CHAPTER III

A BRIEF DISCUSSION OF ETCH PITS AND DISLOCATIONS

A brief review of the experimental results on etching of dislocations is presented in this chapter. The discussion is limited to metals and semiconductors, though references of the work on non-metals are made wherever it has become necessary. An attempt has been made to bring into the text all the significant results on etching. The present day understanding of the theory has also been discussed in brief.

Dislocations are regions of lattice defects in the material which give rise to local strains. It is possible to reveal their presence in a crystal by means of etchants which attack the sites of dislocations preferentially, producing etch pits. Thus a general conclusion has been made that such pits indicate the sites where dislocations emerge on the surface.

In 1940, Burgers[1] proposed a dislocation model for the boundary between two crystals differing in orientation by a small angle θ, about an axis in the boundary. This model consists of a series of edge dislocations, equally spaced, lying in the boundary and running parallel to the axis. If D is the dislocation density and b the Burgers vector then

$$D = b/\theta \qquad (1)$$

The first substantial proof that the presence of dislocations can be revealed by etching was made by Vogel _et al._[2] through their study on the lineage boundaries on germanium. It was the verification of eq:(1) by them that presented the first quantitative evidence in support of the theory of dislocation etch pits. They observed that on etching the lineage boundaries on the (100) plane of germanium, a series of regularly spaced conical etch pits were revealed. The implication was strong that these denoted the edge dislocations. The misorientation angle θ was determined by X-ray method and the distance D between dislocations was calculated using eq:(1). Excellent agreement was obtained between the observed and calculated values of dislocation spacing.

This discovery evoked the interest of many workers and several significant experiments have been conducted subsequently. This has made possible not only a detailed verification of large chapters of dislocation theory, but also the discovery of several new and unexpected facts. Though there are other techniques for the study of dislocations, the etch pit technique facilitates the study of dislocations in the bulk material. The semiconductor materials, germanium and silicon, being the materials largely required in the form of extremely perfect crystals for their varying applications, have been subjected to a wide range of investigations of this sort.

Etch pit technique has been successfully applied to study the dislocation in plastically deformed crystals. Vogel[3] carried out a study of the bent crystals of germanium. The crystals used were having $\{1\bar{1}1\}$ slip planes inclined at 42° to the neutral plane and slip directions $\langle\bar{1}12\rangle$ at an angle of 42° with the neutral axis. The crystals were sectioned to reveal the (111) plane, most nearly normal to the axis of bending and were etched in CP-4 solution and examined to verify the Nye formula

$$\rho = \frac{1}{rb\cos\theta} \qquad (2)$$

where ρ is the dislocation density, r is the radius of curvature of the neutral plane, b is the appropriate Burgers vector and θ the inclination of the neutral plane to the slip plane. There was general agreement between the observed and calculated dislocation densities, after annealing the specimen at 800°C for three days. On the contrary no such agreement was observed in the crystals which were not annealed. This anomalous behaviour has been explained in terms of work-hardening. This work is an excellent illustration of the way in which etch-pit technique can be used to obtain informations regarding the production of dislocations during plastic deformation and their motion under controlled conditions.

Greiner and co-workers[4,5] studied the rotational slip in semiconductors by twisting the crystals and then etching them. They observed that rotational slip took place only when a $\{111\}$ plane was normal to the torsion axis. They proposed a mechanism by which dislocations multiply.

Breidt _et al._[6] studied the dislocations produced by indenting the $\{110\}$ and $\{111\}$ surfaces of germanium at 400°C. Slip took place around the indentation mark which delineated all the $\{111\}$ planes intersecting the surface. On etching the specimens, pits were observed to form along slip lines. All these observations give support to the view that etch pits correspond to dislocations. However Ellis[7] observed a hundred-fold increase in the number of etch pits when an etchant based on potassium hypochlorite was used instead of CP-4. Pits produced by CP-4 etchant gets over etched by this reagent. These new pits are small in size and show inner details such as terraces and spirals. If the spirals are attributed to the screw dislocations, their Burgers vector must be large, of the order of 1000 A°. The significance of this is still not understood completely. Hobstetter[8] has discussed these observations in detail.

The work of Gilman and others[9-11] yielded detailed information on the dislocation etch pits in the case of LiF crystals. They have attributed the formation of dislocation etch pits by a dilute aqueous solution of ferric fluoride to the preferential nucleation of unit pits at dislocations, and the inhibition of the edges of these pits by ferric ions. The effect of ferric ions has been shown to slow down the motion of the steps across the crystal surface, producing steep, visible etch pits instead of the wide indistinct pits that form in pure water. The ferric ions appear to act by complexing with fluride ions of the crystal surface protecting the surface from dissolution.

Preferential nucleation of unit pits at a dislocation has been shown by these authors to be due to the energy (core energy and elastic strain energy) that is localized there. The authors have further pointed out that the localized energy depends on the nature of the dislocation and the impurity content, and as suggested by Cabrera[12], these factors affect the etching behaviour. The pit nucleation rate has been found to be slightly higher at an edge dislocation than at a screw dislocation. Impurities which segregate at dislocations have been shown to be responsible for the reduction in the nucleation rate.

One of the most elaborate problems that has been solved successfully by an etching procedure probably is the experimental determination of the stress-velocity relationship for individual dislocations by Gilman and Johnston[13]. Grown-in dislocations are found to be immobile as a consequence of pinning by impurities, associated with them. It is believed that the grown-in dislocations play only a passive role in the plastic behaviour of the crystals. Fresh dislocation half-loops were therefore introduced into the crystal and the velocities of dislocations were measured as a function of stress for both deformed and undeformed crystals. The velocity was found to be an extremely sensitive function of the applied stress; for a critical stress it increased rapidly and then approached some limiting value. Etch pit studies of dislocation multiplication in LiF single crystals have led to a simple description of the process[14].

Amelinckx[15] was the first to attempt the use of etching technique in finding correlation between dislocations and slip lines. He found that etch pits grouped along slip lines and certain grain boundaries in addition to a general random distribution. His experiments indicated that dislocations alone were responsible for the localization of etch pits on slip lines. On the other hand, the results of Wyon and Marchin[16] and Wyon and Lacombe[17] indicated that dislocations alone were not sufficient to explain the localization of some etch pits on the slip lines in aluminium single crystals, but the segregation of impurities at dislocations was necessary for the formation of etch pits. Thus it was believed that dislocations in metals could be revealed as etch pits only if they were decorated by impurities. For example, dislocations in zinc were decorated by Cadmium[18] in iron-silicon alloy by carbon[19] and in copper by tellurium[20].

However, in recent years several etchants have been developed that reveal dislocations in metals and semiconductors without decorating them by impurity segregation. Such etchants are usually reliable only for etching low-index planes.

Lovell and Wernick[21] have developed an etchant for copper that reveals undecorated dislocations and have obtained the evidence for dislocation climb. Modifications of their etchant led to two dependable etchants for $\{111\}$ planes, viz. that of Livingston[22] and that of Young[23]. In a series of publications Young has reported an excellent and systematic study of etch pits on copper single crystals. Etchants have been developed which can reveal dislocations on the (111), (100) and (110) faces and can distinguish between 'clean and 'dirty' dislocations with a Cottrell atmosphere, and also between edge and screw dislocations. Crystals were bent under tension and the density and distribution of etch pits were studied, before, during and after the deformation. For crystals of low dislocation density, it was found that a large amount of dislocation multiplication occured prior to yielding. The yield stress was found not to be dependent on the initial density and distribution of the dislocations. Annealed crystals with a dislocation density of 10^3 to $10^4/cm^2$ were bent in a 4-point jig and etched. Dislocation motion and multiplication prior to yield were observed. There was evidence for dislocation interaction and pile-ups at sub-boundaries. The spacing of the pits did not correlate with the expected value in the case of pile-ups. This was attributed to the fact that the plane of etching did not contain the barrier. Young also studied the effect of various doses of neutron irradiation on the density and distribution of dislocations. For these crystals dislocation multiplication before yielding was not observed. This result has been suggested to be due to source hardening.

The results obtained by Ruff[29] on single crystal foils of copper using etching as well as transmission electron microscopy, however, showed that one-to-one correspondence did not exist in these crystals. There were grown-in dislocations which were not etched, suggesting atmospheric effects and formation of etch pits which could not be attributed to dislocations.

Livingston[30] made the astonishing discovery that positive and negative dislocations in copper can be distinguished by etching $\{111\}$ planes with a proper etchant. The two kinds of dislocations were found to produce pits of different depths on the same crystal face and appeared as light and dark pits on the same micrograph. The origin of the difference in etching rate is not well understood now.

Lovell and Wernick[21], Pandya and Bhatt[31] and Kosevich[32] have studied the etching of the (111) cleavage plane of Bismuth. Evidence that the pits were at dislocation sites was obtained from deformation and annealing studies and from density counts on intersecting tilt boundaries by Lovell and Wernick. Shah[33], using an etching reagent that reveals dislocations,has studied the distribution of dislocations around an indentation mark on freshly cleaved (111) planes of bismuth. The observed characteristics have been explained by the author on the basis of the proporties of bismuth and the Frank-Read mechanism of multiplication of dislocation.

Wernick et al.[34], Shigeta and Hiramastu[35], and Pandya and Bhatt[36] were able to produce pits and terraced pits on the (111) cleavage plane of antimony using different reagents. Using modified CP-4 Kosevich[37,38] studied etch pits on the (111) plane of antimony. He observed etch grooves connecting large etch pits whose origin is not yet understood completely. The block boundaries, twin lamella, slip lines and pile-ups were studied by the authors. By employing selective etching Soifer and Startsev[39] studied the motion of dislocations in antimony single crystals. They observed wedge-shaped tracks having point-bottomed pits at their terminations which were interpreted as due to dislocations which were arrested. The tracks coincided with the $\langle 1\bar{1}0 \rangle$ directions which are the lines of intersections of three $\{11\bar{1}\}$ planes with the (111) cleavage plane. Thakar[40] observed dislocation pile-ups in the case of antimony single crystals grown from the melt. The dislocations were revealed by etching with a modified CP-4 reagent.

Etch features on zinc has been investigated by many workers and a number of etchants that reveal dislocations have been developed. The first systematic study of distribution of etch pits in high purity zinc was done by Meleka[41]. A satisfactory agreement between the theoretical and experimental values for the spacing of pits was obtained, assuming every pit to be due to a dislocation of unit strength. However, the density of pits observed by these authors was found to be relatively low even after the crystals were severely deformed, indicating that their etchant did not reveal all the dislocations produced by the deformation. Pits representing low-angle boundaries, polygonization boundaries, pile-up of dislocations, slip bands, etc. were observed by Gilman[42] in high purity zinc single crystals and cast polycrystals of zinc containing 0.1 atomic per cent of cadmium as impurity. Servi[43] observed etch spiral patterns on a plane nearly parallel to the basal plane of zinc. He also observed some complex spirals which were subsequently interpreted by Frank and Vreeland[44] in terms of dislocation theory. George[45] reported an etching technique which produces spirals and star-shaped pits on the (0001) face of zinc crystals grown from vapour and droplets. Dislocations that lie on the (0001) plane cannot be revealed by this technique. Rosenbaum[46] and Rosenbaum and Saffren[47] concluded from their study on the basal plane of zinc crystals that screw dislocations can be revealed without prior decoration. They also studied non-basal slip and twin accomodation in zinc crystals by means of the etching method. The existence of a one-to-one correspondence between etch pits and dislocations have been shown by Bradt _et al._[48,49], from their studies on etching of high purity zinc. Their studies also included the slip on (0001) and $\{1\bar{2}12\}$ planes. Damiano _et al._[50] using the etchant developed by Gilman[42] studied the arrangement of dislocations in zinc single crystals doped with cadmium. The effect of annealing was evidenced by the arrangement of dislocations into low-angle boundaries and hexagonal net works. Pandya and Shah[51] using acetic acid, produced pits on the (0001) planes of zinc, probably on the dislocation sites. Pandya and Balasubramanian[52] used 0.05% iodine in ethyl alcohol to reveal dislocations on the basal plane. They have observed elongated hexagonal pits, suggestive of dislocations inclined to the basal plane.

George[53] studied the etch pattern on vapour-grown cadmium single crystals. Using a saturated solution of picric acid in acetone he observed complex spirals on the basal plane of some of the crystals. In some cases etch patterns closely resembling hexagonal spirals with the dislocation line emerging at the centre were observed. The formation of etch pits has been attributed to the presence of dislocations.

Tyapunina *et al.*[54] used the etching technique to study the relationship between dislocations and concentration of zinc in cadmium. They have shown that the presence of dislocations have an important effect on the distribution of zinc in the cadmium crystals, both within the solubility range of zinc and the two-phase region of the equilibrium diagram.

Tyapunina and Zinenkova[55] have developed chemical etchants for revealing undecorated dislocations in high purity cadmium single crystals. They have studied the dislocations formed during the growth process and deformation of plate-like single crystals of cadmium. Analysis of the etch figures on deformed and undeformed crystals and the results of successive etching have shown that the etch pits are formed at the sites of the emergence of the dislocations. Etch patterns on the surfaces of high purity cadmium single crystals have been recently investigated by Pandya and Thattey[56].

Levinstein and Robinson[57] developed an etch pit technique to reveal the dislocation structure in the (100) and (111) surfaces of silver single crystals. Evidence was presented establishing a one-to-one correspondence between the etch pits and the dislocation line intersecting the surface. The authors showed that the etching technique when used in conjunction with electron microscope replica method, is capable of revealing dislocations having densities upto 10^{10} lines/cm^2. Preliminary data on the structure of slip bands in silver indicated that the slip bands have a cellular structure of the type observed by the transition electron microscopy.

Evidence has been obtained to show that impurities have an effect on the size, shape and density of pits, from the studies on electrolytic and chemical etching of tungsten[58]. Spreadborough *et al.*[59] employed etch pit procedures to gain qualitative information regarding processes involving twins in iron and iron alloys. The etch pit procedure revealed only about 10% of the dislocations thought to be present in the crystals and indicated that the local stresses around twin-twin and twin-grain boundary intersections were at least 20% higher than the over-all stresses at a distance of about 10 microns from the intersections.

Lovell _et al._[60] used a hydrofluoric-nitric-acetic acid mixture to reveal dislocations on $\{10\bar{1}0\}$ faces of tellurium single crystals. Pits, which were distributed randomly, along grain boundaries and around mechanically worked regions, were obtained by the authors. Using hot concentrated sulphuric acid Blakemore _et al._[61,62] produced well-defined pits having two types of shapes on prism planes of laevo- and dextro-type tellurium crystals. The shapes of the pits were not symmetrical about the c-axis and were found to be mirror images of each other. This has been attributed to the structure of tellurium which places it in the enantimorphous class. Blum[63] has reported solutions which reveal dislocations on faces parallel and perpendicular to the c-axis in tellurium.

Koma *et al.*[64] have investigated the shape of etch pits produced by the slow action of hot sulphuric acid on the cleavage planes of tellurium. They have also attributed the characteristic shape of the pits to the peculiar crystal structure of tellurium. A microscopic model is proposed for the formation of the etch pits and the left or right handedness of the spiral chain and the +ve direction of the x-axis are determined on the basis of the proposed model.

Etching the $(10\bar{1}0)$ faces of tellurium with hot concentrated sulphuric acid Shukla[65] has observed that the pit size is markedly temperature dependent. He could observe etch grooves and terraced pits on the prism faces of vapour grown tellurium crystals. The etch grooves are attributed to dislocation lines running almost parallel to the plane of observation. The terraced structure is attributed to non-continuous segregation of impurities at dislocations.

It is known that the strain energy of a dislocation line is small. So a successful etching technique to delineate it must be able to differentiate small differences in energy. The work of Chalmers and King[66] on silver has indicated that thermal etching might accomplish this purpose. They observed that thermal etching at 300°C revealed grain boundaries and at 600°C started becoming etch grooves. Young[67] observed on copper crystals annealed under hydrogen atmosphere and then heated to a temperature of 1050°C under vacuum, spirals around (111) poles which were attributed to the screw dislocations terminating at the surface around the (111) poles. The work of Machlin[68] on the thermal etching of silver crystals showed that the pits were formed at stationary dislocation grooves. They also showed that the screw oriented dislocations could be distinguished from those of the edge oriented ones in that that the pits at the former ones were several times smaller and shallower than that at the edge dislocations. This shows that besides chemical etching the thermal etching can also be used successfully to reveal dislocations.

In thermal etching removal of the atoms occurs mainly at the exit termination of dislocation lines on the surface since the binding strength of atoms is weak in a dislocated region. One-to-one correspondence between the thermal etch pits and dislocations is not yet fully established and a careful study is necessary before any definite conclusions can be made.

The observations presented above indicate that though one-to-one correspondence between etch pits and dislocations is not obtained in all cases, excellent agreement is indeed obtained in certain metals, non-metals and semiconductors. The etch pit technique has proved to be a powerful tool to reveal dislocations and to study the mechanism of plastic deformation in the hands of a careful investigator, as evidenced in the case of copper. On the other hand, the results of Ellis and Ruff seem to warn us that the technique should be used with care. In view of the contradictory nature of the results one has to conclude that only when the pits aggregate in a manner clearly attributable to dislocations or produce distinct orientation effects, can they be safely regarded as dis- locations.

A new approach to the problem of dissolution (or growth) has been made by Frank[69] and Cabrera[70], which is being called the kinematic theory of dissolution (or growth). The basic assumption of this theory is that dissolution (or growth) of a crystal occurs by the removal (or deposition) of atoms at a step, resulting in the progression of the step across the surface. The development of the form of the crystal then takes place by the creation of new steps and the motion of the steps across the surface. The application of the theory to the problem of dislocation etching has been attempted by Cabrera[70]. When the step flux 'J' at a given point on the surface is a function only of the step density 'ρ' at that point, the kinematic theory predicts that points of a given orientation 'ρ' will have straight line trajectories as dissolution proceeds.

Batterman[71] has attempted to explain the appearance of facets on hillocks in the case of germanium in terms of the measured orientation dependence of the etch rate. Using this, he concluded that the $\{322\}$ planes are stable hillock facets on germanium etched with superoxol. Irving[72] in a further analysis of this approach concluded that for the same etchant $\{310\}$ may also be stable hillock facets on germanium. He also extended the work to include pit facets.

Frank and Ives[73] applied the theorems of dissolution developed by Frank to the results of Batterman. Derived dissolution profiles were found to correspond to those obtained in the experiment. The authors inferred that the etch rate for germanium in superoxol is, to a first approximation, only dependent on crystal orientation. Ives[74] was the first to demonstrate the quantitative applicability of the kinematic theory to the dissolution of LiF.

The kinematic theory of crystal dissolution has been applied to a study of dislocation etch pit growth on (111) surfaces of copper undergoing anodic dissolution in solutions containing

HCl and HBr[75]. The dissolution shapes of these pits at successive times have been determined from interference photomicrographs taken as the reaction progressed. The evolutions of the shapes have been shown to be in accord with predictions of the theory.

In an etch pit study of the (0001) surface of zinc dissolved in alcoholic hydrochloric acid solutions, it has been observed by Ives and McAusland[76] that the slope of etch pits are controlled by the dissolution kinetics of the crystal. Pits have been found to widen at a rate independent of the type of defect attacked, while the slopes are dictated by the relative rates of dissolution at the defect sites.

Inspite of the developments in the understanding of the dissolution process, there is still some controversy regarding the details of the mechanisms by which dislocations affect dissolution process and as such there is a genuine need for a better definition of the mechanisms involved.

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