THE STEREOLOGY OF THE INTERGRANULAR SURFACE OF A METAL

David A. Aboav

29 Clements Road, Chorleywood, Hertfordshire, England, WD3 5JS

ABSTRACT

After some introductory remarks on the mathematical properties of a 4-connected, 3D honeycomb, two examples of this structure occurring in Nature --- the foam, and the polycrystal --- are compared and are seen in some respects to differ fundamentally. The stereology of the intergranular surface of a polycrystalline metal is then investigated experimentally, by determining topological characteristics of grains from a specimen of pure aluminium that have been separated from one another by the addition of gallium. It is found that, immediately after the aluminium has recrystal-lized, the faces assume a 'non-random' arrangement when the metal is annealed and grain growth takes place. The new arrangement is quantitatively described by a simple empirical equation.

Key words: arrangement, grain-faces, intergranular surface, metal, nonrandomness.

INTRODUCTION

• A metal generally consists of a honeycomb of polyhedral cells, or 'grains', whose polygonal faces form a 4-connected tessellation in three dimensions. This tessellation of faces --- the intergranular surface --- determines the size and shape of the individual grains, on which the strength of the metal depends. The stereological properties of this surface are therefore matters of practical importance.

The fact that a metal is not transparent makes it difficult to determine the structure of its interior. One may examine the tessellation of its grain-sections at the metal surface, say, or a stereoscopic X-ray photograph of its interior (W.M.Williams & Smith, 1952); or one may detach the grains and measure them one by one (Desch, 1919). But none of these methods is entirely satisfactory; for, as Exner (1972, 1987) has pointed out, one cannot by planar measurements alone establish the true spatial distribution of grains that are not all of the same shape; and three-dimensional shape can be assessed only by tedious experimental work. Nevertheless, the measurement of the shape of a large number of separated grains, though laborious, affords perhaps the most reliable means of determining the interior structure of a metal; and for that reason it is adopted in this investigation.

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The intergranular surface of a metal is often compared to the more easily observed intercellular surface of a transparent soap foam, which in some ways it resembles. But there is a significant difference between the two surfaces, and because of it a certain misunderstanding has arisen through their comparison. We shall therefore look briefly at some of the topological properties of a foam, before turning to those of a metal.

The opportunity recently arose of observing the shape of individual grains, from a specimen of pure aluminium, which had been separated from one another, as Desch (1919) had separated the grains of beta-brass. Measurements of the shape of some hundreds of these grains confirmed what had been suspected from observations of grain sections in the surface of a polycrystal (Aboav, 1970), namely that, during the course of growth, the faces of the intergranular surface, originally arranged at random, subsequently take up a 'non-random' configuration. A promising feature of this new finding is that this rearrangement of faces is quantitatively described by an equation of great simplicity. My main object in speaking to you today is to tell you of that result.

But, before we turn to the experimental findings, let us first take a brief look at some of the mathematical concepts used to describe them.

THE 4-CONNECTED, 3D HONEYCOMB

The aggregate of grains in a metal has the topological structure of an irregular, 4-connected honeycomb, whose slightly curved faces, and edges, meet at equal angles at the edges, and vertices, of the honeycomb, respectively. In practice this honeycomb is often represented by a simpler one having its vertices joined by straight lines, in which case the average value, [θ], of the angle, θ , at which they meet is related to the average value, [N], of the number of faces, N, per polyhedral cell and the average value, [n], of the number of edges, n, per polyhedral cell-face in the honeycomb by the equations

$$[\theta] = \frac{2}{3} \prod \frac{([N] - 3)}{([N] - 2)} , \qquad (1)$$

and

$$12 = [N](6 - [n]) .$$
 (2)

(Coxeter, 1973). Since they are related by two independent equations only, the three quantities [n], [N], and $[\theta]$ are not constant.

For reasons of stability, however, the vertex angles of many physical honeycombs tend to be equal; so that in such systems, although [θ] is not a constant, it nevertheless approaches the constant value, θ_i , equal to \cos^{-1} -1/3, or 109° 28' 16" 23"".... Since this angle is incommensurable with a rightangle, it is convenient to use in its stead an angle, θ_c , commensurable with a rightangle and approximating to θ_i . As good an approximation as any physical system is likely to require is given by

$$\theta_{\rm c} = \frac{253}{416} \prod \tag{3}$$

or 109° 28′ 16″ 9‴ which differs from θ_i by less than a quarter of a

second of arc. It follows from Eqs. (1),(2), and (3) that, in a honeycomb whose average vertex angle has this value, the average values of N and n are

$$[N] = 13^{29}/73 \tag{4}$$

and

$$[n] = 5 \frac{17}{163} \tag{5}$$

There can, however, be few systems in Nature requiring for their description so accurate an approximation. For dealing with the granular structure of a metal the approximation 31

$$\Theta = \frac{31}{51} \Pi \tag{6}$$

or approximately 109° 25', which gives the average values

$$[N] = 13^{1}/3 \tag{7}$$

and

$$[n] = 5^{1}/10 \tag{8}$$

is quite accurate enough. The construction of a 4-connected honeycomb with average vertex angle approximating to $\cos^{-1/3}$ has not, however, the same significance for macroscopic systems as it has, for example, in a molecular theory of chemical structure; and, although the question is habitually referred to in discussing the polycrystalline structure of metals, there is perhaps no real need to raise it there.

Two further problems bearing on the present investigation are the construction of

- (i) a periodic, 4-connected honeycomb whose unit cell is a single polyhedron (Kelvin, 1894); and
- (ii) a 4-connected honeycomb that divides its space with minimal partitional area (Thomson, 1887).

Since to make its cells identical does not necessarily make the partitional area of a honeycomb as small as possible, we are here faced with two distinct problems.

With regard to the first one, a periodic 4-connected honeycomb cannot have as unit cell one of the regular polyhedra, since none has a vertex angle of \cos^{-1} -1/3. But, if we admit also the semi-regular polyhedra and so allow the vertex angles to have more than one value, four such periodic honeycombs can be constructed (Andreini, 1905), of which one has as unit cell a single polyhedron, the truncated octahedron. And if we admit polyhedra with faces that are irregular, or have vertices that are not coplanar, at least one more such honeycomb with a single polyhedron as unit cell can be constructed (R.E. Williams, 1968).

The second problem is not so easily solved. It might be thought that, if all the cell-faces of a honeycomb are of minimal surface area, its total partitional area cannot be made any smaller without diminishing the number of its cells; but the following considerations show that this is not necessarily the case. In Figs.la-c are shown three different, trihedral, 14-hedra of equal volume, and with faces that are everywhere of zero mean curvature, and hence of minimal surface area. They are (a) Williams's 14-hedron (1968), (b) a truncated octahedron, and (c) a truncated, hexagonal, double skew pyramid. The number, R, shown beneath each polyhedron is the ratio of the surface area of that polyhedron to that of a sphere of equal volume, and has not the same value in each case. The problem of determining the polyhedron with the smallest surface area for a given volume and given number of faces is, despite its elementary nature, not easy to solve. Time does not allow us to enter on the matter in any detail, but we here notice the following:

(i) A necessary condition is that the faces of the polyhedron be tangent to a sphere at the centre of gravity of the faces (Lindelöf, 1869); and this condition is sufficient for a polyhedron of given type, if all its vertices are trihedral (Steinitz, 1922).

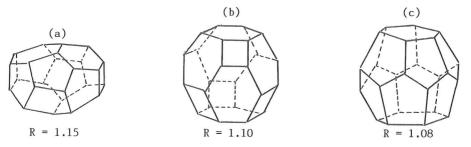


Fig.l. (a) Williams's 14-hedron (1968); (b) a truncated octahedron; and (c) a truncated, hexagonal, double skew pyramid.

(ii) It is conjectured (Goldberg, 1934) that, among the N-hedra of given type, the one with the smallest surface area for a given volume is a 'medial' one --- i.e. that it is a trihedral polyhedron possessing, at most, only n-gonal and (n+1)-gonal faces.

(iii) For N < 16 there is exactly one medial polyhedron for every value of N, except N = 11 and N = 13. Consequently, a trihedral 13-hedron cannot possess pentagonal and hexagonal faces only, but must have at least one of its faces quadrilateral.

(iv) Of the many different trihedral 14-hedra only one, the truncated double skew pyramid of Fig.lc is medial; so that, if Goldberg's conjecture is correct, this polyhedron is the 14-hedron with the smallest surface area for its volume.

These facts suffice to indicate that to construct, in a given volume and with a given number of cells, a 4-connected honeycomb having the smallest possible partitional area is not so simple a problem as appears at first sight.

We turn now to the naturally occurring 4-connected honeycombs, which may conveniently be divided into two types, defined by the two states of matter (Schrödinger, 1944): the fluid (or 'amorphous'), and the crystalline.

THE FOAM

As well as to the above mathematical conditions, a naturally occurring honeycomb is subject to a physical restriction, which limits, often severely, the number of forms it may assume. Broadly speaking this restriction can be of two kinds, depending on whether the honeycomb is in the fluid, or in the crystalline, state. Since the two kinds differ fundamentally in origin, they give rise to structures that differ in important respects. Though it lies outside the field of our enquiry, the fluid honeycomb is here briefly considered, because of the part it has played in past attempts to account for the observed growth of grains in a metal.

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Subject only to internal forces, the cell-faces of a fluid honeycomb, such as a soap foam, move so that their curved edges and surfaces may meet at equal angles, and that their total surface area may be as small as possible. Consequently, their mean curvature tends everywhere to zero (Plateau, 1873). Cell-faces of positive Gaussian curvature (cup-shaped) therefore move towards their centres of curvature (Aboav, 1971) and, by causing adjacent faces to diminish in size, eventually bring about the collapse and disappearance of a cell. Adjacent cell-faces thereupon rearrange themselves (to ensure that the honeycomb remain 4-connected); some other face, or faces, then become cup-shaped; and so the process goes on, until all the cells have disappeared (Smith, 1952). A foam is thus perpetually unstable. That all its cells are destined to disappear in that way is dictated by the severe restriction imposed on the mean curvature of their faces, namely that it tend everywhere and at all times to zero.

Thomson (Lord Kelvin) (1887), reflecting upon the second of our two problems, (v. supra), in a paper entitled 'On the Division of Space with Minimum Partitional Area', opened with the (correct) statement: 'This problem is solved in foam ', but subsequently diverted his attention to the first problem, namely the construction of a periodic honeycomb, and proceeded to tackle the two problems as if they were one and the same. This he was able to do because he did not take into account the topological factor determining the ratio of the surface area of a polyhedron to that of a sphere of equal volume (Goldberg, 1934). Consequently, he arrived at the (erroneous) conclusion that a foam would be in equilibrium, if it consisted of equal cells having the shape of truncated octahedra, with their edges and faces curved to ensure that they met everywhere at the same angles.

In practice the air in a foam diffuses through its cell-walls slowly enough to allow the cells, despite their perpetual instability, to be observed and measured with ease; and this enabled Matzke (1946) to determine in a foam the shape of 600 cells, which were sufficiently far removed from the walls of their enclosure. Table 1 shows $Z_{\rm g}$, the number of these cells that had q quadrilateral faces.

Table 1. Matzke's (1946) foam. Number, Z , of cells with q quadrilateral faces. $\ensuremath{\mathsf{q}}$

q	0	1	2	3	4	5	6
Z_q	120	232	138	85	22	3	0

Matzke, like Desch (1919) before him, expressed surprise that not one of the 14-faced cells had the shape predicted by Kelvin; whereas about one in seven of them had the shape of Williams's polyhedron --- a fact that was later to puzzle Williams (1979), since this polyhedron has an even larger surface area in relation to its volume than the polyhedron of Kelvin (Fig.1b). How, then, are we to account for this seemingly contradictory state of affairs?

Geometry (Eq. (4)) ensures that a cell in a 4-connected honeycomb is most likely to have 13 faces; but a 13-hedron must have at least one of its faces quadrilateral, since topology does not permit a 'medial' polyhedron to have 11 or 13 faces (Goldberg, 1934). Hence, since all but two of Matzke's 600 cells had 12 or more faces, almost all these faces would have been pentagonal or hexagonal, had it not been for this topological prohibition. It is this prohibition that compels at least 10% of the cell-faces to be quadrilateral; and this in turn obliges some, at least, of the 14-faced cells to have one or more faces quadrilateral --- even at the cost of increasing their surface

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area in relation to their volume. Hence Williams's polyhedron, with its two quadrilateral faces and resulting greater relative surface area, occurs in Matzke's foam as well as the truncated skew pyramid, which has the smallest surface area for a 14-hedron; whereas Kelvin's polyhedron is nowhere to be found, since, as the values of Z_q in table 1 imply, it has too many quadrilateral faces.

That the value 1.15 (Malinski, Cwajna, & Maciejny, 1987) of the ratio, R, for Williams's polyhedron (Fig.1a) is greater than it is for Kelvin's (Fig.1b) depends on the fact that the faces of Williams's polyhedron are not tangent to a sphere at their centre of gravity (Lindelöf, 1869). If the polyhedron is transformed to satisfy that condition (Fig.2), its ratio, R, diminishes.

It is, then, the coincidence of the number 13 appearing in two different guises --- one geometrical, the other topological --- that causes a honeycomb of foam cells in practice to assume so perplexing a form. But, amusing though this numerical accident is, we must not dwell upon it; for, as we shall presently see, the behaviour of a fluid honeycomb has little to do with that of a crystalline one.

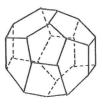


Fig.2. Williams's (1968) polyhedron transformed to satisfy Lindelöf's (1869) condition.

THE POLYCRYSTAL

Neighbouring cells of a foam are separated by a film of liquid, without which their gaseous contents would be indistinguishable; but no such 'second phase' is needed to delineate the cell of a polycrystal, or metal grain, whose solid content is already distinguished from that of its neighbours by its anisotropy. Despite this physical difference, these two honeycombs have in common the geometrical property that their cell-faces and edges meet at roughly the same angles, thus giving their faces a similar doubly curved appearance. The two systems are alike, too, in that in some circumstances the smaller cells of a polycrystal may collapse and disappear, in the manner of the smaller cells of a foam. But there the resemblance between the systems ends. In particular, the movement and the stability of the two intercellular surfaces are found, on closer examination, to be quite different.

The most striking difference between the systems is that a polycrystal, unlike a foam, is not perpetually unstable. Subject only to internal forces, the grains of a metal may sometimes 'grow' as a result of the collapse and disappearance of other grains. But it is a commonly observed fact that, when this occurs, the grains do not go on growing indefinitely; for the movement of the intergranular surface, however vigorously it may take place to begin with, almost always slows down and comes to a halt. In the absence of an externally applied force, a polycrystalline metal is rarely observed to change into a single crystal.

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This cessation of growth is a common and easily demonstrated property of metals. The fact that, in a pure metal at a given temperature, grain growth ceases at a well-defined value of the average grain size, implies that the intergranular surface of a metal is stable, provided that the mean curvature, H, of its individual faces nowhere exceeds a well-defined maximum value, H_T , dependent on the temperature. The restriction imposed on the curvature of cell-faces in a polycrystal is thus less severe than it is in a foam, the condition for stability in the two cases being

for a foam (Plateau, 1873):
$$H = 0$$
 (9)

for a polycrystal:
$$H \nearrow H_{\pi}$$
. (10)

It is therefore to be expected that the mechanism of cellular growth will be more complicated in a metal than in a foam, since the non-zero, maximum permissible mean curvature, H_T , will enable some faces to become stable before others. These faces will be the first to come to rest; and being, it may be supposed, able to act in the intergranular surface as 'anchors', will offer, to the faces that are still unstable, possibilities of spatial arrangement that are denied the foam, all of whose faces are perpetually unstable. The intergranular surface of a metal is therefore free to assume the shape most favourable from the point of view of its stability; and this shape, since it perforce excludes the highly unstable arrangements that arise from the random clustering of unstable faces is *ipso facto* not a 'random' one.

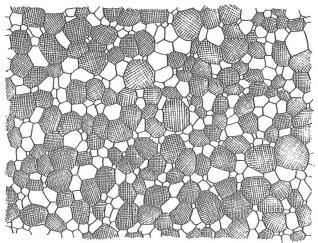


Fig. 3.Polycrystalline magnesium oxide; grain-sections with more than 6 sides shaded.

In a pure metal at a given temperature the growth of its grains, from their initial recrystallised state until they reach equilibrium, generally takes place in two stages. If the temperature is high enough, there is at first a sudden, catastrophic change in the intergranular surface, in the course of which the total number of grains in the metal may diminish by several orders of magnitude (Vogel, 1923). If, thereafter, the metal is kept at the same temperature, the grains continue to grow, but at a rate some orders of magnitude smaller than before. During this, the final stage of growth, the average

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grain diameter approaches asymptotically a stable value, which depends regularly on the temperature (Andrade & Aboav, 1966). This division of the growth into two stages, which does not occur in a foam, gives reason to suspect there may be, as we have supposed, some 'non-random' element in the mechanism of grain growth in a polycrystal.

This suspicion is reinforced by the appearance of the etched surface of a polycrystal during the growth of its grains (Aboav, 1970). Fig. 3 shows in outline the surface of a specimen of polycrystalline magnesium oxide, with the grain sections having more than 6 sides (the 'major' grain sections) shaded, and the others (the 'minor' ones) in white. Similar patterns are observed in the surface of a pure metal. The major and minor sections are seen to be linked together in chains; so that nowhere is a grain-section of one kind completely surrounded by others of the same kind; but, for the reason already given (Exner, 1972), the existence of a corresponding 'nonrandom' distribution of grain size in three dimensions cannot be deduced with certainty from the 2D evidence of a section of the polycrystal. Because of its practical implications, however, it is essential to establish beyond doubt whether such a change --- from a random to a 'non- random' arrangement of its faces --- takes place in the intergranular surface of a metal as its grains proceed to grow; and it was for that reason that the measurements now to be described were made.

EXPERIMENTAL INVESTIGATION

It was decided to determine the shape of the intergranular surface of a metal directly, by measuring individual grains that had been separated from one another. Rhines & Patterson (1982) by adding to it liquid gallium, succeeded in separating several hundred grains from a specimen of pure aluminium, and drew the Schlegel diagram of each of them from within a given volume of metal. Professor Patterson has kindly allowed me to use these diagrams for the purpose of this investigation.

METHOD & RESULTS

The metal used in the experiments was taken from a single ingot of 99.998% pure aluminium. After 50% rolling, and annealing for 100 minutes at 400°C, it was elongated in tension by 6% at -196°C; and recrystallisation and grain growth induced in two chosen specimens, (a) and (b), by further annealing at 600°C. for periods of (a) 4 minutes and (b) 1 hour, respectively. Each specimen was then cleaned with hydrofluoric acid and placed upon a glass plate on an electric hot-plate kept at 50°C. It was found that a piece of gallium placed on a specimen fully penetrated it within five minutes and allowed its grains to be easily separated. The outer grains were removed, to leave a central cluster of about 100 grains, which were then separated from one another.

A Schlegel diagram (Coxeter, 1973) was drawn for every grain of such a cluster and m_n , the average number of sides of grain-faces adjacent to n-sided ones throughout the intergranular surface of the cluster, was determined by counting the number of sides of each of the polygons in contact with every n-sided polygon in the diagrams, and averaging the result for each value of n. Figs. 4a and b show typical diagrams of a grain taken from each of the two specimens. Each has 15 polygons, or grain-faces; but, as far as one can see from them, there is no noticeable difference in the way their polygons are arranged.

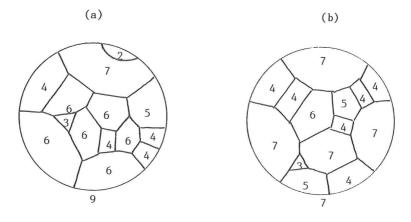


Fig. 4. Schlegel diagrams of 15-faced grains

To detect such a difference, if any, in specimens that have been differently treated, measurements of sufficiently many grains must be made, and their average taken. The average value $[m_n]$ of m_n then approaches a constant value given by

$$[m_n] = [n] + \mu_2 / [n]$$
(11)

where [n] is the average value of n in the same specimen, and $\mu_2 = [(n-[n])^2]$. The variation of m with n, which must in each case be determined empirically, is shown for each of the two specimens, (a) and (b), in Figs. 5a and b.

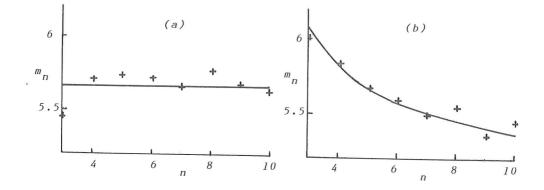


Fig.5. Variation of m with n in aluminium annealed at 600°C. for (a) 4 mins. and (b) 1 hour.

The straight line in Fig. 5a represents the equation $m_n = [m_n]$; the rectangular hyperbola in Fig. 5b represents $m_n = [n] + \mu_2 / n$. Since these curves pass roughly through the points representing the observations, it follows from Eq. (11) that, for the specimen annealed for only 4 minutes, m_n is given approximately by

$$m_n = [n] + \mu_2/[n]$$
 (12)

and is therefore constant; and (b) that, for the specimen annealed for 1 hour, m_n varies linearly with the reciprocal of n , thus:

$$m_n = [n] + \mu_2/n$$
 (13)

In this equation [n] ≈ 5.1 (cf. Eq.(8)), and $\mu_2 \approx 3$, while for specimen (a) [n] ≈ 4.9 , and $\mu_2 \approx 3\frac{1}{2}$; but the total number of grains here measured is too small to establish that this difference is significant.

DISCUSSION

These results show how, in specimens of recrystallised aluminium, annealed for different lengths of time at the same temperature, a difference in the 3D arrangement of the grain-faces, not noticeable at a glance, may be made evident by counting a sufficiently large number of their sides.

Immediately after the metal has recrystallised, the grain-faces, as might be expected, show no preferential arrangement, the average number of sides of faces adjacent to n-sided ones being at this stage independent of n (Eq.(12) Fig.5a). But this condition does not long persist; for, as the metal is heated, the 'random' arrangement soon disappears and is replaced by one in which faces with fewer sides are more likely to be surrounded by ones with more sides than average, and vice versa (Eq.(13), Fig.5b). It is as if the faces with the fewest sides, and hence the least size and greatest curvature, sought to avoid one another; and this is not surprising, when one remembers the extreme instability shown by a polycrystal whose grains are all smaller than a certain size.

That Eq. (13) contains no arbitrary constant leads one to suppose the relation may be universal, and may hence be correlated with the experimentally established fact that, in pure metals in which grain growth has taken place from a state of complete recrystallisation, the form and spread of the resulting distribution of grain size is independent of the metal and of its average grain diameter (Andrade & Aboav, 1966). Naturally, such a general supposition cannot be grounded on the few data at present available. But the question is of considerable practical interest; and, if on further investigation the above effect is found to be of wide generality, it may help to explain how the presence in a metal of a second phase can have so profound an effect on its mechanical properties.

In an alloy of aluminium containing 1.2 atomic percent of tin, for example, the average value of n is the same as for pure aluminium([n] ~ 5.1); but the spread of the distribution of n is smaller: $\mu_2 \sim 1$ (Williams & Smith, 1952), as compared with $\mu_2 \sim 3$ for the pure metal. This suggests that particles of a second phase may, too, act as 'anchors' in the intergranular surface during the rearrangement of its faces. If, then, the grains of this alloy prove to be easily separable by the addition of gallium, we propose next to establish whether the addition of tin, which alters the spread of the distribution of sides per grain-face, also influences the spatial arrangement of the faces; and eventually to make similar measurements with metals of different crystal structure.

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A final remark. We have assumed that the intercellular surface of a polycrystal, unlike that of a foam, is stable if its mean curvature nowhere exceeds a certain maximum value. We cannot, however, account for this difference in behaviour unless we invoke a molecular hypothesis (Aboav, 1971), namely that of the 'crystal lattice', which Hally (1784) postulated to account for the observed anisotropy of crystals.

Since our everyday (Riemann, 1854) concepts of length and angle are not precise enough to define the elements, or molécules intégrantes, of such a lattice, Hally had to turn for its construction to the geometry of the Ancients, whose concepts, by virtue of assumptions they had made about them, possessed the required degree of precision. Among those assumptions was the Fourth Postulate of Euclid (Heath, 1956), whose object was, in effect, to deny the attribute of 'anisotropy' to the solid bodies found in Nature --those res longae latae aut profundae (Newton, 1670, 1706) from which stemmed all their notions of geometry, and without which they could not demonstrate its propositions. Such an assumption was indispensable, if, as experience seemed to demand, the geometry was to be made independent of scale. Thus it came about that, in seeking to account for the observed anisotropy of crystals, Hally unconsciously invoked a principle which itself tacitly appeals to the symmetry properties of naturally occurring solid bodies. This petitio principii on the part of Hally undoubtedly lies at the root of the difficulty that has long hampered our search for a simple description of the mechanical behaviour of crystals.

Clearly, to avoid arguing in a circle, postulates like those of Euclid, which have their origin in the observed properties of naturally occurring solids, must be excluded from any attempt to 'explain' why those solids appear to us as they do. In particular, it is vain to seek, in a geometry grounded on concepts derived from the 'things long, broad, and deep' (Newton, 1706) that surround us, an explanation of that mystery of mysteries, their apparent three-dimensionality. The conclusion seems inescapable that we must look elsewhere for the concepts on which to base a description of the phenomena that are presently the object of our attention.

Until we sever the ties with classical geometry (Menger, 1949) ---- until, that is, we succeed in formulating an adequate geometry that does not depend, for the definition of its basic concepts, on the familiar properties of the solids we see around us --- I do not think we can hope to achieve a satisfactory description of the mechanical properties of matter in the crystalline state. But we may have a long time to wait for that; and we must in the mean time get on with the work! May I, then, propose that we agree for the time being to accept --- as a fact not requiring an 'explanation' --- that the intergranular surface of a polycrystal is in general stable, provided its 'curvature' nowhere exceeds a certain maximum value, dependent on its chemical composition and the temperature.

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