ARTICLES

CRYSTAL STRUCTURE AND FIVE-FOLD DIFFRACTION SYMMETRY OF THE ALUMINIUM–MANGANESE ICOSAHEDRAL PHASE: A REAPPRAISAL

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ABSTRACT

A model is developed for the crystallization of icosahedral phases in Al–Mn, Al–Mn–Si and allied systems, starting from the icosahedral clusters and ordered pentagonal chains of atoms postulated a few decades ago to exist in alloy melts. It is shown that different traditional crystal structures can emerge through the joining together in regular fashion of the pentagonal chains of atoms in the special conditions of rapid solidification. The primary unit for the building of such structures is the primitive *orthorhombic* subcell that forms by joining 13-atom icosahedra in three perpendicular directions, through *vertex* connection along the first axis, *edge* connection along the second axis and *triangular ledge* connection along the third axis. A family of orthorhombic unit cells can be visualized to develop from this model through *complex* ordering involving two or more species of atoms and consequent *multiplication* of the subcell parameters. The orthorhombic unit cell dimensions are arrived at on the basis of this model for the $Al_{80}Mn_{20}$ icosahedral phase and it is shown that the position of X-ray and electron reflections can be satisfactorily explained by this unit cell.

It is also shown that this model for growth of icosahedral phases permits emergence of *orientation variants* of the same crystal 72° to each other as well as random changes in orientation and growth directions on a submicroscopic scale. Each grain or 'crystal' of such icosahedral phases thus turns out to be a complex mosaic made up of five sets of irregular-shaped microcrystallites of nanometre dimensions of a traditional orthorhombic phase, each fully or partly coherent with and oriented 72° to its neighbours. Such a state of affairs is shown to account for the unusual microstructural and some other features of these exotic phases.

INTRODUCTION

A number of interesting models have been proposed[1,2] since 1985 to explain the atomic arrangements in intermetallic phases obtained by rapid solidification of mostly aluminium-based alloys and displaying five-fold symmetry in their X-ray and electron diffraction patterns. All these models generally assume quasiperiodicity of atomic arrangements and deal with non-traditional lattices arrived at by novel approaches like three-dimensional Penrose tiling, inflation–deflation operations, generalized multi-grid methods and strip projection and cut projection from high-dimensional periodic lattices. The location of atoms in the rigid geometrical frameworks thus proposed, i.e. the so-called decoration of the quasilattices, has however presented some difficulties like unacceptable density or peculiar interatomic distances, and no model has been able thus far to explain satisfactorily all observed features of these exotic phases.

As documented earlier[3], a few attempts have also been made[4–7] since 1985 to explain the unusual diffraction effects from these so-called icosahedral phases on the basis of interpenetrating multiple microtwins of traditional crystals. However, the unit cells proposed subsequently for such phases, and particularly for the $Al_{80}Mn_{20}$ icosahedral phase, by Pauling[8], the present author[9], and Khan and Wibbeke[10] have not been supported by convincing arguments or models for their formation, nor accompanied by any detailed description of the actual number and the exact location of the concerned atoms in them to explain satisfactorily *both* positions and intensities of the reflections in diffraction patterns. The case for the presence of twins or orientation variants on a submicroscopic scale in such phases has been reopened most recently in a rather spectacular way by the findings of Vecchio and Williams[11,12]. Studying wedge-like specimens of the Al–Li–Cu icosahedral phase, these workers have not only brought out the existence of

numerous nanometre-scale microcrystallites in micron-size grains, but also demonstrated the absence of five-fold symmetry in very thin sections of their specially prepared specimens. In fact convergent beam electron diffraction patterns showed evidence for *just* single crystals in the thinnest sections examined.

BASIS OF NEW MODEL

A model is developed in this paper for the crystallization of icosahedral phases in Al–Mn, Al–Mn–Si and allied systems, starting from icosahedral clusters[13] and pentagonal chains[14] of atoms in the concerned metallic melts. The existence in liquid metals of icosahedral clusters containing 13 atoms each (figure 1, a–c) and characterized by high density as well as low energy was postulated by Frank[13] four decades ago to explain the marked undercooling recorded by small drops of liquid metals. Despite their high co-ordination number such icosahedra cannot constitute nuclei for the growth of solid crystals with close-packed structures. In the case of *alloy* liquids the Frank icosahedra were postulated almost three decades ago by Hume–Rothery and Anderson[14] to join together through sharing of the vertex atoms (figure 1, d,e) when the sizes, valencies and electronegativity values of the concerned atoms differ suitably to confer stability to the resulting pentagonal chains. The emergence of peaks in eutectic distribution curves, i.e. the stabilization of the liquid state at certain compositions, in binary alloy systems could be explained on the basis of such *ordered* pentagonal chains of atoms.

As elucidated by Hume–Rothery and Anderson[14], if solute B is added to liquid metal A, mixing is encouraged whenever there is a pronounced tendency for B atoms to prefer A rather than B neighbours. Thus pentagonal chains made up of different repeating units in an AB alloy can acquire stability at certain compositions, like the following, through avoidance of B–B contacts:

I. 1A–5A–1B–5A—1A–5A–1B–5A ——— 1/12 or 8.33 at.% B

II. 1B–5A–1B–5A ——————— 1/6 or 16.67 at.% B

III. $1A-\underset{2B}{\overset{3A}{}}-1A-\underset{1B}{\overset{4A}{}}\,1A-\underset{2B}{\overset{3A}{}}-1A-\underset{1B}{\overset{4A}{}}$ ——— 1/4 or 25.00 at.% B.

The present model for icosahedral phase formation starts with the idea that different crystal structures can in fact emerge through the joining together of such pentagonal chains in special circumstances, particularly when appreciable or drastic kinetic undercooling of the alloy melt is brought about by techniques like rapid solidification.

Although novel, the concept advanced here of icosahedra and/or icosahedral chains *themselves constituting nuclei* for the growth of crystals from metallic melts is by no means a startling one. Apart from α-rhombohedral (figure 2a) boron, whose structure is built up of regular icosahedra of atoms in slightly deformed cubic close packing, its α-tetragonal compounds $B_{50}C_2$ and $B_{50}N_2$ (figure 2b) are known to be made up of slightly distorted icosahedra of B atoms linked by C and N atoms respectively[15]. In regard to such structures it has also been recognized that the generation of an infinite three-dimensional lattice with the *perfect* five-fold symmetry of ideal icosahedra is impossible in practice and one should be prepared for distortions, translations, voids, chemical inhomogeneities, lattice parameter variations, etc. in them. Even in alloy systems the growth of complex cubic phases made up of clusters of atoms formed around icosahedra has been known for some time[16]. The cubic phases with the composition $A_{12}B$ in Al–W, Al–Cr and Al–Mn systems seem to be made up of thirteen-atom icosahedra (figure 2c) aligned without any atom sharing to generate their 26-atom body-centred cubic (b.c.c.) unit cells[17].

The occurrence of structural units with icosahedral symmetry, even if they are distorted more often than not, in a large number of complex intermetallic phases has recently been highlighted in a review[18].

It is interesting to record here that the very first model suggested for the structure of the i-Al–Mn phase[19] was based on icosahedra joined by their edges, although not regularly but *randomly* and without any long-range order. This approach was further developed by Stephens and Goldman[20] into the so-called icosahedral glass model with vertex-sharing icosahedra packed randomly while maintaining bond-orientational order. These could explain the sharp peak positions in diffraction patterns, but features like peak intensities could not be satisfactorily accounted for by them.

GROWTH OF ORTHORHOMBIC STRUCTURES

The present model envisages growth of different crystalline phases with orthorhombic or related

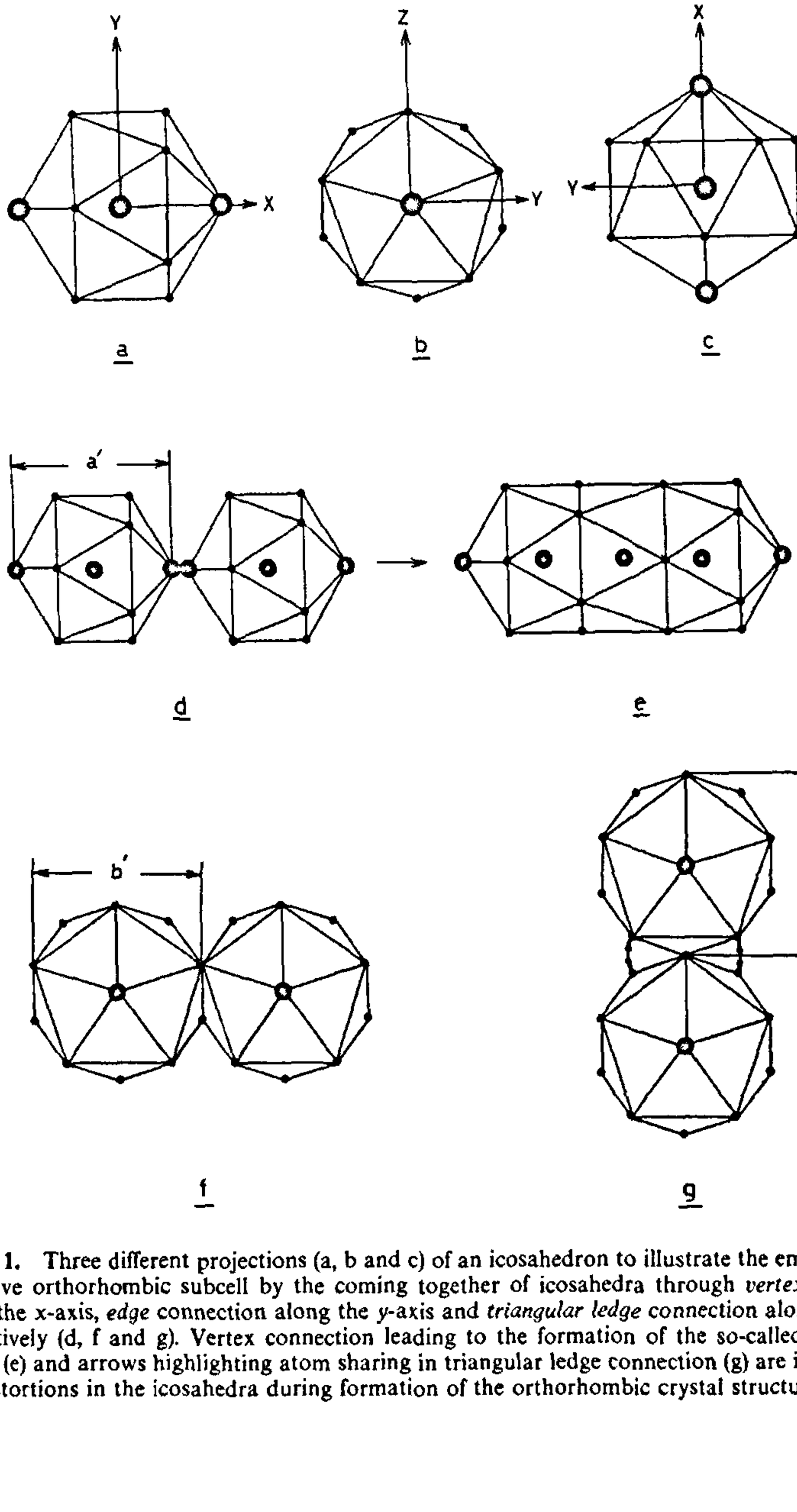
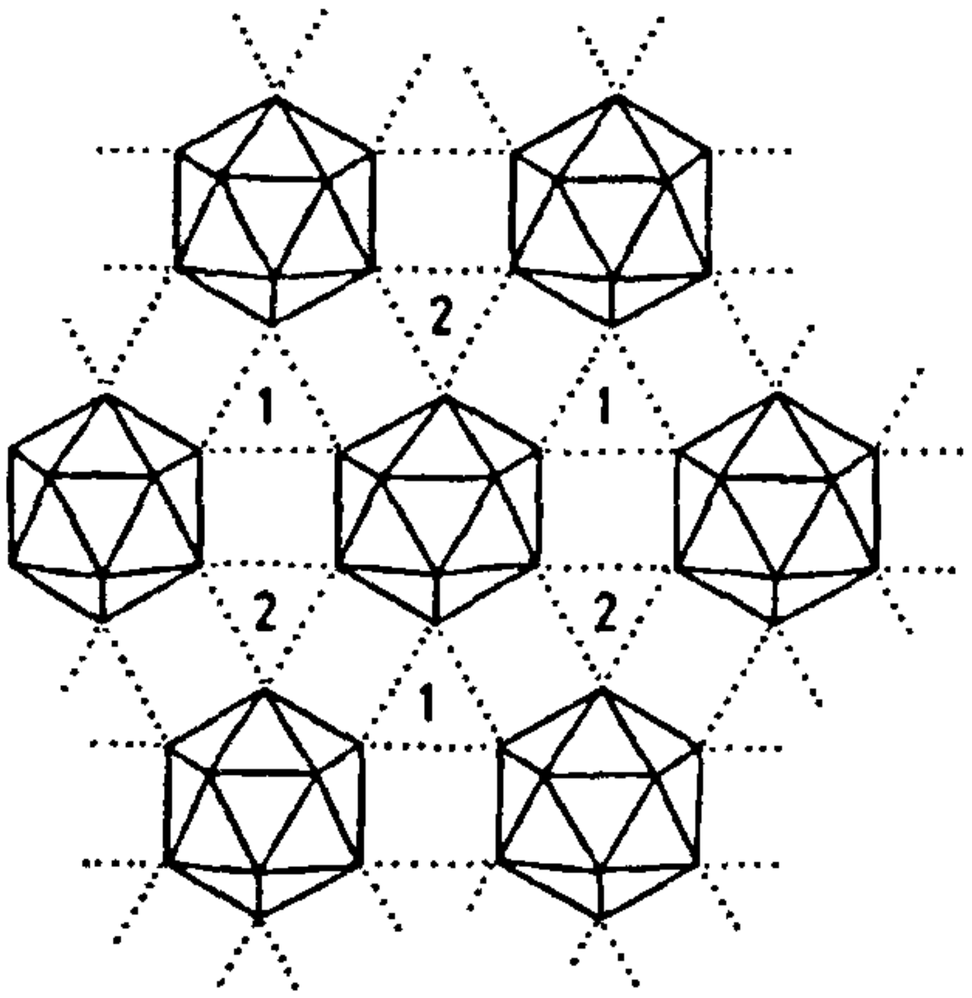
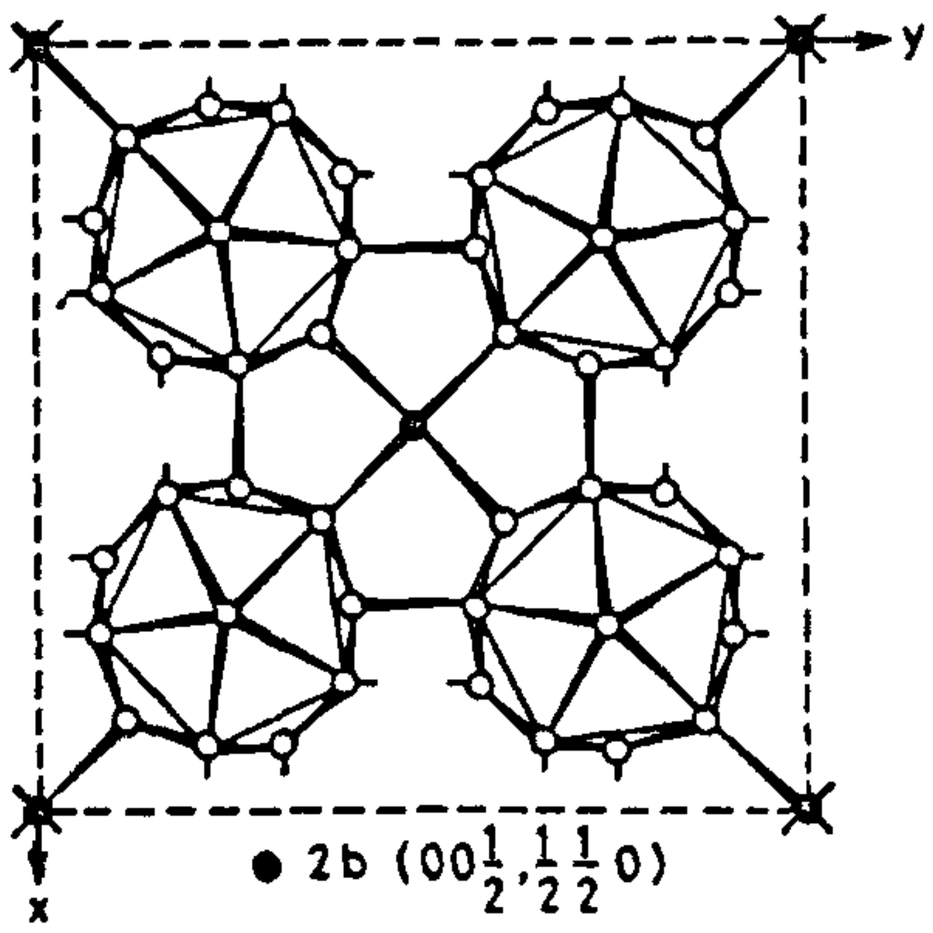


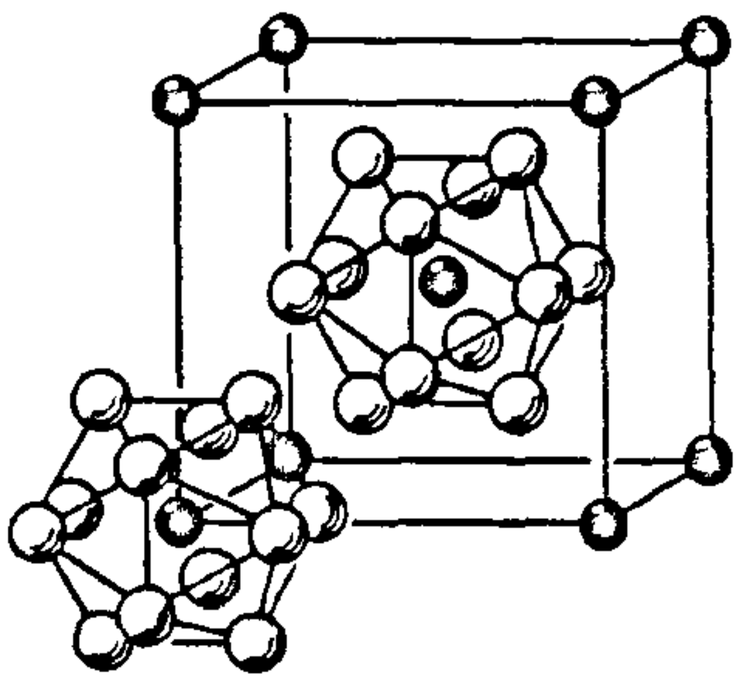
Figure 1. Three different projections (a, b and c) of an icosahedron to illustrate the emergence of a primitive orthorhombic subcell by the coming together of icosahedra through *vertex* connection along the *x*-axis, *edge* connection along the *y*-axis and *triangular ledge* connection along the *z*-axis respectively (d, f and g). Vertex connection leading to the formation of the so-called pentagonal chains (e) and arrows highlighting atom sharing in triangular ledge connection (g) are illustrative of the distortions in the icosahedra during formation of the orthorhombic crystal structure.



a:
α-R Boron



b:
α-T $B_{50}N_2$



c:
$Al_{12}W$ (b. c. c.)

Figure 2. Crystal structures based on geometrical arrangements of the icosahedra: a, α-rhombohedral boron; b, α-tetragonal boron nitride; c, body-centred cubic $Al_{12}W$ intermetallic phase.

crystal structures through the coming together in a *regular* fashion and in all *three* directions of the pentagonal chains (figure 1, d,e) made up of vertex-sharing icosahedra in alloy melts. The primary *unit* for the building of such crystal structures is the primitive orthorhombic *subcell* that forms by joining 13-atom icosahedra (figure 1, d–g) through *vertex* connection along the *x*-axis, *edge* connection along the *y*-axis and *triangular ledge* connection along the *z*-axis. If the length of one of the 30 edges of the icosahedron is *l*, the edge lengths a', b' and c' for this subcell will work out on the basis of geometry to 1.902 *l*, 1.618 *l* and 1.701 *l* respectively, *provided* there is no distortion at all of the icosahedra through the proposed joining and atom sharing. In actual practice, apart from the value of *l* fluctuating in accordance with the *actual* occupancy by A, B, C, etc. atoms of the icosahedron, the sizes, valencies, positions, etc. of the concerned atoms and the nature of atom sharing may be expected to distort the icosahedra and modify a', b' and c' appreciably. It is not difficult, however, to visualize the growth of *several* orthorhombic unit cells, base-centred, body-centred or face-centred, through *complex ordering* involving several species of atoms, and the consequent enlargement of the cell parameters to $m{\cdot}a'$, $n{\cdot}b'$ and $o{\cdot}c'$ respectively, where *m*, *n* and *o* are integers. In some respects such orthorhombic structures can be viewed as a set or family of *polytypes*, the occurrence of which has been strongly suspected in the case of *decagonal* phases of Al–Mn, Al–Fe, Al–Co and related systems[21].

Considering the icosahedral phases of the Al–Mn, Al–Mn–Si and closely related alloy systems, it is possible to arrive at the values of *m*, *n* and *o* and the nature of the orthorhombic unit cell by a careful analysis of X-ray diffraction data, wherein the strong and medium reflections are less than 20, the weak and very weak reflections run into perhaps hundreds, and the highest observed *d* value as close to 1.0 nm. While Pauling[8] has proposed a cubic unit cell with $a = 2.336$ nm, the present author was able to show very good agreement[3] between observed and calculated *d* values of the same $Al_{80}Mn_{20}$ icosahedral phase on the basis of a smaller tetragonal unit cell with $a = 1.653$ nm and $c = 1.736$ nm. The most recent studies[22] on the stoichiometry of Al–Mn-based icosahedral phases have confirmed some previous estimates of around 20 at.% solute elements in these phases, even though the first and most-quoted paper on i-Al–Mn phases[23] had insisted on the formula Al_6Mn for this phase. Among the three types of pentagonal chains referred to earlier, a combination of II and III leads to the following repeating unit of the chain:

$$\left[1\,\text{Mn}\text{–}5\,\text{Al}\text{–}1\,\text{Al}\text{–}\begin{matrix}3\,\text{Al}\\2\,\text{Mn}\end{matrix}\text{–}1\,\text{Al}\text{–}\begin{matrix}4\,\text{Al}\\1\,\text{Mn}\end{matrix}\right.$$

$$\left.\text{–}1\,\text{Al}\text{–}\begin{matrix}4\,\text{Al}\\1\,\text{Mn}\end{matrix}\text{–}1\,\text{Al}\text{–}\begin{matrix}3\,\text{Al}\\2\,\text{Mn}\end{matrix}\text{–}1\,\text{Al}\text{–}5\,\text{Al}\right].$$

This in turn gives a value of 3 for *m* and the generally accepted ~20 at.% Mn for the phase with 7 Mn atoms and 29 Al atoms in the repeating unit. A value of 4 each for *n* and *o* follows from the parameters already arrived at by the author and referred to earlier. Such a large unit cell seems to be *necessary* for the complex ordering between Al and Mn atoms in the structure built around 3-unit pentagonal chains through sharing of ALL but the central atom in each icosahedron by neighbouring icosahedra. The i-Al–Mn and allied phases thus acquire base-centred (A) orthorhombic unit cells made up of 48 ($3 \times 4 \times 4$) primitive orthorhombic subcells and *perhaps* 336 (7×48) atoms in them. The nature of atom sharing, which is obviously complex, will decide whether the proportion between Al and Mn atoms remains the *same* i.e. 29 to 7, as in the seed pentagonal chains.

Table 1 brings out the clear picture that emerges on the basis of the model proposed and the considerations detailed above. We start with the reasonable value of 0.26 nm for the *average* Al–Mn distance, i.e. for the *average* edge length in the icosahedra

Table 1 *Orthorhombic lattice parameters in nanometres for Al–Mn icosahedral phase*

Parameter	$i\text{-}Al_{80}Mn_{20}$
a_i'	0.4946
b_i'	0.4207
c_i'	0.4424
a'	0.5096
b'	0.4137
c'	0.4344
a	1.5288
b	1.6548
c	1.7376

Parameters a_i', b_i' and c_i' are for the ideal subcell, the edge of the icosahedron (*l*) being 0.26 nm; a', b' and c' are for the actual subcell; and *a*, *b* and *c* for the unit cell.

constituting the building block for the orthorhombic crystal structure of i-Al–Mn, i-Al–Mn–Si and related phases. Recent studies, using neutron diffraction and contrast variation effects, of the partial pair distribution functions in i-Al–Mn and allied phases have yielded[24] average Al–Mn distances of 0.255 nm for a co-ordination number of 9.4 and 0.305 nm for a co-ordination number of 2.4. Significantly, no close contacts between Mn atoms could be observed in these studies, justifying the concept of ordered pentagonal chains constituting nuclei for the growth of icosahedral phases in the present model. In table 1 a_i', b_i' and C_i' represent the *ideal* parameters for an orthorhombic subcell based on regular icosahedra with edge length 0.26 nm and with no distortion due to atom sharing . Parameters a', b' and c' are the *actual* parameters of the primitive orthorhombic subcell following distortions through occupancy by atoms of *different* sizes, valencies, etc. and also atom sharing, while a, b and c refer to the actual *lattice parameters* of the i-Al–Mn phase on the basis of the values 3, 4 and 5 arrived at for m, n and o respectively.

It is very significant that despite obvious distortions due to atom sharing, one of the axial ratios, viz. c/b, of the orthorhombic phase almost coincides with the ideal value of 1.0515 for a regular icosahedron (table 1). In fact it was the highlighting of this axial ratio by Mackay[25] in regard to the tetragonal unit cell proposed earlier[3] that led the author to think afresh on this problem and develop the present model. Another point to be highlighted here is the slight expansion in the value of a' in relation to a_i' (table 1) brought about by vertex joining in the pentagonal chain, resulting perhaps in the *equalization* of distances between central atoms and the planes of pentagons. Incidentally, it may be noted in passing that orthorhombic and rhombohedral structures are closely related to *cubic* structures and can be derived from the latter by slight distortions of the cubic unit cell.

The mechanism envisaged in the present model for the growth of icosahedral (and perhaps also decagonal) phases in general permits the emergence of orientation variants of the same crystal 72° to each other, as illustrated in figure 3. In fact, with a large number of pentagonal chains of differing lengths obviously available to serve as seeds for nucleation, not only can the grain sizes be expected to be small (as actually observed), but also random or quasiperiodic changes in orientation and growth directions on a submicroscopic scale can be envisaged. Thus *each* grain or crystal of such icosahedral phases can turn out to be a complex

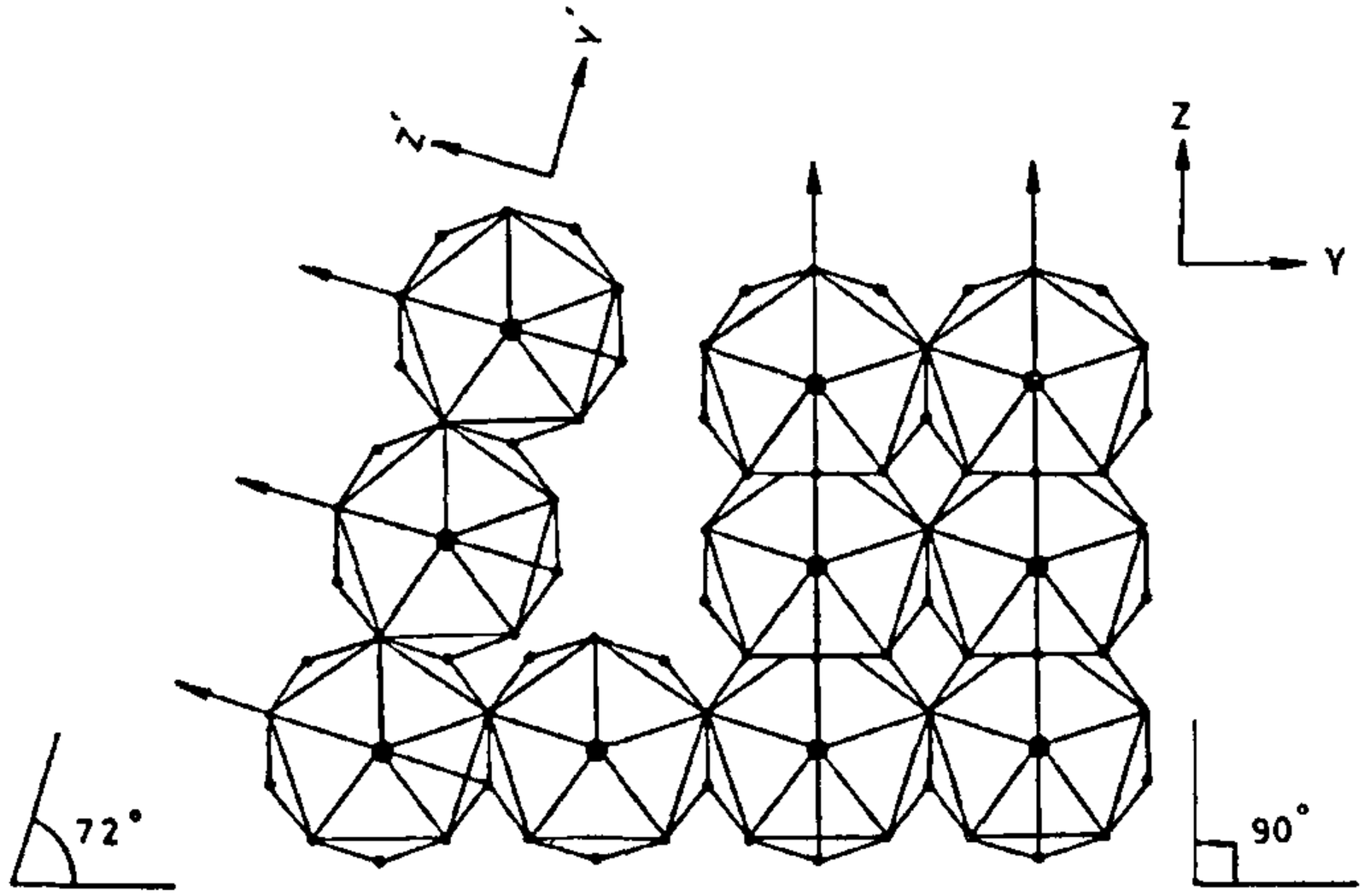


Figure 3. Illustration of a change in orientation during growth of the orthorhombic crystal from pentagonal chains of atoms. Five orientations 72° to each other can be conceived through random changes in growth direction for the nanometre-size microcrystallites constituting a grain of the icosahedral phase.

mosaic made up of five sets of irregular-shaped microcrystallites on nanometre scale of the traditional orthorhombic phase, grown around one or more parallel pentagonal chains, each fully or partly coherent with and oriented 72° to its neighbours. Such a state of affairs has in fact been noted by many workers[4, 11, 26–28] in regard to different icosahedral phases.

ANALYSIS OF DIFFRACTION DATA

Tables 2 and 3 present results of analysis of the X-ray diffraction data obtained for the i-Al–Mn phase with synchrotron radiation[29]. Table 2 shows how most of the strong and medium reflections from i-$Al_{80}Mn_{20}$ can be most satisfactorily indexed on the basis of *both* the subcell and the actual base-centred (A) orthorhombic unit cell, highlighting the fact that a few medium-intensity and most of the large number of weak and very weak reflections are due to the *complex ordering* between the concerned species of atoms during crystallization. Table 3 shows that all 48 reflections recorded thus far and referred to in many publications on i-Al–Mn can again be very satisfactorily accounted for on the basis of the proposed unit cell. It is interesting to note that most of the weak reflections at higher Bragg angles can also be indexed on the basis of the concerned subcell described in table 1. Apart from the excellent agreement between calculation and observation and the strict adherence to extinction rules applicable to such structures (table 3), the most encouraging aspect of the analysis here is that the strong and medium reflections in table 1 display indices that one would expect from a cursory examination of the atom positions in the subcell (figure 1, d, f, g).

Table 2 *Calculated and observed interplanar spacings (d_{cal} and d_{obs}) in nanometres with observed relative intensities (I, rounded to multiples of 5) for the strong and medium X-ray reflections from the i-$Al_{80}Mn_{20}$ phase, which get indexed on the basis of both the subcell (h' k' l') and the unit cell (h k l) described in table 1*

			i-$Al_{80}Mn_{20}$		
No.	*h' k' l'*	*h k l*	d_{cal}	d_{obs}	*I*
1	002	008	0.2172	0.2172	100
2	020	080	0.2069	0.2068	80
3	022	088	0.1497	0.1497	10
4	400	12.00	0.1274	0.1275	20
5	402	12.08	0.1099	0.1101	10
6	004	00.16	0.1086	0.1085	10
7	420	12.80	0.1085		
8	040	0.16.0	0.1034	0.1033	5

Table 3 *Calculated and observed interplanar spacings (d_{cal} and d_{obs}) in nanometres with observed intensities (I) for all, i.e., strong, medium and weak, observed X-ray reflections from i-$Al_{80}Mn_{20}$ phase*

No.	*h k l*	d_{cal}	d_{obs}	*I*
1	013	0.5467	0.542	<1
2	031	0.5257		
3	024	0.3846	0.385	22
4	242	0.3356	0.335	8
5	333	0.3144	0.314	<1
6	404	0.2870	0.286	2
7	026	0.2733	0.274	<1
8	306	0.2518	0.252	3
9	046	0.2373	0.238	<1
10	622	0.2345	Al	—
11	008•	0.2172	0.217	100
12	640•	0.2170		
13	337	0.2069	0.207	78
14	080•	0.2069		
15	644•	0.1941	0.194	1
16	175	0.1939		
17	606	0.1913	0.191	<1
18	800	0.1911		
19	375	0.1825	0.183	<1
20	480	0.1819	0.182	<1
21	735	0.1753	0.175	2
22	093	0.1753		
23	804	0.1749		
24	664	0.1719	0.172	<1
25	448	0.1718		
26	557	0.1665	0.166	<1
27	268	0.1665		
28	680•	0.1606	0.160	1
29	806	0.1595		
30	088•	0.1498	0.150	11
31	577	0.1494		
32	775	0.1457	0.145	3
33	00.12•	0.1448		
34	0.12.0•	0.1379	0.137	<1
35	04.12•	0.1367		
36	884	0.1336	0.134	<1
37	12.00•	0.1274	0.127	20
38	60.12•	0.1259	0.126	<1
39	984•	0.1257		
40	12.04•	0.1223	Al	—
41	6.12.0•	0.1213	0.121	1
42	80.12	0.1154	0.114	1
43	90.12•	0.1102	0.110	6
44	12.08•	0.1099		
45	00.16•	0.1086	0.109	7
46	12.80•	0.1085		
47	68.12•	0.1075	0.108	3
48	0.16.0•	0.1034	0.103	2

Al, Possible overlap of Al reflections. *hkl*•, such *hkl* values are also indexable on the basis of the subcell. Subcell and unit cell parameters as in table 1.

Figure 4 brings out, through computer simulation of the five possible orientation variants of the orthorhombic phases under discussion, the emergence of the familiar electron diffraction patterns with five-fold symmetry, wherein *most* of the spots are due to dynamic double diffraction, as first suggested by Ball

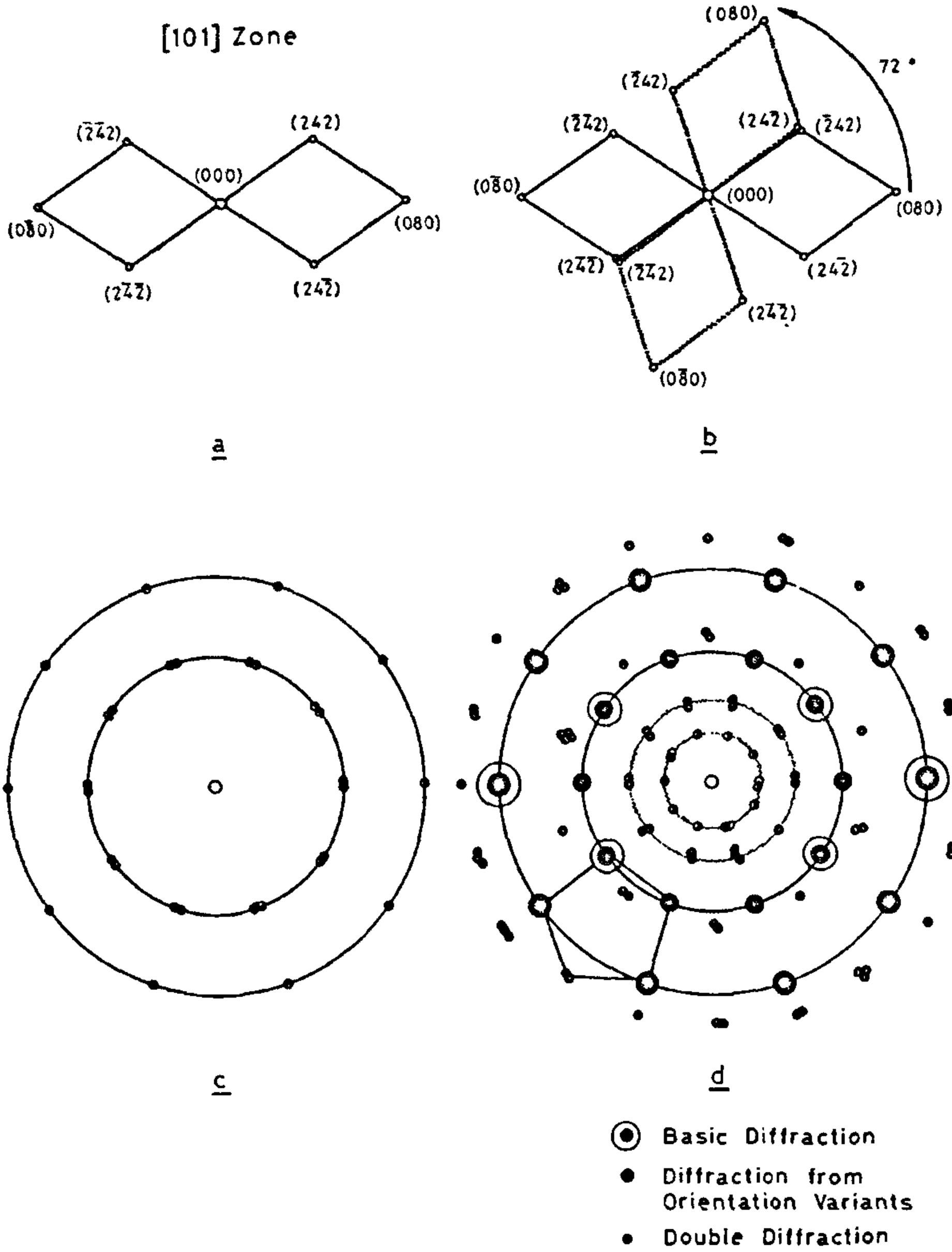


Figure 4. Computer-simulated electron diffraction pattern (d) consisting of basic diffraction, orientation variant diffraction and dynamic double diffraction spots for i-Al–Mn phase. The starting point here (a) is the single crystal pattern from the [101] zone. Two orientation variants and all five orientation variants lead to patterns b and c respectively, ignoring double diffraction.

and Lloyd[4] with regard to the i-Al–Li–Cu phase. The starting point in this rather striking demonstration is the single crystal diffraction pattern (figure 4a), as computer-simulated for the [101] zone of the orthorhombic i-Al–Mn phase. Starting with just *two* families of reflections, viz. {080} and {242}, the highly impressive and by now famous five-fold pattern emerges (figure 4d), with *dozens* of reflections, some due to orientation variants but *most* due to double diffraction. Because the interplanar distances of the permitted 242-reflection and the forbidden 050-reflection, viz. 0.334 and 0.331 nm respectively, are *very close* to each other, the so-called Fibonacci sequence of reflections, viz. (020), (030), (050), (080), and (0.13.0), appears fortuitously because of double diffraction, conjuring thereby illusory visions of quasiperiodicity in atomic arrangements for these phases.

Further implications of the present model and the explanation for many other observations related to icosahedral and decagonal phases will be discussed elsewhere.

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