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K. L. Bates

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AN INVESTIGATION OF FERROMAGNETIC DOMAINS IN SURFACES
INCLINED TO THE (110) PLANE OF CRYSTALS OF SILICON-IRON

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K.L. Bates.

ABSTRACT

The magnetic domain arrangements, in surfaces slightly inclined to (110) planes of Si-Fe crystals, have been studied by the Bitter pattern technique. The relationship between the pattern and the inclination of the surface to the (110) plane was examined by observing the patterns on crystals cut from thin sheets and then measuring the surface orientation by means of a two-circle goniometer. The results were checked by observing the changes in patterns as the surface of a thick and a thin crystal were ground through successive small angles to increase the inclination of the surface.

It is concluded that as the inclination between the surface and the (110) plane is increased up to angles of about 10° - 12° the resulting magnetostatic energy is minimised by the formation of reverse lozenges. The lozenges increase in number as the angle increases and eventually form a lace pattern which completely obscures the underlying domain arrangement. A pattern of reverse lozenges arranged in rows perpendicular to the $[001]$ direction, a criss-cross pattern and a zig-zag pattern were frequently observed but could usually be removed by annealing. It is suggested that these patterns indicate the presence of strain.

An arrangement of tadpole-like deposits has been investigated. The results suggest that this is a surface arrangement in which the vectors point along the $[100]$ and $[010]$ directions at $\approx 45^{\circ}$ to the surface. The structure is considered to be a 'transverse' domain which is formed during demagnetisation and plays an important part in the nucleation process.

Chapter 1

INTRODUCTION

1.1. Formation of Domains

A ferromagnetic substance is characterised by the fact that it can be magnetised to saturation in relatively weak fields and that, below the Curie temperature, it can have a finite intensity of magnetisation even when the applied field is zero. The first satisfactory explanation of the spontaneous magnetisation was given by Weiss who assumed that an internal field, proportional to the intensity of magnetisation of the specimen, acted on each atomic dipole. This field would tend to align the magnetic dipoles parallel to each other though the alignment would be opposed by thermal agitation. A statistical treatment showed that a finite intensity of magnetisation was possible with zero field and also gave a relationship between this intensity and the temperature which was in close agreement with experiment.

In 1928 Heisenberg showed that, according to the quantum theory, an 'exchange interaction' of electrostatic origin should

exist between atoms. In the case of the ferromagnetics, the result of the interaction is that minimum exchange energy is achieved only when the spins of electrons in the incompleted subshells - and, therefore, the magnetic moments of the atoms - are aligned parallel to each other.

If this were the only energy factor involved, a single crystal of a ferromagnetic substance would be permanently magnetised in one direction. Magnetisation curves for iron, nickel and cobalt (Honda and Kaya, 1926) clearly indicate that the crystals can be more easily magnetised in one direction than another. In other words, the crystals are magnetically anisotropic and it would appear that, for minimum energy, the electron spins must be aligned parallel to some preferred direction within the crystal lattice. The origin of this magnetocrystalline anisotropy is not completely understood but it is possible to represent the energy involved by suitable empirical expressions. For instance, in iron, which has a cubic lattice, the preferred directions are along the cube edges. If α_1 , α_2 and α_3 are the direction cosines for any magnetic vector, relative to the cube edges, the energy must be represented by a function of α_1 , α_2 and α_3 in which the terms are interchangeable. The energy may therefore be represented by

$$E = K_0 + K_1 (\alpha_1^2 \alpha_2^2 + \alpha_1^2 \alpha_3^2 + \alpha_2^2 \alpha_3^2) + K_2 \alpha_1^4 \alpha_2^4 + \dots$$

where K_0 , K_1 and K_2 etc. are defined as the anisotropy constants.

A single crystal permanently magnetised along an easy direction would have minimum anisotropy and exchange energies, but could have very large magnetostatic energy due to the 'free poles' existing on at least two of its faces. These 'free poles' must give rise to a demagnetising field within the specimen, the magnitude of

which would be determined by the shapes of the specimen. In the case of a sphere of iron magnetised to saturation this demagnetising field would have a value of about 7000 Oersted and, since this is sufficiently large to overcome the anisotropic effect, the arrangement must be unstable.

The total energy of the system may be considerably reduced if

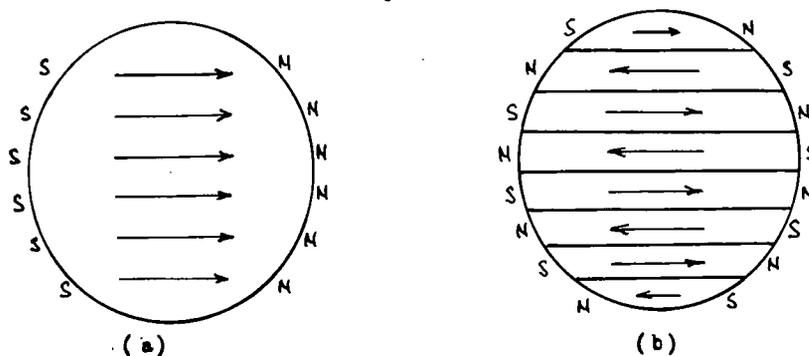


Fig. 1. Sphere of iron; (a) magnetised uniformly and (b) divided into domains.

the specimen is divided into regions magnetised consecutively in opposite directions as shown in fig. 1. The conditions that the magnetic vectors must be aligned along certain preferred directions for minimum anisotropy energy and parallel for minimum exchange energy are still fulfilled within any region - or domain- but not at the boundary between two domains. The boundary is called a domain boundary or wall and within it the magnetic vectors gradually rotate from one domain direction to the next. Thus, since the minimum exchange and anisotropy energy conditions cannot apply within the wall, the wall must possess a net amount of energy.

The contribution to this wall energy arising from the exchange interaction between neighbouring atoms will be an inverse function of the wall thickness since the misalignment between neighbours

will decrease as the wall thickness increases. On the other hand, the anisotropy energy will increase with thickness since more electron spins are twisted from the easy direction in a thicker wall. Consequently, there will be an optimum thickness giving minimum wall energy. For an iron crystal, this optimum thickness for a wall between two domains magnetised in opposite directions is approximately 2×10^{-5} cm. and the wall energy is between 1 and 10 erg/cm².

The orientation of a domain wall is determined principally by the need to ensure flux continuity through the wall - a condition which is fulfilled only if the normal components of the magnetic vectors on opposite sides of the wall are equal. If the flux is not continuous, 'free poles' will be formed on the walls and give rise to demagnetising fields which, in turn, will tend to rotate the wall until the 'poles are eliminated'. The plane of the wall must therefore contain the line which bisects the angle between the domain vectors on its opposite sides. This condition, however, is obviously insufficient to completely determine the plane of the wall and, subject to it being fulfilled, the wall will lie in the plane for which its magnetocrystalline and exchange energy is a minimum.

In a stable condition the total energy of the crystal must be a minimum and the crystal would divide into domains as described above providing that the increase in wall energy is less than the consequent reduction in magnetostatic energy.

If more than one easy direction exists within the crystal, small subsidiary domains may form at the ends in such a way as to decrease still further, the magnetostatic energy. In iron, for instance, it is easy to imagine subsidiary triangular domains magnetised

perpendicular to the main domains to bring about complete flux closure as shown in fig. 2. These subsidiary domains are known as 'closure domains'. In the case shown, magnetostriction effects

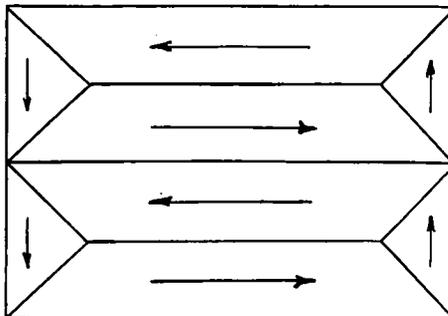


Fig. 2. Triangular closure domains.

will tend to elongate the main and closure domains in perpendicular directions (along the direction of their respective vectors) and the system must therefore have an associated strain energy. This magnetoelastic energy will be a function of the volume of the closure domains and is approximately 500 erg cm^{-3} for the case shown. Clearly, therefore, closure domains are in fact only possible if the reduction in magnetostatic energy is greater than the increase in wall and magnetoelastic energy.

Generally, then an unstrained crystal of ferromagnetic material will consist, in the absence of an applied field, of an arrangement of domains. Each domain will be magnetised along some preferred direction in the crystal and the arrangement of the domains will be one of those for which the sum of the magnetostatic, magnetoelastic and wall energies is a minimum.

1.2. Process of Magnetisation.

The magnetisation of a specimen by an applied field can be accounted for in terms of a rearrangement of the domains in its crystals. In an iron crystal there are six possible directions of magnetisation and, when the specimen is unmagnetised, there will be some domains magnetised in each of these directions. This is called a six-phase state. When a field is applied, it will tend to rotate the vectors into the three easy directions nearest to its own direction. This is, in fact, brought about by a rotation of the atomic vectors, giving an effective wall movement in such a way as to cause the domains magnetised in the three directions to grow at the expense of the others. The specimen is then said to be in a three-phase state and this must be the condition when the net field within the specimen - i.e. the resultant effect of the applied and demagnetising fields - is not zero. As the effective field is increased, the domains will pass, by the same process, first to a two-phase state with vectors along the two easy directions nearest to the field and, finally, to a one-phase state. In the one-phase state, the crystal will be a single domain the vectors will be in the easy direction nearest to the field or, in higher fields, will be twisted out of this direction towards the field direction.

1.3. Process of Demagnetisation - Nucleation

If a crystal in an applied field is magnetised to a two or three-phase state and the field is removed the specimen might be expected to become demagnetised by an exact reversal of the process described above. That the crystal does not actually do so, but

shows remanence and coercivity, is attributed to the fact that strain and non-magnetic inclusions make some wall movements irreversible. A wall may be driven over an 'energy hump' by an applied field but will not return over it when the field is removed.

If, however, the crystal is saturated and the field then removed, there are no reverse domains to grow by wall movements. The reverse domains could only be formed by some of the magnetisation vectors rotating from one easy direction to another through a difficult direction. This point was made by Brown (1945) in a criticism of the domain theory and he showed that, because of the energy involved, an ellipsoidal specimen with its long axis along an easy direction would not split up into domains. Kittel (1949) has shown that to produce a reverse domain by rotation of the magnetic vectors in a uniaxial crystal would require a reverse field of the order of $2K/I_s$ (where K is the anisotropy constant and I_s is the intensity of the spontaneous magnetisation). On this basis, the coercivity of an iron crystal would be expected to be several hundred times that found by experiment. It has been suggested by Bates and Martin (1953) that even when the reverse field acting on the specimen is very small, a very high field may exist in the region around an imperfection due to the formation of 'free poles' there. The imperfection may thus act as a nucleus for a reverse domain and this domain will be limited to a small region around the nucleus since its formation will remove the high reverse field. Once formed, however, the reverse domain will be very susceptible to small changes in applied field and will grow rapidly to demagnetise the specimen when the field is reduced.

1.4. Experimental Study of Domains.

There is now a great deal of experimental evidence in support of the domain theory, most of which has been obtained from a study of carefully prepared crystal surfaces. The direction and magnitude of the magnetisation varies from domain to domain and these variations must give rise to localised magnetic fields at the specimen surface. A number of methods have been devised for studying the pattern of these variations and, from the patterns, information about the domain structure within the crystal may be deduced.

The simplest and most commonly used method was devised by Bitter (1932). A colloidal suspension of a ferromagnetic powder is placed on the polished surface of the specimen and the small particles are attracted, due to the field gradient, into the regions of high fields - particularly on the domain walls and in scratches which run across the direction of magnetisation of the main domains. Thus the outline of the domains intersecting the surface is marked by a heavy colloid deposit and the pattern can be studied under the microscope. Although the method has the great advantage of simplicity, it can only be used over a limited range of temperature and on crystals for which the anisotropy is high enough to produce very concentrated localised fields.

Other methods of studying the pattern include one based on the deflection of an electron beam by the stray fields at the specimen surface and another based on the rotation of the plane of polarisation of a beam of light reflected from the surface. The various methods are discussed in detail by Craik and Tebble (1961).

Chapter 2

PREVIOUS WORK

2.1. Flux-closure and Reverse Domains.

When the surface of a specimen is inclined to an easy direction, 'free poles' will appear on the surface but the resulting magnetostatic energy may be reduced, as already described, by the formation of subsidiary flux-closure domains. Using a thin crystal of iron whose surface was within a few degrees of a (100) plane, Williams, Bozorth and Shockley (1948) found that these subsidiary domains have a fir-tree arrangement. The main domains are magnetised alternately along the $[010]$ and $[0\bar{1}0]$ directions and are separated by 180° walls which are perpendicular to the surface. The fir-tree branches are shallow surface domains, attached to the main walls at 45° so that the magnetic vectors in the branches are perpendicular to those in the main domains. The magnetisation vectors in the branches therefore lie in the easy directions $[001]$ and $[00\bar{1}]$ parallel to the surface and so form no free poles on the surface. It can be shown that the resultant decrease in magnetostatic energy is greater than

the increase in magnetoelastic and wall energy so that the fir-tree structure is preferable.

If a thin specimen of Si-Fe has a surface parallel to a (110) plane, the magnetisation vectors will be parallel to the surface and along the $[00\bar{1}]$ and $[00\bar{1}]$ directions, giving a main arrangement of anti-parallel domains. The walls within the crystal will be 180° walls, inclined at an angle of about 32° to the specimen surface as shown by Graham and Neurath (1957) and intersecting the surface in straight lines parallel to the $[100]$ direction. If the surface is inclined at an angle θ to the (110) plane to form a (11n) plane, free poles will be formed on the surface. The easy directions, other than those in which the main domains are magnetised, will be at approximately 45° to the surface and subsidiary domains, formed to decrease the magnetostatic energy, might be expected to be small regions of reverse magnetisation rather than flux-closure arrangements. Results of observations on such surfaces have been published by Bates et al and by Paxton and Nilan.

Bates and Mee (1952) reported investigations of the pattern on the (110) (side) face of a Neel cut crystal when the crystal was magnetised in the $[1\bar{1}0]$ direction. In such a case, the body of the crystal consists of narrow, laminar domains parallel to the $(1\bar{1}0)$ face, magnetised alternately in the $[100]$ and $[0\bar{1}0]$ directions. Flux-closure domains are necessary on the (110) face to minimise the energy due to the free poles which would otherwise be produced. Bates and Mee found an interesting lace pattern on this (110) face, from which they deduced a closure structure of the form shown in fig 3a or 3b.

Bates and Mee (1952b) also reported a kind of tree pattern observed

on thin Si-Fe specimens in which the specimen face was slightly inclined to the (110) plane. The pattern consisted of line deposits, parallel to the $[001]$ direction and indicating the 180° .

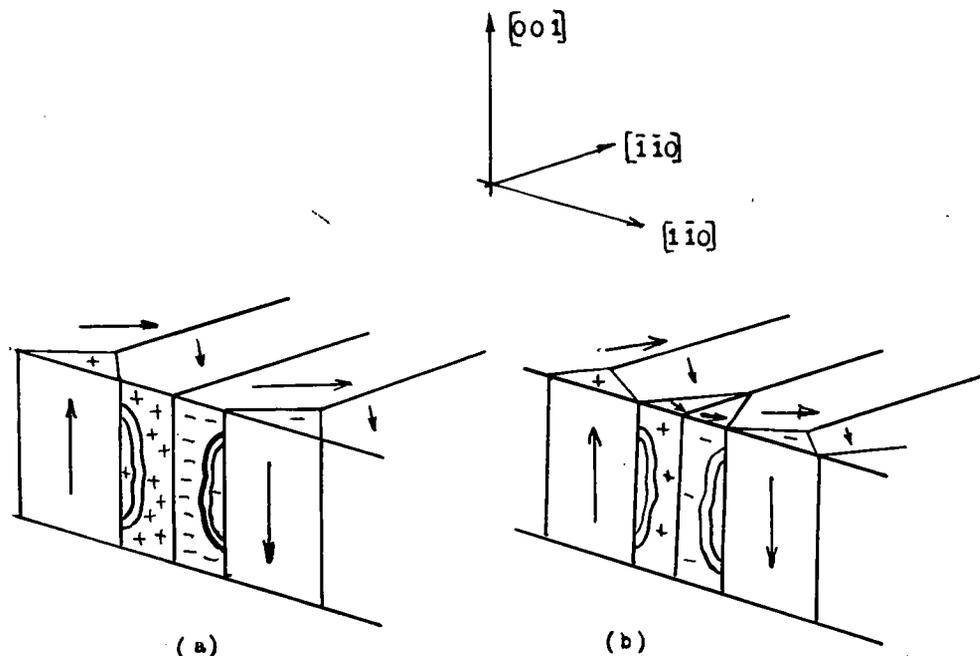


Fig. 3. Pattern on (110) face of a Neel out.

walls, with the tree branches attached to these lines. They concluded that the branches were a flux-closure structure similar to that observed by Williams, Bozorth and Shockley on surfaces inclined at a few degrees to a (100) plane. It was found that the branches were inclined at about 32° to the 180° walls and it was concluded that, for flux continuity across the walls, the vectors within the branches would be parallel to the surface and at 64° to the $[001]$ direction. These vectors would not, however, be in an easy direction.

Bates and Hart (1953) compared the patterns on the (110) faces of single crystals and poly-crystal specimens, the specimens being cut from thin sheets. In zero field, the pattern was found to

consist of parallel line deposits indicating 180° walls along the $[001]$ direction, with daggers pointing in the $[00\bar{1}]$ direction and arranged in rows between the 180° walls.

In fields applied parallel to the $[1\bar{1}0]$ direction, three pattern types were observed:-

- (a) a lace pattern similar to that observed by Bates and Mee and described above,
 - (b) a complicated, discontinuous pattern consisting of short lengths of unconnected lace patterns
 - and (c) a pattern of tadpole-like deposits and short wavy walls.
- The pattern (c), they reported, started as a dagger structure which broke up in the applied field and eventually formed a lace pattern. No detailed explanation of the patterns was given but it was concluded that the patterns were basically the same for single and polycrystal specimens.

Paxton and Nilan (1955) investigated the variation of pattern with inclination of the specimen face to the (110) plane. The

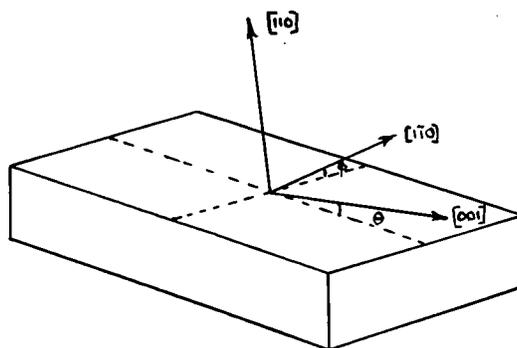


Fig. 4. Orientation of specimen face.

polycrystal specimens were punched from Si-Fe sheets and the orientation of the surface relative to the (110) plane was expressed in terms of two angles - θ in a plane parallel to the $(1\bar{1}0)$ plane

and ϕ in a plane parallel to the (001) plane - as shown in fig 4. They found that for values of θ between 0° and 2° the pattern consisted of 180° walls running parallel to the $[001]$ direction. Between 2° and 4° , spikes were observed between the 180° walls, starting at one crystal edge and pointing down the slope. For inclinations between 4° and 6° the pattern consisted of uniform rows of small daggers arranged perpendicular to the $[001]$ direction and, at higher inclinations, a lace or maze pattern was observed. The pattern was found to be independent of ϕ , at least over the range $0^\circ - 10^\circ$. For values of θ up to 4° the patterns were explained in terms of a main arrangement of domains magnetised alternately in the $[001]$ and $[00\bar{1}]$ directions, the spikes or daggers being regions of reverse magnetisation caused by free poles on the surface. No attempt was made to explain the patterns for higher angles.

2.2. Nucleation.

The process of nucleation for single- to two-, single- to four- and two- to four-phase changes have been studied by Bates and Martin (1953 & 1956) and by Martin (1953). Changes in pattern on a (110) face were observed as a field was reduced and, in another case, as the specimen was rotated in a field. In every case it was found that demagnetisation commenced with the formation of transverse spikes at imperfections in the surface of the parent domain(s), the spikes being magnetised parallel to the surface and perpendicular to the surrounding domain. In the one- to two-phase change, daggers of reverse magnetisation were formed when the transverse spike encountered an inclusion and then grew rapidly as the field was further reduced.

Similar observations for a one- to two-phase change were reported by Bates and Davis (1956) for the (110) face of a crystal initially saturated in the [001] direction. As the applied field was reduced, tadpole arrangements were first observed, running across the surface at about 35° to the [001] direction. At lower fields, reverse lozenges were formed and grew rapidly to form the large reverse domains. The pattern changes were not explained in detail but the tadpole arrangement was considered to be the intersection with the surface, of a transverse spike of the Bates-Martin type formed within the specimen.

2.3. Purpose of This Work.

This paper describes the results of further observations of patterns on surfaces slightly inclined to the (110) plane. The variation of pattern with inclination has been re-examined and more information has been obtained about the nucleation processes in single- to two- phase changes of magnetisation.

Chapter 3

EXPERIMENTAL DETAILS

3.1. Selection of Specimens.

Surface domain structures were observed by the powder pattern technique on crystal specimens cut from thin transformer sheets of 3% Si-Fe and on one large single crystal of Si-Fe. The thin sheets of transformer steel were 0.5 mm thick, of (110) texture, and contained extra-large crystals - some being several centimetres across. The (110) planes of the crystals in such sheets are within a few degrees of the sheet surface and the [001] direction is approximately parallel to the rolling direction.

To select a suitable specimen, the sheet was first etched for a few minutes in a slightly acidified ferric sulphate solution at a temperature of approximately 80°C. The solution consisted of 100g ferric sulphate and 10ml sulphuric acid in one litre of water. A black deposit formed on the surface during the etching and this was removed by swabbing with a 10% aqueous solution of sodium nitrate to which a few drops of nitric acid had been added. The

outline of the larger grains could then be easily traced. Small rectangular specimens, approximately 0.25 cm^2 in area and with their edges parallel and perpendicular to the $[100]$ direction, were marked in pencil on the surface of suitable grains. These specimens were then cut out with a small hacksaw, the sheet being firmly clamped between two wood blocks to prevent any distortion. There was evidence in the surface powder patterns (page 31) of some strain being produced by this method of cutting but this was limited to a region within 0.2 mm of the cut edge and was always removed during annealing. A spark erosion machine was constructed and used successfully to cut crystals for other work. Such a machine gives a good strain-free cut although the process can be rather messy since the cutting must take place under oil. The machine would have required modification for use in cutting crystals for this work and it was considered that the simpler hacksaw method was justified in spite of the small amount of strain that resulted.

3.2. Preparation of Specimen Surface.

For the observation of domain arrangements by the powder pattern technique, the specimen surface must be flat and free from strain. Surfaces were first mechanically polished down to 000-grade emery and this was followed by a further mechanical polish on a 1μ diamond impregnated disc until all scratches had been removed. To remove the strained surface layer produced during the mechanical polish, the specimens were then electrolytically polished in a chromic-acetic acid solution made up as follows:-

800 cm^3 glacial acetic acid, 38 cm^3 distilled water and 150 g CrO_3

If, after this treatment, the powder patterns on the thin specimens indicated residual strain, the specimens were annealed at 800 - 900°C in a nitrogen stream. The specimen was placed in a silica-glass tube situated in a tube furnace and the nitrogen stream passing over the specimen first flowed over a coil of iron wire to remove traces of oxygen. Since after this annealing process the surfaces were slightly tarnished, it was usually necessary to carry out a further light diamond and electrolytic polish.

In an electrolytic polish, the specimen forms the anode in a cell containing a suitable electrolyte. It is generally considered that the polishing effect is due to the high resistance film of electrolyte which forms over the specimen surface. It is therefore necessary to maintain, during the process, a steady flow of liquid over the specimen face, fast enough to remove any bubbles which may be formed but not so fast as to disrupt the high resistance film.

The method used in these experiments gave very good results and the arrangement is shown diagrammatically in fig. 5. The specimen was fixed in a horizontal position about 1 cm above the centre of a circular iron cathode of 2.5 cm diameter. The steady flow of electrolyte over the specimen face was achieved by rotating the assembly at approximately 60 rev/min so that the anode and cathode moved together about a 4 cm radius.

The thin specimens were attached to the end of a small bar

magnet which, in turn, was held in position during the polish by the Terry clip (T). The Terry clip was connected to the positive

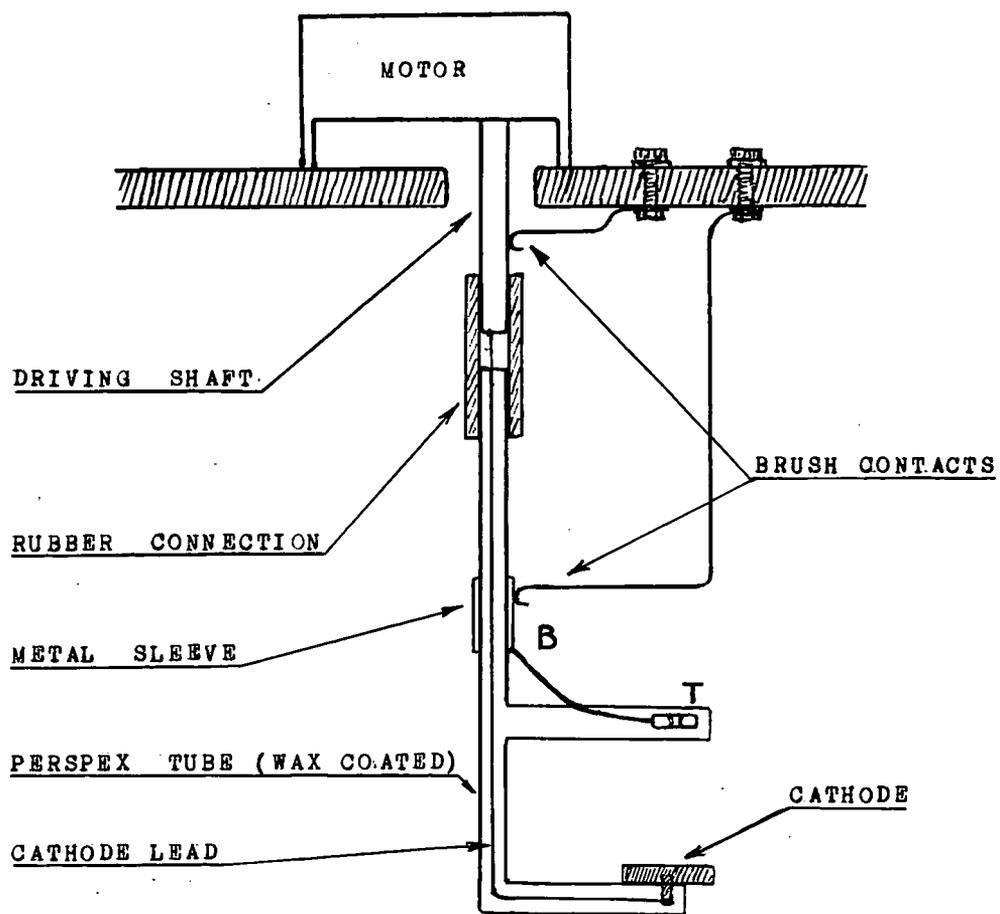


Fig 5 Polishing apparatus

side of the power supply via the brush contact (B) and the magnet served as the electrical contact with the specimen. The exposed part of the rear face of the specimen and the surface of the magnet immersed in the electrolyte were covered with a thin film of paraffin wax which also helped to hold the specimen flat against the face of the magnet.

The large Si-Fe specimen was mounted in perspex to facilitate

grinding. For the electrolytic polish, the mount was bolted to a perspex holder which could be held by the clip (T). Fig. 6 shows how electrical contact was achieved. When in position, the

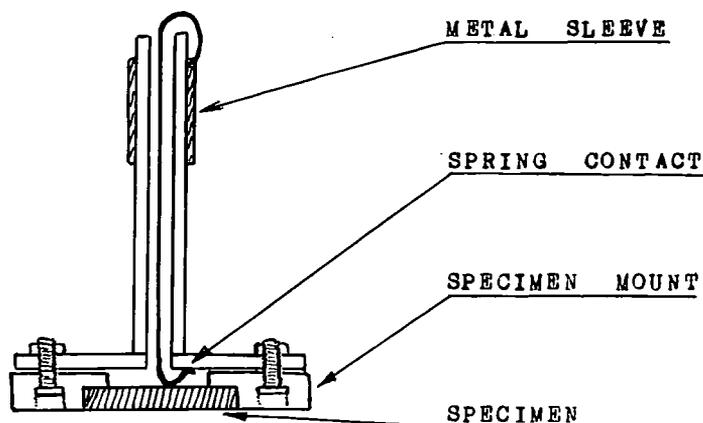


Fig 6 Holder for larger specimen

metal sleeve on the holder made contact with the clip and was also connected by means of a central wire to a spring which pressed against the back of the specimen.

It was thus, in each case, a fairly simple matter to remove and wash the specimen with its holder immediately after polish.

The method gave excellent results though in some cases, particularly when the specimen was large, only part of the face was polished and the rest showed a matt grey finish. Observations seemed to suggest that this could be due to interruption of the electrolyte film but this was not investigated in detail. A similar matt grey finish was produced over the whole of the specimen face when the electrolyte had aged and a fresh solution had then to be made up before a good polish could again be achieved.

The optimum cell voltage for polishing is that for which the cell resistance is a maximum. The resistance was measured by current and voltage readings or by a Wheatstone net method. The circuit arrangement for the Wheatstone net measurements was as

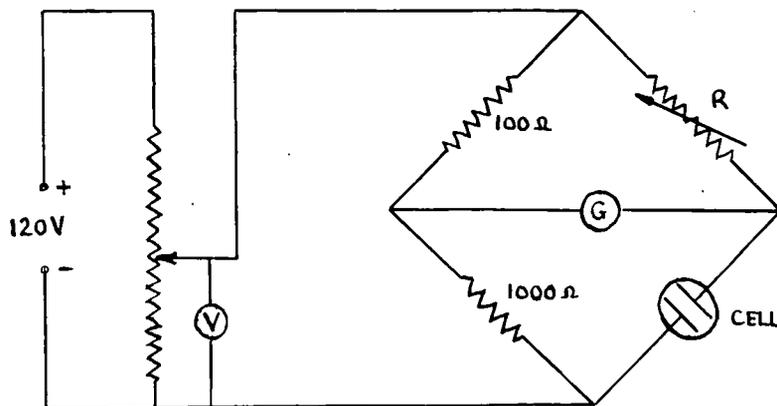


Fig. 7. Circuit for cell resistance measurement

shown in fig 7. For various applied voltages, measured on V, the resistance R was adjusted to give zero deflection on the galvanometer G. The cell resistance was then $10 \times R$ and a maximum cell

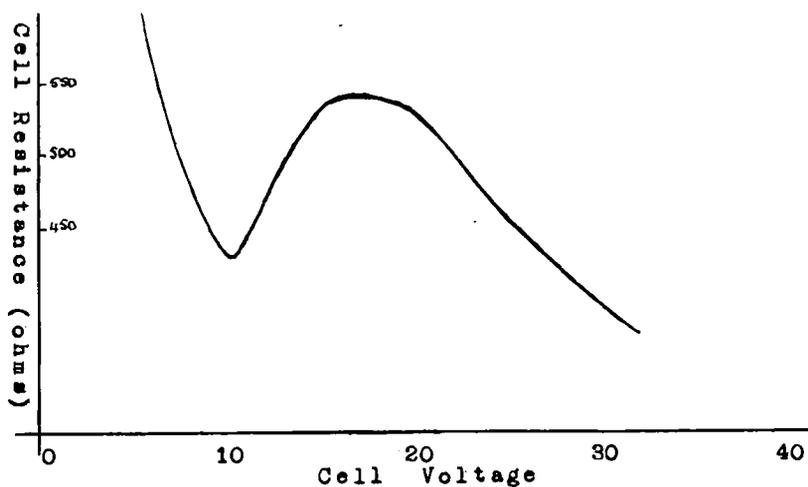


Fig. 8. Polishing characteristic

resistance was indicated when R had to be reduced to balance the circuit. The cell voltage for this condition was, of course, 10/11 of the reading on V.

Fig 8 shows a typical voltage-resistance characteristic for the cell, using the chromic-acetic acid solution. It can be seen that the optimum cell voltage was about 20 volts and specimens were generally polished at this voltage for 15-20 minutes.

3.3. Observation of Patterns.

Most of the observations were made using a Vickers projection microscope. In this instrument, the specimen platform is above the objective and the specimen is placed face downwards over a central hole in the platform. The position of the objective is fixed and the focusing control adjusts the height of the platform. Fine adjustment controls enable the specimen to be moved along in two perpendicular directions in the plane of the platform and the movement can be measured on vernier scales. The specimen platform can also be rotated about a perpendicular axis and the angle of rotation measured to within 5 minutes of arc. The instrument has built-in illumination with a Pointolite source. The image can be viewed through an eye-piece or cast on to a ground-glass observation screen. When the image is to be photographed the glass screen is replaced by a plate holder containing $2\frac{1}{4}$ " x $3\frac{1}{2}$ " plates.

For the experiments in which the specimen was rotated in a magnetic field, the projection microscope could not have been used without some modification to support a stationary magnet just above

the rotating specimen table. Observations in this case were therefore made with a bench microscope as described on page 50. To photograph the image the eye-piece was replaced by a fixed (xl) magnification camera. Ilford G30 plates were used with both microscopes.

The surface domain structure was made visible by the powder pattern technique, using the colloid described by Elmore (1938). A small drop of the colloid was placed on the polished surface of the specimen and covered with a microscope cover slide so that a very thin film spread over the surface. The cover slide was essential to prevent too large a drop of colloid from forming and fouling the microscope objective and also to prevent the liquid from drying quickly on the surface. After applying the colloid it took about 30 seconds for the particles to collect on the walls sufficiently to form a visible pattern. The colloid usually stayed fluid for up to 30 minutes so that variations in pattern due to changes in applied field could be observed and photographed. If left much longer than this, however, the particles tended to coagulate to form large brown patches on the surface or the colloid tended to dry on the surface and 'freeze' the pattern.

3.4. Measurement of Surface Orientation.

The orientations of specimen faces were found by etch pit reflections, using a two-circle goniometer constructed from an old prism spectrometer. The instrument is shown in fig. 9a (plate). The spectrometer telescope was removed and replaced by an arm which could be rotated about a horizontal axis parallel to the telescope

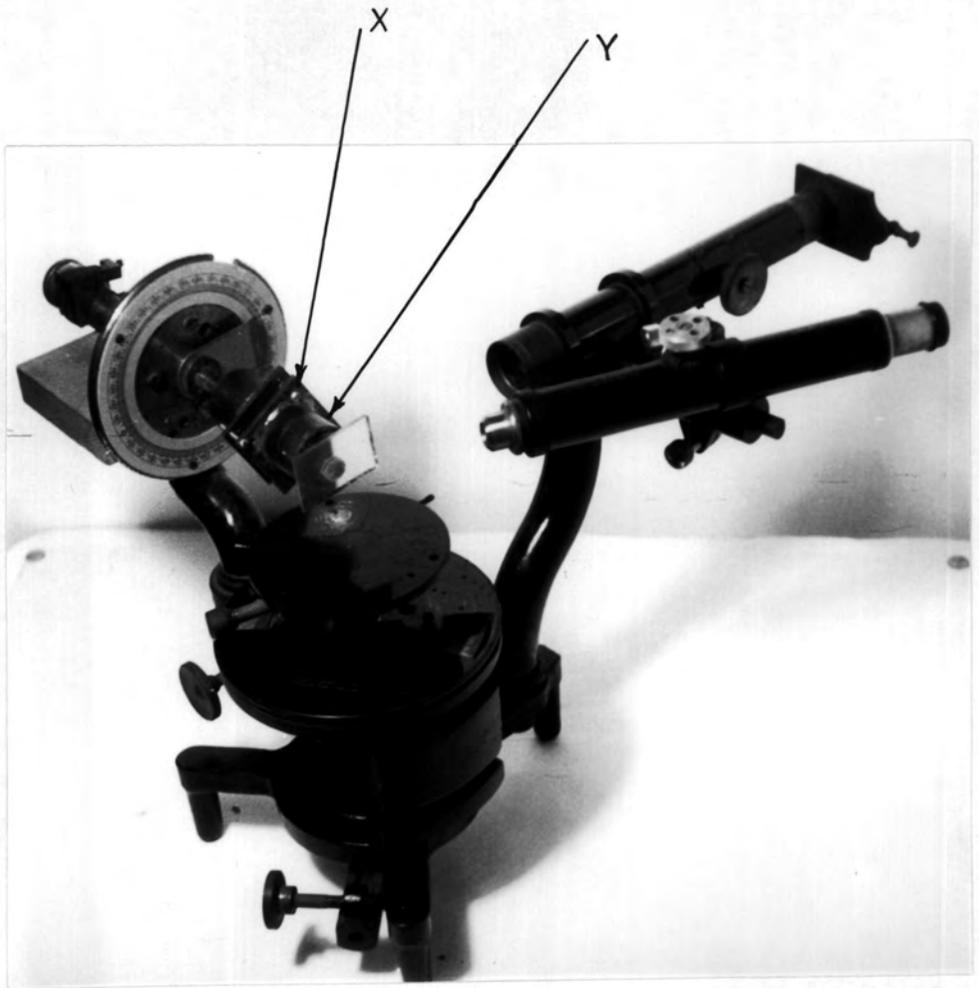


Fig. 9a.

support. This arm terminated about 4 cm short of the vertical axis of the instrument and a 2.5 cm diameter brass cup (Y) was attached to this end by three adjusting screws (X). The rotation of the arm and cup about the horizontal axis was measured by a pointer which moved over a vertical scale calibrated in degrees, scale readings being taken to the nearest half-degree. Since, in practice, the position for maximum reflection could not be estimated more accurately than this, it was not necessary to improve the sensitivity of the scale.

The collimator of the spectrometer was retained but the slit was replaced by a small circular object hole 2 mm in diameter. The specimen was mounted centrally in the cup (Y) and was observed through a microscope on magnification x25 which was clamped to a rigid support attached to the collimator arm. The microscope and collimator were inclined at an angle of about 30 degrees to each other in the horizontal plane.

Before using the instrument, the following adjustments were made.

(a) A pin was mounted horizontally in plasticine at the centre of cup Y and adjusted until, when viewed perpendicularly to the horizontal axis, it showed no lateral movement when this axis was rotated. The pin and horizontal axis would then be coincident. The centre of the horizontal axis at its end remote from the vertical axis was then marked by two other pins for two positions of the horizontal arm 180° apart. The horizontal arm was then set mid-way between these two positions and the three pins checked for alignment. The horizontal arm levelling screws were adjusted until, after the

procedure just described, the three pins were found to be in line and the two axes of the instrument were then known to be perpendicular.

(b) A 2.5 cm diameter bakelite cylinder with a plane mirror attached to its front face was mounted in cup Y so that the mirror face was close to the vertical axis of the instrument. The screws X were then adjusted until the mirror was accurately perpendicular to the horizontal axis. This was achieved by reflecting the image of a small object on to a screen and adjusting the screws until the image showed no movement when the horizontal (mirror) axis was rotated. The horizontal arm was then turned about the vertical axis until it was in line with the collimator. A small pointer was placed centrally at the object hole of the collimator and illuminated from the side so that its image (formed by the collimator lens and the mirror) could also be seen at the hole. The collimator levelling screws were then adjusted until the pointer and its image just coincided at the centre of the object hole. The axis of the collimator was then parallel to the horizontal axis of the instrument.

(c) The microscope was adjusted on its support until it was directed at the centre of rotation of the mirror. The image of the mirror surface at the centre of the field of view then remained stationary when the horizontal axis was rotated. The microscope was firmly locked in this position.

To measure the surface orientation for a thin specimen, part of the surface was first etched for two minutes in the ferric sulphate solution described on page 15 and the black deposit was swabbed off with cotton wool soaked in the sodium nitrate solution. In all

cases but one, very good etch pits were formed and, in this odd case, it was necessary to further etch the specimen for one minute. The crystal was then fixed to the mirror with plasticine so that it was on the axis of rotation with its etched face approximately parallel to the mirror. The collimator hole was illuminated by a Pointolite source and reflections from the outer edges of the mirror were focussed on to a screen to check that the mirror face was still perpendicular to the horizontal axis.

The specimen was then observed through the microscope and rotated about the two axes until the positions of maximum etch-pit reflection were found. In most cases it proved possible to set the crystal consistently to within one degree on each scale though, in some cases, to assist in the setting, the microscope was set slightly out of focus by increasing its length. The images of the pits then appeared as a series of rings of light and the arm was set in the position for which this fringe system was symmetrical. There were four positions of the specimen for which light was reflected by etch pits into the microscope and the horizontal and vertical scale readings were taken for at least ten separate settings in each position. From the average value of the scale readings for each of these four positions, the orientation of the (110) plane relative to the horizontal axis was calculated as follows.

In fig. 9, let an etch pit be at the point S. Suppose SA and SB to be the normals to the two exposed planes (100) and (010) and SC to be the $[110]$ direction. SY and SX are the two axes of rotation.

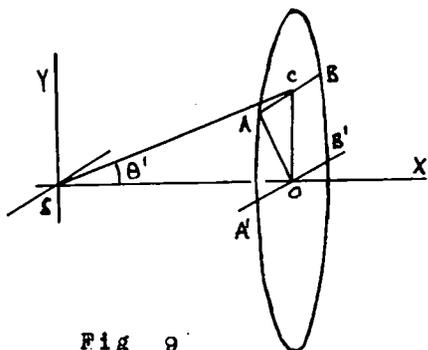


Fig 9

$$\tan AOA' = \tan OAC = OC/AC$$

$$AC = SC \text{ since angle } ASC = 45^\circ$$

$$OC = SC \cdot \sin \theta'$$

$$\tan OAC = \sin \theta'$$

Hence, if θ' is small, $AOA' = \theta'$

and, similarly, $BOB' = \theta'$.

Suppose that the specimen is rotated about SX in an anticlockwise direction through an angle θ' and then turned about the horizontal axis until light is reflected into the microscope from the etched face perpendicular to SA. To then reflect light into the microscope via the face perpendicular to SB, the specimen must be rotated in a clockwise direction through $2\theta'$, bringing SB into the plane ASB, and then rotated about the vertical axis through approximately 180° . Hence, θ is approximately half the difference between the vertical scale readings for maximum reflection into the microscope. θ was assumed to be half of the difference and, since θ was less than 10° , the resulting error will be less than about nine minutes of arc.

If the specimen is now rotated through 180° about the horizontal axis the test can be repeated and another value of θ obtained. The average of the two values will give a value of θ corrected for the possibility that SX may not lie accurately in the plane SAB (i.e. the plane of the collimator and microscope).

The angle θ is the inclination of the $[110]$ axis of the crystal to the axis SX of the instrument. When this had been found for

any specimen it was then necessary to find the inclination of the specimen face to the axis SX before the orientation of the face could be deduced. To do this, a small portion of the polished surface was left unetched. After making the measurements described above and with the specimen still in position on the mirror, the horizontal axis was turned to a position mid-way between the vertical scale readings so that the $[001]$ direction was vertical. Rotating the specimen about the vertical axis and observing the reflection from the mirror and the polished part of the face in turn, the inclination of the face to SX, in a plane parallel to the (001) plane, was determined. The horizontal axis was then turned through 90° and the inclination in a perpendicular plane was determined.

From the three angles thus measured, the orientation of the specimen face relative to the (110) plane was computed and expressed in terms of the two angles θ and ϕ mentioned on page 10.

For the large specimen, shaped as shown in fig. 10, the procedure was somewhat simpler. The crystal was etched in the solution already mentioned and then mounted in plasticine at the centre of

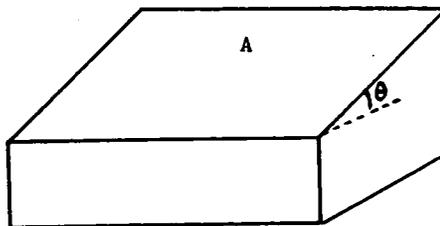


Fig 10 Shape of larger crystal

cup Y with its test surface (A) as near as possible parallel to the horizontal axis. Very good reflections were obtained from the

etched (001) and (00 $\bar{1}$) faces and from the specimen face itself. It was therefore possible, using the goniometer, to measure the orientation of the face (A) directly relative to the (001) and (00 $\bar{1}$) planes.

Chapter 4

RESULTS AND DISCUSSION

4.1. Strain Patterns.

As already mentioned, it is necessary to ensure that the specimen face is flat and free from strain before the domain structure can be studied by the powder pattern technique. The presence of strain will alter the domain arrangement and, therefore, the pattern produced. During the observations described here, it was noted that more than one pattern type was often seen at different points on the same surface at the same time. In addition to what was assumed to be the 'true' pattern, three common types were observed and these are shown in plate 1. These were all assumed to be strain patterns and were generally removed by annealing the specimen.

Pattern 1a consists of a series of largely unconnected zig-zags running approximately along the $[001]$ direction. Although the pattern was generally considered to indicate the presence of strain it was also observed in different circumstances on a strain-free

surface and is referred to below (page 51).

The second pattern, lb, is a criss-cross arrangement, rather complicated but having a basic regularity with rows approximately parallel to the $[00\bar{1}]$ direction. It is very similar to a pattern induced by Corner and Mason (1964) and also by Dijkstra and Martius (1953) on (110) faces when subjected to strain. An explanation of the arrangement has been given by Corner and Mason.

All rectangular specimens cut with the hack-saw from thin sheets showed this pattern, after polishing but before annealing, to a depth of about 0.2 mm along edges perpendicular to the $[00\bar{1}]$ direction. On a narrow band along edges parallel to the $[00\bar{1}]$ direction, the pattern always consisted, before annealing, of clear walls running parallel to the edge of the specimen. These 'edge' patterns were always removed by annealing and this suggests that the strain and domain deformation due to the cutting process did not extend very far into the crystal. It is also interesting to note that the presence of the criss-cross pattern only along the edge perpendicular to the $[00\bar{1}]$ direction confirms Mason's observation (1961) that this pattern could be produced by scratching a (110) surface across the $[00\bar{1}]$ direction.

The third pattern, lc, consists of daggers arranged in rows between parallel walls running perpendicular to the $[00\bar{1}]$ direction. This pattern is very similar to the pattern described by Paxton and Nilan for faces inclined at 4° to 6° to the (110) plane - though, in their observations, the walls do not seem to have been visible. It is also similar to the pattern induced by strain on (110) faces

by Dijkstra and Martius when the strain is perpendicular to the $[00\bar{1}]$ direction, though their patterns did not show the daggers. Dijkstra and Martius concluded from their observations that, under strain, the crystal consisted of laminar domains parallel to the (001) plane, magnetised alternately in the $[01\bar{0}]$ and $[10\bar{0}]$ directions and having triangular flux closure domains in the (110) surface. The flux closure domains will be magnetised in the $[00\bar{1}]$ and $[00\bar{1}]$ directions and if the specimen surface is slightly inclined to the (110) plane, the formation of reverse daggers would reduce the resulting magnetostatic energy. The arrangement could be as shown in fig. 11. The fact that the pattern was usually removed by annealing and was observed on faces at various inclinations supports the suggestion that the pattern was, in these experiments, due to strain.

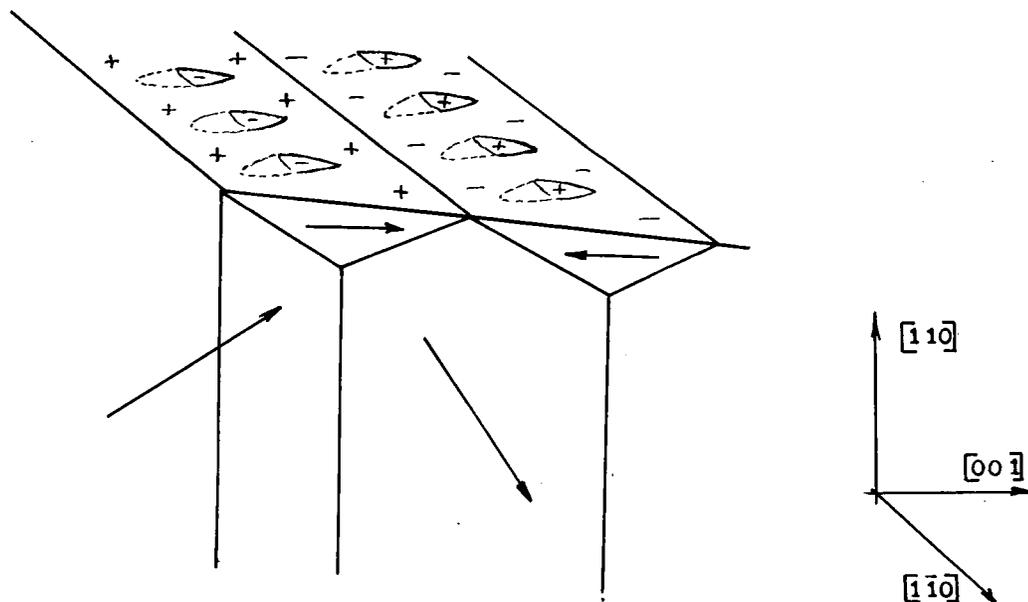


Fig. 11. Possible domain arrangement in a strained crystal.

Plates 2a, 2b and 2c show an interesting case in which this pattern was removed in stages from a specimen after two anneals. In this particular case, the specimen consisted of a rectangle cut from a large crystal in the sheet and containing a small included crystal approximately 1 mm across. After polishing, the pattern at the edge of the inner crystal was similar to that shown in plate 1c whilst a parallel wall arrangement along the $[001]$ direction was observed at places on the outer crystal. No pattern was observed on the rest of the surface due to pitting produced during polishing. The specimen was re-polished and annealed, after which the pattern on the outer crystal was seen to be a parallel wall arrangement starting at the edges and fading away to a pattern of faint parallel lines perpendicular to the $[001]$ direction at the centre of the specimen. The pattern on the included crystal then showed a large number of daggers arranged along the direction of the daggers - i.e. along the probable $[001]$ direction - but merging, towards the centre, into the arrangement shown in plate 1c. Plates 2a and 2b show the pattern after this first anneal and it can be clearly seen and AA' that the change in pattern for each crystal occurs across a line AA' running straight through the crystal boundaries. After further annealing and polishing the specimen, the parallel wall pattern and the new dagger arrangement on the outer and inner crystals respectively, covered the whole of the surface as shown in plate 2c. It would seem that the strain was relieved to the line AA after the first anneal and completely relieved after the second anneal. The parallel wall pattern on the outer crystal and the daggers running

parallel to the $[100]$ direction on the inclusion would then represent the strain-free condition.

The angles of inclination of the crystal faces to the (110) planes, θ and ϕ , were 1.5° and 8° respectively for the outer crystal and 4.5° and 3° respectively for the inner crystal.

4.2. Variation of Surface Arrangement with Inclination.

During the course of this work patterns were observed on a very large number of specimens. To study the variation in pattern as the surface inclination varied, however, a batch of 20 specimens was used. These were cut from the thin sheet and prepared as described above. A few were spoiled during the preparation processes but reasonably good patterns were observed on 15 of them with inclinations ranging from about zero to 8° . Seven of the 15 specimens consisted of a single large crystal with a smaller inclusion and in these cases patterns on both crystals were investigated. The remaining eight were substantially single crystals.

The patterns on each specimen were observed before and after annealing. Thirteen of the large crystals showed more than one pattern after the first polish as mentioned above. Patterns on these were improved by annealing, the zig-zag and criss-cross patterns being removed so that a uniform pattern was obtained over the whole of, or a large part of, the face. The remaining two large crystals showed a single uniform pattern after the first polish and this was not affected by the anneal. The final patterns were divided into five types which are shown in plates 3a - 3e.

Of the inclusions, only four out of the seven showed any pattern change after annealing. All inclusions, after the first polish, showed a pattern of wavy walls and daggers similar to plate 1c running across the expected $[001]$ (rolling) direction and some of them showed, in addition, the criss-cross and zig-zag formations. In the case of the four which showed a pattern change after annealing, the new patterns resembled very closely one of the patterns shown in plates 3. The fact that the included crystals were less likely to show improvement in pattern when annealed may have been due to the fact that the original strain in these was much greater than in the larger crystals or that the strain, removed during annealing was re-introduced during cooling. The $[001]$ directions in the included crystals were inclined to the $[001]$ directions in the surrounding crystals at angles ranging from 6° to 15° but there was no evidence of a correlation between this angle and the improvement of the pattern.

The five patterns in plate 3 form a logical sequence. 3a shows clear 180° walls running parallel to the $[001]$ direction. Where daggers appear, they are obviously associated with some imperfection at the surface and they point in both directions parallel to the main walls. In patterns 3b the 180° walls are still the predominant feature but several daggers can be seen between the walls, pointing in one direction only. In 3c the daggers have increased in number and the walls are almost, but not quite, indistinguishable. Patterns 3d and 3e show a much greater increase in the number of daggers, still arranged along the $[001]$ direction. In these cases, the

walls are completely obscured and the daggers form a complex lace pattern.

Measurement of the inclination of the specimen face to the (110) crystal plane revealed a strong correlation between pattern type and inclination. Table 1 shows the angle θ , without brackets, and the angle ϕ , within brackets, for each specimen.

TABLE 1 - INCLINATION BY PATTERN TYPE

<u>Pattern</u>	<u>Inclination (degrees)</u>
3a	$\frac{1}{2}(10)$, $\frac{1}{2}(15)$, $\frac{3}{4}(8)$
3a/3b	$\frac{1}{2}(8)$, $\frac{1}{2}(20)$
3b	$1\frac{3}{4}(8)$
3b/3c	$1\frac{1}{4}(3)$
3c	3(3), 3(1)
3c/3d	3(1)
3d	$4\frac{1}{2}(3)$, $4\frac{1}{2}(8)$, $8\frac{1}{2}3(13)^*$, $5\frac{1}{2}(3)$, $4\frac{3}{4}(3)$
3e	7(1), 7(5), 6(6)

* Accurate readings could not be obtained for this crystal since the inclination (ϕ) to the side was very high and reflections from etch pits were poor on one side. Using the [100] direction as indicated by the pattern and one accurate setting for reflection from etch pits on the 'good' side, the value of θ was found to be 6° .

None of the specimens in the sample had values of θ greater than 7° but plates 4a and 4b show the pattern observed on another crystal before and after grinding the surface to increase θ by

about 3° . The value of θ for 4a was found to be about 8° and for 4b about 11° . These patterns were observed at an early stage in the work and the value of ϕ was not recorded. Nor was this specimen annealed.

Taken together, these results indicate that the pattern consists of parallel 180° wall arrangements for inclinations (θ) below 1° . Between 1° and 4° daggers appear between the walls, arranged parallel to the $[001]$ direction and always pointing down the slope. The number of daggers increases as θ increases until the walls become almost indistinguishable when the inclination is about 4° . Between 4° and 8° the daggers combine to give a lace pattern which, at even greater angles, takes on a herring-bone appearance. This herring-bone pattern is apparent in one place in plate 3e and would seem to be due to the heavy colloid deposit on parts of the surface not covered with daggers.

There are two important differences between these results and those of Paxton and Nilan. In the first place, daggers, which are considered to be domains of reverse magnetisation decreasing the free-pole energy on the surface, were found by Paxton and Nilan to be formed at the crystal boundaries. In these observations, however, the daggers were found to exist over the whole of the surface. It is possible that, at lower values of θ , the few reverse daggers are attached to non-magnetic inclusions but it is difficult to imagine that the very large numbers at higher inclinations are. In these cases, the daggers are probably produced by the intersection of the surface and a reverse lozenge.

Secondly, these observations indicate that the pattern maintains a $[00\bar{1}]$ alignment at all angles θ up to at least 10° providing the strain has been removed. The arrangement of daggers running in rows perpendicular to the $[00\bar{1}]$ direction, found by Paxton and Nilan to represent faces with inclinations between 4° and 6° , was not characteristic of any inclination in these experiments. It must be pointed out, however, that the Paxton and Nilan results relate to polycrystalline specimens rather than single, large crystals and it is interesting to note that the rows of daggers perpendicular to the $[00\bar{1}]$ direction observed in these experiments and associated with strain, could not always be removed from the small included crystals. Further, Paxton and Nilan did not consider it necessary to anneal their specimens because of the consistency of the patterns.

The Paxton and Nilan pattern has been discussed by Goodenough (1956) who has suggested that the thin specimen of thickness 'h' with opposite faces inclined at an angle θ to the (110) plane should be considered as a crystal of effective thickness $h/\sin\theta$ (i.e. a relatively thick crystal). He has shown that for such a crystal, in which the main domains are magnetised along the $[00\bar{1}]$ and $[00\bar{1}]$ directions, the 180° walls should take on a rick-rack form at the surface, with the amplitude of the wave dependent on the thickness. If the effective thickness is great enough, the pattern becomes one of parallel 180° walls with rows of reverse spikes between them as shown in fig 12. It is suggested that the Paxton and Nilan pattern for θ between 4° and 6° , represents the intersection of the reverse spikes with the sloping surface as in fig 13. It must be noted,

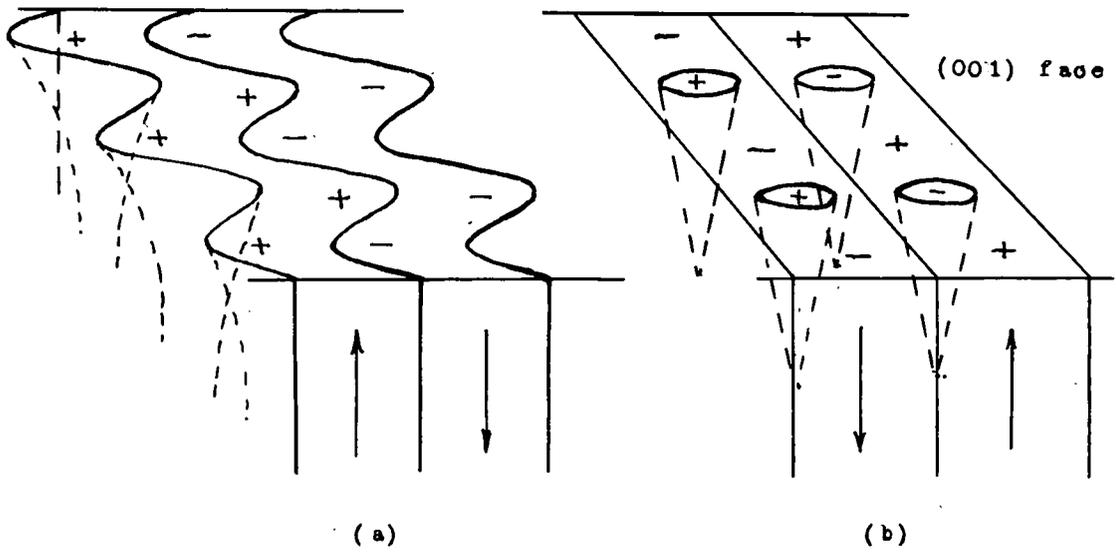


Fig. 12. Rick-rack walls in a thick crystal. Arrangement (a) tends to (b) as thickness increases

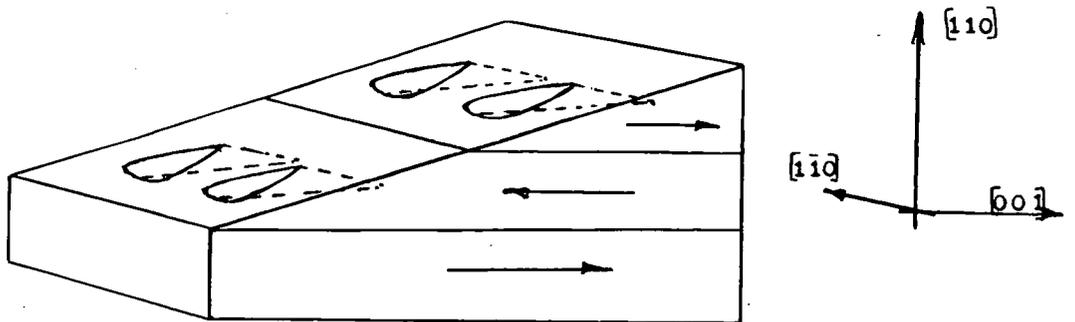


Fig 13 Formation of daggers on face inclined to a (110) plane

however, that the resulting daggers would point up the surface inclination to the (110) plane whereas all observations in these experiments indicated that the daggers point down the slope.

As shown above (page 32), the pattern could be easily explained in terms of the domain arrangement deduced by Dijkstra and Martius for a strained specimen. It is considered that this, and the evidence discussed in the last section (page 32) is sufficient to justify the assumption that, at least in the experiments described here, this particular pattern is due to strain entirely.

Paxton and Nilan found that the surface pattern did not appear to depend on the variation in ϕ over several degrees. This is to be expected since changes in ϕ produce no change in the inclination of the surface to the easy direction of magnetisation and, hence, no need for a change in an arrangement designed to reduce the free-pole energy. The results in table 1 seem to confirm this but, as a further check, one of the specimens for which θ was 7° was ground with a rounded edge so that ϕ varied over 10° whilst θ remained substantially constant. The pattern on the surface showed no noticeable change with ϕ .

The results described above could be checked by grinding a crystal surface through successive small angles, gradually increasing θ , and observing the consequent change in pattern. This method has the advantage that the successive inclinations can be accurately compared, relative to a fixed polished surface, by optical reflection. The method was attempted, using thin specimens cut from the sheet, but the specimens were usually so thin after the first polish that,

in the process of grinding through a small angle, they broke from the mount. One specimen, however, was satisfactorily ground through successive 2° changes in θ and the variation in pattern is shown in plate 5.

After polishing and observing the pattern, the specimen was attached to a cylindrical mount by sealing wax and a small piece of glass was cemented to the rear face of the mount. The angular separation of the two faces was measured by optical reflection, using a prism spectrometer. The specimen face was then ground, as described below (page 42), so as to increase the inclination to the (110) plane by about 2° . The actual change in θ was determined by again measuring the angular separation of the specimen and glass reference faces. The specimen was then removed from the mount for a final electro-polish. The new pattern was observed and the specimen was remounted to increase θ , in the same way, by a further 2° . The pattern after this second grinding was not so consistent over the specimen face as it had been for the previous polishes and showed signs of strain patterns in places. This was to be expected since the specimen - particularly at one edge - was extremely thin and would be susceptible to any slight stress produced in handling it. The patterns do, however, agree closely with the results mentioned above for faces inclined at angles between 0° and 4° to the (110) plane.

In an attempt to overcome the difficulties associated with grinding thin specimens to vary θ , observations were carried out using a single large crystal. This specimen, measuring approximately

10 x 10 x 5 mm, was donated by Drs Corner and Mason. It was cut mechanically by Mason from a very large crystal, using a milling machine and taking off only 10^{-3} in per cut so as to minimise any induced strain. The finished specimen had its faces within a degree or two of the (001), (00 $\bar{1}$), (110), ($\bar{1}\bar{1}$ 0), ($1\bar{1}$ 0) and ($\bar{1}$ 10) planes. It was mounted in perspex with the larger (110) face exposed.

To vary θ , the specimen mount was bolted to a support with the [001] direction as near as possible vertical. The support could be moved in horizontal, machined tracks, perpendicular to the face of a 320 grit grinding wheel so that the specimen could be pressed lightly against the flat grinding surface. By means of a fine adjustment screw the specimen could be turned, on the support, about a horizontal axis parallel to the grinding wheel and it could then be locked in position. It was thus possible to set the specimen and grind the exposed face to increase θ by a desired amount. After grinding on the wheel, the specimen and mount were removed from the support and polished mechanically and electrolytically as already described. The specimen face was ground in this way to increase θ by successive increments of about 2° and patterns were observed at angles ranging from 0° to about 12° . The inclination of the face relative to a small piece of glass, cemented to the rear of the mount, was measured in each case by optical reflections. The inclination of the final face was checked by means of etch-pit reflections, using the two-circle goniometer.

It proved very difficult to obtain a good polish on the (110) face of this crystal and the surface usually showed a large number of

very small pits. Since the surface finish never seemed to match that obtained on the thin specimens - even when polished under similar conditions - it is possible that the electro-polish may have been affected by small amounts of impurity in the crystal. The patterns observed on the large crystal were also poorer than those on the thin specimens and were never completely free from patches of strain. Two attempts to anneal the specimen at 850°C failed to improve the patterns. Under these circumstances, too much weight ought not, perhaps, to be attached to the results except to say that they tend to confirm the conclusions of the observations on the thin specimens. The variation in pattern as θ increased from 0° to 12° is shown in plate 6. It can be seen that over the whole range the pattern maintains a $[001]$ alignment and all the patterns could be interpreted in terms of an increasing number of daggers on the surface of underlying 180° domains.

The patterns shown were all observed in zero field or in a field much too small to produce a three-phase state of magnetisation. In these circumstances, domains in such a large crystal would not be expected to point only in the $[001]$ and $[\bar{0}01]$ directions. Some domains within the crystal would have vectors pointing in the $[010]$, $[0\bar{1}0]$, $[100]$ and $[\bar{1}00]$ directions and this fact could, of course, affect the patterns on the (110) surface.

4.3. Transverse Domains

The interesting structure shown in plate 7 was observed on several specimens, particularly - but not always - at higher values of θ . This structure closely resembles the arrowhead domains

reported by Bates and Mee (1952b) and consists of a series of tadpole-like deposits arranged so that the 'heads' lie in a row at approximately 30° to the $[001]$ direction. The outline of the tadpoles, though not marked by any colloid deposit, has a definite dagger form which may or may not be terminated by a dagger of reverse magnetisation along the $[001]$ direction. Tadpoles were also reported by Paxton and Nilan to occur on (110) faces when fields were applied. The structures were examined in detail in this work, firstly during the process of magnetisation and demagnetisation and, secondly, whilst the specimen was rotated in a magnetic field.

The changes in pattern during magnetisation and demagnetisation in a field approximately parallel to the $[001]$ direction are shown in plate 8. The field was produced by a small U-shaped electromagnet, the specimen being placed horizontally across the flat poles and in contact with the poles. The applied field was varied by adjusting the current through the magnet coils and a one-phase state could be achieved in the specimen with a current of less than one ampere. The domain wall pattern was then replaced by striations of colloid across the $[001]$ direction. The saturating field was then reversed several times to bring the specimen into a definite hysteresis cycle and was then gradually reduced.

The colloid deposits remained unchanged until the field strength had fallen to a certain value, when small tadpole arrangements suddenly appeared (plate 8a). Further slight reduction in the field strength caused these tadpole arrangements to grow in size. All of the arrangements were found to point in the same direction, down the

surface inclination to the (110) plane and to the same side of the $[001]$ direction. It was found that if the specimen was rotated slightly in this condition, so that the $[001]$ direction moved from a few degrees on one side of the field direction to a few degrees on the other side, the arrangement also moved to the other side of the $[001]$ direction. As the specimen was turned, the tadpoles faded away and, in several cases, reformed in the new direction with the base at the same point. This would suggest that the arrangements are associated with some inclusion at - or just below - the specimen surface and that they are surface structures. It was not possible with such a simple arrangement of apparatus to position the specimen with its $[001]$ direction accurately parallel to the field or to investigate in detail how the formation and growth of the tadpoles varied with inclination of the field and $[001]$ directions. Observations did, however, suggest that the tadpoles are less likely to form as the field direction approaches the $[001]$ direction and it is possible that they do not form at all if the field is accurately parallel to the $[001]$ direction at all points.

As the field is further reduced, daggers of reverse magnetisation, pointing along the $[001]$ direction, appear on the tadpole arrangements. These reverse daggers grow out of the tadpoles and two or three may be associated with each arrangement as shown in plate 8b. Demagnetisation is finally achieved in reduced and reversed field by the relatively rapid growth of these reverse daggers.

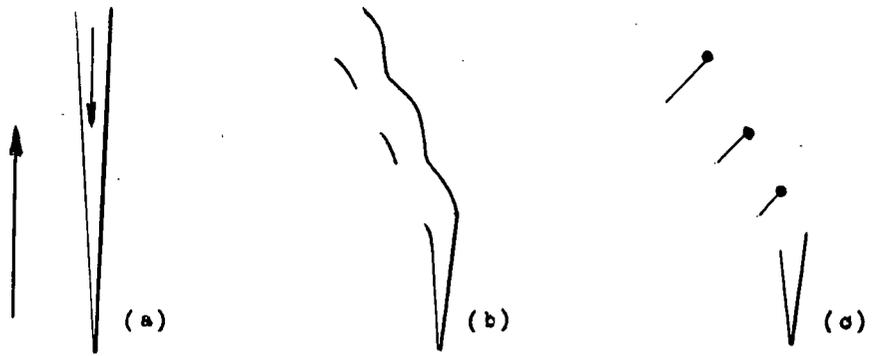


Fig. 14. Reverse dagger (a) becomes stepped dagger (b) then steps close to form tadpoles as in (c).

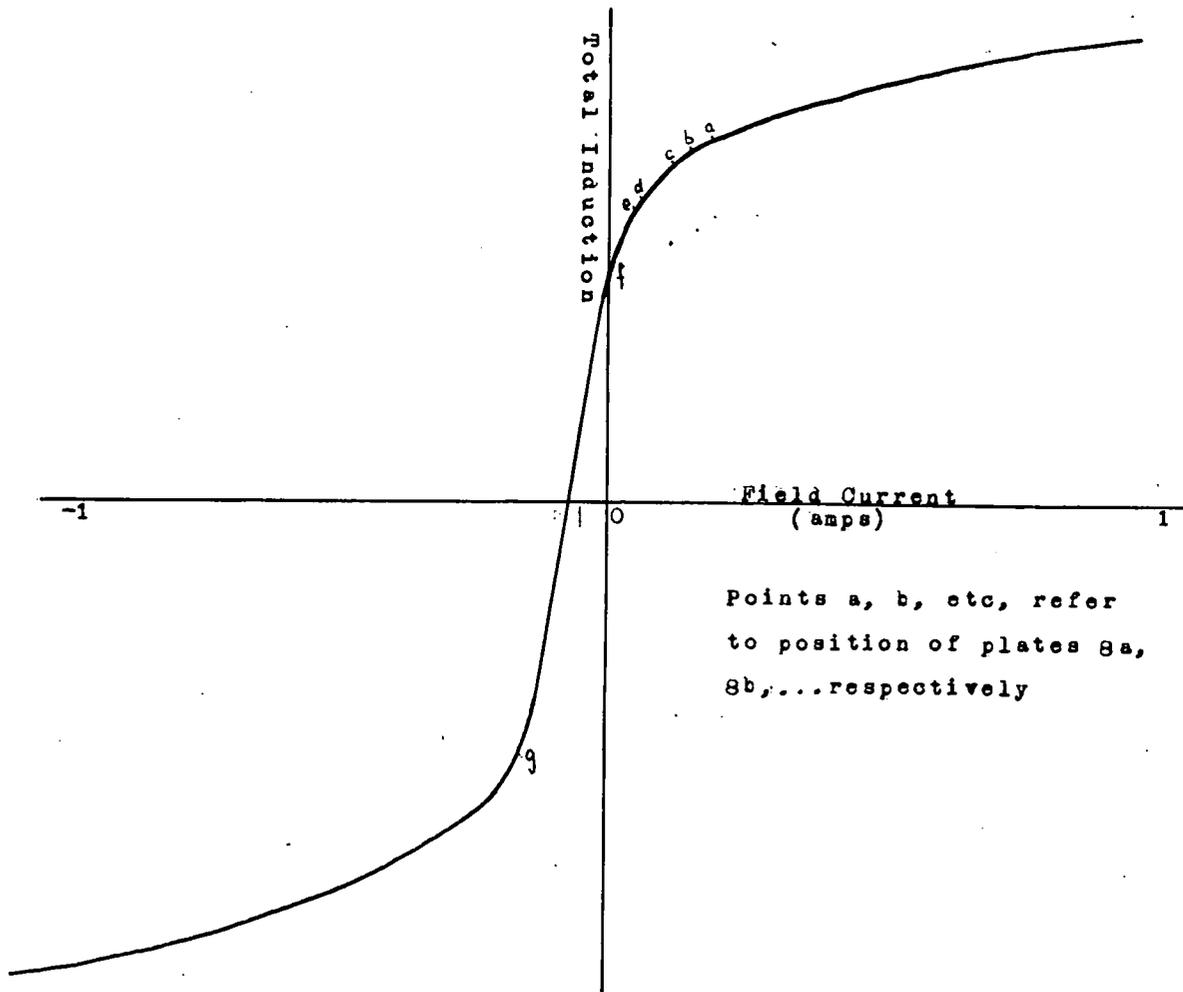


Fig. 15. Variation of magnetic induction with field current.

A gradually increasing field in the opposite direction causes a reverse process. The main 180° walls move so that the alternate domains, magnetised in the opposite direction to the field, shrink and form reverse daggers. The daggers decrease in size as the field strength is increased, though the process is not continuous and the changes occur by sudden jumps so that it is difficult to follow the changes in a particular dagger. Eventually, the reverse daggers begin to move side-ways across the field direction to form a stepped arrangement as shown in fig 14. The steps then suddenly split off from the dagger, one by one, and rapidly close to form a tadpole. This lateral movement of the daggers must be associated with the growth of a new structure, probably just below the surface.

The variation of the total magnetic induction with magnetising current was measured for the specimen shown in plate 8. The specimen was wound with about ^{turns} 70/of 36 gauge cotton-covered copper wire and fastened with sellotape across the pole pieces of the magnet. The flux changes in the specimen were then measured on a Pye scalamp fluxmeter as the current was varied in steps. The variation in magnetic induction as the current is reduced from the saturation value is shown in fig 15. A comparison of the curve with plate 8 shows that the tadpole arrangements occur between saturation and the 'knee' whilst the rapid changes of magnetisation indicated by the steep part of the curve are brought about by the growth of the reverse daggers.

These observations support those of Bates and Martin (1953 and 1956) and Bates and Davis (1956). They would seem to confirm

the suggestion that the tadpole arrangement is a transverse structure and probably the equivalent for the (110) face of the transverse spikes observed on (100) faces by Bates and Martin. The structure would, however, seem to be a surface arrangement rather than an intersection of a transverse spike with the specimen surface - a fact which is supported by the shape of the arrangement. The outline of the tadpoles forms a distinct dagger which always points, like the reverse daggers, down the sloping face as shown in fig 16. The

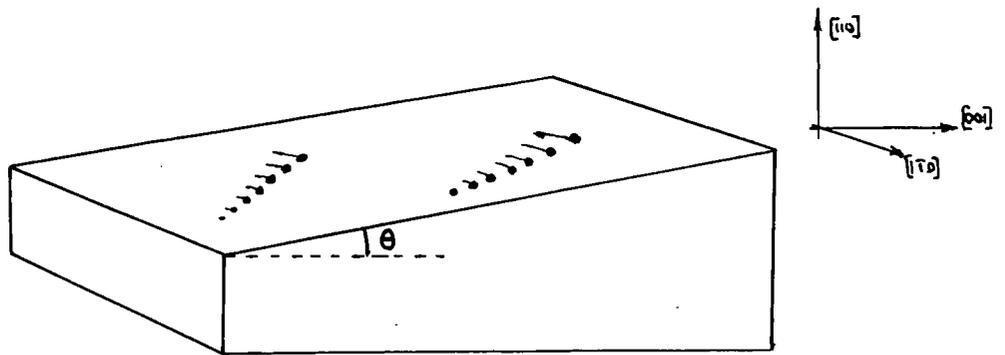


Fig 16 Tadpoles on face inclined to a (110) plane

intersection of a transverse spike with the surface would be expected to produce a much more symmetrical shape - i.e. an ellipse with its major axis at 45° to the $[001]$ direction. Further, the line joining the tadpole heads makes an angle of about 32° with the $[001]$ direction. The angle of inclination for a wall between domains magnetised in the $[001]$ and $[010]$ directions should be 35° if the wall is perpendicular to the specimen face and if there is to be flux continuity across the wall. The wall of the tadpole structure could be perpendicular to the surface in the case of a shallow surface structure but not

in the case of a transverse spike of the type observed by Bates and Martin.

Bates and Martin found that reverse daggers were never created directly at an inclusion but always grew from a transverse spike. As mentioned above, however, there was some evidence in these observations on the (110) face that the reverse domains may be produced directly when the applied field is parallel to the $[001]$ direction. When the specimen face is a (110) face, the vectors in the transverse domain will be at 45° to the surface. These domains cannot, therefore, be expected to be so energetically favourable as in the case when the specimen face is a (001) plane and the transverse vectors are parallel to the surface. The transverse domains are energetically possible in the (110) plane only when the effective field does lie to one side of the $[001]$ direction. This would explain the fact that the tadpole arrangement moves from one side of the $[001]$ direction to the other when the field direction moves through the $[001]$ direction.

Although the heads of the tadpoles form a straight line at 32° to the $[001]$ direction, there was never any indication of any colloid deposit along this line. There would appear to be no wall intersecting the specimen surface here. In this case, the direction of the vector between the tadpoles should be the same as that in the main domain. An attempt to verify this by the scratch technique proved inconclusive.

The surface of the specimen was scratched, after polishing, by means of a sapphire record-stylus. The stylus was first fixed to

a pivoted arm so that the point rested lightly on the surface of the specimen and the surface was scratched by drawing the specimen along beneath the point. Small loads were added to the pivoted arm to vary the pressure exerted by the stylus on the crystal surface. The conditions were found for which a fine scratch was produced which could be seen under the microscope but which did not show signs of the strain pattern along its length.

If the scratches run perpendicular to the $[00\bar{1}]$ direction, colloid will collect in them where they cross the main domain surfaces. Where a scratch crosses a tadpole arrangement, it should be free of colloid if the vectors point in the $[010]$ or $[\bar{1}00]$ directions at 45° to the surface but should collect colloid if the vectors point in the same direction as in the main domain. The results were not consistent. In some cases the colloid cleared from the scratch when a tadpole arrangement formed across it and, in other cases, a colloid deposit appeared on scratches passing through tadpole arrangements. It is possible that, even if the vectors between the tadpoles are parallel to those in the main domains, the colloid is sometimes removed from the scratches because of other intense local fields at the surface. The very heavy deposit on the tadpole head, for instance, does suggest the existence of such intense fields.

To further investigate the direction of the vectors in the arrangement, the specimen was rotated in a magnetic field from a position in which the field was parallel to the $[00\bar{1}]$ direction to a position in which the field was perpendicular to the $[00\bar{1}]$ direction. With the field parallel to the $[00\bar{1}]$ direction, the specimen

was magnetised to just above the 'knee' of the magnetisation curve. When the specimen was rotated to bring the field approximately perpendicular to the $[00\bar{1}]$ direction, the pattern changed to one very similar to that observed by Bates and Mee on the (110) (side) face of a Néel cut, indicating a two-phase condition with vectors along the $[01\bar{0}]$ and $[10\bar{0}]$ directions. The origin and growth of this new pattern was observed.

The source of the field was a small permanent magnet, from a moving coil ammeter, which was bolted to the stage of a bench microscope so that the centre of the field gap was below the objective. The field was then applied to the specimen by simply placing the latter over the gap and in contact with the poles. With a little care, the specimen could be rotated to keep the same spot at the centre of the microscope field of view. It was not considered necessary to make an accurate measurement of the field acting on the specimen.

The change in the pattern as the specimen was rotated through 90 degrees is shown in plate 9. When the field is approximately along the $[00\bar{1}]$ direction, reverse daggers, with a few tadpole arrangements, are observed. As the specimen is rotated, the reverse daggers disappear whilst the tadpole arrangements grow in size and number - a process which is not always continuous, in some cases the tadpoles actually decreasing in size. After 25° rotation, all of the reverse daggers have disappeared. The tadpole arrangements, however, continue to increase until the specimen has turned through about 50° and the tadpoles are approximately parallel to the field.

The tadpoles then begin to link up to form wavy walls running across the surface, approximately parallel to the field as shown in plates 9b - 9d. In some cases (not shown in the plates) parts of these walls bear a distinct resemblance to the zig-zag strain pattern mentioned above (page 30).

As the rotation continues, the wavy walls begin to open out (plate 9f), rotating with the specimen so that they remain along the same direction relative to the crystal face. Plate 9g shows the pattern on the face after a rotation of about 70° , the dark bands having opened out to form a distinct lace pattern and the bands then being at about 30° to the field. As the specimen is rotated through the final 20° to bring the field parallel to the $[\bar{1}\bar{1}0]$ direction, the lace pattern rotates on the surface until the broad bands are also perpendicular to the field. The pattern is then identical to that observed by Bates and Mee (1952).

The polarity on the surface was checked by applying a small vertical field by means of a probe. An iron needle was placed horizontally with one point above the specimen and below the microscope objective. The needle was then magnetised by simply bringing one pole of a bar magnet near to the free end so that the free pole induced above the specimen face gave a small vertical field there. This field was easily reversed by reversing the bar magnet (which was, itself, always far enough away from the specimen to have no effect on the pattern). Plate 10 shows the result - (a) without an applied vertical field, (b) when a vertical field is applied and (c) when the vertical field is reversed. It can be seen that one

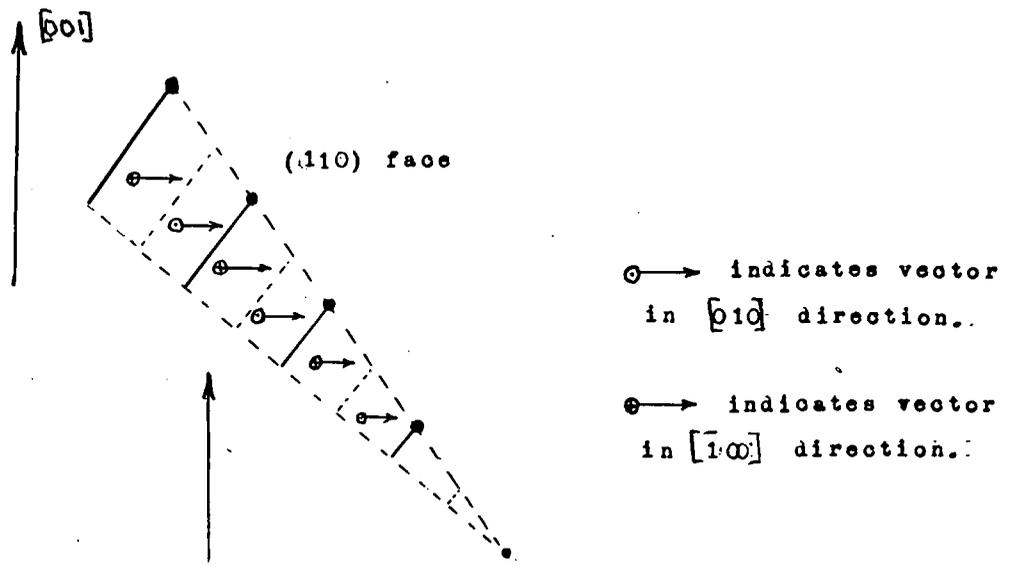


Fig. 17(a)

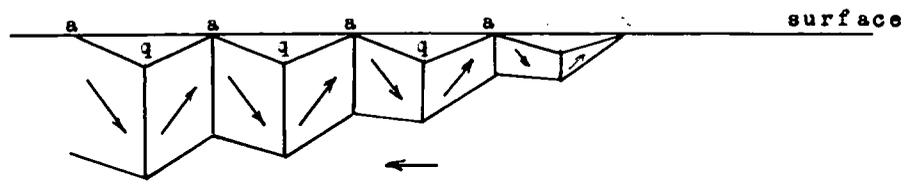


Fig. 17(b). Section perpendicular to surface and along dagger. Tadpoles appear at points 'a'. Vectors in 'q' domains in same direction as in main domain.

side of the broad dark band is cleared when the vertical field is in one direction and the other side is cleared when the field is reversed. This suggests that one half of the dark band has free north poles and the other half has free south poles at the surface and this is in full agreement with the results of Bates and Mee. The domain arrangement is probably as shown in fig 3.

On this basis, the wavy wall originating from the tadpole and eventually becoming the broad band of the lace pattern must, in fact, indicate a boundary between two regions magnetised in the $[010]$ and $[\bar{1}00]$ directions. Bearing in mind that the results mentioned above suggested that the tadpole arrangement was associated with a surface irregularity, the structure could be as shown in fig 17. It could be described as a lozenge, consisting of thin layers, perpendicular to the long dimension, magnetised alternately in the $[010]$ and $[\bar{1}00]$ directions. During the rotation of the specimen in a field, these slices are the nuclei from which the new main domains grow and, in the process of demagnetisation, the walls between the slices are the sources of the reverse daggars.

Chapter 5

CONCLUSIONS

The results of these experiments are in basic agreement with the many observations of (110) face patterns reported by other workers. The experiments do, however, suggest some differences of detail in the interpretation of the patterns.

Patterns observed on the faces before annealing, often bore a very close resemblance to the strain patterns induced by others. These were frequently removed by annealing and it is considered that there is sufficient evidence to indicate that the presence of strain is responsible for the patterns in these experiments.

The results indicate that, as the inclination of the specimen face to the (110) plane is increased up to about 10° , the magneto-static energy is reduced by the formation of reverse lozenges. These reverse lozenges increase in number as the inclination increases, always having an alignment parallel to the direction in

which they point - i.e. along the $[001]$ direction. This seems to be in agreement with the results of Paxton and Nilan except that they found that the lozenges were arranged in rows perpendicular to the $[001]$ direction when the inclination of the surface was between four and six degrees. In these experiments, however, this perpendicular arrangement was quite clearly due to strain.

The tadpole arrangement is a transverse structure and obviously plays an important part in nucleation processes during demagnetisation. These experiments suggest that the structure is associated with some irregularity (e.g. non-magnetic inclusion) in, or near to, the surface. It is rather more complicated than the simple intersection, with the surface, of a transverse spike of the type observed by Bates and Martin on (100) faces.

To say that this pattern indicates a surface structure would not necessarily mean that this arrangement is not formed inside the specimen as well. Similar domains within the specimen would not intersect the surface and, therefore, would not be seen if the spike lay in a plane parallel to the (110) plane. However, it is difficult to imagine that such a structure inside the specimen could be more energetically favourable than the single spike lying in the (010) or (100) plane and inclined at about 45° to the specimen surface. For instance, for the same volume, the wall energy of the tadpole arrangement would be greater but none of the other energy terms would be less. Further, if the specimen face is inclined slightly to the (110) plane, the free poles on the surface just before nucleation would produce a field component within the specimen

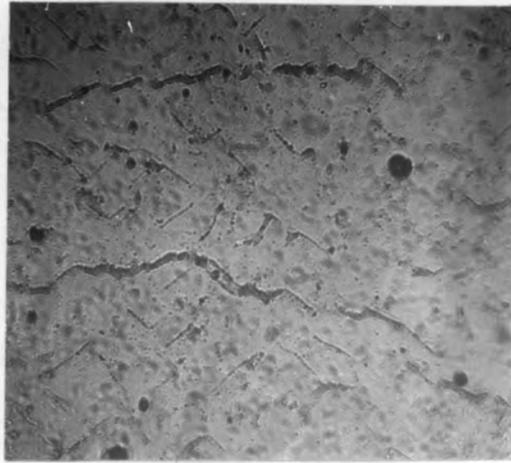
perpendicular to the surface. This field would tend to favour a transverse spike magnetised in one of the $[010]$, $[100]$, $[0\bar{1}0]$ or $[\bar{1}00]$ directions rather than the complex structure magnetised in two of these directions.

If the tadpole arrangement is a surface structure, there remains no evidence of transverse domains, formed inside the crystal, intersecting the (110) surface. Bates and Martin did report that a few reverse daggers on (100) faces were not clearly associated with transverse spikes and they suggested that this may in fact indicate the intersection of an internal transverse spike with the surface. They also found cases of transverse spikes formed at the edge of the specimen intersecting the (100) surface. The fact remains, however, that the patterns on (100) and (110) faces do not offer convincing evidence of the formation of transverse spikes within the specimen. On the other hand, the reduction in magnetic induction occurring between the one-phase state and the formation of reverse daggers is too great to be accounted for in terms of surface transverse structures alone.

The factors affecting the formation of transverse daggers and the extent to which these daggers are surface or volume effects would be worthy of further study and would give some indication of the importance of the surface in nucleation and demagnetisation processes.

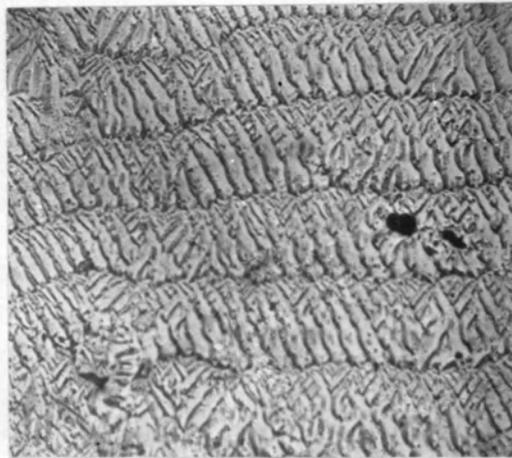
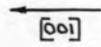
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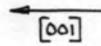


1 (a)

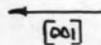
Mag. 370x



1 (b)



1 (c)



001 ↑



2 (a)

Pattern after
first anneal



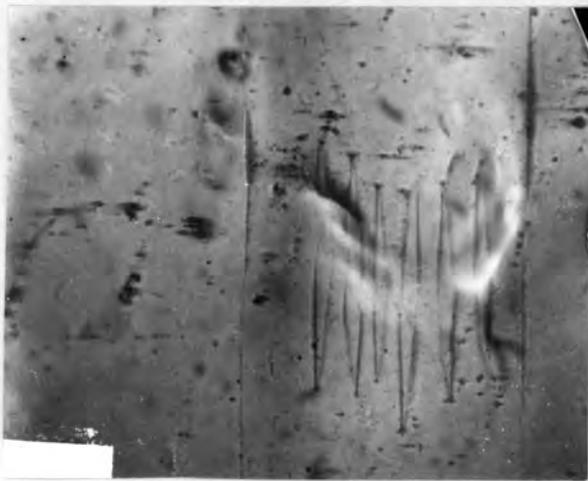
2 (b)

Mag. 70x

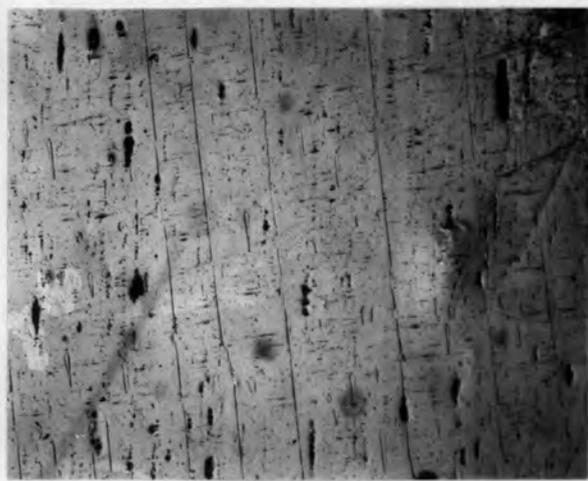


2' (c) Pattern after
second anneal

Mag. 56x



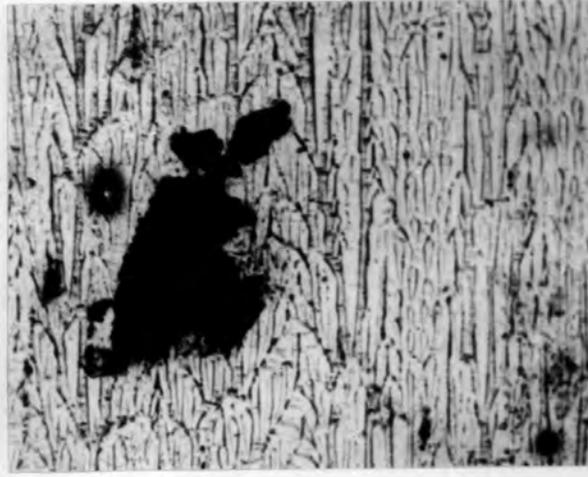
3 (a) Mag. 250x \uparrow [001]



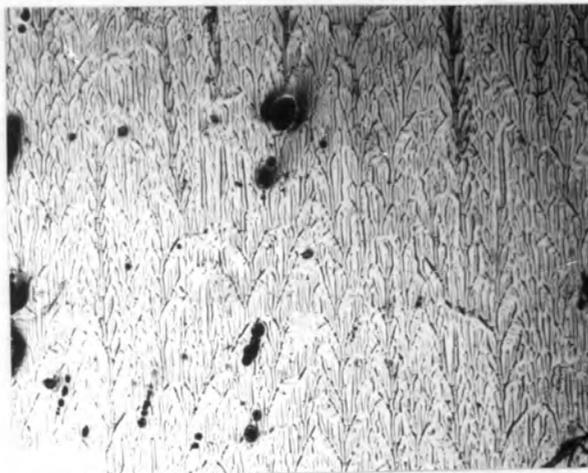
3(b) Mag. 45x \uparrow [001]



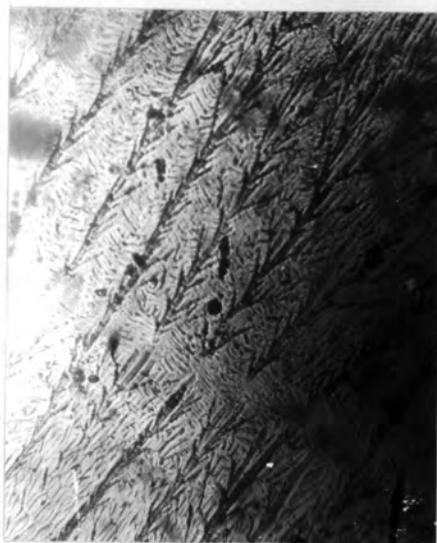
3(c) Mag. 45x \uparrow [001]



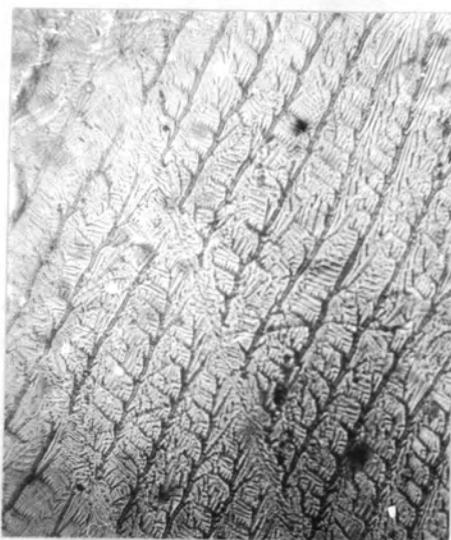
3(d) \uparrow [001]



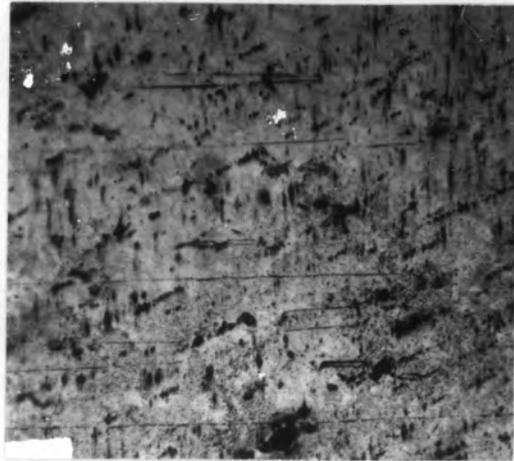
3(e) Mag. 90x \uparrow [001]



4(a) Mag. 150x

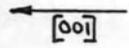


4(b) Mag. 150x



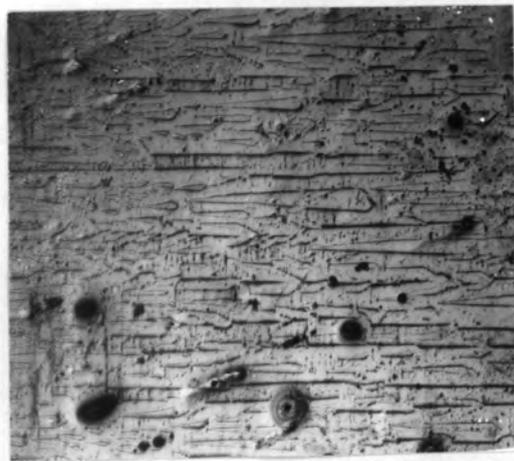
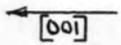
5(a)

Mag. 75x



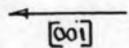
5(b)

Mag. 45x



5(c)

Mag. 70x

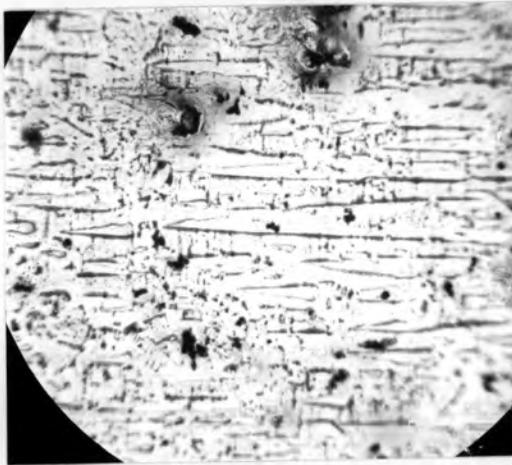




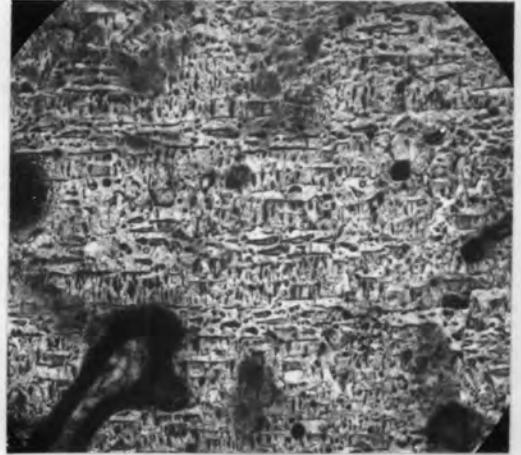
6(a). $\theta = 0^\circ$. Mag 250x



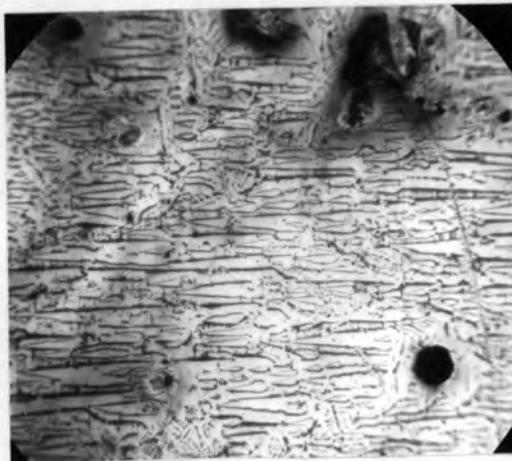
6(b). $\theta = 1^\circ-2^\circ$. Mag 150x



6(c). $\theta = 2^\circ-4^\circ$. Mag 150x



6(d). $\theta = 4^\circ-6^\circ$. Mag 90x



6(e). $\theta = 7^\circ$. Mag 150x



6(f). $\theta = 12^\circ$. Mag 150x

601

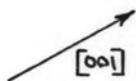
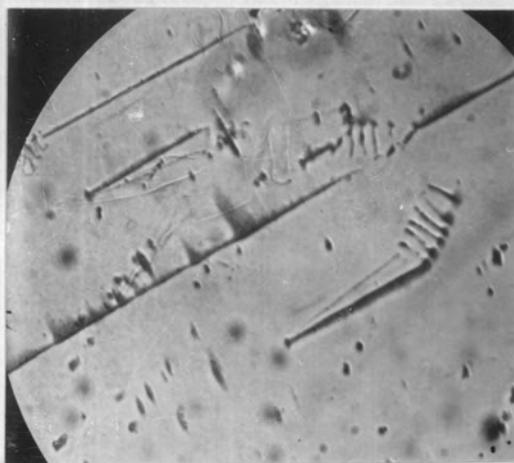


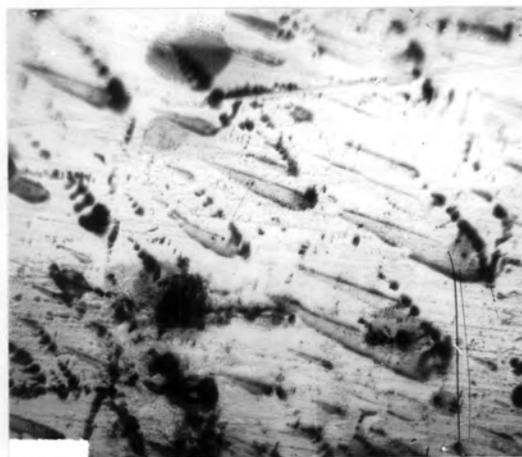
PLATE 7



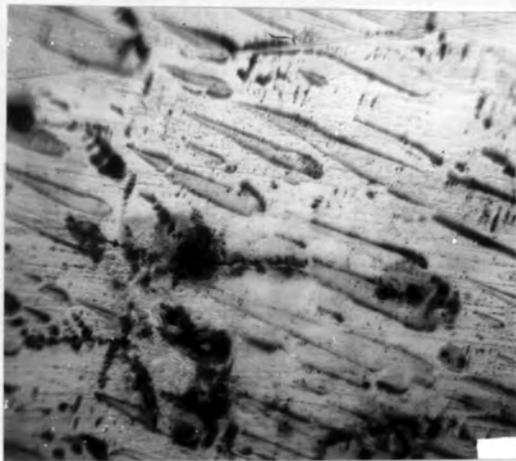
8(a) Mag 150x



8(b) Mag 150x



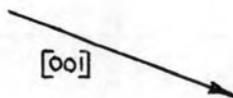
8(c) Mag 150x



8(d) Mag 150x



8(e) Mag 150x



8(f) Mag 150x



8(g) Mag 150x

PLATE 8 (see also facing page)

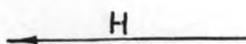
Field approximately parallel to 001 direction, gradually reduced from 8(a) to 8(f) and reversed for 8(g).



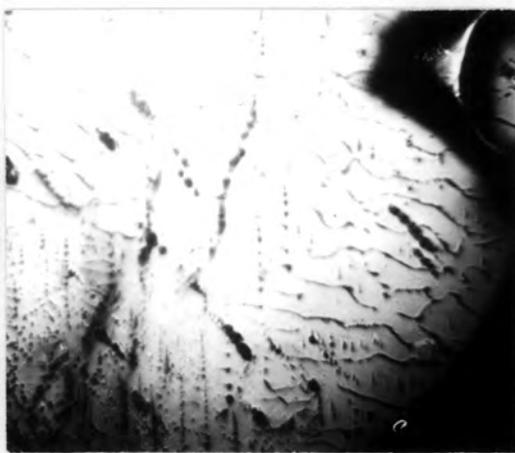
9(a) Field and $[001]$
approximately parallel



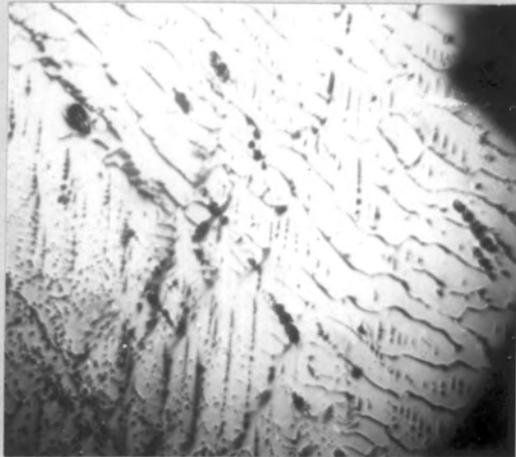
9(b) Rotation 25°



9(c) Rotation 40°

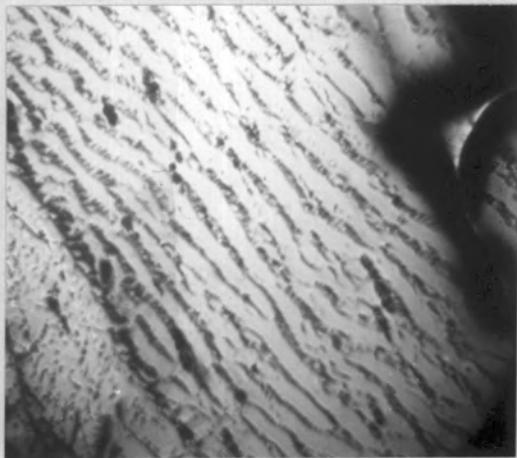


9(d) Rotation 50°



$\theta(e)$

rotation 55°



$\theta(f)$

rotation 60°



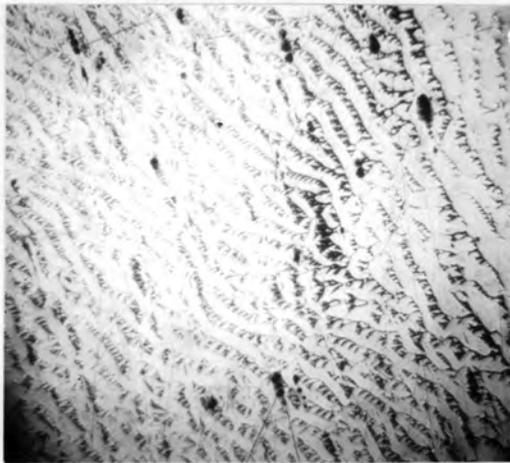
$\theta(g)$

rotation 70°

PLATE 9 (see also facing page)

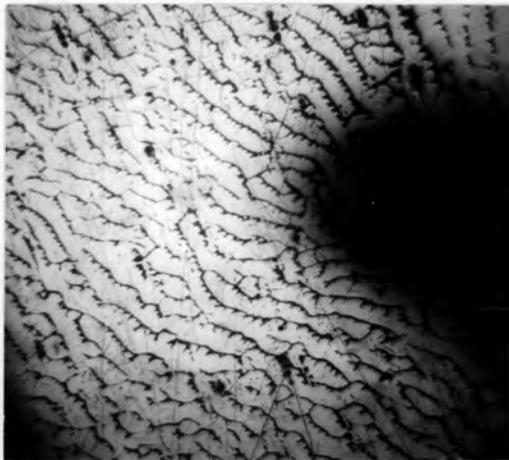
Specimen rotated in applied field.

Mag 90x



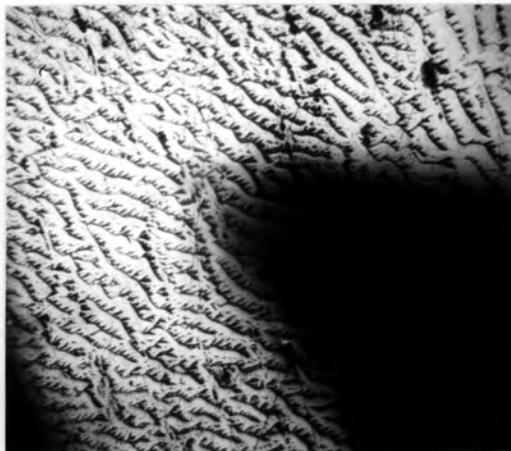
10(a) Mag 90x

Zero vertical field



10(b) Mag 90x

Vertical field downwards



10(c) Mag 90x

Vertical field upwards

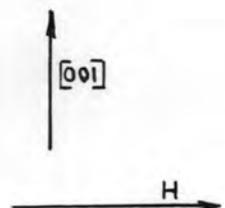


PLATE 10

