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The development of techniques for the detection of inhomogeneities of deformation in nickel and the study of the effect of temperature

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The Development of Techniques for the Detection of Inhomogeneities of Deformation in Nickel and the Study of the Effect of Temperature

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Summary:

The processes by means of which, features present in a deformed metal or alloy may be revealed by etching are reviewed.

Etchants have been developed which make possible a comparison of the deformation behaviour of nickel with that of copper and copper alloys, previously investigated by other workers.

At room temperature the etchant develops periodic markings parallel to and associated with slip bands, but of wider spacing.

Such markings are much better defined in nickel specimens deformed at elevated temperatures.

Specimens deformed at room temperature, ultimately exhibit very extensive coarse scaled inhomogeneity of deformation but there is very little evidence of deformation banding. After deformation at elevated temperatures the coarse scaled inhomogeneity is not exhibited in significant amount. Deformation bands are present, but there are fewer the greater the temperature of deformation.

Markings found by Samuels and other workers in copper and brass were not observed in nickel deformed at room and elevated temperatures, but were readily found in copper nickel alloys deformed at $-183^\circ C$.

Possible reasons for the above observations are discussed and it is suggested that the markings found at $-183^\circ C$ are most probably arrays of stacking faults on closely spaced planes or are heavily faulted twins.

Only a loose correlation has been found, between the fraction of the grains containing these markings and the stacking fault energy. Possible reasons for this are considered.
Introduction:

In recent years a considerable study has been made of markings developed by etching, in deformed copper and brass and these markings have been used to investigate the deformation behaviour of these materials.

The present work was initiated to develop similar techniques for nickel and thus to permit a comparison between the behaviour of nickel and the above materials.

Two etchants were selected and developed to the point at which they sensitively revealed deformation markings in nickel. However, during this work it became evident that the etching behaviour of nickel is essentially different from that of copper and brass and that these differences would be expected in view of the different character of the metals, particularly in regard to their stacking fault energy or stacking fault probability.

As a result of this observation the work was extended to include a series of copper nickel alloys ranging in composition from pure nickel to pure copper.

The literature relating to the deformation behaviour of metals is very extensive indeed and there is a very considerable literature dealing with etching behaviour.

Accordingly the literature review has been restricted to those aspects necessary to a coherent view of the effects of deformation and of the probable behaviour of etchants on the prepared surface of a deformed metal.
Review of the Literature

1. **Slip Deformation**

   The major part of the deformation produced in metals occurs by glide or slip of crystal lamellae, on surfaces within the crystal. Such a surface is called a slip or glide plane; slip occurs on planes of a preferred type and in preferred directions, in a particular crystal structure (1, 2).

   Slip is achieved by the movement of dislocations (3) on the slip plane. The passage of a dislocation line through a plane produces in the crystal, slip of the magnitude of the Burgers' vector of the dislocation. The passage of the line out through the surface of the crystal, produces a slip step in the surface.

   Repetition of the operation may ultimately cause the step to be visible either in a microscope field or even to the naked eye. Slip may occur on a large number of planes and produce fields such as those of figs. (1-4).

   A dislocation line may be halted within a crystal by some obstruction and then divides slipped from unslipped material.

   The plane and direction of slip, the slip system, involved in a process of single slip, is that on which the shear component of the deforming force exceeds the stress for initiation of slip.

   In the face centred cubic metals there are twelve slip systems on planes of the type \( \{111\} \) and directions \( \langle 110 \rangle \)
Fig. 1.
Single slip in 70/30 alpha brass, X1000. Unetched, (20).

Fig. 2.
Multiple slip in aluminium, X150. Unetched, (5).

Fig. 3.
Cross slip in 70/30 alpha brass, X1000. Unetched, (20).

Fig. 4.
Cross slip in 70/30 alpha brass, X1000. Unetched, (20).
2. **Deformation by Slip**

2.1 **Slip in Single-crystals:**

In a single-crystal, slip initiates on that slip system for which the resolved shear stress is the greatest. In suitably oriented crystals, slip on a single system may continue to quite large strains.

During this 'easy-glide' stage (4) slip continues with little increase in stress. It is thought that during this stage, dislocations pass right out of the crystal; slip lines, therefore, appear right across the full width of the crystal.

The resultant surface may appear as shown in fig. 1, or as in that portion of fig. 6 near the lower right hand corner.

As deformation continues, slip may eventually occur on a second system.

Multiple slip is associated with a stage of 'rapid' or 'linear' hardening, (4, 5). New slip steps formed in this stage are progressively shorter than those previously formed, because of obstruction by earlier slip, (6). Multiple slip is illustrated in fig. 2 in which some slip lines are seen not to pass across the whole field.

Following this, a stage of 'cross' or 'connecting' slip, (5), occurs. Cross-slip is illustrated in figs. 3 and 4; it permits obstructions to be by-passed by the slip process and as a consequence the rate of hardening diminishes. This is a stage of 'dynamical recovery', (4).
Fig. 5.
Slip in poly crystalline brass, X100. Unetched, (8).

Fig. 6.
Slip bands in aluminium extended 7%, X125. Unetched, (26).

Fig. 7.
Resolution of a slip band in aluminium extended 5%. Approximately edge on view of lamellae. Replica X 2 8000, (12).

Fig. 8.
Schematic illustration of the terminology adopted for slip markings, (5).
2.2 The Effect of Grain-boundaries

2.2.1 Boundaries of Poly crystals:

Grain boundaries are effective obstacles to slip; their influence extends into the immediately adjacent areas of the grains and visible slip lines are frequently seen to stop either at the grain boundary or in its immediate vicinity; such behaviour is seen in fig. 5.

There is, therefore, no stage of easy-glide in poly crystals, although only one slip system may be observed to operate in a grain.

However, at low strains, a short stage of linear hardening has been observed (5).

2.2.2 Annealing-twin boundaries:

In some face-centred cubic metals annealing twins are common. The twin boundary is the trace of a \{111\} plane.

A twinned region may be an obstacle to slip, but very often slip lines cross a twin boundary with only a change in direction, fig. 5.

"Twin boundaries are a powerful obstacle to crossing slip lines when they do not contain the slip direction" and "are a weak obstacle when the slip direction is contained in them", (7).
3. **Factors Influencing Slip**

3.1 **Introduction**

The work of Ewing and Rosenhain \(^{(9,10)}\) in 1898, first drew attention to the existence of slip steps in the surface of deformed specimens. Subsequently, the nature of these steps has been very extensively investigated using the techniques of optical microscopy. An important part of this work has been reviewed by Brown, \(^{(11)}\).

Using the electron microscope, Heidenreich and Shockley \(^{(12)}\) were able to reveal slip structures very much finer than could be observed by the use of the optical microscope. Work in this field has continued and there is to-day an extensive literature devoted to the field.

The technique is now such that in pure metals and alloys the finest structures produced by slip can be resolved.

The slip features resolved by the optical microscope as single lines, by the electron microscope have frequently been revealed to contain several finer 'slip lines'. The total height of the microscopic slip step or 'slip band' is determined by the smaller steps associated with the slip lines.

Between the slip bands and between the slip lines in a band, is a finer structure only revealed by the electron microscope. Brown and Honeycombe, \(^{(13)}\), called this 'microslip'. It consists of sub-microscopic slip steps.
In the earliest stages of deformation only microslip is found; at a later stage slip bands appear. The geometrical nature of the features is illustrated in fig. 8 due to Clareborough and Hargreaves, (5).

The purpose of the present review of slip is to establish as fully as possible the form of the slipped surface so that at later stages in the thesis an easy reference to the characteristic form of slip markings can be made.

The chief findings are summarised in Table I at the end of the next section.

3.2 The Effect of Strain

3.2.1 Slip in Pure Metals

Heidenreich and Shockley, (12), using single crystals of pure aluminium, were able to resolve lines unresolved by the light microscope fig. 6 and to show that these were in fact composed of a number of small steps produced by slip on planes of the order of 100 atoms apart,(fig. 7). Each lamella so formed had undergone a shear movement of about 2000 Å.

Brown (14) in single crystals of aluminium found that the fine slip lamellae were little affected by the amount of strain, the temperature, or the rate of deformation.

At very small strains slip of approximately 2000 Å occurred on lines spaced "by distances of the order of tens of microns". With increasing strain fresh slip of the same amount occurred on new lines
Fig. 9.
Microslip, (elementary structure), in super-purity silver, 27% elongation. Replica, X 20,000, (7).

Fig. 10.
Slip bands and microslip, (elementary structure), in super purity silver, 27% elongation. Replica, X 20,000, (7).

Fig. 11.
Slip band like array of slip lines in poly crystalline alpha brass, 5% elongation. Replica, X 20,000, (7).

Fig. 12.
A slip free region between clustered slip lines of greatly varying depth in alpha brass, 5% elongation. Replica X 20,000, (7).
between those already formed. The spacing of the slip lines decreased with increasing strain until the whole of the slipped area contained closely spaced slip lines.

Thereafter new slip lines clustered close to lines already formed; with further straining the cluster grew in width and in addition, new clusters formed, (11).

At 15% elongation an aluminium single crystal contained well developed clusters spaced $\frac{1}{2} - 10 \mu$ depending on temperature. The spacing of lines in the clusters ranged from $200 - 800 \AA$ ($0.02 - 0.08 \mu$) which was beyond the resolution of the light microscope.

Tolansky and Holden (15) using light interferometry were able to observe the growth of the lamellar steps and found that the terminal shear on a slip plane was close to $2000 \AA$.

Brown and Honeycombe (13) have given the name "micro-slip" to the fine slip that is a component of slip bands in aluminium; Wilsdorf and Kuhlman-Wilsdorf (16) have called it the "elementary structure" of slip.

Kuhlman-Wilsdorf and Wilsdorf (7) examined the surface of deformed poly crystals of aluminium, copper and silver all of high purity or of super purity.

In aluminium they found that the shear on a slip step varied continuously from a few, to $1200 \AA$ but was never observed to be as large as $2000 \AA$; the average slip on a line was $300 - 400 \AA$.

The lines were clearer and deeper in copper and silver than in aluminium and the average line spacing was respectively 300, 250, and $400 \AA$, (fig. 9).
Continuation of straining caused lines to appear in greater numbers and ultimately, localised grouping into bands commenced, (fig. 10).

An important observation by the authors related the strain for the onset of banding and the strain due to the elementary structure. By making counts of grains showing bands in the optical microscope, the authors were able to demonstrate that the strain due to the elementary structure was greater in copper and silver than in aluminium, an observation consistent with that two paragraphs above. As a consequence the authors observed slip bands in aluminium in 50% of grains at an elongation of $2 - 3\%$, but not in copper, until a deformation of more than 15% had been induced.

Yamaguchi (17) found that in aluminium single crystals, the slip on a band and the slip band density increased with strain.

From the work described above it appears that the behaviour of all the pure metals reviewed is essentially the same.

Seeger (18) observes that "metals give essentially the same surface patterns" and that stacking fault energy (Section 6) has no effect, "provided that comparison is made at the same stage of behaviour". Similar structures will not be observed if metals are not compared at the same stage, for example the appearance of slip bands is associated with the onset of Stage III of slip behaviour, i.e. the stage of dynamical recovery, (4).

Stage III is also characterised by the appearance of cross slip and this has lead to theories of slip band formation in terms of a cross slip mechanism, (18).
3.2.2 Slip in Alloys:

What appears to be a characteristic difference between pure metals and alloys is exemplified in the work of Kuhlman-Wilsdorf and Wilsdorf (7) using poly crystalline (70/30) alpha brass.

They observed that slip lines "do not assemble together to form bands", that their spacing is variable and that they are of greatly varying depth.

There were, however, occasional groups of slip lines closely spaced and of more or less uniform depth, (fig. 11), resembling slip bands, but never so uniformly spaced as in aluminium.

The finest lines in brass compared in height with those in aluminium, but the shear on the most active might exceed 6000 A°. There were regions 1 - 2 μ wide (1 - 2 x 10^4 A°) at elongations of several percent, in which no trace of slip appeared, (fig. 12), but with increasing deformation these spaces filled with lines. The smallest line spacing found, however, was 120 A°.

Slip depth in a line was influenced by grain size. At 0.05 - 0.3 mm grain diameter, slip depth was 200 - 500 A°; at a diameter of 1 mm the slip depth was 500 - 1000 A°.

Thus the important difference from pure metals observed in brass was irregularity of surface features; slip lines of the lightest and the heaviest shear existed side by side and spacing was quite variable.

Although slip bands such as are revealed in pure metals by the electron microscope, (fig. 10), were not revealed in brass, a brass specimen
Figs. 13a & b. The multiplication of slip bands (21);

a) Alpha brass single crystal; unetched X4. Unresolved slip clusters, widely spaced, shear 0.006.

b) Alpha brass single crystal; unetched X4. Unresolved slip clusters, closely spaced, shear 0.056.

Fig. 14.
Alpha brass single crystal, unetched X 200.
Slip clusters resolved, (21). Reduced X 0.75 in reproduction. Shear 0.056.
Figs. 15a - c.
The growth of slip cluster during increasing shear. Deflection of horizontal scratch is a measure of the shear displacement in the band, X 800. Unetched. Reduced X 0.75 in reproduction, (21).
viewed by the optical microscope, "gave the usual impression of being covered with slip bands", (7).

The form of the slipped surface in 70/30 brass has been studied by Maddin, Mathewson and Hibbard, (19, 20), using the optical microscope.

In single crystals of brass slip 'clusters', or 'bands' appeared almost instantaneously with observed deformation. Clusters that appeared as single lines at 4 x figs. 13a & b, were resolved at 200x and were seen to contain a number of fine lines, (fig. 14).

The authors called the clusters, 'macrolines'; the fine lines of which they were composed were more irregularly spaced than those in the bands in aluminium. Their spacing was upward of 1000 Å. Shear on a line was about 5000 Å but was again not so regular as in aluminium.

Fine lines were also observed, of the size of those forming macrolines, but appearing in positions isolated from them, (fig. 14).

Treuting and Brick, (21), observed that on their first appearance the 'macrolines' were widely and irregularly spaced, but with increasing strain, their density and uniformity of spacing increased, (figs. 13a & b). With increasing strain the density of lines in clusters increased and clusters increased in width by the addition of lines at intervals of about 2000 atomic diameters (≈ 6000 Å); shear on resolved lines was 700 atomic diameters (≈ 2000 Å).

The growth of a cluster is shown in figs. 15a - c; the displacement of the horizontal scratch is a result of the shear in the band.

Burghoff and Mathewson (22) in 70/30 brass single crystals, during early creep at room temperature found an approximately linear relationship
between slip band density and strain. Fourie and Wilsdorf (23) in 80/20 brass observed many of the features referred to above (7, 19-21) they also observed an average fine slip height 270- 450 \( \AA \).

Although they are irregular in depth and spacing and can be resolved by the optical microscope, it appears that the fine slip lines on brass are equivalent to the micro-slip lines on pure metals.

They are "the boundaries of elementary slip lamellae and the constituents of slip bands" and cannot further be resolved using a technique with a resolving power of 200 - 300 \( \AA \), Brown (11).

The formation of slip bands in pure metals, as indicated earlier, is associated with stage III of slip because cross slip then occurs and it is believed (18) that bands occur by a cross slip mechanism.

It is observed (18) that single crystals of alpha brass do not show stage III behaviour at room temperature until a very large strain is imposed.

A low stacking fault energy (Section 6) is thought to inhibit cross-slip; the low stacking fault energy of alpha brass may, therefore, explain why such slip bands have not been seen in this alloy under the experimental conditions used.

However, the question is complicated by observations made on other alloy systems and the above explanation may not be adequate, (Seeger (18) p. 314), but the stated objective of this review, makes it inappropriate to consider this question further.
Key to Table 1.
Table I

Slip Parameters, Pure metals and alpha-Brass; Room Temperature

'a' slip line spacing, 'b' slip band width, 'c' slip band spacing, 'd' slip step height

(see key drawing opposite)

<table>
<thead>
<tr>
<th>Metal</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
<th>Source</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al</td>
<td>~ 100 atoms</td>
<td>-</td>
<td>-</td>
<td>2000 Å</td>
<td>12</td>
<td>Strain unstated, single crystals</td>
</tr>
<tr>
<td>Al</td>
<td>10's of µ</td>
<td>-</td>
<td>2 µ</td>
<td>2000 Å</td>
<td>14</td>
<td>Very small strain, spacings decreased with increasing strain, single crystals.</td>
</tr>
<tr>
<td>Al</td>
<td>200-800 Å</td>
<td>-</td>
<td>½-10 µ</td>
<td>-</td>
<td>11,14</td>
<td>15% elongation; single crystals.</td>
</tr>
<tr>
<td>Al</td>
<td>-</td>
<td>-</td>
<td>2 µ</td>
<td>-</td>
<td>11,14</td>
<td>40% elongation; single crystals.</td>
</tr>
<tr>
<td>Al</td>
<td>400 Å</td>
<td>-</td>
<td>300-400 Å</td>
<td>(average)</td>
<td>7</td>
<td>Strain unstated; microslip height</td>
</tr>
<tr>
<td>Cu</td>
<td>300 Å</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>7</td>
<td>Strain unstated; poly crystals.</td>
</tr>
<tr>
<td>Ag</td>
<td>250 Å</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>7</td>
<td>Strain unstated; poly crystals.</td>
</tr>
</tbody>
</table>

NB - The parameter 'b' has relevance in Section 3.3, The Effect of Temperature.
<table>
<thead>
<tr>
<th>Composition</th>
<th>a</th>
<th>b</th>
<th>c</th>
<th>d</th>
<th>Source</th>
<th>Remarks</th>
</tr>
</thead>
<tbody>
<tr>
<td>70/30</td>
<td>200-500 Å</td>
<td>-</td>
<td>-</td>
<td>300-400 Å</td>
<td>7</td>
<td>Grain diameter 0.05 - 0.3 mm largest slip step height 6000 Å. Some regions 1 - 2 wide free of slip. Poly crystalline.</td>
</tr>
<tr>
<td>70/30</td>
<td>500-1000 Å</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>7</td>
<td></td>
</tr>
<tr>
<td>70/30</td>
<td>1000 Å</td>
<td>-</td>
<td>-</td>
<td>5000 Å</td>
<td>19, 20</td>
<td>Slip line depth and spacing, cluster depth and spacing variable. Single crystals.</td>
</tr>
<tr>
<td>70/30</td>
<td>6000 Å</td>
<td>-</td>
<td>-</td>
<td>2000 Å</td>
<td>21</td>
<td>Slip line depth and spacing, cluster depth and spacing variable. Single crystals.</td>
</tr>
<tr>
<td>80/20</td>
<td>-</td>
<td>-</td>
<td>-</td>
<td>275-450 Å</td>
<td>23</td>
<td>Slip line depth and spacing, cluster depth and spacing variable. Single crystals.</td>
</tr>
</tbody>
</table>
### 3.3 The Effect of Temperature

Yamaguchi and Togino (24) by averaging the resolved shear deformation over all observed bands found that in aluminium the slip movement per band was 25% less at liquid air temperature. They also found that bands formed at high temperatures were quite coarse; a microscope was needed to see bands formed at low temperatures. Deformation at liquid air temperatures produces a much greater density of slip. Polycrystalline specimens were used, of grain size large enough to easily contain the specimen gauge length.

Brown (14) found fewer, broader bands at high temperature; at liquid oxygen temperature, bands were fine and closely spaced. The same deformation at high temperature was distributed over fewer bands.

At low temperature the many bands contained fewer lamellae; at high temperature, there were few bands containing many lamellae. For 10% shear strain the spacing of bands was respectively $2 \mu$ and $6 \mu$.

At very low temperatures there was a significant reduction in the shear movement produced by a given strain as shown below for 20 - 30 measurements per specimen, but at higher temperatures, the magnitude was fairly constant.

<table>
<thead>
<tr>
<th>Deformation temperature</th>
<th>Slip distance (mean) per lamella</th>
<th>Limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>180°C</td>
<td>1500 A°</td>
<td>1200 - 1800 A°</td>
</tr>
<tr>
<td>20°C</td>
<td>2000 A°</td>
<td>1600 - 2200 A°</td>
</tr>
<tr>
<td>250°C</td>
<td>2200 A°</td>
<td>1700 - 2500 A°</td>
</tr>
</tbody>
</table>
The following values relate to observations made by Brown on aluminium (single crystals) with a 40% shear strain.

<table>
<thead>
<tr>
<th>Deformation temperature</th>
<th>Distance between neighbouring bands ((\ell))</th>
<th>No. of lamellae per band</th>
</tr>
</thead>
<tbody>
<tr>
<td>-180°C</td>
<td>(\frac{1}{2} - 1) approximately</td>
<td>1 - 2</td>
</tr>
<tr>
<td>20°C</td>
<td>2 approximately</td>
<td>3 - 4</td>
</tr>
<tr>
<td>250°C</td>
<td>4 approximately</td>
<td>5 - 6</td>
</tr>
<tr>
<td>500°C</td>
<td>10 approximately</td>
<td>12</td>
</tr>
</tbody>
</table>

If the number of lamellae in a band is divided by band spacing measured normal to the slip plane without regard to the mode of clustering, the result can be called 'slip lamella density'.

It is seen that the figures above support those previously given in requiring at 250°C approximately 1.45 times the shear movement per lamella as at -180°C.

Initially, at all temperatures a few bands are formed, widely spaced; with increasing deformation new bands form at low temperature but at higher temperatures existing bands broaden.

Rosi and Mathewson (25) for temperatures -196 to 80°C found slip band density for 0.25% extension to increase with decreasing temperature.

Cahn (26) found "intimate cross slip" to be more frequent at low and "prominent cross slip" at high temperatures. His definition of prominent cross slip appears to require prominent interconnecting slip, as distinct from intimate cross slip which does not.
3.4 **Effect of Rate of Deformation**

Crussard (27) found that slip bands in aluminium rapidly deformed, appear fully developed and remain unchanged as load is increased; at a low rate of straining bands increased in depth on subsequent increase of load, or segments of lines appeared fully formed, then grew in length. This has found support in Rosi and Mathewson, (25). Brown (28) found the spacing between bands increased with decreasing strain rate; there was an increase in slip lamellae per band but the slip displacement per lamella remained constant. This finding was supported elsewhere, (29) and, in addition, slip bands were found to be more irregular after slow straining.

Parker and Smith (30) in single crystals and polycrystals of copper deformed at various rates in tensile impact found that the number of slip lines in a band increased with impact velocity. Slip bands appeared closer together in specimens broken at higher velocity but there was also a general increase in total deformation at fracture.

Information about the effect of rate of deformation is often incidental to the main subject of the experiment which may be creep or impact. Very much of the information about the deformation of polished specimens is also for single crystals. There is a considerable fund of information concerning slip in metals due to those who have examined deformed and etched specimens.
Deformation bands in an aluminium single crystal extended 7%, X4. Etched, (26).

Deformation bands in an aluminium single crystal extended 20%, X4. Etched, (26).

Early slip unobstructed, later slip obstructed by kink bands. Aluminium extended 20%, X 500. Unetched, (34).
4. Deformation Bands

4.1 The General Nature of Deformation Bands:

During the deformation of single crystals, in tension for example, it is generally found that the rotation of the crystal due to slip (5) is not uniform throughout the specimen, but that even at low strains, banded regions develop, in which the rotation is different from that in the specimen as a whole, (26).

It is most likely that in single crystals in tension the development of these bands is due to the presence of bending moments, introduced by constraints at the grips.

In the polished surface of a specimen undergoing deformation, such bands become apparent by reflected light, due to the surface tilting that accompanies their formation. They occur in both single crystals and poly crystals and in the latter case constraints due to grain boundaries and in some cases due to the boundaries of annealing twins, are presumably responsible.

These features are given the general name, "deformation band", (31). Single crystals may contain bands a millimetre or so in width; in the grains of poly crystals, they may be microscopic in size, (2).

Once formed they increase in intensity as deformation increases, (32, 26), due to the continued surface tilting.

Most of the bands form early in deformation and persist but other, usually weaker bands, may form between, (26), or within them, (33), as deformation continues.

Fig. 16 shows deformation bands as dark lines running horizontally
Deformation band in aluminium extended 10%. Bend zones can be observed, with between them a portion disoriented with portions outside, X 250. Unetched, (26).

Kink band in aluminium extended 25%. Disorientation in the band causes slip on new system, X 32. Unetched, (26).
across the field in a specimen deformed 7%. With increasing deformation, the bands do not increase in width, but may become wavy, (fig. 17). The bands are narrow, compared with the width of the material between them and have been found to contain a range of orientation amounting to 2°. There is little or no lattice distortion in the material between the bands.

In the early stages of its formation, a deformation band does not cause great obstruction to slip. With continued deformation, the lattice rotation in the band causes it to completely obstruct slip, (Honeycombe, 34). This effect is illustrated in fig. 18.

The simplest deformation bands are generated when a portion of the lattice is rotated about an axis lying in the slip plane and perpendicular to the slip direction, (26, 32, 34-38).

Continuity with the lattice outside the band is maintained by regions of curvature in the lattice, which lie in the interface between the band and the material between the bands, (fig. 19). These regions of curvature lie on a pair of surfaces initially plane and parallel, called 'bend planes', (35), which together with the lattice in the band, rotate from their initial position as deformation continues. Ultimately the rotation of the lattice in the band, may cause slip in the band to initiate on another system, (fig. 20).

The term 'kink band' has been used, (34) to describe a deformation band having a 'sharp curvature' in the bend planes.

Cahn (26) has observed that "there is often much curvature in deformed
poly-crystalline specimens in which the most strongly bent regions have been called deformation bands".

The term 'deformation band' is therefore seen to describe an extensive range of phenomena. It describes forms such as those above, that are relatively simple, sometimes because the strain is small or because single crystals are used.

In heavily deformed and poly-crystalline specimens it may describe very complex bending and variation of orientation.
Figs. 21a & b. Schematic illustration of the transformation of a region of curvature into a polygonised region, (39).

a) Regions of curvature

b) Polygonised regions

Fig. 22. Polygonisation in nickel deformed 1.4% at room temperature and annealed 15 min. at 950°C, X 375. Etched Polygonisation boundaries delineated by pits, (40).
5. **Polygonisation**

5.1 **General Characteristics:**

Curvature in a lattice implies instability, as a result of high dislocation density and of the associated elastic strain.

As a consequence, suitable conditions will permit dislocations to move and interact so as to produce annihilation in many instances, together with a diminution of lattice curvature. One means to diminish lattice energy is that edge dislocations of the same sign should assemble one above the other in a surface perpendicular to the slip direction; a proper distribution on this surface may require dislocations to climb along it from one plane to another. Such events can create small segments of lattice unbent or substantially so, separated by a low angle interface. The result is a polygonised lattice, (6).

The higher the temperature at which polygonisation occurs, the smaller the residual curvature and the smaller the dislocation density within a segment of lattice. Diminution of lattice energy is very rapid about a certain temperature, above which the metal may be considered fully polygonised, (figs. 21a & b). The separate segments of a polygonised lattice are called sub-grains and the interfaces between, 'sub-grain' or 'low angle' grain boundaries.

In a poly crystal, many sub-grains may be contained within one ordinary grain.

Polygonisation has been studied by various authors (39-46), who have been able to detect it in a variety of ways.
It may be readily detected, in Laue diagrams when the distorted spots characteristic of the deformed metal appear to change to a number of discrete spots associated with the formation of the sub-grains, (46).

Sub-grains may also commonly be revealed, fig. 22, by the use of the appropriate etchants, (39–45). Polygonisation may also produce surface effects due to dislocation movements involved. These are illustrated in fig. 21.
6 Deformation - twinning

6.1 Introduction:

"Crystals are said to be twinned if they are composed of portions that are joined together in definite mutual orientation", (2).

This definition is made more specific by the observation that in a particular case there exists a plane in which the lattice of the one portion appears as a mirror image of the other, (1, 47). This plane is called the "twinning plane" and across it, the lattice of one portion is coherent with that of the other, (48).

Twins may arise 1) by a process of growth, 
or 2) as a result of deformation, (47)

Deformation twins alone will be discussed here.

In the simplest case, the twinning operation is described macroscopically by a simple shear movement parallel to the twinning plane; other cases, in addition require small individual adjustment in the position of the atoms, (2).

The generation of a homogeneous twinned lattice requires that in the twinned volume, the twinning operation occurs on each lattice plane in turn without exception, so as to relate each plane to its predecessor in identical fashion, (49).

The plane and the shear direction involved in the twinning operation are called the 'twinning elements'. The following are the twinning elements for metals and alloys in the most common crystal structures:
Figs. 23a & b. Atom sites in (111) plane of F.C.C. lattice.

Perfect slip vector, with twinning and untwining vectors as components. Movement of extended dislocation, (6).

a) Atom sites, in layers 'a', 'b' and 'c'.
b) Partial dislocations and stacking fault.

Fig. 24. Twinning in F.C.C. lattice. Twinned sites in full line.

After Schmid and Boas "Plasticity of Crystals" F.A.
Deformation twinning has been studied for a considerable period, yet, until quite recently "it was not thought that twinning caused a significant part of the deformation in plastically deformed face-centred cubic metals", (2).

However there is now abundant evidence (48, 50 and others) for twinning in metals and alloys having this structure particularly at low temperatures.

In the face centred cubic structure, the twinning shear is 1/6 of the lattice vector, \( \frac{1}{6} \mathbf{a} \langle 112 \rangle \).

Lattice planes \( \{111\} \) are laid down in a sequence of three so that the fourth plane is equivalent to the first and so on. Each plane is displaced relative to the last by a movement \( \frac{a}{6} \langle 112 \rangle \).

As a result, if the atoms in the base layer lie at "a", (fig. 23a),
those in the second and third layer lie at "b" and "c" respectively.

The (111) layer sequence is ABC ABC' ABC indefinitely repeated. If layer C' and all above be shifted \( \frac{a}{6} [211] \) the sequence becomes ABC ABA' CA \( \ldots \ldots \ldots \) and a four layer segment of hexagonal close packed lattice is introduced.

This is a (single) faulted sequence and contains an 'intrinsic' stacking fault, which is equivalent to the removal of one lattice layer.

A consecutive shift of layers B and above by \( \frac{a}{6} [211] \) produces the double faulted sequence ABC ABA' ABA' ABC \( \ldots \ldots \ldots \), called an 'extrinsic' stacking fault or "twinning fault" which is equivalent to the insertion of an extra lattice layer at \( \hat{A} \), (51). This fault is the simplest twin, with twinning planes at \( \hat{B} \) and \( \hat{C} \); it is one layer (\( \hat{A} \)) thick and is produced by a proper reiteration of single faulting.

It is clear that the same shear on all planes above \( \hat{C} \) produces a twin ABC ABACBCA \( \ldots \ldots \ldots \), two layers thick and so on.

In this context the shear \( \frac{a}{6} [211] \) can be called 'the twinning shear'.

These notions are illustrated in fig. 24.

Slip on the (111) plane can be produced by a movement \( \frac{a}{2} [110] \); this can be more readily achieved by two movements \( \frac{a}{6} [211] \) and \( \frac{a}{6} [121] \), (12). As a result, an atom in a "b" layer, (fig. 23a) moves to a "c", then back to a "b" position, executing a slip translation.

The dislocation \( \frac{a}{2} [110] \) can lower its energy, (12, 6) by the reaction:

\[
\frac{a}{2} [110] = \frac{a}{6} [211] + \frac{a}{6} [121]
\]
producing the new dislocations referred to above. These dislocations are called 'partial dislocations'.
because, alone, they cannot produce one perfect lattice translation, (6).

Slip is produced by these partials when they move in a pair, one leading the other through the lattice. The leading dislocation \( \frac{a}{6}[\bar{2}11] \) has the twinning shear just discussed; the trailing dislocation, \( \frac{a}{6}[\bar{1}21] \), produces the "untwining shear", (50). Between the pair is a ribbon of stacking fault, (6) produced by the leading partial, fig. 23b. The partial dislocations and the stacking fault between, constitute an "extended dislocation". Passage of these partials in pairs produces slip; the passage of an odd number produces faulting, (53).

The separation of the partial dislocations, due to the repulsion between them, is limited by the interfacial energy, (\( \gamma \)) in the stacking fault, (6) and ribbons of stacking fault tend to an equilibrium width. They are wider in metals and alloys having a low value of \( \gamma \), (stacking fault energy), so that in copper, (\( \gamma \approx 40 \text{ ergs cm}^{-2} \)) they are \( \sim 35 \text{ A}^0 \) wide and in aluminium, (\( \gamma \approx 200 \text{ ergs cm}^{-2} \)) only 7 \( \text{ A}^0 \) wide, (52).

Stress is capable of causing an increase in area of stacking fault if the untwining dislocation can be strongly anchored; an appropriate application of stress might then produce a considerable area of stacking fault, (50).

However, the partials referred to above, both move in the (111) plane with considerable ease, limiting the area of faulting. The twinning partial is therefore unlikely to be able to produce twinning, (50).

The dislocation \( \frac{a}{2}[011] \) which does not readily move in the (111) plane can readily reduce its energy, (12, 6), by the reaction:
Fig. 25. Twinning mechanism of Ookawa, (57). The line 'ab' is a locked segment of dislocation line of Burgers' vector $\frac{a}{2} 011$ lying in the (111) plane; 'a' and 'b' are the points of emergence into the plane.

Fig. 26. Twinning mechanism of Cohen and Weertman, (56).
\[
\frac{a}{2} [011] = \frac{a}{3} [111] + \frac{a}{6} [\bar{2}11], \quad (54).
\]

Movement of the twinning partial, \(\frac{a}{6} [\bar{2}11]\), in the (111) plane, under stress, is easy. However, the partial \(\frac{a}{3} [111]\) is strongly anchored, (Sessile (57)), in that plane. Thus, movement of the partial, \(\frac{a}{6} [\bar{2}11]\), may here produce an indefinitely large area of fault.

Proposed twinning mechanisms which enable a twin of finite thickness to be formed by the above process are:

1) The pole mechanisms, related to that of Cottrell and Bilby, (49) and modified for face-centred cubic metals by Ookawa (57) and Venables (54), by which the twinning dislocation, having acted once, climbs out of the affected plane onto successive twinning planes.

2) The mechanisms that require a fresh twinning source to operate on each affected plane; Cohen and Weertman, (56).

Ookawa, fig. 25, supposes that a dislocation \(\frac{a}{2} [011]\) by the reaction above, produces a dislocation \(\frac{a}{6} [\bar{2}11]\), which is capable of sweeping in the (111) plane. He further proposes that a segment of the dislocation line lies in the (111) plane and that its points of emergence act as "poles" for the sweeping dislocation, \(\frac{a}{6} [\bar{2}11]\).

Single action of this dislocation on a (111) plane produces an intrinsic fault on the plane. On reaching the [\(\bar{2}11]\] direction the partials recombine to form the dislocation \(\frac{a}{2} [011]\) which climbs one lattice layer along the (011) plane, there to repeat the operation that occurred on the previous plane. Indefinite reiteration of the operation produces a volume of twin.
Cohen and Weertman, (56) in a proposal of the second kind, imagine that after extensive deformation there are heavy pile-ups of extended \( \frac{a}{2} [011] \) dislocations on the \((11\bar{1})\) plane. The pile-ups occur, for example, at Cottrell-Lomer barriers, (3, 6).

The partial dislocations, \( \frac{a}{6} [112] \) and \( \frac{a}{6} [\bar{1}21] \), piled-up at barriers, recombine subsequently, to react again as follows:

\[
\frac{a}{2} [011] = \frac{a}{6} [\bar{2}11] + \frac{a}{3} [11\bar{1}];
\]

the escape of the twinning dislocations, \( \frac{a}{6} [\bar{2}11] \) from the piled-up groups through the \((11\bar{1})\) planes forms an intrinsic fault on each affected plane, (fig. 26).

The authors observed that the twins formed would be faulted, a fact of observation, (58) and that the mechanism would operate best in systems of low stacking fault energy.

6.4 Stacking-fault Energy and Stacking-fault Probability

6.4.1 Introduction:

Stacking fault energy has not been directly determined; values are obtained by the measurement of other properties of deformed metals, thought to be related to it.

These include line broadening and line peak displacement in powder patterns of a metal or alloy, (48, 59), the bowing apart and curvature of partial dislocations, (60) and behaviour in creep, (60).

There is often poor agreement between values obtained by different methods. Frequently, however, the magnitude of the values obtained for a group of materials, places them in the same order whichever method
of determination is used.

Another quantity frequently used is "the stacking fault probability, \(\phi\)". This is defined as the fraction of \(\{111\}\) type planes faulted by deformation. It can be determined from x-ray measurements, but the calculation assumes that faulting occurs on only one family of planes and that a faulted surface crosses the full width of the crystal, (53).

The stacking fault probability has an inverse relationship to the stacking fault energy, (61).

Values of stacking fault energy for a number of metals and alloys are summarised in Table II at the end of this section. It will be seen, as noted previously, that the values for pure metals differ widely.

Furthermore, alloying has a considerable effect on stacking fault energy.

If the foregoing mechanisms for twinning are accepted at least in general terms, it is clear that stacking fault energy is closely related to the formation of deformation twins, i.e. materials with low stacking fault energy twin more readily than those with high stacking fault energy.

This relationship is discussed in detail by a number of authors, (48, 50, 51, 54 and others) and will be referred to later.

6.4.2 Stacking fault Energy and Faulting in Alloys:

Davies and Cahn (63) found that in the alpha solid solution alloys of the univalent, face-centred cubic metals copper and silver, the addition
Fig. 27
Stacking fault probability for solutes in the same period as the solvent, (63). Fig. 27 as a function of composition and fig. 28 as a function of electron concentration.

Fig. 28
Size factors:
A: Cu-Zn, 1.04
Ag-Cd, 1.03
B: Cu-Ga, 1.06
Ag-In, 1.13
C: Cu-Ge, 0.96
Ag-Sn, 1.05

Fig. 29
Stacking fault energy and probability for the Cu - Ni system. (59).
of a solute of greater valency caused an increase in stacking fault probability, (Section 6.4.1).

The addition to either solvent metal, of a solute in its own period, would either at the same atomic percentage, (fig. 27) or at the same electron concentration, (fig. 28), produce very nearly the same stacking fault probability, if in each case, the solute had the same valency.

For the same atomic percentage, (fig. 27) or for the same electron concentration, (fig. 28), the solute with the greater valency produced a greater stacking fault probability.

There was conflicting evidence, (63, 61), that for a particular solute valency the further apart in period the solvent and solute were, the greater was the stacking fault probability, ($\alpha$), at the same electron concentration.

The influence of the size factor was not mentioned and in fact the size ratio was always near unity in this work.

Foley, Cahn and Raynor, (64) were able to support these findings in part, as a result of a study of stacking fault probability in the alpha phase alloys of the systems Cu/Si, Cu/Ge, and Cu/Ge/Si. Their results showed that the stacking fault probability increased with electron concentration in a fashion similar to that found by Davies and Cahn, (63).

At an electron concentration of 1.3 the stacking fault probability of the alloy in each of the binary systems, was about twenty five times that of pure copper.

The stacking fault energy and stacking fault probability of alloys in
Figs. 30a and b. Categories of stress strain curve, (48).

a) without load drop; no twinning  b) with load drop and twinning

Fig. 31. Initial twinned region, twinned on one plane, X 48.
Unetched, (48).

Fig. 32. Twinning on primary and conjugate slip planes, X 150.
Unetched, (68).
the copper nickel system has been determined by Henderson, (59), for compositions ranging from pure copper to pure nickel, (fig. 29). These data will again be referred to later.

6.5 Twinning and Faulting in Different Materials

6.5.1 Pure Metals:

Blewitt, Coltman and Redman, (48), have reported twinning in single crystals of pure copper, \((\gamma = 40 \text{ergs cm}^{-2})\), deformed in tension at 4.2°K and 77.3°K. By x-ray diffraction the twinning plane was found to be the \(\{111\}\) and the direction \(\langle112\rangle\).

Twinning occurred after heavy extension in slip, for a limited range of initial orientations. At the onset of twinning there was a marked load drop, (fig. 30b), thereafter twinning continued at a lower level of stress, "with no evidence of work hardening". If twinning did not occur, there was no load drop prior to fracture, (fig. 30a).

Fig. 31 shows the initial twinned region of a specimen with twinning occurring on a single plane. For certain initial orientations, twinning occurred "on the primary and the conjugate slip planes", (fig. 32).

The authors did not consider that there was a critical shear stress for twinning. However, Thornton and Mitchell, (62), found evidence for a critical stress for twinning in copper.

In a note to the paper, (48), the authors reported twinning in silver \((\gamma' = 30 \text{ ergs cm}^{-2})\) and gold \((30 \text{ ergs cm}^{-2})\) but never in aluminium, \((\gamma = 200 \text{ ergs cm}^{-2})\), even under favourable conditions.
Figs. 33a – c. Effect of temperature on twinning habit in specimens of an alloy, gold 25 silver 75 atomic percent, (50). (See fig. 34).

a) twinning in one plane only; near upper temperature of twinning; $193^\circ K$.

b) twinning on primary and conjugate slip planes at adjacent sources; $77.3^\circ K$.

c) twinning on primary and conjugate slip planes concurrently; $20.4^\circ K$. 
Suzuki and Barrett, (50), found mechanical twinning in gold and silver and in their alloys; in silver twinning could be produced by slow extension even at $0^\circ C$; gold did not twin at $0^\circ C$ but did at $77.3^\circ K$ and probably a little higher.

The authors considered that in this system, initial orientation was important in permitting, for favourable orientations, a critical resolved twinning stress to be reached in advance of failure by slip.

The orientations permitting twinning were similar to those found elsewhere, (48).

Haasen, (4), in nickel, $(\gamma = 90 \text{ ergs cm}^{-2})$ single crystals, deformed in tension at $4^\circ K$ and $20^\circ K$, found no load drop, but found metallographic evidence of twinning in the necked portion of the specimens. A similar finding was made, (48), in conditions unfavourable to twinning, in copper extended at $77.3^\circ K$.

The necked region of a specimen may experience stresses several times larger than those elsewhere, (4).

6.5.2 **Alloys:**

For all compositions in the silver gold system, Suzuki and Barrett in the work (50) referred to in the previous section, found an upper temperature for twinning near which the alloys twinned on a single plane, (fig. 33a).

Silver and silver rich alloys showed additional modes of twinning at lower temperatures, (figs. 33b and c).
Fig. 34. Stress-strain curves associated with twinning at different temperatures, (50). (See figs. 33a–c).
The orientation of the stress axis in each specimen is as shown in the stereographic triangle.
Fig. 35. Apparent stacking fault energy as a function of composition in the system Cu - Zn, (65).

Fig. 36. Twinning and faulting behaviour of alpha-phase Cu - Al alloys as a function of stacking fault energy and twinning stress, (58).
For these alloys, as temperature of twinning was reduced, propagation from a single source on one plane (fig. 33a) was replaced by propagation from several adjacent sources on either the primary or the conjugate slip plane, (fig. 33b).

This behaviour was succeeded at still lower temperatures by the appearance in a given area, of twinning concurrently occurring on both the primary and the conjugate slip plane, fig. 33c.

In these alloys it was also observed that as the deformation temperature decreased, so did the load drop at onset of twinning, (fig. 34).

Thornton and Mitchell (62) studied the deformation twinning behaviour of copper and its alpha phase alloys containing 5, 10, 20 and 30 percent zinc deformed at temperatures between 4°K and 300°K. It was known, (60, 65), that as zinc content increased, the stacking fault energy decreased at first rapidly, then more slowly, (fig. 35).

They found that copper behaved as reported elsewhere, (48); there were always large load drops on twinning.

As the temperature of an alloy decreased, the load drop at onset of twinning also decreased, (62, 50). The load drop at the highest temperature for twinning also decreased with alloying. It was found that the 70/30 alloy gave no visible evidence of twinning at any test temperature used, nor was there evidence of load drop. However, the authors noted that Venables had found evidence of "prolific, but fine twinning", in electron micrographs of this material under the same conditions.

These findings suggested that twinning becomes more minute in
character as stacking fault energy and temperature decrease. This is consistent with the findings of Suzuki and Barrett (50) referred to above.

The experimental results of Thornton and Mitchell (62), indicated that there was a critical shear stress for twinning, which decreased linearly with decreasing stacking fault energy. The authors quoted current work by Venables, showing that for the same stacking fault energy, the twinning behaviour of the alpha alloys of the systems Cu/Zn and Cu/Al was similar.

Venables (58) examined the twinning and faulting behaviour of the alpha phase alloys of the copper aluminium system, to determine the effect of stress, orientation of the tensile axis, temperature and stacking fault energy.

The temperature and the orientation of the tensile axis were found to have subsidiary influence. Stress and stacking fault energy were found to be important in determining twinning and faulting behaviour. Using stacking fault energy values due to Howie and Swann (60), data were plotted to yield fig. 36; the main body of the data used were for results at 77°C.

Twinning with load drop occurred for low alloy, (higher γ ) compositions. It occurred without load drop for compositions of low stacking fault energy.

Fig. 36 shows a critical stress for twinning and/or faulting, that decreases with stacking fault energy, whereas with alloying, there is an increase in the critical shear stress for slip, (3).

At high stacking fault energies, twinning occurred at high stress,
<table>
<thead>
<tr>
<th>add + 3g/ml</th>
<th>5</th>
<th>15</th>
<th>30</th>
<th>60</th>
<th>120</th>
<th>60</th>
</tr>
</thead>
<tbody>
<tr>
<td>add + 4g/ml</td>
<td>5</td>
<td>15</td>
<td>30</td>
<td>60</td>
<td>120</td>
<td>60</td>
</tr>
<tr>
<td>add + 5g/ml</td>
<td>5</td>
<td>15</td>
<td>30</td>
<td>60</td>
<td>120</td>
<td>60</td>
</tr>
</tbody>
</table>

Note: Concentrations are given in g/ml.
### Table II

*Deformation twinning* of metals and alloys:

<table>
<thead>
<tr>
<th>Metal or Alloy</th>
<th>γ ergs cm⁻²</th>
<th>Behaviour</th>
<th>Source</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silver</td>
<td>~ 30</td>
<td>Twinning at 20.4°K to 273°K</td>
<td>50</td>
</tr>
<tr>
<td>Gold</td>
<td>~ 30</td>
<td>Twinning at 20.4°K to 77.3°K</td>
<td>50</td>
</tr>
<tr>
<td>Copper</td>
<td>~ 40</td>
<td>Twinning at 4.2°K and at 77.3°K</td>
<td>48</td>
</tr>
<tr>
<td>Nickel</td>
<td>~ 90</td>
<td>Twinning only in neck of specimens fractured in tension at 4°K and 20°K</td>
<td>4</td>
</tr>
<tr>
<td>Aluminium</td>
<td>~ 200</td>
<td>Twinning not found</td>
<td>48</td>
</tr>
</tbody>
</table>

**Alpha phase alloys**

<table>
<thead>
<tr>
<th>Cu/Zn + Cu/Al</th>
<th>~ 40</th>
<th>As for pure copper T = 77°K Twins with load drop</th>
<th>58, 62</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu/Zn + Cu/Al</td>
<td>~ 33</td>
<td>Similar to pure Cu T = 77°K Twins with load drop</td>
<td>58, 62</td>
</tr>
<tr>
<td>Cu/Zn + Cu/Al</td>
<td>~ 22</td>
<td>Twins with and without load drop. T = 77°K</td>
<td>58, 62</td>
</tr>
<tr>
<td>Cu/Zn + Cu/Al</td>
<td>~ 8</td>
<td>Twins without load drop; some faulting</td>
<td>58, 62</td>
</tr>
<tr>
<td>Cu/Zn + Cu/Al</td>
<td>~ 3</td>
<td>Faults and some twins (Approx. 70/30 composition in brass)</td>
<td>58, 62</td>
</tr>
</tbody>
</table>
with load drop and irregularity in stress behaviour. Each irregularity was associated with the production of stacks of fine twins which formed only in localised areas of the specimen. The stacks were about 5000Å thick and contained 50% of twinned material.

Wide twin bands visible in the optical microscope, therefore contained many lamellae, (figs. 33a - c).

Alloys of lowest stacking fault energy initially formed stacking faults only. These were detected by the electron microscope. At higher stresses these alloys formed stacking faults and twins.

Alloys of higher stacking fault energy formed small amounts of twin in marginal or slightly unfavourable low stress conditions.

6.5.3 Summary

Table II summarises data contained in sections 6.5.1 and 6.5.2.

The data suggest a close relationship between stacking fault energy and twinning behaviour; deformation twinning appears more likely in metals of low stacking fault energy.

Silver and gold however, with the same stacking fault energy, have different upper temperatures of twinning. It therefore appears that stacking fault energy alone is not a sufficient criterion of deformation twinning tendency.

The values determined, (50), for the critical shear stress for twinning at 77.3°K, show that gold has a critical twinning shear stress of ~ 9.5 kg mm⁻² and silver ~ 6.5 kg mm⁻². Temperature has not a
significant effect on these quantities, (50).

The authors (50) observed that where a specimen did not twin, the resolved twinning shear stress never reached the critical value for the composition concerned, before necking and fracture occurred.

It appears likely that the different behaviour of silver and gold arises from the difference in the critical twinning stress.

6.6 Rate of Deformation and Twinning:

C.S. Smith, (66), using high rates of stress loading was able to produce twinning in copper at room temperature. Under similar conditions, Appleton, Dieter and Bever (67) were able to produce twinning in an alloy containing gold 82.6 and silver 17.4 weight percent, which had been found, (50), not to twin above about 120$^{0}$K by slow loading in tension. Nolder (68) under similar conditions produced deformation twinning in nickel at room temperature, but failed to find it after tensile deformation in liquid helium. At the same temperature Haasen (4) had found twinning only in the necked portion of nickel specimens fractured under similar conditions.

These authors (66 - 68) used explosive shock loading in compression. The deformation produced never exceeded 5 - 7% and was generally half this, but the change in physical properties in copper was such as accompanies a 95% reduction by rolling, (66).

Explosive loading of the specimen caused "the passage of a shock wave at pressures which are very much greater than the yield strength".
The pressure rose almost instantaneously as the wave passed a point and continued to act for a few microseconds only. The pressure was then released almost as rapidly, (67).

It was found, however, that there was a minimum pressure (66 - 68) below which twinning did not occur. In nickel, this pressure was approximately 350 kilobars, (≈ 5 \times 10^6 \text{ lbs in}^{-2}).

It has long been known, (69), that impact, even at lower rates of loading than those cited above, is more likely to produce twinning, than is slow straining.
The Use of Etching to Reveal Evidence of Deformation

Features Revealed by Etching

Introduction:

Cumming and Moore in a review of etching, (70) state that the revelation of metallographic structure is possible because the rate of dissolution is different from point to point in the surface etched. Among the factors determining the rate of solution are:

1) The orientation of the specimen lattice relative to the surface to be etched and

2) The emergence of dislocations in that surface.

As a consequence of these and of the effect of deformation upon them, a pattern of active sites exists in the surface; at these dissolution is more rapid than it is elsewhere. Dissolution tends to expose the relatively inactive close packed planes, (70) by removal of metal from steps at the edges of incomplete close packed planes or at the sites of emergence of dislocations with a component of their Burgers vector (3) normal to the surface etched, (71).

The active sites become anodic to others, (72), which consequently become cathodic.

During etching, ions of various kinds diffuse to anode or cathode and may, by deposition, 'colour' or 'stain' them. For example anodic areas on a copper specimen may be coloured by the deposition of a sulphide film, (72) and cathodic areas on nickel specimens, by a complex ion derived from the ferric chloride used as etchant, (Corson, 73).
Saubestre (74) refers to several different ions of this kind, positively or negatively charged, formed during the etching of copper by ferric chloride.

If in addition, these ions can be deposited as an optically anisotropic epitaxial film, the surface may very sensitively reveal in polarised light, phenomena such as change of orientation (75, 76) from grain to grain in a polycrystalline specimen or changes of orientation within a grain due to deformation.

Polarised light is also critically reflected (77, 78) from the facets of geometrical etch pits (79, 80) and from steps or terraces produced by etching (70, 77). This phenomenon serves to sensitively reveal, similar features to those revealed through the existence of an optically active epitaxial film.

The foregoing observations form the general basis of the following brief review of etching.

7.1.2 The Effect of Lattice Orientation

Planes other than close-packed planes can be considered as being composed of steps whose surfaces are themselves close packed planes, (70).

In the general case a surface may therefore be rapidly attacked at these steps, exposing close packed planes.

Terraces so produced, by etching a grain of uniform orientation, serve to distinguish it from another of different orientation.
Etch pits can be nucleated at sites of dislocations. Since a particular etchant may expose a particular low index lattice plane, (70) the facets formed in geometrical etch pits, may by reflection serve to differentiate one grain from another in a polycrystalline specimen.

Where sufficient variation of orientation occurs in an individual grain, a change in geometry of the features referred to above, will indicate its presence.

Planes of high indices are more active than planes of low indices or than close packed planes, because of a greater density of active steps (70). There will thus be a difference in rate of dissolution from grain when a suitable etchant is used and grains of different orientations will be distinguished from one another by associated differences of relief in the etched surface.

Sufficient variation of orientation within a single grain will similarly create a corresponding profile of relief during etching.

7.1.3 The Detection of Single Dislocations:

Etch pits may be produced at the sites of suitably oriented dislocations, whether the dislocation be of the edge or the screw type (3). In particular a screw dislocation is capable of aiding solution "by maintaining steps in the surface", (71).

Young (71) recognises three classes of etch pit:

1) Those formed at 'clean' dislocation sites,
2) Those formed at 'dirty' or 'decorated' sites, i.e. the sites of dislocations with an "atmosphere" (3),

3) Those formed at sites where impurity segregation has been sufficient to permit "precipitation there of a discrete phase".

Pits are nucleated at clean dislocations only when the etched surface differs by only a few degrees of angle from a close packed surface, (71). The pit is nucleated at a surface step caused by the dislocation.

Etching causes the step to move away from the source exposing close packed layers; the pit therefore has a geometrical outline, which in the face-centred cubic structure is one of the sections of a cube, (79, 80).

The formation of well defined pits is favoured by the presence of suitable amounts of certain constituents, (84, 71) such as bromine for example, in copper etchants, (71). Pits at the sites of 'dirty' dislocations are nucleated in part at least, due to "the difference in chemical nature", (71), in the immediate environs, caused by the atmosphere, (3). Once nucleated however, a geometrical etch pit is formed provided the surface etched is close to having a close packed configuration, (71).

Where a discrete phase is precipitated at a dislocation, the dislocation may be considered "as a line source of this impurity" and a suitable etchant may etch such a site more actively than it does the bulk of the crystal, (71).

Such a site is detected with facility in all surfaces of section, whatever the indices.
Fig. 37. Aluminium (99.998% pure) deformed 40.2% at -72°C by slow compression. Kink bands almost vertical, undulant slip markings diagonally across the field, X1000. Etched, (88).
7.1.4 The Detection of Slip:

At the lowest strains slip may be revealed by the etching of dislocation sites associated with slip surfaces.

Slip is therefore revealed by etch pits or groups of etch pits lying in the trace of the slip band (82 - 84).

This is not the mechanism in all cases, particularly in metals of low stacking fault energy since Swan and Nutting, (86) in 70/30 brass and in the alloy copper 92 aluminium 8 weight percent, (each of very low stacking fault energy), found that after deformation, dissolution caused preferential attack at stacking faults, causing "corrosion slots".

Slip lines may also be revealed in some alloys after deformation, by a precipitation treatment. Gayler (98) in the aluminium-copper alloy containing 4 percent by weight of copper, revealed markings that at low magnification appeared as lines, but at higher magnification could be resolved "as rows of particles of very fine precipitate".

McKinnon (99) in a deformed supersaturated alloy of silver in aluminium was able to demonstrate similar features.

There are occasions when, after a considerable amount of deformation, undulations parallel to the slip trace are produced by etching. They are obviously regions of relief attack. Fig. 37 shows their development in the etched surface of aluminium deformed 40.2% at -72°C (Bannejee, 88). They were not observed after less than 12% deformation.

Bannejee thought they were due to the association of stacking faults with the slip process. However, in view of the very high stacking energy
of aluminium, it appears probable that some other mechanism should be considered. For example as will be considered subsequently in connection with the present work, slip markings of this kind may well be revealed by preferential attack due to systematic local curvature, or due to an associated grouping of dislocations.

7.1.5 The Detection of Twinning:

The definition of twinning given in Section 6.1 shows that the twinning plane separates regions of different orientation. Etching will therefore reveal twinning as a change in orientation.

In deformation twinning the situation may be more complex due to the existence of faulting. Deformation twins are referred to again in the following sections.

7.1.6 The Detection of Deformation-bands:

As has been discussed in Section 4, deformation bands are regions of a crystal having an orientation different from that of the adjoining parts of the crystal, an etchant that reveals differences in orientation (Sections 7.1.1 to 7.1.3) can be used to reveal their presence.

Such etchants can be very sensitive, for example, in lead a difference in orientation of $2^\circ$ of angle may be detected, (89) in an etched specimen, by use of polarised light. Dayton (78) has shown that differences in orientation can be sensitively revealed in polarised light due to the
Fig. 38. Slip line indications in tough pitch copper compressed 2.5% at room temperature. Electrolytic thiosulphate etch, X500, (87).

Fig. 39. Slip line indications in 70/30 alpha brass, compressed 2.5% at room temperature. Electrolytic thiosulphate etch, X500. (87).

Fig. 40. Slip band indications at grain boundaries and occasionally in the body of the grain. Slip line indications elsewhere in the grain. Relief etch X500. 70/30 alpha brass, (87). 10% compression.

Fig. 41. Slip band indications in one system. Bands at grain boundaries and crossing grain. Slip line indications in two other systems, X500. 70/30 alpha brass, (87). 10% compression.
changing orientation of etch pits in the etched surface.

Using a technique capable of laying down an optically anisotropic epitaxial film, Hone and Pearson, (75) have revealed differences in orientation in aluminium and Woodard (76) in monel metal.

An etchant capable of relief attack may also reveal them; see Section 7.1.2.

7.2 The Use of Etching to Reveal Features due to Deformation.

7.2.1 Etch-markings Associated with Slip in Copper and Alpha-Brass:

The metallographically prepared surface of a deformed specimen frequently reveals by etching, markings that have been reliably related, (87), to the slip deformation of the metal. Because of the ease with which these features can be developed in copper and copper based alloys, considerable work has been devoted to their study in these alloys. The extensive literature has been reviewed by Samuels (87) and by others.

Samuels' work on copper and the alpha brasses led him to define two kinds of etch marking associated with slip:

Type I. Slip-line Indications - "systems of parallel grooves or lines of etch pits, oriented according to the crystallographic planes of the particular grains in which they occurred. Developed at low deformations". Figs. 38 - 40.

Type II. Slip-band Indications - "relatively wide bands oriented similarly to those of Type I and apparently a development of them. Developed after low and medium deformation". Figs. 40, 41.
The markings have been studied in copper and brass; particular attention has been paid to the high brasses. Slip line indications in copper are illustrated in fig. 38, those in 70/30 brass in figs. 39 and 40. Slip band indications in 70/30 brass are seen in figs. 40 and 41.

There have been many reports of etch markings seen after about 20% deformation or more, (87) and fewer at lower deformations. However, there has been considerable disagreement in the observations even in the same material.

Samuels (87) was able to show that this confusion could be rationalised if it were recognised that many of the 'standard' etchants used were capable only of displaying "slip band indications" and not 'slip line indications'. He also showed that the threshold deformation for appearance of both 'slip band' and 'slip line indications' depended on composition.

Samuels using poly crystalline specimens, found that indications first appeared (after deformation by a large number of hammer blows), after the following deformations:

<table>
<thead>
<tr>
<th>Zinc %</th>
<th>Deformation %, type 1.</th>
<th>type 2.</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>1.5</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>3.0</td>
<td>50</td>
</tr>
<tr>
<td>10</td>
<td>2.5</td>
<td>9</td>
</tr>
<tr>
<td>15</td>
<td>1.0</td>
<td>7</td>
</tr>
<tr>
<td>20</td>
<td>0.5</td>
<td>4.5</td>
</tr>
<tr>
<td>30</td>
<td>0.1</td>
<td>2</td>
</tr>
</tbody>
</table>
Fig. 42. Slip band indications in 70/30 alpha brass compressed 20% at room temperature. The markings are narrow and of uniform width and are dark etching. Etched - acid ferric - chloride, X500. (87).

Fig. 43. Deformation indications in 85/15 brass compressed 20% at room temperature. Indications are often lenticular in form and composed of finer 'laminations'. Electrolytic thiosulphate etch, X500. (87).

Fig. 44. Deformation indications in 90/10 brass compressed 20% at room temperature. Indications composed of fine lenticular markings. Clusters broader than in 85/15 brass. Different systems may etch with colour contrast. Ferric-chloride etch, X500. (87).

Fig. 45. Deformation indications in tough pitch copper compressed 29% by one blow at -183°C. Ferric chloride etch, X500. (97).
Using the most sensitive methods for their detection (90) markings in 70/30 brass were not observed until after a significant slip deformation had occurred, (83). Jacquet (91, 82) has resolved them into rows of etch pits in the vicinity of grain boundaries.

In copper, "slip line indications" appeared at first "as systems of parallel lines of etch pits and usually extended completely across the grain", (fig. 38). They became "more numerous and more marked" at higher deformations and eventually appeared as definite lines. In 70/30 brass they were "generally similar to those found in copper", they appeared in brasses of other compositions but at higher deformations.

Slip band indications first appeared in 70/30 brass as short, dark lines developed along slip line indications in areas adjacent to the grain boundaries and twin boundaries, (figs. 40, 41). They rapidly grew in number and in length as deformation increased and appeared to be closely related to and an extension of slip line indications.

Lightly etched they could be resolved as "a series of fine parallel lines", translated at intersections with those of other systems. By heavier etching with "colouring reagents" they appeared as unresolved coloured bands, (fig. 42). By heavy relief etching they appeared as "marked grooves", (fig. 40).

Slip band indications grew broader as zinc content decreased, (figs. 42 - 44), their breadth being greatest in the alloy containing 10% zinc.

The broader markings were observed to etch light coloured against a dark etching matrix and frequently in other grains the contrast was reversed.
Fig. 46. Deformation indications in 70/30 brass compressed 19.8% at room temperature, (20°C). Compression produced by a single blow. Electrolytic thiosulphate etch, X1000. (33).
In 70/30 brass, markings were mainly of uniform width, and were substantially straight (fig. 42), they became laminate and composed partly of matrix material as the zinc content of the alloy decreased. They also then tended to taper toward their termination which might be at the grain boundary or at their contact with similar markings in another system. They were often curved and their outline defined some other surface than the (111), figs. 43 and 44.

Alloys containing less than 10% of zinc showed these broad markings only after extensive deformation, they were then fewer in number and narrower and unlike those in the higher brasses quite commonly terminated in the central portion of the grain.

"Slip-band indications" were not observed at any deformation in copper deformed by repeated hammer blows at room temperature but they have been observed (Hatherly 92) in tough pitch copper after deformation by 29% at one blow at -183°C, (fig. 45).

In 70/30 brass broad markings were produced (92) by deformation at elevated temperature, (500°C); they have also been seen, (33) after 19.8 percent deformation at 20°C by one blow, (fig. 46). The significance of the observations just recorded, is considered in a later section.

Particular reference has been made to the works of Samuels and Hatherly because of their thoroughness and because they are quite recent publications.

Others over the years have done similar work, but Samuels (87) has shown that this literature is confused by the lack agreement about the identity of the markings described and by lack of detailed knowledge
of the capacity of etchants to reveal the various features.

7.2.2 The Nature of the Etch-markings found in Copper and Copper-based Alloys:

Markings similar to those discussed, have been examined in 70/30 brass by Vogel, (19, 21), who thought they were mechanical twins because of their similarity to Neumann bands. Adcock (97) observed them in the alloy, copper 80 nickel 20 weight percent, heavily reduced by rolling. Mathewson, (69), thought they were twins because they were more readily developed by impact, than by slow strain; Samans (93) by x-ray diffraction found evidence of twinning, his etch markings were similar to those in fig. 46.

The idea that twinning might be the cause, has been supported and denied. In particular Brick (95), Barrett and Steadman (96) and Burke and Barrett (94) examined cold worked 70/30 brass by x-ray diffraction and other techniques without detecting any trace of twinning. It is observed that where they are shown, their markings are not similar to those of Samans, (fig. 46) but rather like those of fig. 39.

However, work done in recent years (48, 50, 58, 62) and reviewed in Section 6.5 appears to give new support to the possibility of twinning in face-centred cubic metals and alloys.

It is therefore possible that the markings described above and shown to be associated with twinning are indeed due to fine twins and stacking faults.
Consideration of the markings discussed above is aided by the use of Samuels' (87) classification of them.

Slip line indications in copper, compressed 2.5% at room temperature, by sensitive etching are revealed as rows of rounded etch pits or clusters of etch pits, never quite losing their identity even when assembled into a (discontinuous) line.

These points are observed in fig. 38, which also shows that the pits are in some instances assembled on very many adjacent planes, (on a slip band). However, Samuels (87) noted that at higher deformations, the individual pits in copper become indistinguishable.

It seems reasonable to assume that the individual etch pits are associated with emergent dislocations.

At 0.03% compression, 70/30 brass (83) shows a few fine "slip line" indications near a twin boundary and these are plainly seen to be rows of etch pits closely "piled-up" near the twin boundary but more widely spaced at a distance from it. Similar observations have been made by Jacquet (82).

Although etch pits in brass are apparently associated with emergent dislocations, the preferential attack related to the slip markings may also be due to another cause. By sensitive etching, 70/30 brass compressed 2.5% reveals fields such as fig. 39. If this field is considered in association with the observation, (86), that in deformed 70/30 brass, dissolution "produces corrosion slots", (Section 7.1.4), it seems entirely possible that the mechanism associated with the attack of slipped regions is often different in brass from that in copper.
Figs. 40 and 41 show 70/30 brass compressed 10%; in these, slip band indications appear. Samuels (87) observed that slip band indications can be developed by light etching, "as a series of fine parallel lines".

Slip band indications observed by the optical microscope appear to be of constant width throughout their length and wider than slip line indications.

In the view being developed here, the transition from slip line indications to slip band indications occurs as a result of an increase in width of the faulted region associated with slip.

The etch markings just discussed appear to undergo an important change when the composition of the specimens is changed, or the temperature of deformation, or the rate of loading is altered.

The effect of change of composition is illustrated in figs. 42 - 44, for specimens deformed at room temperature.

What are called slip band indications in 70/30 brass become or are superseded by laminated clusters which may be tapered or curved in outline.

Each lamination in a cluster has certain of the properties of a twin, for example, it tapers to a point at its termination and may also appear in different colour contrast in grains of different orientation.

Clusters of this kind are not seen in 70/30 brass deformed at room temperature by hammering, (87), but apparently appear, (33) under conditions of greater impact. They did not appear in copper deformed by a single blow at temperatures above -76°C but readily appeared in copper similarly deformed at -183°C, (92). They were not found in 90/10 brass deformed above 200°C, but were found in 70/30 brass deformed at 500°C, (92).
When considered with those, (50, 58, 62) reviewed in Section 6 and with that (86), reviewed in Section 7.1.4, these observations suggest the following provisional explanation of the markings:

**Type I, Slip-line Indications:** Etch patterns that at lowest strains are rows of etch pits, perhaps extending across the whole width of the grain.

The same features at higher strains may become continuous or discontinuous lines or bands. The second phase of development occurs at a lower strain in high brass, (stacking fault energy low), than in copper.

These features lie in the trace of the slip planes in the deformed metal or alloy.

**Type II, Slip-band Indications:** Etch patterns that apparently are an extension of type I. They appear readily in high brass, (stacking fault energy low) on a broader band of planes than do type I.

They never appear in copper, although banded rows of etch pits as wide as slip band indications do, (fig. 36). These have been called slip line indications.

The etching of stacking faults in high brass has been suggested as being sufficient to explain the differences seen between the markings in brass and those in copper.

Since even slip line indications are defined, (87), in terms of observations made with the optical microscope, they may involve activity on very many adjacent planes; it is therefore probably a matter of convenience to distinguish between type I and II.
Fig. 47. Deformation bands in 70/30 brass compressed 11.8% at 20°C. Banded appearance traverses grain boundaries, X100. (33). Ferric chloride etch, polarised light.

Fig. 48. Deformation bands in 70/30 brass, compressed 11.8% at 20°C. Bands intersect narrow twins; etch contrast reversed at passage through twins; twins heavily bent. Ferric chloride etch X100. Polarised light. (33).

Fig. 49. Deformation bands in 70/30 brass, intersected by "slip band indications"; bending of indications reveals kinking in band boundaries, X500. (33). Ferric chloride etch, polarised light.
Type III, Twin-indications: Etch markings that appear under conditions thought to favour the production of twins, (50, 58, 62 and 66 - 68, Sections 6.5.1, 6.5.2 and 6.6).

It is probable (Venables 58) that the twin laminations in a cluster are heavily faulted and contain considerable material in the matrix orientation; there is obviously a lot of matrix material between the laminations.

These terms will be used where relevant in discussion of results of the experimental work.

7.3 Deformation-bands:

Deformation bands have been observed in a wide variety of metals and alloys, (2).

Hatherly and Samuels (33) in 70/30 brass deformed by a single blow, have shown them as differentially etched band-like regions. As deformation increased "they became more band-like, (fig. 47) and the differentiation between the band and the parent grain became sharper".

Where they intersected narrow annealing twins at an angle near 90°, the twins were often bent and there was a reversal of contrast between band and twin, (fig. 48).

Frequently, "bands in one grain appeared to induce banding in the adjoining grain", fig. 47. Usually only one system of primary bands appeared in a grain but these might contain secondary bands, (fig. 48).

By their curvature, "slip band indications", where they passed through
the boundary of a band, indicated a curvature of the lattice which, "in most cases was gentle", (fig. 49).

"Slip band indications" might delineate a band in this way, or by their existence in parallel array throughout its whole length.

The authors considered that these were 'kink bands' and that 'bands of secondary slip' (34) were unlikely in poly-crystalline specimens.

They found that the development of bands was not greatly affected by the temperature of deformation but the bands became narrower as the deformation temperature rose.
8 Experimental

8.1 Specimen materials

8.1.1 Nickel:

All nickel specimens were prepared from nickel rod 0.5" in diameter with the following analysis*:

- Carbon: 0.07 weight percent
- Silicon: 0.03 weight percent
- Copper: 0.02 weight percent
- Iron: 0.09 weight percent
- Manganese: 0.20 weight percent
- Magnesium: 0.10 weight percent
- Chromium: 0.02 weight percent
- Titanium: not detected

As supplied, the material had a grain size of about 0.15 mm diameter and contained fine inclusions believed to be graphite. The rod was cold drawn to 0.375" diameter. Specimens 0.375" long were cut from the cold drawn rod under running water, using an abrasive cut-off wheel.

Before use the specimens were annealed in vacuum (< 0.01 mm Hg) for 30 minutes at 740°C to give a uniform grain size of 0.04 mm.

* This nickel was kindly donated and the analysis supplied by the International Nickel Company Pty. Ltd.
8.1.2 **The Copper Nickel Alloys:**

A specimen of nickel of the kind described in Section 8.1.1 and a specimen of copper (purity < 99.95%, O.F. H.C. copper BS 1861: 1952) were used in this set.

The copper was received as 0.5\" diameter rod, and was cold drawn to 0.375\" diameter before use; samples 0.375\" long were used.

Copper - Nickel alloys were made by melting together under vacuum, copper of the above specification and nickel of the following typical composition (109):

<table>
<thead>
<tr>
<th>Element</th>
<th>Weight Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nickel (plus Cobalt)</td>
<td>99.4</td>
</tr>
<tr>
<td>Carbon</td>
<td>0.10</td>
</tr>
<tr>
<td>Silicon</td>
<td>0.05</td>
</tr>
<tr>
<td>Sulphur</td>
<td>0.005</td>
</tr>
<tr>
<td>Copper</td>
<td>0.10</td>
</tr>
<tr>
<td>Iron</td>
<td>0.15</td>
</tr>
<tr>
<td>Manganese</td>
<td>0.20</td>
</tr>
</tbody>
</table>

Accurately weighed amounts of the metals were melted to produce an ingot approximately 1\" in diameter and of the nominal compositions shown below. The ingot was annealed at 950°C until homogenised, then cold forged to a section 0.5\" square. Samples 0.375\" long were removed.

All specimens used were annealed for 30 minutes at an appropriate temperature between 750°C and 1050°C, capable of producing a uniform grain size of 0.15 mm.

* Kindly donated by Metal Manufactures Ltd., Port Kembla.*
The compositions of specimens in this set were:

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Copper</th>
<th>Nickel</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>99.95%</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>95%</td>
<td>5%</td>
</tr>
<tr>
<td>3</td>
<td>90%</td>
<td>10%</td>
</tr>
<tr>
<td>4</td>
<td>85%</td>
<td>15%</td>
</tr>
<tr>
<td>5</td>
<td>75%</td>
<td>25%</td>
</tr>
<tr>
<td>6</td>
<td>60%</td>
<td>40%</td>
</tr>
<tr>
<td>7</td>
<td>20%</td>
<td>80%</td>
</tr>
<tr>
<td>8</td>
<td>Nickel</td>
<td>99.4%</td>
</tr>
</tbody>
</table>

NB All annealing was carried out in a vacuum of <0.01 mm Hg.

8.2 The Deformation of the Specimens

8.2.1 Nickel:

A series of specimens was prepared in order to study the surface features produced by deformation at room temperature.

Specimens of nickel were therefore sectioned on a diametral plane, then polished on this surface. They were then deformed by a large number of hammer blows alternately at each end.

It was found possible by this procedure, to compress a specimen with little or no "barrelling" and to keep the top and bottom surfaces plane and parallel to within ±0.002".

Care was taken in manipulation, to ensure that the polished surface of the specimen received no casual damage during handling.

* Specimens were sectioned by abrasive wheel under flowing water.
The deformations produced in this set of specimens were as follows:

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Compression</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0%</td>
</tr>
<tr>
<td>2</td>
<td>0.5%</td>
</tr>
<tr>
<td>3</td>
<td>1.5%</td>
</tr>
<tr>
<td>4</td>
<td>2.5%</td>
</tr>
<tr>
<td>5</td>
<td>3.4%</td>
</tr>
<tr>
<td>6</td>
<td>5.2%</td>
</tr>
<tr>
<td>7</td>
<td>7.1%</td>
</tr>
<tr>
<td>8</td>
<td>10%</td>
</tr>
<tr>
<td>9</td>
<td>15%</td>
</tr>
<tr>
<td>10</td>
<td>20%</td>
</tr>
<tr>
<td>11</td>
<td>30%</td>
</tr>
<tr>
<td>12</td>
<td>41%</td>
</tr>
</tbody>
</table>

*Reported values of compression are accurate to 0.5%.

The lower limit of deformation was set by the difficulty of accurately producing less deformation by a number of hammer blows; the upper, by the observation that due to severe surface rumpling much of the field of view was already outside the depth of focus at 40.7% compression.

The effects of deformation revealed by etching, were examined in another set of specimens. After deformation these were sectioned on a diametral plane, metallographically polished and etched.

As before, this set of specimens were deformed at room temperature by a large number of hammer blows at each end.

* See footnote page 77.*
* Temperatures quoted are the furnace temperatures measured by a thermocouple junction in contact with the specimen. No attempt was made to record temperature during deformation, in order to permit rapid deformation.
The following deformations were produced:

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Compression</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0%</td>
</tr>
<tr>
<td>2</td>
<td>1.8%</td>
</tr>
<tr>
<td>3</td>
<td>3.3%</td>
</tr>
<tr>
<td>4</td>
<td>7.0%</td>
</tr>
<tr>
<td>5</td>
<td>10.6%</td>
</tr>
<tr>
<td>6</td>
<td>16.9%</td>
</tr>
<tr>
<td>7</td>
<td>20.4%</td>
</tr>
<tr>
<td>8</td>
<td>31.7%</td>
</tr>
<tr>
<td>9</td>
<td>40.2%</td>
</tr>
</tbody>
</table>

A further set of nickel specimens was deformed at elevated temperatures. For this series a large number of hammer blows were rapidly delivered at one end only; the specimens were then quickly quenched in cold water.

This procedure was adopted throughout, because some of the specimens were deformed at temperatures exceeding the recrystallisation temperature of nickel. Therefore rapid deformation and quenching were essential to retain as far as possible, the structure produced by the deformation.

There was no evidence of recrystallisation in any specimen deformed at elevated temperature.

The temperatures of deformation, and the deformations produced in the specimens are recorded below:

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Compressed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>29.3% at 980°C</td>
</tr>
<tr>
<td>2</td>
<td>27.9% at 980°C</td>
</tr>
<tr>
<td>Specimen</td>
<td>Compressed</td>
</tr>
<tr>
<td>----------</td>
<td>------------</td>
</tr>
<tr>
<td>3</td>
<td>17.9%</td>
</tr>
<tr>
<td>4</td>
<td>9.7%</td>
</tr>
<tr>
<td>5</td>
<td>5.5%</td>
</tr>
<tr>
<td>6</td>
<td>2.86%</td>
</tr>
<tr>
<td>7</td>
<td>25.5%</td>
</tr>
<tr>
<td>8</td>
<td>17.8%</td>
</tr>
<tr>
<td>9</td>
<td>13.3%</td>
</tr>
<tr>
<td>10</td>
<td>9.80%</td>
</tr>
<tr>
<td>11</td>
<td>4.34%</td>
</tr>
<tr>
<td>12</td>
<td>20.9%</td>
</tr>
<tr>
<td>13</td>
<td>15.8%</td>
</tr>
<tr>
<td>14</td>
<td>10.8%</td>
</tr>
<tr>
<td>15</td>
<td>6.10%</td>
</tr>
<tr>
<td>16</td>
<td>18.4%</td>
</tr>
<tr>
<td>17</td>
<td>10.8%</td>
</tr>
<tr>
<td>18</td>
<td>5.43%</td>
</tr>
<tr>
<td>19</td>
<td>2.52%</td>
</tr>
</tbody>
</table>

A further set of specimens was specially prepared to examine polygonisation in lightly deformed nickel.

Three specimens of nickel were taken and all were annealed 1 hour at 950°C.

Specimens 1 and 2 were compressed at room temperature 1.5% by light hammer blows, specimen 3 was left undeformed. Specimens 1 and 3 were annealed at 950°C for 15 minutes.
All three specimens were then etched for metallographic examination using an etchant due to Guard (100).

8.2.2 Copper - Nickel specimens:

With this series it was considered that the results would be more informative if detailed comparison with earlier work on copper, (92), could be made. This required that all specimens should be deformed as accurately as possible, to a final compression of 30%, at the temperature of liquid oxygen.

A simple drop hammer was constructed and calibrated by a preliminary series of experiments. A further set of experiments established that the temperature of deformation was not more than 7°C above the temperature of liquid oxygen.

The following are the actual compressions produced:

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Compression</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>29.4%</td>
</tr>
<tr>
<td>2</td>
<td>28.8%</td>
</tr>
<tr>
<td>3</td>
<td>30.9%</td>
</tr>
<tr>
<td>4</td>
<td>26.4%</td>
</tr>
<tr>
<td>5</td>
<td>30.3%</td>
</tr>
<tr>
<td>6</td>
<td>30.5%</td>
</tr>
<tr>
<td>7</td>
<td>27.2%</td>
</tr>
<tr>
<td>8</td>
<td>28.0%</td>
</tr>
</tbody>
</table>

The deformed specimens were* sectioned in a plane parallel to one of

* See footnote page 77.
the side faces and passing through the specimen axis.

Etching of the metallographically polished specimens was carried out with acid ferric chloride solution of a composition found satisfactory for copper deformed in this way, (92).

8.3 Metallographic preparation; Grinding and Polishing:

It is known, (13), that surface deformation in a specimen, may alter the habit of surface slip during subsequent deformation. Experimental evidence also exists, (101, 102), that due to the accompanying deformation, mechanical polishing may produce in the polished surface, on etching, features bearing no relation to the condition of the bulk of the specimen.

A procedure, (103) was therefore used, that together with electrolytic or chemical polishing would produce surfaces free from polishing deformation. The operations involved were, (103):

1) Grinding on wet or dry paper of successively finer grit. The final paper used was 600 #; wet-grinding was used at all stages. The 600 # paper was followed by a 600 # wax lap.

Initial polishing was performed successively on a pad carrying an emulsified dispersion of 4-8 μ diamond powder then on another carrying a similar dispersion of 0-1,μ diamond powder.

Each step in this procedure was performed as prescribed, (103).

Final polishing of nickel was achieved electrolytically by use of the following electrolyte, due to Wensch, (107):

\[
\text{Sulphuric acid (conc)} \quad 573 \text{ ml.}
\]
This electrolyte is a low resistance type and operates at approximately 1.3 volts. A specimen develops a satisfactory surface after an electropolish of 5 minutes duration.

The final polishing of copper was performed electrolytically by use of an electrolyte, (due to Jacquet, 1945), containing 900 g of orthophosphoric acid per litre of aqueous solution. It was also found to operate effectively at 1.3 volts, but required an elapsed time of 20 minutes to produce an effective polish.

Final polishing of the copper - nickel alloys was best performed by immersion in a chemical polishing solution of the following composition:

- Acetic Acid (conc.) 26 ml.
- Nitric Acid (conc.) 14 ml.
- Hydrochloric Acid (conc.) 2 drops

derived from a chemical polish for nickel, due to Fox and recorded by Jacquet, (108).

The total thickness of metal removed during preparation for etching was approximately 60 μ.

8.4 Etching

8.4.1 Preliminary Survey of Nickel Etchants:

A review of literature cited in a bibliography, (104), on the metallography of nickel and nickel based alloys, led to the selection of a number of etchants for trial.
Of those tested, among the most promising was one used in the etching of copper and copper based alloys.

A basic composition of this etchant is:

Potassium Dichromate 5 g  Concentration 0.284 - 0.156 molar.
Sulphuric Acid (conc.) 8 ml.  Concentration 2.4 - 1.33 molar.
Water 50 ml.  Concentration -
Hydrochloric Acid (conc.) 2 - 50 ml.  Concentration 0.37 - 5.5 molar.

Another such etchant was the acid ferric chloride solution used for the same purposes, (106):

Ferric Chloride 10 g  Concentration 0.41 molar.
Hydrochloric Acid (conc.) 30 ml.  Concentration 2.2 molar.
Water to make 120 ml.  Concentration -

The aqueous solution of ferric chloride, also had interesting properties, (73).

8.4.2 The Dichromate Etchant:

After initial trial on deformed specimens the dichromate etchant was used to etch a set of specimens deformed at room temperature by amounts ranging from 0 - 40.2%.

The etchant composition used was:

Potassium Dichromate 4.4 g  Concentration 0.30 molar
Sulphuric Acid (conc.) 6.7 ml. Concentration 2.4 molar
Hydrochloric Acid (conc.) 0.3 ml. Concentration 0.066 molar
Water to make 50 ml. Concentration —

It was found that this etchant produced considerable relief before significant detail was developed. Etching times varied from 30 - 120 second, the time being shorter for the specimen with the greater deformation.

In view of the excessive relief a systematic study of the effect of etchant composition was undertaken using the specimen deformed 40.2%.

The criteria adopted to assess the value of a particular composition were:

1) that an etchant should produce a minimum of relief consistent with a proper display of features.

2) that it should be as selective as possible in its action so that a sharp alteration of attack would result from a change in the condition of deformation in the specimen surface.

It was recognised that the etchant was an oxidising agent, (70) and that the effectiveness would be influenced by the concentration of the dichromate and the hydrogen ion. The chloride ion might also influence behaviour, (105).

The etchant was therefore studied to determine:

a) the effect of altering the chloride ion concentration,
b) the effect of altering the hydrogen ion concentration,
c) the effect of altering the dichromate ion concentration.
Etchants listed in Table III were used in the first part of this study:

<table>
<thead>
<tr>
<th>Etchant number</th>
<th>K₂CrO₇ (molar conc)</th>
<th>H₂SO₄ (molar conc)</th>
<th>HCl (molar conc)</th>
<th>H⁺ (normal conc)</th>
<th>Cl⁻ (molar conc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.298</td>
<td>2.42</td>
<td>0.067</td>
<td>4.90</td>
<td>0.067</td>
</tr>
<tr>
<td>2</td>
<td>0.294</td>
<td>2.36</td>
<td>0.216</td>
<td>4.95</td>
<td>0.216</td>
</tr>
<tr>
<td>3</td>
<td>0.288</td>
<td>2.32</td>
<td>0.423</td>
<td>5.06</td>
<td>0.423</td>
</tr>
<tr>
<td>4</td>
<td>0.278</td>
<td>2.24</td>
<td>0.815</td>
<td>5.05</td>
<td>0.815</td>
</tr>
<tr>
<td>5</td>
<td>0.250</td>
<td>2.02</td>
<td>1.83</td>
<td>5.96</td>
<td>1.83</td>
</tr>
</tbody>
</table>

Etchant number 1, is that referred to just above.

Observation showed that a specimen etched with a specular appearance and 'rounding' of detail, when the chloride ion concentration was low. An increasing chloride ion concentration ultimately caused and extended the deposition of a stain which was initially very selectively deposited.

However, continued increase of the chloride ion eventually reversed this process and caused a specular etch, poor in detail.

The best revelation of fine detail was apparently associated with the initial appearance of staining. Accordingly in subsequent compositions, the chloride ion concentration was kept at about 0.7 molar.

The following etchant compositions were used to study changing acidity. Sodium chloride was used instead of hydrochloric acid, as the source of...
the chloride ion. Acidity could therefore be altered independently of the chloride ion concentration.

Table IV

The Variation of Acidity of the Etchant

(Concentrations expressed as shown)

<table>
<thead>
<tr>
<th>Etchant</th>
<th>K₂Cr₂O₇ (molar conc)</th>
<th>H₂SO₄ (molar conc)</th>
<th>NaCl (molar conc)</th>
<th>H⁺ (normal conc)</th>
<th>Cl⁻ (molar conc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>6</td>
<td>0.287</td>
<td>1.02</td>
<td>0.650</td>
<td>2.05</td>
<td>0.650</td>
</tr>
<tr>
<td>7</td>
<td>0.280</td>
<td>1.43</td>
<td>0.635</td>
<td>2.86</td>
<td>0.635</td>
</tr>
<tr>
<td>8</td>
<td>0.261</td>
<td>2.47</td>
<td>0.610</td>
<td>4.95</td>
<td>0.610</td>
</tr>
<tr>
<td>9</td>
<td>0.249</td>
<td>2.36</td>
<td>0.823</td>
<td>4.72</td>
<td>0.823</td>
</tr>
</tbody>
</table>

It appeared from the use of the above etchants that optimum etchant was about 2.5 molar in sulphuric acid and about 0.6 molar in sodium chloride.

The effect of varying dichromate concentration was studied in solutions of constant acid and chloride content.

<table>
<thead>
<tr>
<th>Etchant</th>
<th>K₂Cr₂O₇ (molar conc)</th>
<th>H₂SO₄ (molar conc)</th>
<th>NaCl (molar conc)</th>
<th>H⁺ (normal conc)</th>
<th>Cl⁻ (molar conc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>0.262</td>
<td>2.47</td>
<td>0.615</td>
<td>4.95</td>
<td>0.615</td>
</tr>
<tr>
<td>11</td>
<td>0.185</td>
<td>2.47</td>
<td>0.615</td>
<td>4.95</td>
<td>0.615</td>
</tr>
<tr>
<td>12</td>
<td>0.132</td>
<td>2.47</td>
<td>0.615</td>
<td>4.95</td>
<td>0.615</td>
</tr>
<tr>
<td>13</td>
<td>0.0925</td>
<td>2.47</td>
<td>0.615</td>
<td>4.95</td>
<td>0.615</td>
</tr>
<tr>
<td>14</td>
<td>0.0462</td>
<td>2.47</td>
<td>0.615</td>
<td>4.95</td>
<td>0.615</td>
</tr>
</tbody>
</table>
It was found by use of these etchants, that initially a decrease in dichromate content made little difference to etching characteristics. However, compositions from 13 onward showed continuing loss of detail, the fine detail associated with slip dissappearing first and deformation bands later.

Etchant 16 did little more than attack the grain boundaries.

The final composition chosen is shown below:

- Potassium Dichromate 0.26 molar concentration
- Sodium Chloride 0.60 molar concentration
- Sulphuric Acid 2.5 molar concentration

8.4.3 The Ferric Chloride Etchants:

The use of dilute aqueous Ferric Chloride has been recommended by Corson (73), as a nickel etchant. It has been used here on specimens to be viewed by polarized light.

However the composition below, commonly used with copper, revealed good detail and was generally preferred. There were, however, certain concentrations which caused staining.

The composition, (106), of this etchant is shown below:

Ferric Chloride 10 g concentration 0.41 molar
Hydrochloric Acid
(conc) 30 ml. Concentration 2.20 molar
Water to make 120 ml. Concentration -

The composition was systematically varied to determine the best composition; The specimen was that used in the previous section. The compositions used appear in Table VI.

| Table VI |
|----------------------|----------------------|
| The Variation of Composition of Acid–Ferric Chloride |
| Etchant | Ferric Chloride (molar conc) | Hydrochloric Acid (molar conc) |
| 17 | 1.23 | 2.20 |
| 18 | 0.82 | 2.20 |
| 19 | 0.41 | 2.20 |
| 20 | 1.23 | 1.10 |
| 21 | 1.23 | 0.55 |
| 22 | 1.23 | 0 |

The systematic reduction of ferric chloride in the continued presence of a high acid content caused a progressive loss of detail in the etched surface.

As in the case of potassium dichromate, fine detail is first affected, while coarse detail such as deformation bands is last affected; at lowest ferric chloride content, the grain boundary etch still remains.

The systematic reduction of acid content, in the continued presence of high ferric chloride content, caused a reduction in fine detail, but
ultimately with the onset of selective staining fine detail again increased.

However, staining may increase to the point at which it is objectionable.

A critical assessment of the results of etching, led to the selection of etchant 20 as the best in this group.

Detail was revealed by a selective relief attack, yet no significant staining occurred.

A comparison of the results obtained with both dichromate and ferric chloride etchants, showed no particular advantage for either using normal illumination. However the aqueous ferric-chloride etchant used by Corson, (73), produced a surface in which such features as deformation bands were very sensitively revealed, due to the improved contrast produced by using polarised light.

In view of this the ferric chloride etchant has been used throughout the final part of the work.

8.5 Method of Counting Twin-indications in the Copper - Nickel Alloys

After metallographic preparation, the specimens were traversed at a magnification of 500x, across their length and the number of grains intercepted and the number of grains showing twin-indications, counted.

The specimen was traversed at $\frac{1}{4}$, $\frac{1}{3}$, $\frac{2}{3}$ and $\frac{3}{4}$ of the width and approximately 115 grains were counted for each traverse, so that approximately 450 grains were counted for each specimen.*

An indication of the reliability of the results may be obtained by

* For most specimens duplicate counts were made.
comparing for the different traverses of a specimen the proportion of grains containing twin indications.

For alloys of high copper content, the proportion of grains showing indications was high and the variation between traverses was never greater than 19% of the mean and was generally less than half of this.

However for the alloy of low copper content, where only few indications occurred, it is not particularly meaningful to make this comparison.
PLATE I

Nickel X 500

Fig. 1. Unetched
Compression 0%

Fig. 2. Unetched
Compression 1.52%

Fig. 3. Unetched
Compression 2.52%

Fig. 4. Unetched
Compression 2.52%
Results:

The results of the experimental work will be considered in three sections, those for nickel deformed at room temperature, those for nickel deformed at elevated temperatures and those for the copper-nickel alloy system.


9.1 Introduction:

To facilitate comparison, photomicrographs of the deformed polished specimens, for various deformations, are presented together with the corresponding photomicrographs of the etched specimens.

In all cases the magnification is 500 X, and the axis of compression of the original specimen is parallel to the narrow edge of the photograph.

9.2 Features Observed in Deformed Polished Specimens.

9.2.1 Slip:

Fig. 1 plate I is a field in the undeformed nickel; it contains a number of annealing twins but otherwise no feature worthy of comment at this point.

A specimen compressed 0.51% contained no observable evidence of deformation. However a deformation of 1.52% caused a few grains to display fine multiple slip.
The fine slip bands were diffuse in appearance, uniformly spaced and in a particular system, of uniform intensity; they were straight and apparently parallel, with a spacing of approximately $1.5 \mu$. They appeared to run without apparent obstruction across the full width of a grain. Fig. 2 illustrates these observations.

In fig. 2 the beginnings of a characteristic feature appear. A fine scale surface rumpling can be seen in a small triangular grain near the bottom right hand corner of the field. The rumpling becomes more prominent as deformation increases, as will appear in following fields. The rumples are parallel or approximately parallel to slip bands and it is thought that they are a consequence of the slip process.

At 2.52% compression slip bands were more plainly displayed; they were still uniformly spaced, but superimposed on the system of fine clusters was the fine scale rumpling referred to above.

The rumpling is seen in the large triangular shaped grain of fig. 3; the characteristic spacing is about $3.0 \mu$. Rumpling varies in intensity from one part of the grain to another and is generally less well developed near the grain boundaries.

Fig. 4 shows a field photographed slightly out of focus to facilitate the viewing of this feature. The slip bands are not focussed in this field yet the rumpling is resolved in fair detail. It is therefore concluded that this is indeed a coarser feature, not only in spacing but also in depth.

In fig. 4 they are seen to be of approximately uniform spacing as before, but sometimes tapering to a point and disappearing or running
Fig. 5. Unetched
Compression 3.36%

Fig. 6. Unetched
Compression 3.36%

Fig. 7. Dichromate Etch.
Compression 3.30%
Fig. 8. Unetched
Compression 5.17%

Fig. 9. Unetched
Compression 7.08%

Fig. 10. Dichromate Etch.
Compression 7.40%
together and uniting. It is also observed that in one portion of a grain they may be essentially parallel to one another but not parallel to similar features elsewhere in the same grain.

The first extended surface curvature appeared at 3.36% compression. It is seen in fig. 5 plate II, in the upper portion of the large central grain and in the adjoining twinned region. Further evidence of change in orientation is seen in the inhomogeneity of slip behaviour in the large grain. In addition, inhomogeneous deformation is demonstrated by the appearance of curved slip bands in fig. 6, where they run parallel to the twin boundary in the large central grain.

Additional evidence of extensive surface rumpling is the variation in brightness of the field in the immediate vicinity.

In fig. 8, plate III, 5.17% compression, further examples of the features discussed above can clearly be seen in the large grains near the right and left hand sides of the field.

Similar features are also seen at 7.08% compression. Fig. 9 plate III is of particular interest because in it, is fine scale rumpling running parallel to the twin boundary in a large central grain. This particular feature will be referred to again, by way of comparison with a feature in an etched specimen.

If the graphite inclusions in fig. 9 are considered, it is apparent that bands intersect them and pass on apparently unaffected by their presence. The form of the bands is apparently unaffected even in the immediate vicinity of the inclusions.

Similar observations can be made in other fields and at other
**Fig. 11.** Unetched
Compression 10.1%

**Fig. 12.** Unetched
Compression 10.1%

**Fig. 13.** Dichromate Etch.
Compression 10.6%
Fig. 14. Unetched  
Compression 15.3%  

Fig. 15. Unetched  
Compression 15.3%  

Fig. 16. Unetched  
Compression 15.3%  

Fig. 17. Dichromate Etch.  
Compression 16.9%
Fig. 18. Unetched  
Compression 20.0% 

Fig. 19. Unetched  
Compression 20.0% 

Fig. 20. Dichromate Etch.  
Compression 20.4%
Fig. 21. Unetched
Compression 30.5%

Fig. 22. Dichromate Etch.
Compression 31.7%
Fig. 23. Unetched
Compression 40.7%

Fig. 24. Unetched
Compression 40.7%

Fig. 25. Dichromate Etch.
Compression 40.2%

Fig. 28. Aqueous Ferric Chloride Etch
Compression 39.6%
compressions, hence it can be concluded that the graphite inclusions had no significant effect on the observed features of deformation.

At 10.1% there is very general surface rumpling; this is apparent in figs. 11 and 12 plate IV.

Features associated with slip, continue to develop in figs. 14 - 16 plate V, 18 and 19 plate VI, 21 plate VII, and 23 and 24 plate VIII.

In figs. 18 plate VI and 24 plate VIII the intersection of two systems of fine scale rumpling produces fine mounds in the surface; these are revealed in the oblique illumination. The appearance of these mounds again supports the idea that where these features intersect the surface, they cause a fine scale rumpling in the surface.

9.2.2 Deformation bands:

At the higher deformations, markings with the characteristic appearance of deformation bands were frequently present, plates V - VIII.

Apart from such features with relatively well defined boundaries, the surface became progressively more rumpled on a coarse scale, (plates IV - VIII), until finally the surface became too complicated for detailed interpretation.

A feature with the characteristics of a deformation band is seen in the large central grain of fig. 19 plate VI. In this grain the slip passes through a region in which it is disoriented with that outside the region. At the boundaries of the region slip bands are curved. These are the properties of a deformation band.
Fig. 26. Polygonised and Etched (100)
Compression 1.5%

Specimen annealed 1 hour at 950°C, compressed 1.5% by light hammer blows, then annealed for 15 minutes at 950°C.
Etchant: Aqueous solution of nitric and acetic acids (40).

Fig. 27. Polygonised and Etched (100)
Compression 1.5%
Fig. 23 plate VIII contains a large central grain in which the slip curves sharply in three places in the grain; this again is characteristic of deformation bands.

9.3 Features Seen in Etched Surfaces of Section of Deformed Specimens:

The metallographically prepared and polished set of nine specimens listed in Section 8.2.1 was etched for a suitable time in etchant 1 table III, Section 8.4.2.

The undeformed specimen and that compressed 1.8%, were indistinguishable one from the other at a magnification of 500 X in oblique bright-field illumination. However it is of interest to note that inhomogeneous deformation is quite well developed at these low strains.

This can be seen from the pattern of disoriented regions produced by polygonisation in a specimen deformed 1.5% at room temperature by repeated hammer blows.

In fig. 26 plate IX a large central grain is divided into roughly parallel sections by the pattern of polygonisation. This pattern is consistent with significant variation in slip deformation between the approximately parallel sections. Disorientation on a finer scale is apparent in the bottom right hand corner. Fig. 27 contains more patterns of a similar kind but in most instances, apparently on a single system.

The specimen compressed 3.3%, showed the first features of interest. These are shown in fig. 7 plate II. In many of the grains, there are faint parallel markings with approximately the same spacing as that of
the fine scale rumpling first clearly observed in a polished specimen at 2.52% compression, see fig. 4 plate I and fig. 6 plate II. These etch features are seen in grains near the lower right hand corner of fig. 7. They are more plainly developed in a specimen deformed 7.4% (fig. 10 plate III) and should be compared in this field, with the fine scale rumpling, running parallel to the twin boundary fig. 9. The features revealed in fig. 10 are considered to be equivalent to fine scale rumpling.

The etched surface of a specimen deformed 10.6% is shown in fig. 13 plate IV. Features similar to those just discussed are plainly seen in several of the grains, but in addition it is now possible to observe relief attack on a coarser scale and in addition, the rumpling is not uniformly developed over the whole area of a grain.

These remarks apply to the field of fig. 17 plate V, 16.9% compression, and to fig. 20 plate VI, 20.4% compression.

It is increasingly possible to identify the etch features of figs. 7, 10, 13, 17 and 20 with the fine scale rumpling of the polished specimens. However, for specimens deformed 31.7% and 40.2% the identity of the markings revealed by etching, with those seen on the deformed surface is even more apparent.

In fig. 22 plate VII, 31.7% compression and fig. 25 plate VIII, 40.2% compression, it is possible to see typical features showing a marked similarity to those in the polished specimen, due to fine scale rumpling and extended surface rumpling.

Relief attack developed on an extended scale, observed in the specimen
deformed 10.6% and in subsequent specimens more heavily deformed, is consistent with the existence of corresponding variations in orientation and strain energy produced by the deformation and revealed in the surface of section.

Deformation Bands: Fig. 28 plate VIII shows a field in a specimen compressed 39.6% at room temperature, by repeated hammer blows.

The prepared surface of section was etched in aqueous solution of ferric chloride and photographed using polarised light.

Changes in orientation are sensitively revealed, (75, 76) by corresponding changes in contrast and the large grain at the bottom of the field, in this way reveals change of orientation on an extended scale. The grain above this, reveals deformation bands by the marked and abrupt changes of contrast it contains and by the diffuseness of the boundaries of the bands, (see fig. 47, Review Section).
Fig. 29. Acid Ferric Chloride

Compression 2.52% at 400°C.

Fig. 30. Acid Ferric Chloride

Compression 2.86% at 980°C.
PLATE XII Compressed Nickel X 500. Acid Ferric Chloride Etch.

Fig. 36. 10.8% at 400°C.

Fig. 37. 10.8% at 400°C.

Fig. 38. 10.8% at 600°C.

Fig. 39. 9.8% at 800°C.

Fig. 40. 9.7% at 980°C.

Fig. 41. 9.7% at 980°C.
Fig. 42.
Compression 15.8% at 600°C.

Fig. 43.
Compression 13.3% at 800°C.

Fig. 44.
Compression 17.9% at 980°C.
Fig. 45. Acid Ferric Chloride Etchant.  
Compression 18.4% at 400°C.  

Compression 20.9% at 600°C.

Fig. 47. Acid Ferric Chloride Etchant.  
Compression 25.5% at 800°C.  
P.T.O.

Fig. 48. Acid Ferric Chloride Etchant, (see next page)  
Compression 27.9% at 980°C.
Fig. 49. Aqueous Ferric Chloride Etchant (100)
Polarised light
Compression 27.9% at 980°C.

Figs. 49 and 50 are views of the same field.
Specimen is that for Fig. 48.

Fig. 50. Aqueous Ferric Chloride Etchant, (400)
N.I. Illumination
Compression 27.9% at 980°C.
10. **Nickel Deformed at Elevated Temperatures**

Figs. 29 - 50 plates X - XIV are representative of fields for specimens deformed at elevated temperatures. They show that the etched surface of section exhibits features related to the presence of slip and deformation bands.

At all deformations parallel markings associated with the presence of slip and similar to those described in the previous section were seen, (see figs. 36, and 38 - 40 plate XII).

At the lower temperatures (400°C and 600°C) these markings, although similar to those formed at room temperature, are more sensitively developed, that is a specimen compressed 2.52% at 400°C shows them about as well does a specimen deformed 20 to 30% at room temperature, (see figs. 29 plate X, 20 plate VI and 22 plate VII). In the specimen deformed at 400°C they also occupy a larger fraction of the field.

With increasing temperature of deformation the markings are increasingly well defined (figs. 36 and 38 - 40 plate XII).

Deformation bands are more common in specimens lightly deformed at 400°C than in those similarly deformed at 980°C, figs. 29 and 30 plate X.

As deformation is increased deformation bands become more numerous at all temperatures of deformation but at a similar deformation they are always less numerous in a specimen deformed at the highest temperatures.

Specimens deformed at 800°C and 980°C are characterised by a flatness of appearance in the etched surface as compared with those deformed at 400°C and this is even more apparent when a specimen deformed at 980°C is compared with one deformed at room temperature (see figs. 48 plate XIV
and 22 plate VII).

Deformation bands; Figs. 49 and 50 plate XIV contain deformation bands; they are reproductions of the same field, fig. 49 by polarised light and 50 by normal incident illumination for a specimen compressed 27.9 at 980°C.

In fig. 49 which sensitively reveals changes in orientation the identity of the bands is shown by the change of orientation of the material within them and by the diffuseness of their boundaries. The lightly etched surface of the specimen revealed in fig. 50 shows detail associated with fig. 49 and also shows the similarity of the deformation bands to those more heavily etched in fig. 47.
Acid Ferric Chloride Etch (92)
PLATE XVI  Copper 20, Nickel 80, deformed 27.2%; -183°C. X 500

Fig. 56.

Fig. 57.

Fig. 58.

Fig. 59.

Fig. 60.  Acid Ferric Chloride Etchant, (92).
PLATE XVII  Copper 20, Nickel 80, deformed 27.2%; -183°C. X 500

Fig. 61.

Fig. 62.

Acid Ferric Chloride Etchant, (92).
PLATE XVIII  60 Copper 40 Nickel deformed 30.5%; -183°C.  X 500

Fig. 63.

Fig. 64.

Acid Ferric Chloride Etchant, (92).
PLATE XIX  Copper 85 Nickel 15, deformed 26.4%; -183°C.  X 500

Fig. 65.

Fig. 66.

Acid Ferric Chloride Etch, (92).
PLATE XX    Copper 90 Nickel 10 deformed 30.9%: -183°C. X 500

Acid Ferric Chloride Etch, (92).
PLATE XXI  Copper 95, Nickel 5, deformed 28.8%; -183°C. X 500

Fig. 71.

Fig. 72.

Fig. 73.

Fig. 74.

Acid Ferric Chloride Etch, (92).
PLATE XXII  Copper deformed 29.4%; -183°C. X 500

Fig. 75.

Fig. 76.

Fig. 77.

Fig. 78.

Fig. 79.

Fig. 80.

Acid Ferric Chloride Etch, (92).
11. The Examination of Deformed Specimens in the Nickel Copper System.

11.1 Features Observed.

11.1.1 Slip:

The etched surface again displays parallel markings similar to those observed in etched specimens of nickel deformed at room temperature and at elevated temperatures, that is, markings thought to be associated with fine scale rumpling due to slip. These are seen in figs. 52 – 54 plate XV for pure nickel; their spacing varies from fine to very coarse in the context of previous work.

They are also seen in pure copper, in the light etching grains of figs. 77 and 78 plate XXII, but in copper they have never been observed to be so coarse as some of those in nickel.

Fig. 73 plate XXI for a specimen containing 5% of nickel also shows them in the light etching grains near the right hand side of the field.

They generally appear to be less well defined for alloys richer in nickel, but are nevertheless seen, as for example, in the bottom left of fig. 58 plate XVI for 80% nickel.

The nominal deformation of all specimens in this series is 30%, so that this feature is associated with extensive deformation.

11.1.2 Slip-line Indications:

Fig. 74 plate XXI contains etch pits in two parallel systems. It can be seen that these pits lie along fine parallel black lines and are
extended a little in length in the direction of these lines.

Similar lines, dark etching and sharply resolved, are exhibited in the field of fig. 57 plate XVI (80% nickel) but here the lines are more prominent and generally sharper than before. In this field as well as in others, for example figs. 58 - 60 plate XVI and fig. 62 plate XVII it is possible to see that the lines are associated with and parallel to the undulant parallel markings that have been related to slip and are discussed above.

The fine lines are more sharply resolved than the latter and more prominent.

The extension in length of the small dark etching pits lying along the fine lines just discussed, by their habit are suggestive of faulting in the slip plane. They appear to have the same character as the fine lines themselves, but to be an extension in width. They are seen elsewhere in fig. 63 plate XVIII (40% nickel), fig. 66 plate XIX (15% nickel) and fig. 70 plate XX (10% nickel).

Examination of the series of specimens suggests that the lines become less well defined with increasing copper content. However, with increasing copper content, well formed twin indications become increasingly prominent.

11.1.3 Twin Indications:

Twin indications are rarely seen in pure nickel under the experimental conditions used, but with increasing copper, become numerous and well developed.
Fig. 81. Fraction of grains showing "twinning indication" and stacking fault energy, as functions of composition in the Copper-Nickel System.

The two points lying below the average curve are plotted for completeness. However, since in both cases accidental variations in experimental procedure resulted in a total compression 10% less than the desired value of 30%, it is considered reasonable to neglect these points in drawing a curve showing the general trend.

As the nickel content is increased the fraction of grains showing twinning indications decreases and with it the precision of the fraction based in 400 grains also decreases. Limits are shown for each value.
### Table VII

**COPPER-NICKEL ALLOYS DEFORMED IN IMPACT COMPRESSION AT -183°C.**

**Fraction of Grains Containing ‘Twin-indications’**

*(See page 90)*

<table>
<thead>
<tr>
<th>No.</th>
<th>Nickel % (nominal)</th>
<th>Deformation</th>
<th>$\times$ Mean per Count</th>
<th>$\gamma$ Mean of Counts</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>29.4%</td>
<td>0.318</td>
<td>0.347</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.376</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>5</td>
<td>28.8%</td>
<td>0.285</td>
<td>0.289</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.293</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>10</td>
<td>30.9%</td>
<td>0.357</td>
<td>0.342</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.327</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>15</td>
<td>26.4%</td>
<td>0.278</td>
<td>0.268</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.259</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>25</td>
<td>30.3%</td>
<td>0.283</td>
<td>0.305</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.327</td>
<td>0.304</td>
</tr>
<tr>
<td>6</td>
<td>40</td>
<td>30.5%</td>
<td>0.278</td>
<td>0.234</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.189</td>
<td></td>
</tr>
</tbody>
</table>
### Table VII Cont.

**COPPER-NICKEL ALLOYS DEFORMED IN IMPACT COMPRESSION AT -183°C**

**Fraction of Grains Containing 'Twin-indications'**

(See page 90)

<table>
<thead>
<tr>
<th>No.</th>
<th>Nickel % (nominal)</th>
<th>Deformation</th>
<th>Mean per Count</th>
<th>Mean of Counts</th>
</tr>
</thead>
<tbody>
<tr>
<td>7</td>
<td>80</td>
<td>27.2%</td>
<td>0.195</td>
<td>0.122</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.064</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.127</td>
</tr>
<tr>
<td>8</td>
<td>100</td>
<td>28.0%</td>
<td>0.091</td>
<td>0.022</td>
</tr>
</tbody>
</table>

(averaged on 400 grains)

**X** Mean for each 450 grains.

**Y** Mean for total grains counted including duplicate counts where appropriate.
This is shown in fig. 81 in which the fraction of grains showing twin indications is plotted as a function of composition. Fig. 81 faces Table VII in which the same data are tabulated. It will be noted that whereas only 2.2% of grains showed twinning, (9 grains in 400) in 35% pure nickel, in copper about $\frac{2}{3}$ of grains showed twinning.

An example of twinning in pure nickel is shown in fig. 55 plate XVI.

The character of twin indications changes with increasing copper content; in low copper alloys the indications tend to occur as isolated examples within the grains (see fig. 56 plate XVI) whereas with higher copper contents they occur as clusters of fine twin indications, (see figs. 68 plate XX and 71 plate XXI).

It should be noted that in pure copper, (figs. 76 and 77 plate XXII) the twin indications etch darkly in light etching grains and vice versa.

In all clusters it is apparent that there is significant amount of material of the matrix orientation interleaved between the fine twins.

11.1.4 Deformation-bands:

In all specimens deformation bands were observed. There does not appear to be a characteristic variation of the habit of the bands with composition, (see figs. 61 plate XVII, 64 plate XVIII and 78 and 80 plate XXII).
12. Discussion of Results.

12.1 The Etchant Used:

As stated earlier, the etchants found most promising for use with specimens to be examined by normal bright field illumination were the acid dichromate solutions and the acid ferric chloride solutions.

In the compositions preferred, the properties of the two were not greatly different. Each was capable of producing relief etching and each was certainly sensitive to differences in orientation, see fig. 7 plate II and fig. 10 plate III; however the ferric chloride composition used for the major part of the work had been adjusted to minimise surface relief and to produce an optimum selectivity of attack.

Another factor in the production of etch relief in the surface of a deformed specimen may be an associated pattern of strain energy.

12.2 Polished Specimens of Nickel Deformed at Room Temperature:

These specimens showed the ordinary sequence of events found in face-centred cubic poly crystalline metal specimens as deformation is increased.

At the lowest deformations, slip bands appeared, followed later by inhomogeneity on an extended scale.

It is significant to the discussion to follow, that the continued development of slip was apparently associated with the appearance of a coarse parallel feature called "fine scale surface rumpling" whose spacing did not apparently change with increasing deformation, but whose development was plainer as the deformation increased.
This was particularly obvious at 30% and 40% compression and evidence has been advanced that suggests that the fine scale rumpling is associated with curvature in the surface as deformation increases, see fig. 14 plate V and figs. 21 plate VII and 24 plate VIII.

In specimens deformed at room temperature there was only minor surface evidence of deformation bands even at 40% compression; however, surface curvature on an extended scale was ultimately very prominent.

12.3 Deformation Features in Nickel Examined in an Etched Surface of Section.

12.3.1 Specimens Deformed at Room Temperature:

In a specimen deformed 3.3%, etching revealed first observed indication of deformation. Spacing was approximately equal to that of the fine scale rumpling observed in the polished specimens.

The etchant's observed sensitivity to orientation change and the observation that in a polished surface, fine scale rumpling is a coarser feature than the slip bands with which it is associated, (figs. 3 and 4 plate I), suggests that the etchant is detecting in fig. 7, variations of orientation associated with fine scale variations in deformation accompanying the fine scale rumpling and and revealing markings parallel with slip. This proposal is supported by the observation that the parallel markings associated with slip are increasingly well developed by etching as deformation increases, (see figs. 10, 13, 17, 20, 22 and 25 plates III - VI) and that a similar development of surface rumpling accompanies increasing
deformation, (see figs. 8, 12, 16, 19 and 24 plates III – VIII). Etch markings of similar spacing are exhibited in the polygonised fields of figs. 26 and 27 plate IX.

Figs. 20, 22 and 25 plates VI – VIII also show extended regions of etch relief of the same order of size as that of the grains. Extended regions of curvature of a similar size are also seen in figs. 19, 21 and 24 plates VI – VIII.

These features should be compared with the field of fig. 28 plate VIII which shows patterns of etch contrast known to be related to variations in orientation.

Similar patterns are already present in fig. 26 in the large central grain, at 1.5% compression, before it is possible to observe any evidence of similar deformation in the polished specimen.

These observations suggest that there is deformation on a coarser scale than that discussed above and that with this is associated variation in orientation to which the etchant is sensitive.

It is suggested that these changes of orientation are detected because the etchant then readily produces corresponding surface relief, (figs. 7 plate II and 10 plate III show differently oriented grains etched in marked relief).

However, it is possible that associated patterns of strain energy are at least in part responsible for the relief etching produced.

It therefore appears that apart from systematic parallel markings associated with slip, the etchant also detects coarse irregular patterns of inhomogeneous deformation whose size is of the same order as that of a grain.
No markings similar to those described by Samuels (87) and called by him "slip band indications" were detected. It is thought that this is good evidence for their absence since as will appear in Section 12.4 the same etchant detected such markings in nickel deformed at low temperature.

12.3.2 Specimens Deformed at Elevated Temperature:

Markings associated with slip: A conspicuous feature of those markings at elevated temperatures corresponding to those features associated with slip at room temperature is that they appear better defined and become increasingly well defined with increasing temperature, (see figs. 17 plate V and figs 42 - 44 plate XIII).

There is considerable evidence in the literature (14, 24, 25) that at elevated temperatures the strain due to slip is mostly concentrated in slip bands that are more widely spaced than those at room temperature.

It is not clear how far such evidence can be extended to specimens deformed by impact at elevated temperature, but it might be that a similar effect is present. If so it follows that an etchant could differentiate the regions arising from fine scale rumpling more sharply than those produced at room temperature.

Since the polished and deformed surfaces of specimens deformed at elevated temperatures were not examined, it is not possible to compare the closeness of relationship between the etched and deformed surface for room and elevated temperature.

Coarse Etch Features: An important observation about the specimens deformed at elevated temperatures is that inhomogeneity of deformation
on an extended scale is never apparent to the extent found at room

temperature. As a result, the etch relief found in fields such as those

in figs. 22 and 25 plates VII and VIII does not develop even in the most

heavily deformed specimens. Such a development seems possible only if

grain boundary constraints are progressively relaxed with increasing
defformation temperature.

From general experience of metals deformed at elevated temperatures,
as in creep* it might be that some form of 'viscous' behaviour at the grain
boundaries is responsible for the suggested relaxation in constraint due
to the boundaries.

However, here again, there is some reservation about extending the
results of such experiments to specimens deformed by impact.

If as has been argued constraints at the grain boundaries are reduced
at elevated temperatures in the present experiments, it might be anticipated
that for the same compression, the effect of the slip process in deformation
would become progressively simpler, (see figs. 36, 38 and 40 plate XII).

12.3.3 Nickel Deformed at the Temperature of Liquid Oxygen.

Slip: In the section dealing with nickel deformed at room temperature
and elevated temperatures, attention has been drawn to parallel markings
in the etched surface and these have been associated with fine scale
rumpling due to slip.

Similar markings are seen in nickel deformed at \(-183^\circ C\), (see figs. 53

*See "Creep in Metals" Sully, Butterworth 1949, p. 71*.
and 54 plate XV). They are more prominent and better defined than those seen at room temperature. In terms of the suggested mode of operation of the etchant, that is an ability to etch by relief, regions of slightly different strain due to slip, it is not clear why the markings at \(-183^\circ C\) are better defined than those at room temperature.

Deformation Bands: Coarse scale disorientation within different grains is seen at all temperatures. At room temperature complicated disoriented regions arise and deformation bands with relatively well defined boundaries are not observed.

However at elevated temperatures relatively simple and well defined deformation bands are seen. If as previously argued, a reduction in grain boundary constraint occurs at elevated temperatures this effect might be expected.

At \(-183^\circ C\) the etch markings which have been interpreted as deformation bands are simpler than those at room temperature. Here again, as in the case of the markings associated with slip it is not clear why this is so.

12.4 Deformed Specimens in the Copper-Nickel System

Slip: In the etched surface of the pure metals and also in the alloys, systematic markings were exhibited similar to those already described for nickel deformed under other conditions and which have been associated with slip, (see figs. 53 and 54 plate XV for nickel and figs. 77 and 78 plate XXII for copper.

In view of the close similarity to markings previously discussed, it
is proposed that they are due to the same cause. Thus it is assumed that wherever they occur extensively, considerable slip has occurred.

Slip line indications, twinning and faulting: Parallel to the markings described above and associated with them, fine sharply etched lines are sometimes seen; these are particularly well developed in plates XVI and XVII for a specimen containing 80% of nickel.

It will be noticed that some fields contain lines that are very fine, (for example the field of fig. 60 plate XVI) and it is possible that there are some of these unresolved. Other fields contain lines that are relatively quite broad, (see fig. 57 plate XVI).

There is therefore a continuity of development of these lines, which at their finest may be submicroscopic in thickness but which can apparently develop by growth in width. There is a similarity between these lines and those called slip line indications by Samuels (87).

Fig. 57 plate XVI also contains markings that suggest that the lines can develop by a local increase in width, and its extension, into the very wide markings seen in this figure and elsewhere (see fig. 56).

These broad markings are similar to those called twinning indications elsewhere, (see Section 7.2.2). Development into the broader markings is predominantly at the grain boundaries; this suggests that locally high values of stress are required for their lateral growth.

These observations suggest that all the markings in the field of fig. 57 are twinning indications in different stages of development.

Although these markings are called twinning indications their precise nature can not be decided. In copper Blewitt, Coltman and Redman (48)
considered similar markings to be twins; the same conclusion was reached by Suzuki and Barrett (50) for markings found in the silver-gold alloys.

However Hatherly (92) has reviewed the literature relating to these markings in connection with similar markings found in brass and has concluded that no definite decision can be made.

Recent work by Malin (110) appears to support the view that they are probably not simple twins, since in deformed single crystals of copper, twins observed in all specimens by the use of the electron microscope could not be related to markings similar to twinning indications found only in specimens deformed in certain orientations at the temperature of liquid nitrogen.

In the review (Section 6) it was pointed out that numerous authors have suggested that ease of twinning should be correlated with variations in stacking fault energy or stacking fault probability. In the present work only a loose correlation has been found, (see fig. 81). This suggests that other factors are operative in determining the frequency of occurrence of twinning.

However, it is clear that stacking fault energy or stacking fault probability, should be most directly correlated with the formation of stacking faults and if the indications discussed above are in fact arrays of stacking faults, that all such indications should be included in any count of grains.

In the present work, however, only well defined markings were counted. It is clearly not possible to include all the fine markings and indeed many such relevant indications might not have been resolved.
This possibility makes it unlikely that a close correlation with stacking fault probability would be found. Thus apparent anomalies such as different frequencies of occurrence of markings for alloys with the same stacking fault probability, may be due to this cause.

Deformation Bands: Etch patterns interpreted as being associated with the presence of deformation bands are exhibited throughout the range of composition; no apparent relationship between occurrence and composition could be derived.
**Conclusions:**

The following conclusions have been derived:

1. Etchants have been developed which reveal characteristic markings in deformed nickel by producing relief effects associated with periodic variations in orientation.

2. Periodic markings parallel to and associated with slip bands are developed by the etchant with varying degrees of detail depending on the condition of deformation, but having a wider spacing than the slip bands.

3. The markings associated with slip are much better defined in nickel specimens deformed at elevated temperatures, possibly because the deformation is concentrated in more widely spaced bands at elevated temperatures.

4. No markings comparable with those described by Samuels (87) for copper and brass were found in specimens deformed at room and elevated temperatures.

5. Markings similar to those described by Samuels (87) were found in the copper-nickel alloys deformed at \(-183^\circ\text{C}\). They are referred to as twinning indications but are probably not simple twins and may be arrays of stacking faults on closely spaced planes or heavily faulted twins.

6. No close correlation between stacking fault probability and frequency of occurrence of twinning indications has been found for the various specimens in the copper-nickel system, but this may be due to the experimental impossibility of including all the relevant indications.
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