CHAPTER IX

A STUDY OF PLASTICALLY DEFORMED SINGLE CRYSTALS OF ZINC

In this chapter, are presented the results of etching crystals deformed by various methods. The deformation studies have been carried out extensively by various workers and the interest of the present author in this problem is limited to an examination of how far the various etching reagents are useful in revealing dislocations generated and redistributed as a result of plastic deformation. This problem is of utmost importance, because the major role which dislocations have to play is in the deformation of metals. The various deformations studied are (1) Non-basal slip (2) Twinning (3) deformation caused by indenting. The redistribution of dislocations resulting from the annealing of deformed crystals are also studied using the etch method.

Meleka[1] was the first to study the dislocations in plastically deformed zinc single crystals. From the distribution of the etch pits along the slip line on the surface of the crystal resulting from the basal slip, it was possible to conclude that pits are formed at dislocation sites. The prominent slip occurs on the basal plane (0001) in the $\langle 11\bar{2}0 \rangle$ directions. However, if a zinc single crystal is deformed in such a way that the basal slip is prevented, then the crystal is forced to slip in other planes. Gilman[2] has shown that when single crystals of zinc are pulled in tension parallel to basal planes at 250°-400°C slip occurs on the first order $\{10\bar{1}0\}$ prism planes in the $\langle 11\bar{2}0\rangle$ directions. Bell and Cahn[3] performing similar experiments at room temperature found that slip occurs on the second order pyramidal planes i.e., $\{11\bar{2}2\}$ in the $\langle\bar{1}\bar{1}23\rangle$ directions. Rosenbaum[4] using the etch pit technique has studied the non-basal slip and twin accommodation in zinc single crystals. The pattern of dislocations that intersect the basal plane of the deformed crystals has been revealed using an etching reagent consisting of 0.5 M. HBr in ethenol for 5 seconds. Observations of Bell and Cahn that at room temperature non basal slip occurs on the $(11\bar{2}2)$ pyramidal plane, has been confirmed by the author. Observations have also been made on the $(10\bar{1}2)$ mechanical twin that go partially through the crystal.

Brandt[5] et.al. have developed a method of etching $\{10\bar{1}0\}$ planes of high purity zinc without external solute decoration. Using this method quantitative agreement with Nye formula has been obtained. Observations on tilt boundaries and on specimens twisted about the [0001] axis also has shown good agreement between theory and experiment. The results of the studies on etched $\{10\bar{1}0\}$ planes, therefore indicate a strong one to one correspondence between etch pits and dislocations in high purity zinc.

Sinha and Beck[6] also have developed a reagent to reveal dislocations in $(10\bar{1}0)$ plane of high purity zinc, alloyed with small amounts of copper and aluminium. The crystals were bent and the polygonization phenomena was investigated. It was observed that these impurities do not have any effect on the polygonization. They do not have any effect on the mechanical polygonization also.

Unisovskaya and Stepenova[7] have studied deformation of single crystals of zinc alloyed with 0.1% cadmium, under compression parallel to the basal plane at various temperatures. The results indicate that the occurrence of slip system $(1\bar{2}12)$ $\langle 1\bar{2}13 \rangle$ predominates at room temperature, and those of the other systems namely $(10\bar{1}0)$ $\langle 1\bar{2}10 \rangle$ and $(11\bar{2}2)$ $\langle \bar{1}\bar{1}23 \rangle$ at higher temperatures. The dislocation pattern were revealed by the etch technique.

While the study of deformed crystals using etch techniques offers a wide scope of work for a careful investigator, the interest of the present author was limited to the adaptability of this technique to the dislocation pattern resulting from the deformation of the crystals. Therefore in the present study the results of the earlier workers are freely utilized within reasonable limits. The results

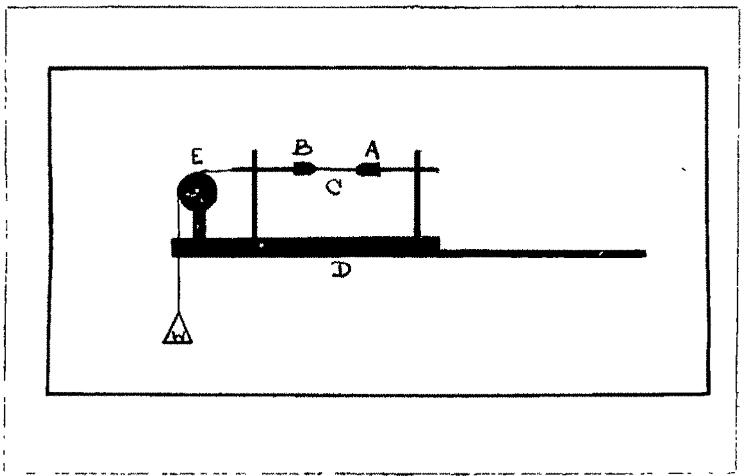


Fig. IX -1

obtained show qualitative agreement with the ealier results.

Crystals used for deformation under tension were 5 cms long and 0.6 cms in diameter with the basal planes parallel to the crystal axis. These crystals were cleaved carefully at liquid air temperature so that a specimens of rectangular cross-section are obtained after grinding the curved surfaces. The crystals were deformed under tension parallel to the basal plane. This was done using a device constructed for the purpose. The line diagram of this device is shown in fig.IX-1. A and B are two chucks fixed horizontally on two vertical blocks. A is fixed on a platform D, while B is movable along a groove in D. The arrangement is such that the blocks containing A and B always remain vertical and parallel so that no torque is exerted on the crystal during deformation. E is a pulley over which a wire connected to B is passing. The other end of the wire carries a scale pan to which the load can be added. The crystals were pulled by adding weights to this pan so that small amounts of deformation could be introduced. By careful observation of the surface the marking of the non basal slip can be observed on the basal plane. No quantitative measurements were made on the specimen. The crystals, after deformation, were etched with the various reagents listed in Table VIII-1. Fig.IX-2 shows the distri-

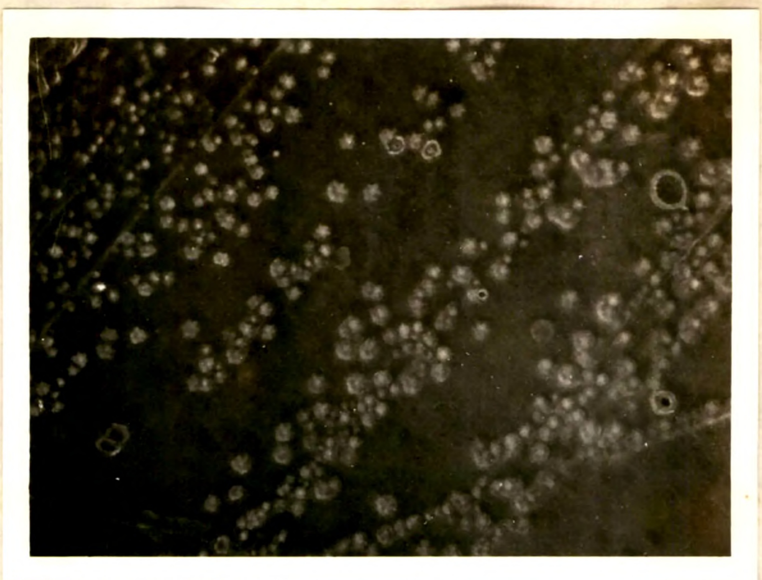


Fig. IX -2   X 360

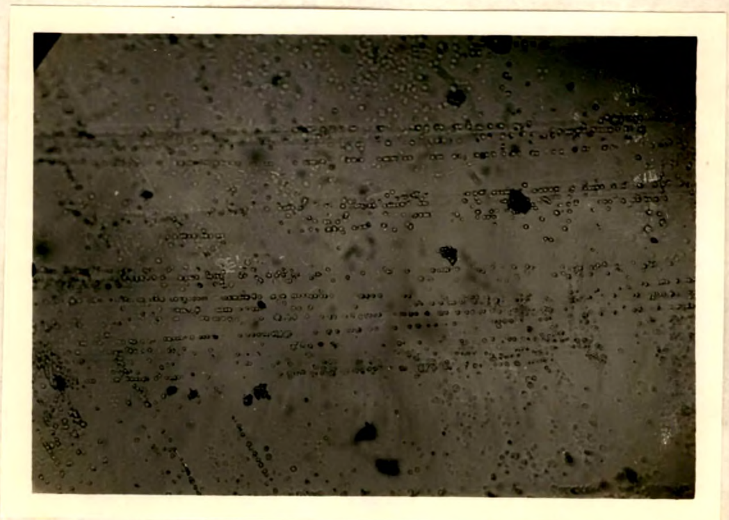
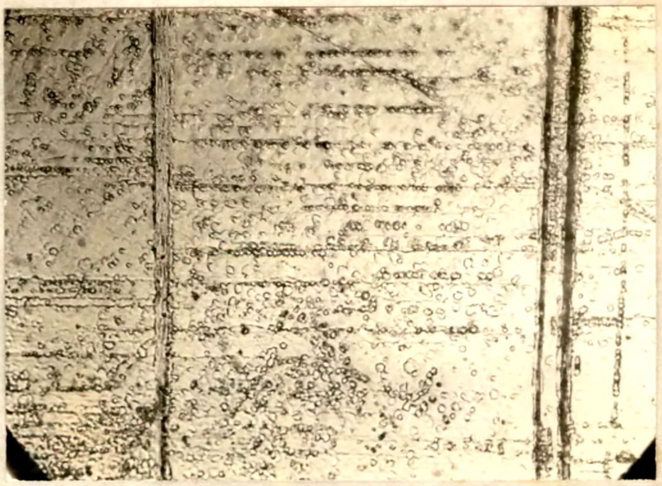


Fig. IX -3   X170

bution of etch pits along the non basal glide bands revealed by etching the specimen with HCl in Ethenol in the ratio 1:5. The same reagent has been used by Rosenbaum and Saffren[8] to reveal dislocation on the basal plane (Reagent J, Table VIII-1) and they have shown that the pits produced by this reagent are oriented with their edges parallel to $\langle 10\bar{1}0 \rangle$ direction. The pits in fig.IX-2 are oriented along the slip bands, the edges being parallel to the bands. Therefore, the bands lie along the $\langle 10\bar{1}0 \rangle$ directions which is consistent with the pyramidal slip in the $\{11\bar{2}2\}$ planes along $\langle \bar{1}\bar{1}23 \rangle$ directions. According to Rosenbaum and Saffren, these pits correspond to screw dislocations. The observation therefore suggests that screw dislocations formed as a result of plastic deformation can be revealed by etching without deliberate solute decoration and also that fresh dislocations resulting from this deformation can be revealed using suitable reagents. All the reagents listed in Table VIII-1 were tried on different specimens deformed under tension parallel to the basal planes. Iodine and none of the etchants containing chromic acid listed in Table VIII-1 was found to be suitable for the purpose. Reagent developed by Kosevich and Soldatov was found to be similar in nature to solution of HCl in Ethenol. Results obtained using bromine and HBr were similar to those of Rosenbaum. Superoxol and CP-4 were also useful. Fig.IX-3 shows the etch pits along non basal slip bands obtained by etching with superoxol.



Fig, IX -4 X 170

Fig. IX -5 X 360

Deformation twinning and the associated lattice movements have attracted the attention of various workers. Twinning in zinc crystals occur on the first order pyramidal planes $\{10\bar{1}2\}$. Twin bands appear as a result of this deformation when twinning does not go throughout the crystal, according to Jillson[9], the lattice will bend about an axis parallel to the twin. Such bending occurs by the formation of a kink or low angle tilt boundary perpendicular to the basal plane. Such kinks have been observed by various workers and is a common feature of twins. The boundary running parallel to the twin traces has already been shown to consist of dislocations as in fig.VIII-19. In addition to this, rows of etch pits running perpendicular to the twin traces are also observed. These etch pits are formed along the secondary kinks reported by Pratt and Pough. Rosenbaum has suggested that these are the pyramidal slip bands. Fig.IX-4 shows a twin band with a row of etch pits running parallel to the band and a number of rows running perpendicular to the twin band. In accordance with the views of Rosenbaum these bands are oriented along the $\langle 10\bar{1}0\rangle$ direction and these can reasonably be considered as slip bands of the $\{11\bar{2}2\}$ $\langle\bar{1}\bar{1}23\rangle$ system. The etch pits correspond to screw dislocations running perpendicular to the basal plane. Pits along such bands are shown by the various reagents. The particular case in fig.IX-4 is the result of etching with superoxol.

The low angle boundaries running parallel to the twin bands were not seen to develop etch pits in the Rosenbaum's work. This is explained by the author as due to dislocations lying in the basal plane. However when this accommodation kink is intersected by the pyramidal glide dislocations a row of dislocations parallel to the twin results. The non basal slip dislocations are partially stopped by the kink. It has been suggested that the row of dislocations may be the slip dislocations piled up at the kink. An alternative explanation is that the character of the slip dislocations is altered at the kink. The pyramidal glide dislocations with the $[11\bar{2}3]$ Burger's vector may react with the $[11\bar{2}0]$ type dislocations of the accommodation kink to give dislocations of $[10\bar{1}1]$ type on the twinning plane. However it has been possible in observations such as being presented in fig.VIII-19 that etch pit row running parallel to the twin bands are seen even when no secondary kinks and the associated non basal glide dislocations are observed. This therefore suggests that the dislocations in the primary kink need not always be parallel to the basal plane, even though cases similar to those observed by Rosenbaum are also seen. Therefore one has to be careful in inferring such interactions.

Another interesting feature associated with the twin band is the appearance of the rows of etch pits emanating from the tip of the twin. Fig.IX-5 shows such a row revealed by etching with bromine. Rosenbaum observes that these pits are due to dislocations lying in the $\{10\bar{1}2\}$ planes of twinning of the metal. The nature and origin of these dislocations is not known. But reasoning indirectly from the shear stress caused by the twin, it appears that the Burger's vectors lie in the shear direction of the twin i.e. $\langle 10\bar{1}1\rangle$ so that pits produced correspond to screw dislocation. Also it is not known whether these precede the twin while it grows through the strained lattice or whether they occur as the interface comes to rest to accommodate stresses at the tip of the twin. Kosevich and Bashmakov[10] have studied the elastic relaxation of the mechanical twins that pass partially through the crystal and conclude that if the twin is to remain inside the crystal after load is removed, then the stresses must be relieved. This view is supported by the observations on the twin bands, visible on the cleavage surfaces of the crystal. The tip of the twin is always characterised by dislocations in different distributions. In addition to the rows of etch pits observed on the tip of the twins shown in fig.IX-5, other features, such as seen in fig.VIII-16 are also quite prominent. Very often cleavage lines are seen to be nucleated and many a time a concentration of etch pits at the tip of the twin is also observed. Fig.VIII-16 is a case where all these are seen together and what is more striking is the repetition of the pattern in a mirror image fashion. This type of striking symmetry is seldom observed. The twins in the crystal are formed during cleavage and therefore these features suggest the symmetrical distribution of the stress resulting from the impact during cleavage.

Specimens 4x0.5x0.2 cm were cleaved out of a single crystal having the basal plane parallel to their axis. A number of these crystals were bent by pressing around brass cylinders of different diameters. The formation and growth of twin bands during the process has been studied. The twins appear as broad bands and pass throughout the crystal when the radius of curvature is not large. Such bands are shown in fig.IX-6. More often when the axis of bending is parallel to the $[10\bar{1}0]$ axis the crystals fracture along the $(10\bar{1}0)$ plane, when the radius of curvature is small. For large radius of bending they appear as thin bands and these do not often pass throughout the specimen. The distribution of etch pits after this deformation has been studied. The surface countour of these specimens remain same even after annealing. Wei[11] has shown that the crystal size has a decisive effect on the initiation of the mechanical twins. A crystal about 200 microns thick can be bent about the $[10\bar{1}0]$ axis a number of times without twinning. This observation has been confirmed in the course of this work and it has been found that specimens thinned to very small thickness by chemical polishing do not show twins after bending.

Fig. IX -6   X 90

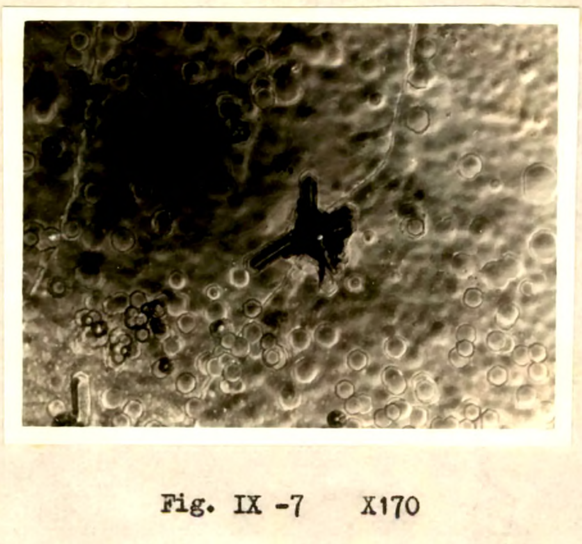


Fig. IX -7   X170

Verification of Nye formula offers the best qualitative test to establish the one-to-one correspondence between etch pits and dislocations. However, bending experiments in zinc become complicated by twinning. It is well known that zinc single crystals with their basal planes parallel to one of the bounding faces do not bend smoothly around any axis lying in the basal plane. Brandt[5] et.al. have determined the etch pit densities by bending the $[\bar{1}2\bar{1}0]$ axis of the crystal about the $[10\bar{1}0]$ direction. The resulting etch pattern on the $(10\bar{1}0)$ plane of the crystal satisfy Nye formula within the limits of experimental error. Since dislocations formed by bending the crystals in this way lie in the basal plane their presence and distribution can be studied only by etching the prism plane and therefore noné of the reagent made from halogens listed in Table VIII-1 useful for the purpose. It has already been shown that superoxol is effective on the$(10\bar{1}0)$ plane. However, the experiments on bending have not given satisfactory results. Brandt et.al. have obtained good agreement with Nye formula. Nye formula predicts a dislocation density given by $\rho = (rb)^{-1}$ where r is the radius of curvature and b the Burgers' vector which is 2.66A^{o}, (the lattice parameter in the $[1\bar{2}10]$ direction). For a curvature of 7.5 cms the expected dislocation density is 51×10^{5} cm^{-2} while Brandt et.al have obtained a value $54 \pm 10 \times 10^{5}$ cm^{-2}. In the present case the initial etch pit density was 5×10^{6} cm^{-2} and after deformation and etching the pit density is about $20 \times 10^{6} cm^{-2}$. In many cases the resolution of the etch pits is very difficult and surface presents a corroded appearance. The observed values are not uniform and very high. This may be due to the high initial dislocation density in the crystals used in the present work. It should therefore be admitted that no definite conclusions could be drawn from such studies.

Deformation of the crystals by indentation has also been studied. The indentation was done in a microhardness tester attached to the Vickers Projection microscope. A very characteristic feature of all the crystals deformed in this way is the appearance of twin bands emnating from the indented region. Subsequently, crystals have been etched with various reagents to reveal the dislocation configuration around these marks. In general it has not been possible to observe any concentration of etch pits around these marks. Fig.IX-7 shows a region which has been idented in this way, and subsequently etched with HCl in Acetic acid. The twin bands have been etched and a single etch pit is seen at the tip of the band. The results suggest that the crystal deforms during indentation by twinning and the bending of the surface. The surface around the four sides of the indentation mark is distorted heavily. The dislocation associated with these bends may be lying in the plane of the crystal

Fig. IX -8   X 170

Fig. IX -9   X 170

and hence are not revealed by etching. However, in fig.IX-8 after etching with an alcoholic solution of HI pits are seen around the indentation. But the pattern is not symmetrical and hence cannot be the result of indentation. Fig.IX-9 an indentation made at the edge of a twin band. The sharp bending of the twin band can very clearly be seen. On the sides opposite to the twin band a series of kink bands are also seen. Subsequent etching with HCl in acetic acid has not revealed any concentration of etch pits around indentation mark. The results can therefore be interpreted as that the accommodation of the indentor on the surface is facilitated by twinning and the resultant accommodation bending of the surface.

The results are in agreement with those of Rosenbaum. The deformation introduced by the above author was more macroscopic because of the large size of the indentor, which is a steel ball of 0.68 cm diameter. As a result of this the twin bands produced are larger in size and number. Even in this case no concentration of etch pits around the indentation is observed.

Effects of annealing the bent crystals at various temperatures and times have been studied. The annealing was done in an atmosphere of hydrogen at 250°C, 310°C, 350°C and 390°C for periods varying from 8 hours to 96 hours.

In all cases the phenomena of polygonization was observed. Figs.IX-10 and IX-11 show the polygonized are as on the basal plane after annealing at 310°C for 12 hours. Fig.IX-10 is the bent area of the crystal etched in superoxol after annealing. The pits in the different grains are arranged in linear arrays. Fig.IX-11 is the region heavily deformed by pressing in the vice. The number of blocks in this case is more their size being smaller than in the previous case. The etch pit distribution in the different subgrains are not the same. Fig.IX-12 show the intersection of three sub boundaries after etching in Bromine dissolved in acetic acid. The three regions can be easily differentiated. One grain is free from any observable pits. The results indicate that the reagent is very sensitive to the small change in the orientation of the subgrains caused by polygonization. The structure manifests itself as rectangular blocks on the $(10\bar{1}0)$ face of the crystal. The resultant etched surfaces are shown in figs.IX-13,IX-14, and IX-15. The fig.IX-15 is very similar to the polygonisation boundaries observed by Sinha and Beck.

The results presented above indicate that under suitable conditions etch pattern can be readily used to study the dislocation structure after deformation at least qualitatively. It has not been possible to show clear one-to-one correspondence between etch pits and dislocation.

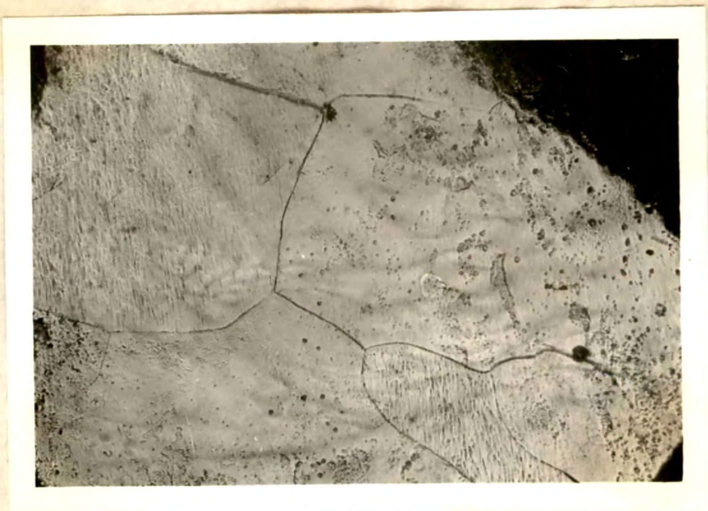


Fig. IX -10 X 170

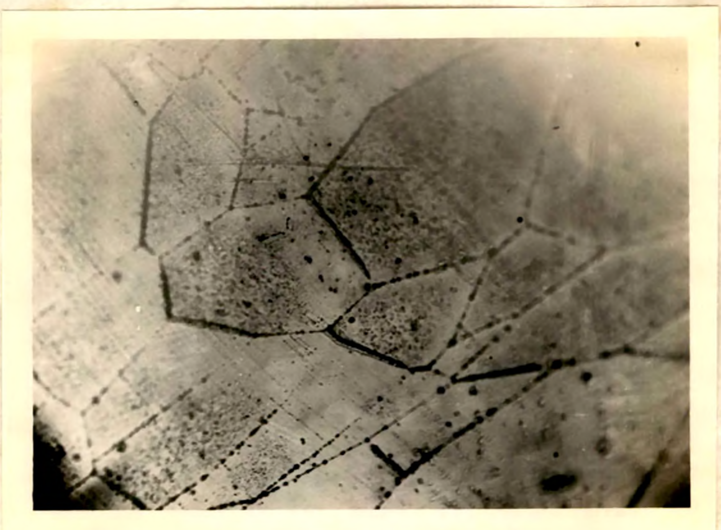


Fig. IX -11 X 170

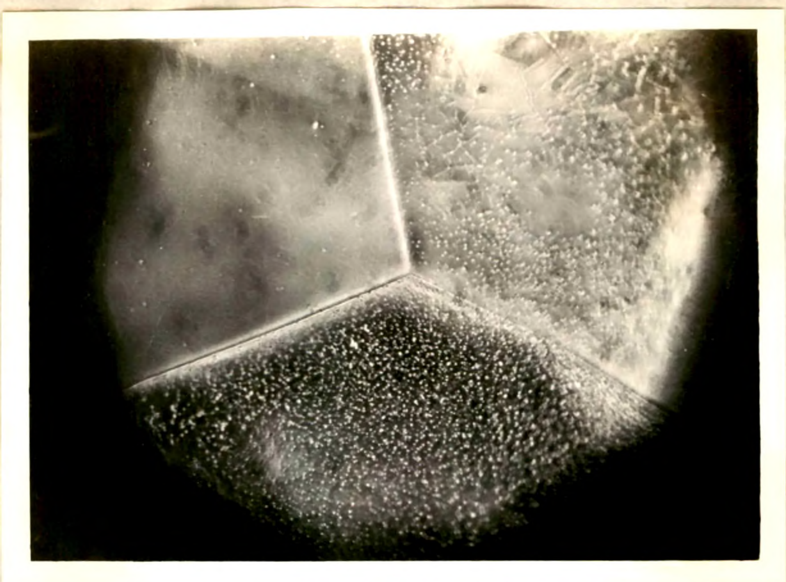


Fig. IX -12 X 170

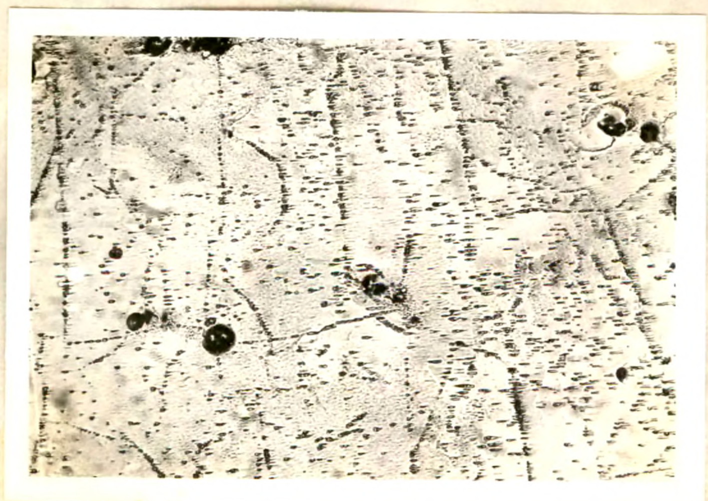


Fig. IX -13 X 170

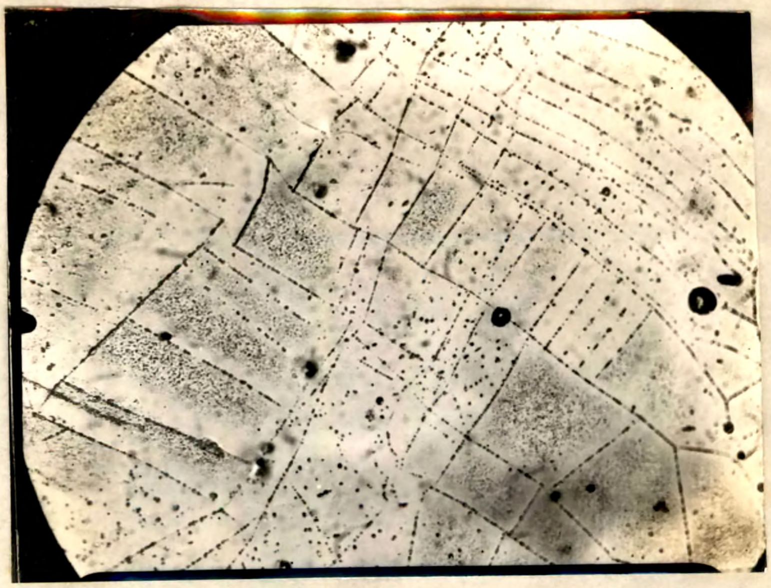


Fig. IX -14 X 170

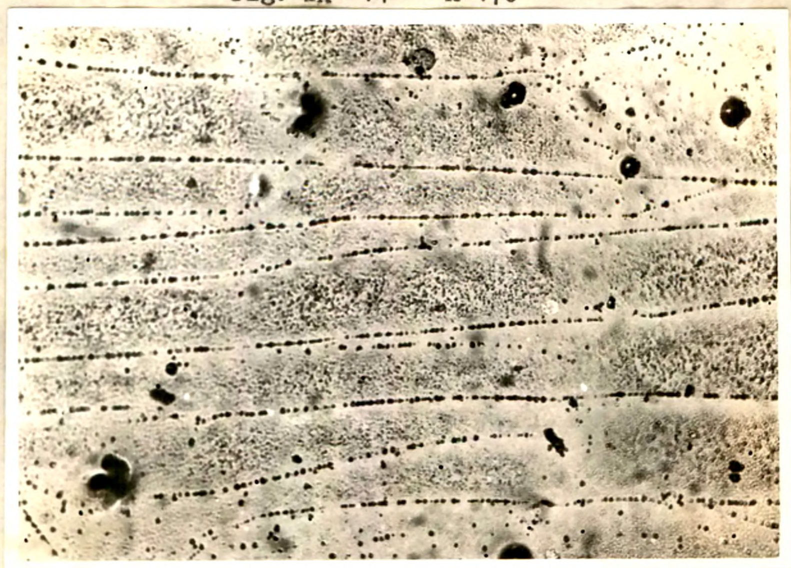


Fig. IX -15 X 170

The work reported in this chapter is neither exhaustive nor comprehensive but is only indicative of the fact that the etch phenomena can be used to study the dislocations in deformed crystals.

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