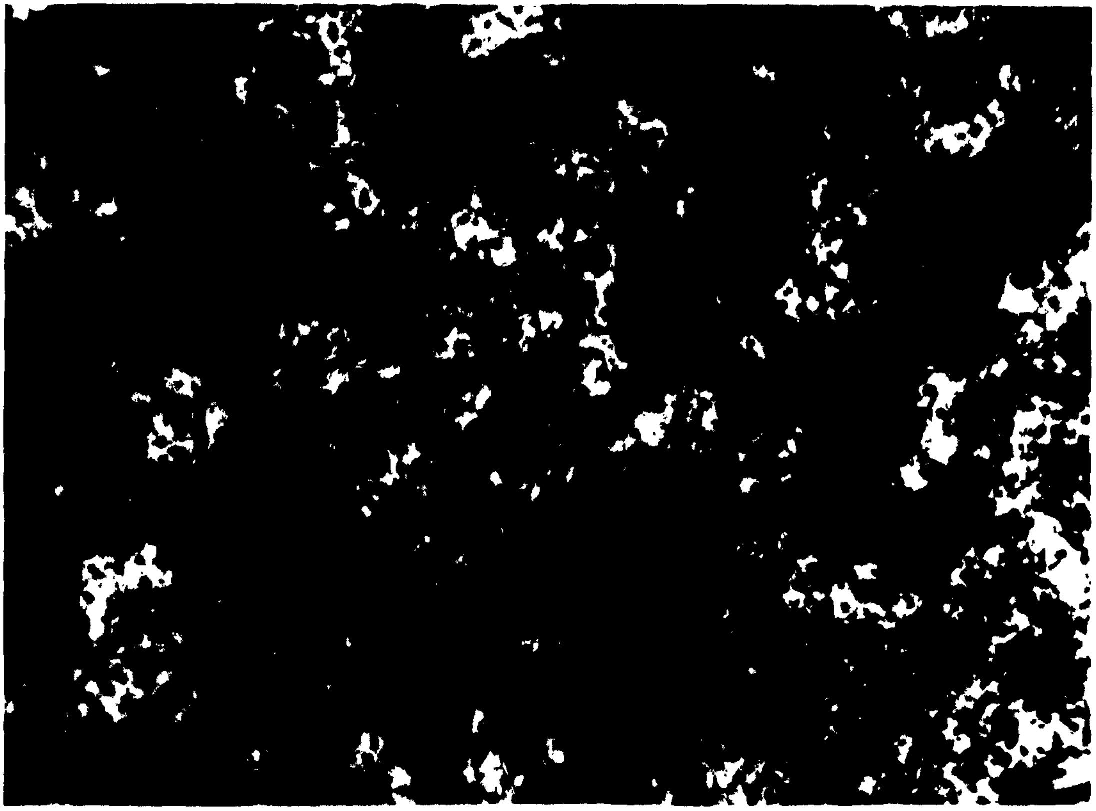

Figure 14 Plate No. 54562 X160,000

Figure 15 Plate No. 29181 X32,000

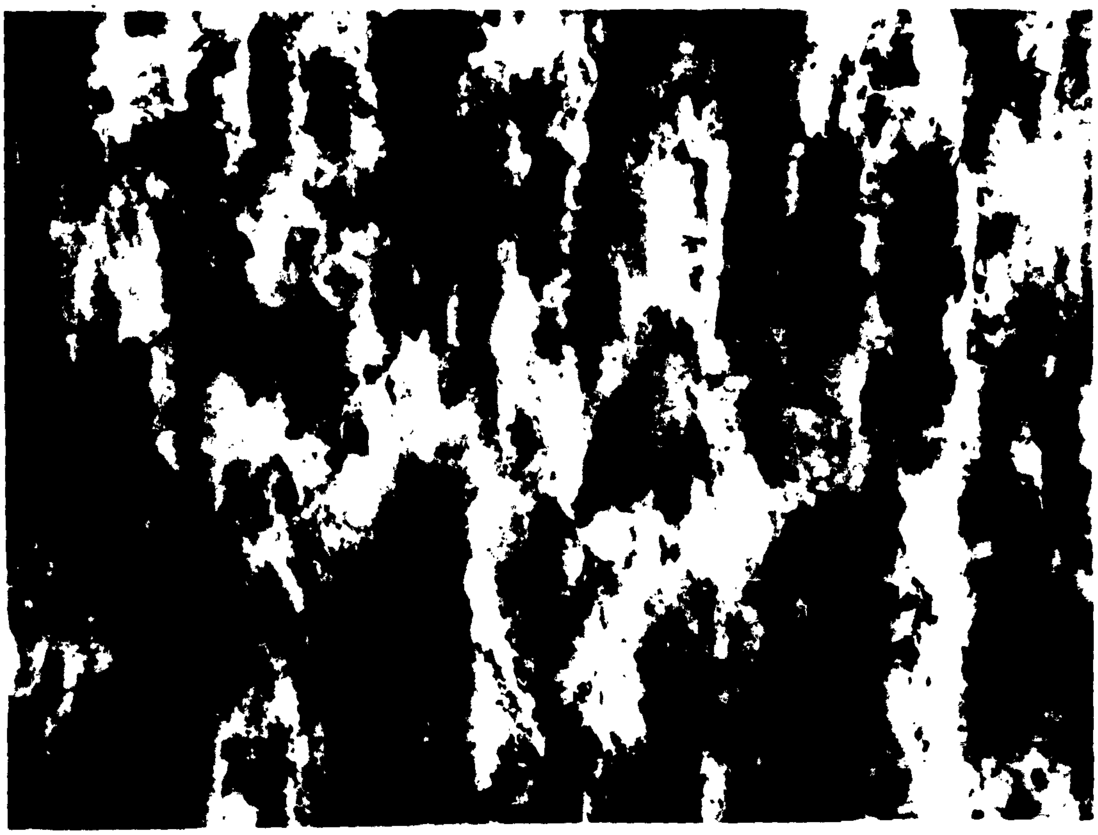

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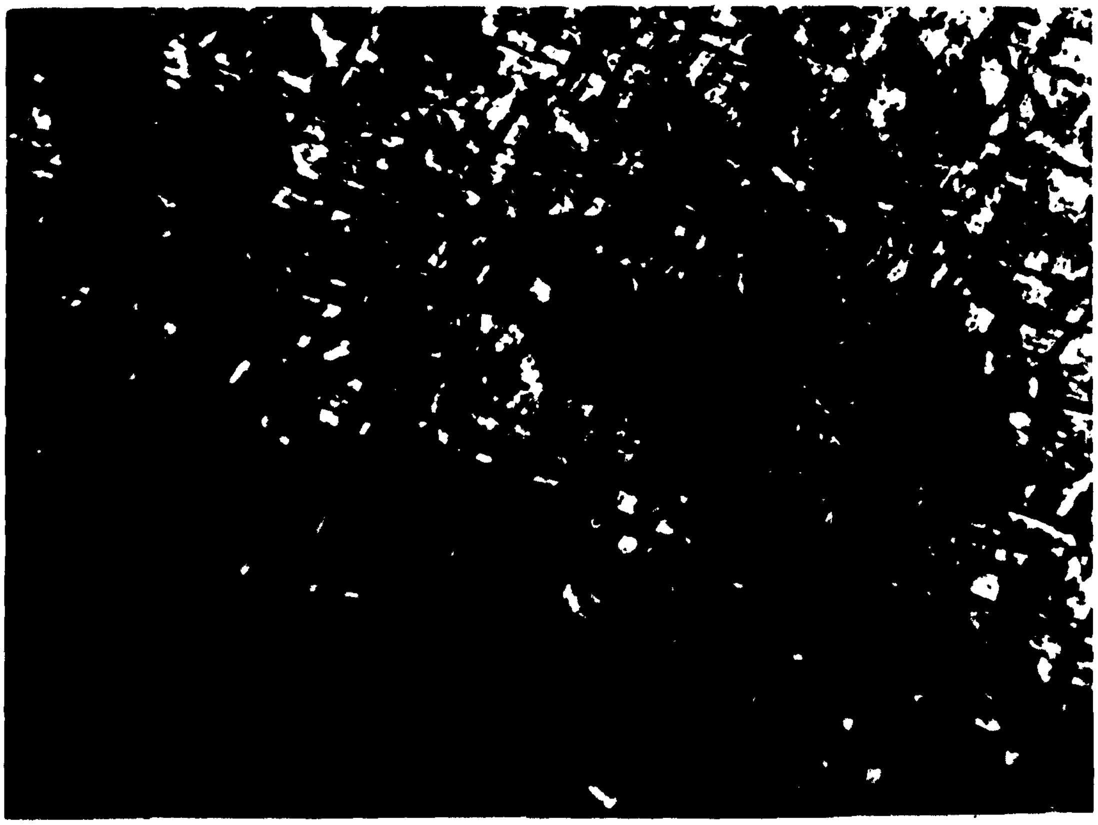
Figure 17 Plate No. 54871 X40,000



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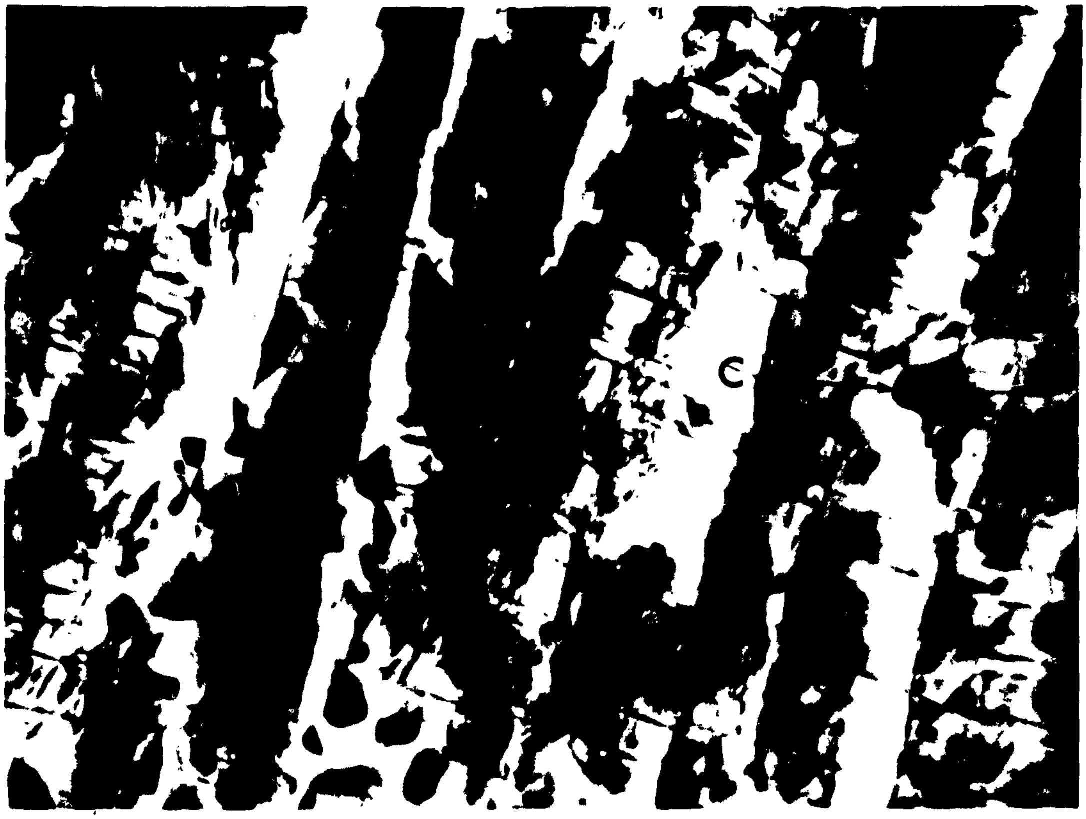

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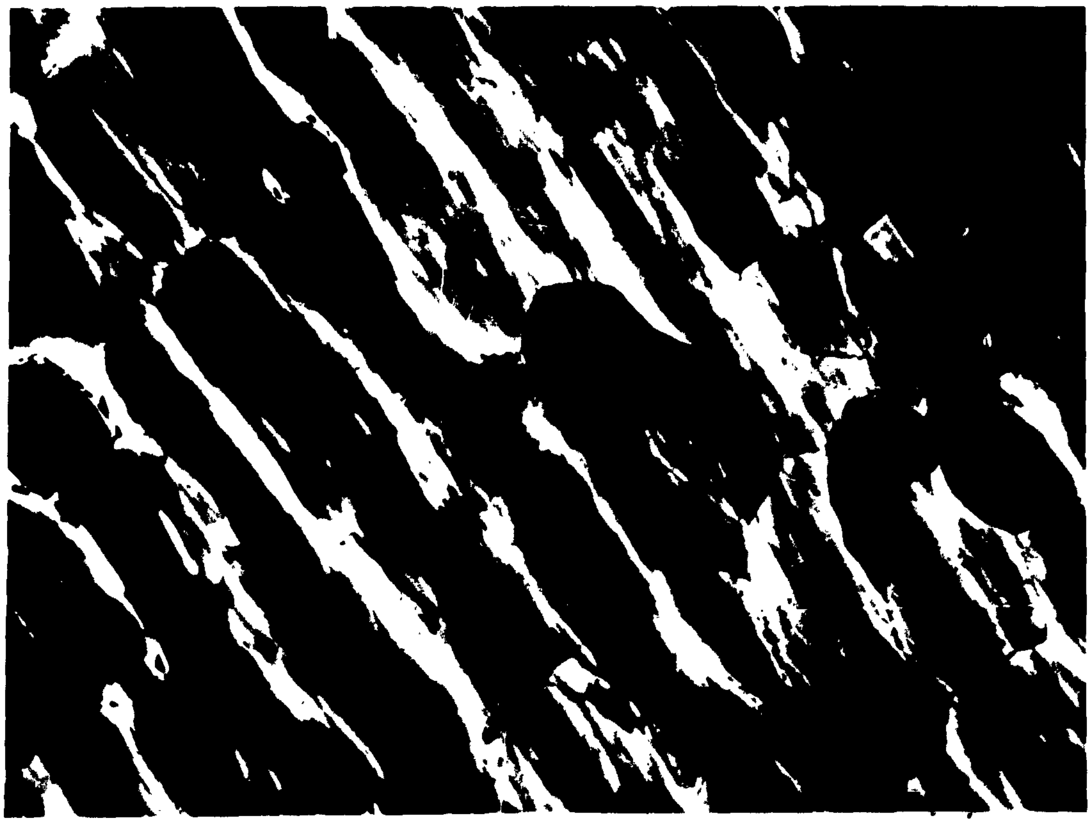

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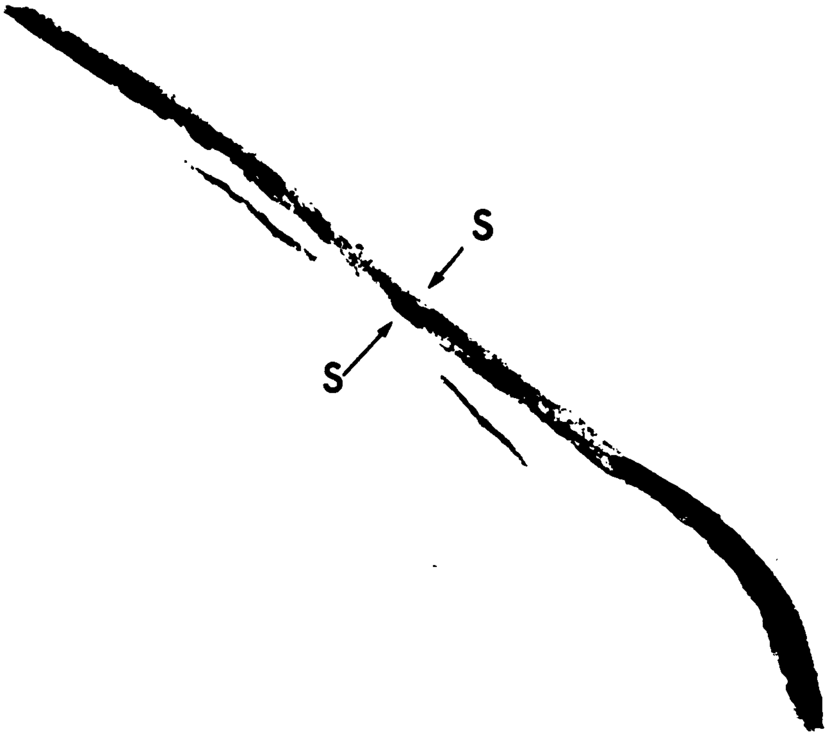

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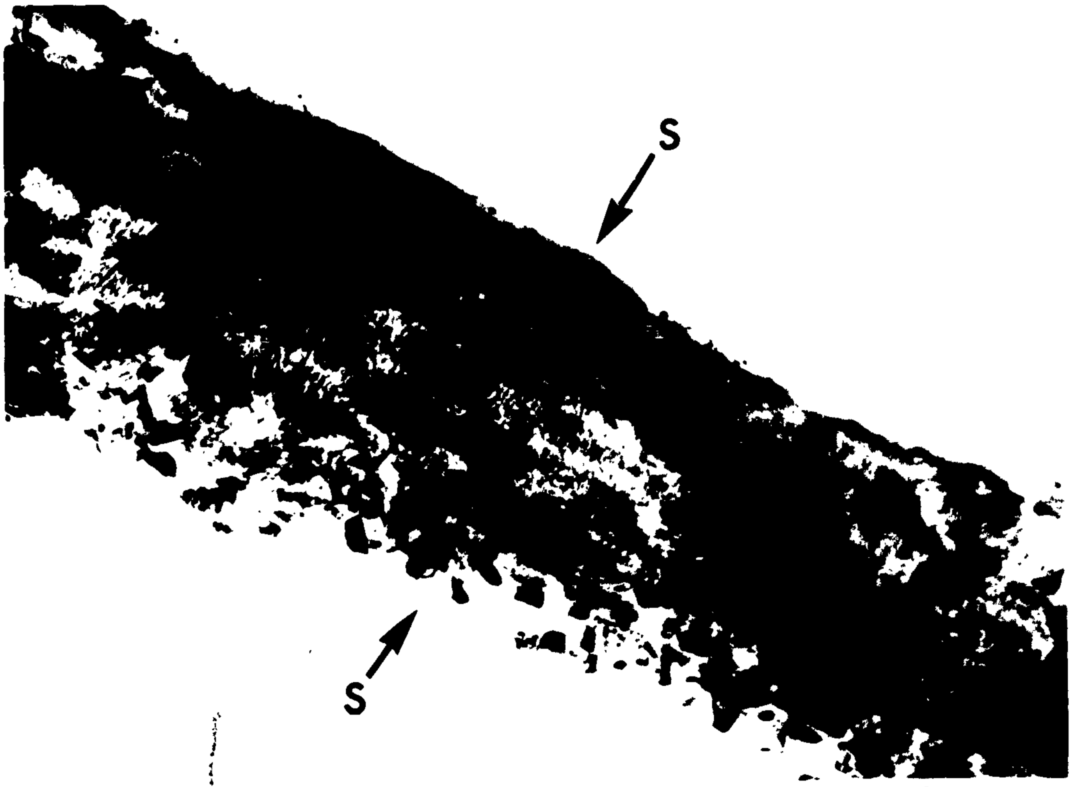

Figure 22 Plate No. 55066 X60,000

Figure 23 Plate No. 55072 X32,000

Figure 24 Plate No. 55068 X32,000