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FROM FRANK-KASPER PHASES TO THE ICOSAHEDRAL QUASICRYSTAL

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Abstract – This is a resume of works carried out recently on the structure of icosahedral quasicrystal. Part I deals with a microdomain structure of several pentagonal Frank-Kasper phases displaying 5 symmetry but no ordinary translation order, a structure quite close to the two dimensional quasicrystal. Part II describes how a new icosahedral phase with the point symmetry $m\bar{3}5$ was found in NiTiV alloys by rapid solidification of alloys with a structure of or akin to a pentagonal Frank-Kasper phase. Part III discusses the structure models of the two- and three-dimensional quasicrystals derived from their close structure relationship, especially the rhombohedral $MgCu_2$ unit, to the pentagonal Frank-Kasper phases.

I – FIVEFOLD DIFFRACTION PATTERNS OF PENTAGONAL FRANK-KASPER PHASES

In discussing the structure of liquids, later extended also to glasses, Frank/1/ considered the case of the icosahedral clusters with 12 atoms surrounding a central one as its first coordination shell. Instead of the fcc and hcp packings, the icosahedral arrangement was proved to have a significant lower energy, at least for simple Lennard-Jones pair potentials. Not only being highly symmetrical with a point symmetry $m\bar{3}5$ (see Fig.5d), the icosahedron composed of 20 tetrahedra has only tetrahedral interstices while the cuboctahedron (fcc) and anticuboctahedron (hcp) have tetrahedral as well as octahedral ones. Later Frank and Kasper/2,3/ studied the building principles of the intermetallic compounds of tetrahedrally close-packed structures which were called since then Frank-Kasper (FK) phases. It was shown that the pentagonal FK phases consist mainly of layers of juxtaposed icosahedra of the same or similar orientation. For example, in a unit cell of Cu_4Cd_3 with 1124 atoms, there are no less than 568 icosahedra in it/4/. Pentagonal FK phases are common in transition metal systems, such as the μ , C, Laves phases, etc/5-7/. Fig.1 is the (110) projection of the structure model of the C14 Laves phase with a nonbasal fault. Both perfect and faulty regions consist of pentagon-triangle sheet at height of 0 and $\frac{1}{2}$ (pentagons superposed antisymmetrically) as the primary or main layers. The atoms at $\pm\frac{1}{4}$ are located inside the pentagonal antiprisms forming the secondary layers. These pentagonal antiprisms together with the atoms inside them form in turn parallel chains of interlocked icosahedra. At the (111) domain boundary a single slab of the μ structure is formed, which, together with the C14 Laves structure on both sides of it, forms a narrow slab of the C phase/7/. These intergrown structures have two most prominent features. First, the match at the interphase boundary is good and this explains why these phases can intergrow easily. Second, the icosahedral chains in these phases and also in the interphase boundaries have the same orientation.

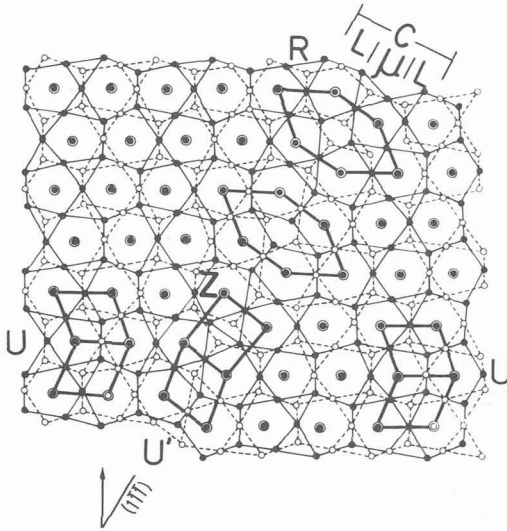


Fig.1 Structure model of the C14 Laves (L) with a μ or C slab at the $(1\bar{1}\bar{1})$ domain boundary. All these phases are composed of icosahedral chains with the same orientation. The basic building blocks are the outlined MgCu_2 (U and U'), Zr_4Al_3 (Z) or their compound (R) units.

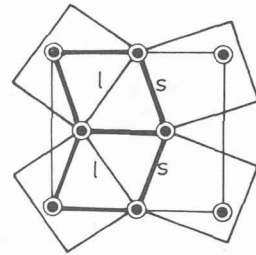


Fig.2 Schematic diagram shows the growing of differently oriented Zr_4Al_3 (or μ or C) units on a C14 Laves unit (thick lines). Only the centers of the icosahedral chains are shown.

Thus, the structures of various FK phases are characterized by the secondary layers whose atoms represent the centers of these pentagonal antiprisms. Fig.2 shows the various configurations of interfacing a rectangular Zr_4Al_3 unit on the C14 Laves unit which itself consists of two C15 MgCu_2 parallelogram units in twin position/8/. This is possible because the short (s) and long (ℓ) sides of the Zr_4Al_3 rectangle are of about the same lengths as the sides (s) and diagonal (ℓ) of the MgCu_2 parallelogram. Since both the structures of the μ and C phases are composed of these units, Fig.2 represents also the various possible ways of formation of these phases on the existing C14 Laves phase unit.

Fig.3 is the high resolution image of microdomains of these pentagonal FK structures. Computer image simulations of the μ , C14 Laves and C phases as well as the $(1\bar{1}\bar{1})$ fault model of the C14 Laves phase all confirmed a one-to-one correspondence between the bright dots in the high resolution image and the tunnels inside the icosahedral chains (or the atoms on the secondary layers). The arrangements of these icosahedral chains in different domains can thus be read directly from the observed image. In Fig.3 at least two rotation domains of the C14 Laves (labeled L' and L''), two rotation domains of the C15 Laves or the MgCu_2 type of structure (labeled U and U') and several C (C), μ (μ), Zr_4Al_3 (Z) domains can be recognized. These domains intergrow intimately, and in some cases microdomains only a few unit cells in width exist. However, no matter how complex or faulty the structure is, the underlying building block is the same, i.e. the icosahedral chains of the same orientation, and their arrangement is fairly simple, being either rectangular, rhombic or a combination.

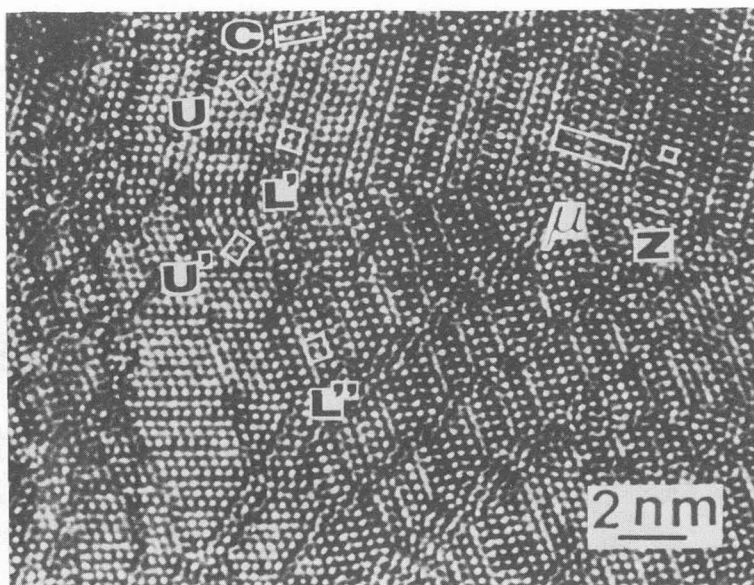


Fig.3 Structural image of an aggregate of various $Zr_4Al_3(Z)$, C14 Laves (L), C15 Laves (U), μ and C microdomains in intimate intergrowth/8/.

Figs.4a-4c are the EDPs of the μ , C14 Laves and C phases, respectively, taken with the incident beam parallel to the juxtaposed icosahedral chains. The distribution of diffraction spots is a representation in the reciprocal space of the space lattices in the real space. Since these phases have different space lattices (μ : rhombohedral; C14: hexagonal; C: monoclinic), it is natural that their EDPs look quite different. However, the outermost ten spots in them (marked with arrows) show a five fold distribution, and those strong ones all occur at about 8 nm^{-1} , though their indices are quite different. This is indeed quite unexpected. What is more striking is the EDP of a heavily faulted specimen containing many microdomains of these FK phases as shown in Fig. 4d. A crossgrid of spots corresponding to a reciprocal plane no longer can be detected, but the fivefold symmetry now becomes more obvious with spots located on concentric circles with the outermost ten spots at about 8 nm^{-1} , though much stronger now, remaining at the same positions as the corresponding ones in Figs.4a-4c. This gives a clue for solving this "anomaly" by superposing EDPs from four differently oriented C and three C14 Laves phases as shown in Fig.4e. The resemblance between Figs.4d and 4e is remarkable, and this proves that the former is indeed the EDP of an aggregate of FK phases with many C and C14 domains whose orientations follow the intergrowth requirements shown in Fig.2. One can see from Fig.3 that these domains in general are only a few nm in size, and this is a kind of "ultrafine grain" structure where long-range order is limited to less than ten unit-cells. Nevertheless, that such a "mosaic" structure can still give a quite clear fivefold diffraction symmetry is astonishing, and this must result from the icosahedral chains of the same orientation in these FK phases. To extend this argument one step further, the Fourier transform of an icosahedron was calculated and is shown in Fig.4f. Both the diffuse halos and fivefold maxima agree well with the EDPs shown in Figs.4d and 4e and this proves beyond any doubt that the fivefold distribution of electron diffraction spots of the pentagonal FK phases comes from the

pentagons and that they are ten in number from the two antisymmetrically superposed pentagons or from the icosahedron.

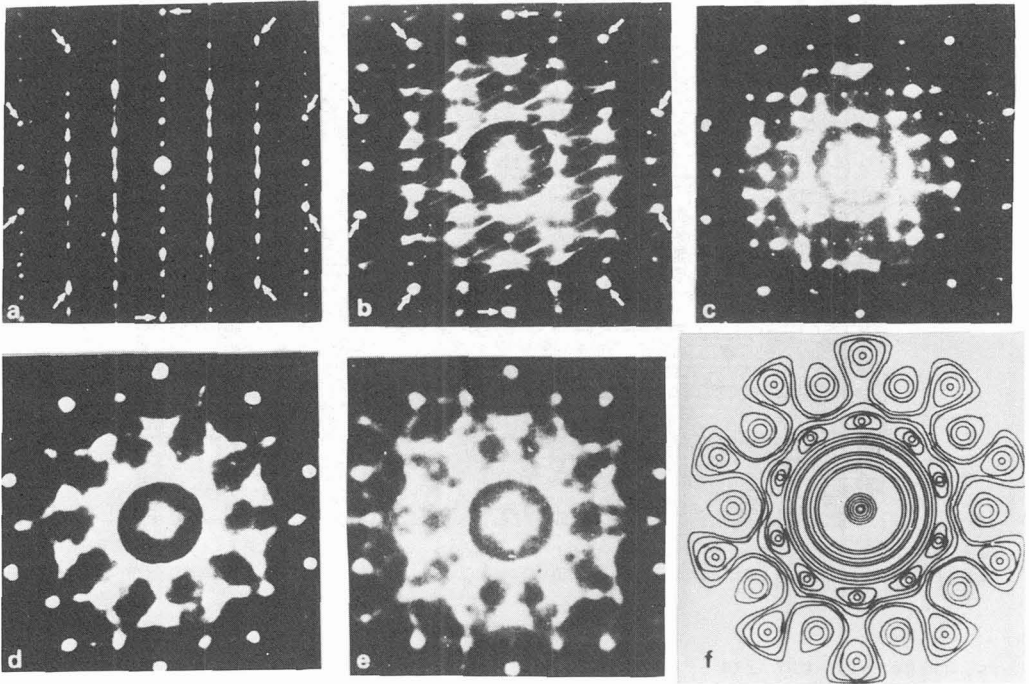


Fig.4 EDPs of pentagonal FK structures with fivefold symmetry: (a) μ : outermost ten spots are marked with arrows; (b) C14 Laves: outermost ten spots are marked; (c) C: fivefold symmetry becomes clearer; (d) mosaic structure with many microdomains: only fivefold distributed diffuse spots appear; (e) composite EDP of differently oriented C and C14 Laves phases: similar to (d); (f) Fourier transform of a single pentagonal antiprism: diffuse halos and fivefold diffuse maxima resembling (d) and (e).

From the above discussion it becomes clear that the orientation order of icosahedral chains is manifested by the fivefold symmetry of the EDP and their lacking of long range translation order in a plane normal to the icosahedral chains by the absence of a reciprocal plane lattice in Fig.4d. This is a structure rather close to that of a two dimensional quasicrystal. As regards to other aperiodic structures with fivefold symmetry, see a recent review by van de Waal/9/.

II - A NEW ICOSAHEDRAL PHASE WITH $m\bar{3}5$ SYMMETRY

Recently Shechtman, Blech, Gratias and Cahn/10/ reported a metastable phase with a composition Al-14 at%Mn having only the point group symmetry $m\bar{3}5$ but no ordinary translation symmetry. They called it an icosahedral phase. In rapidly quenched $(Ti_{1-x}V_x)_2Ni$ alloys ($x=0.0-0.3$) we have found a similar phase/11/. The presence of such an icosahedral phase was verified by selected-area electron diffraction and the EDPs (Fig.5) as well as the angles among the five-, three- and twofold axes are in agreement with those reported by Shechtman et al.

In order to show the characteristics of this kind of icosahedral structure, HREM examinations were made. Figure 6 is such an image taken along the fivefold axis and processed optically in a Fourier transform apparatus. It is obvious that 10 bright dots, lying on a circle with 5 symmetry, occur abundantly. Some of these sets of 10 bright dots are marked with small black dots, others are marked with white stars in their centres. It should be noted that in some cases the 10 bright dots are not regularly distributed and this may serve as an indication of distortions existing in the icosahedra. Although these bright dots more or

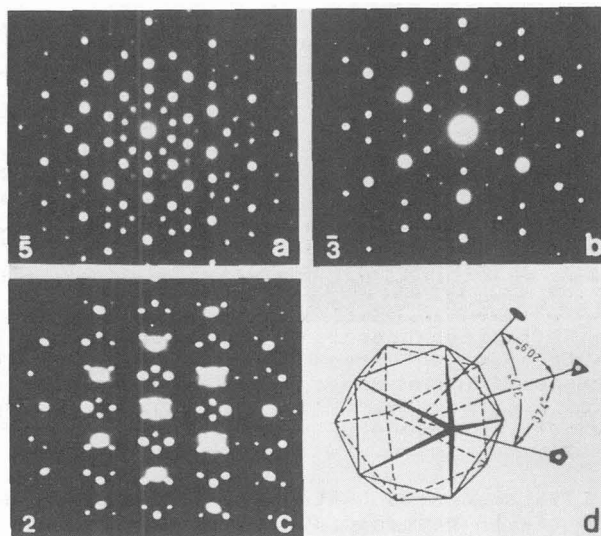


Fig.5 EDPs of the $(Ti_{0.9}V_{0.1})_2Ni$ icosahedral phase displaying (a)5, (b)3, and (c)2 fold symmetries complying with (d) the $I_h \bar{3} 2 5$ point group of an icosahedron.

less lie on parallel lines, as shown by the arrows in Fig.6, no periodicity could be found. An optical diffraction pattern has been obtained from the original HREM image, shown as an inset in Fig.6, and again it shows the fivefold symmetry. No two-dimensional reciprocal lattice can be derived from it. The inner ring of 10 spots of the optical diffraction pattern corresponds to 0.38 nm while that in the EDP (second ring of 10 spots in Fig.5a) corresponds to 0.36 nm. From these observations it seems that this icosahedral phase has a long-range orientation order of fivefold symmetry but no ordinary translation symmetry.

However, a detailed examination of Fig.6 will show that the bright dots are arranged as a two-dimensional Penrose pattern/12/ or rather on a Penrose lattice. Figure 7 is a portion of the two-dimensional Penrose pattern consisting of two kinds of rhombus with angles 72° and 144° . Mackay was the first to call this pattern a quasi-lattice/13,14/. Filled circles have been placed on some quasi-lattice points of this two-dimensional pattern to highlight the local fivefold symmetry (A in Fig.7). It is interesting to note that the bright dots, marked by small black dots around A in Fig.6, agree exactly with the solid circles around A in this pattern.

In this case the rings of 10 bright dots or solid circles touch each other, having two dots in common. There are other cases where the rings

of 10 dots are interlocked, as a function of the golden-section hierarchy of the local pattern examined and as schematically shown by open circles around B in Fig.7. However, neglecting local variation in the contrast, five such interlocked rings displaying a local fivefold symmetry have in fact been observed around B in Fig.6. In this case there is a five-member ring of bright dots around the fivefold axis marked with a black star.

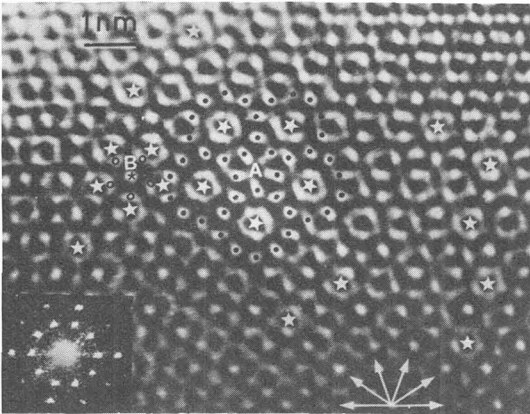


Fig.6 HREM of the NiTiV icosahedral phase showing the frequent occurrence of 10-member rings of bright spots with white stars at their centres. Local fivefold symmetry at A and B (compare with Fig.7).

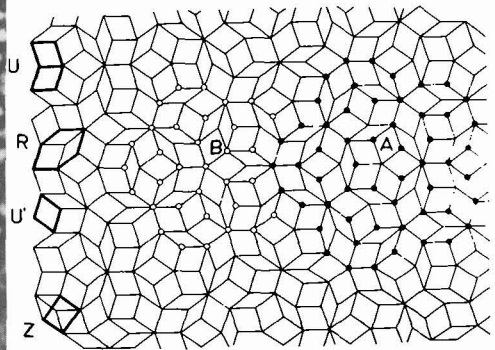


Fig.7 A portion of the Penrose lattice. Solid and open circles show the 10-member rings in regions A and B respectively (compare with Fig.6). U, Z and R units are outlined (see Fig.1).

In some EDPs of NiTiV specimens heated to 400°C an even distribution of diffraction spots with commensurate spacings along one or several directions has also been observed [16]. Figures 8a and 8b show such a phenomenon in one and two directions (marked with arrows, compare with Fig.5b) and Fig.8c shows such a regular distribution of spots in all three directions that it is quite similar to a three- or sixfold EDP of a crystalline phase. In other words, the original $\bar{3}$ symmetry has been modified and therefore these EDPs are called pseudo threefold patterns.

In Fig.9a, only the spots in one of the five directions of this fivefold EDP have nearly the same spacing (marked with arrows) and the inner ten-member ring of strong diffraction spots in Fig.5a becomes now an ellipse with the long axis in the same direction as the regularly distributed spots. Owing to this "distortion", the spots on other twofold axes no longer lie on straight lines. However, pentagons, though somewhat distorted, can still be easily recognized, though there are many extra spots either due to the effect of multiple diffraction or to the modification of the icosahedral structure. Similar situation has also been found in the twofold EDPs. Tilting experiments show that the angular relationship among the pseudo 2, $\bar{3}$ and $\bar{5}$ axes are in good agreement with that dictated by the point group $m\bar{3}5$. This indicates that in the main this structure is icosahedral, but some modification has to be introduced.

A one-dimensional regular distribution of electron spots implies some kind of one dimensional translation order in the real space. For this reason a two-dimensional Penrose lattice is drawn with a number of nodes displaced in the direction of one of the twofold axes to give a succession of three equal spacings, some of these are shown by dotted lines in Fig.9b. In other words, the original Fibonacci sequence with

at most only two equal spacings in succession has been slightly modified. The optical diffraction pattern is shown in Fig.9c with an evenly distributed spots (marked with arrows) in one direction only. They are in identical position as those in Fig.9a and, moreover, the ten-member ring of strong spots, analogous to that in Fig.9a, also becomes an ellipse. This is, in fact, a structure with many one-dimensional microdomains or clusters existing in an icosahedral matrix.

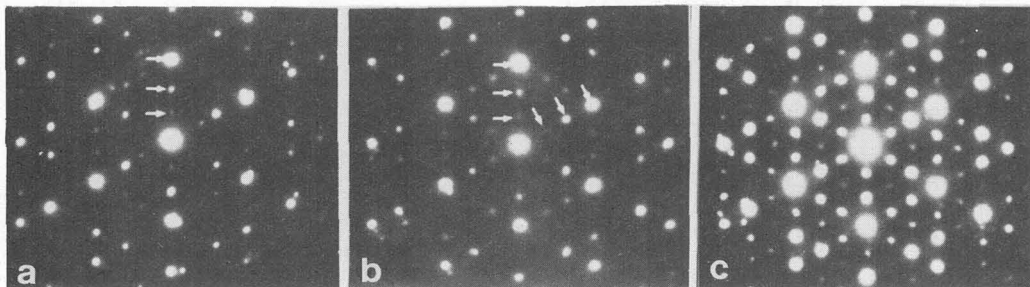


Fig.8 Pseudo-threefold EDPs of the transition structure with an evenly distributed diffraction spots in one (a), two (b) and three (c) directions.

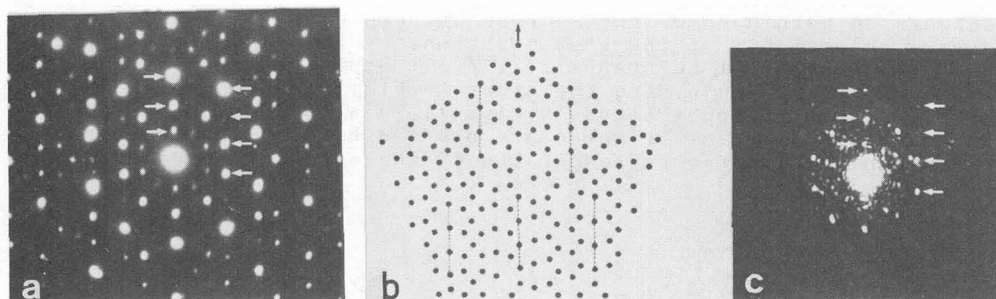


Fig.9 Pseudo-fivefold EDP of the transition structure (a) with an evenly distributed diffraction spots in one of the five twofold directions. (b) shows the structure model based on the Penrose lattice with some three successive equal spacings (dotted lines) and (c) its optical diffraction pattern quite similar to (a).

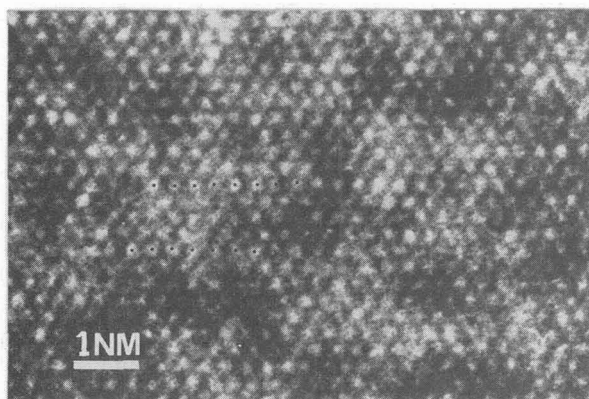


Fig.10 High resolution image along the $\bar{3}$ axis of the icosahedral phase showing some regular arrangement of bright dots.

By mainly electron diffraction analysis, we have presented above evidences of the existence of translation order of a few nm in the icosahedral phase.

hedral phase. Only the Penrose lattice has been slightly modified as in Fig.9b and nothing about the atom positions has been touched. This is a rather naive model of the transition structure. How can this translation order be accommodated to the non-crystallographic icosahedral symmetry? How far can an icosahedral structure tolerate this kind of translation order before a break between them takes place? Will this transition stage be an embryo of the formation of the equilibrium NiTi_2 phase? All these have to be answered in order to get a somewhat detailed picture of the transition of the icosahedral phase to an equilibrium one. We have also studied this by means of HREM and the existence of translation order in the icosahedral phase can clearly be seen in Fig.10.

III - STRUCTURE MODELS OF ICOSAHEDRAL QUASICRYSTALS

Looking at Figs.1 and 7 carefully, one will find, to his great excitement, that a one-to-one correspondence exists in many places between the pentagonal FK phases, such as the C14 Laves phase in Fig.1, and the icosahedral phase in Fig.7. For example, the MgCu_2 units U and U' as well as the compound unit R outlined both in Figs.1 and 7. This, and also the same distance of about 0.4 nm between two nearest image points or two icosahedra sharing a face, leads us to the conclusion that the image points of the icosahedral phase along the $\bar{5}$ axis or the nodes of the two-dimensional Penrose lattice, shown schematically in Fig.7, could be the centres of icosahedra. Figure 11 is the result of placing icosahedra on each node of the Penrose pattern with some slight but necessary adjustments of the atom positions by reference to the structure of the pentagonal FK phases/16,17/. It is interesting to notice that the dashed lines joining the atoms in Fig.11 reproduce exactly the original Penrose tiling (Penrose's Fig.4/12/) with pentagons, diamonds, boats and stars (the latter three are denoted by d, b and s, respectively, in Fig.11). The two representations, the original Penrose tiling

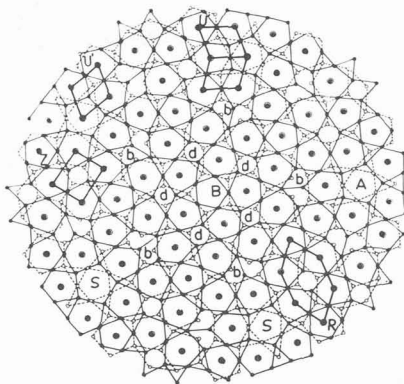


Fig.11 A structure model of the 2D quasicrystal with the icosahedra somewhat distorted and placed in juxtaposition as in the pentagonal FK phases on the lattice points of the Penrose lattice (Fig.7). Dashed lines are the Penrose tiling with pentagons, diamonds (d), boats (b) and stars (s).

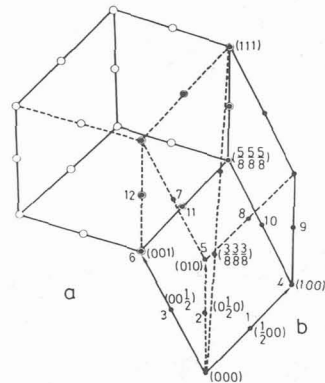


Fig.12 A structure model of the 3D icosahedral phase showing the obtuse (a) and acute (b) rhombohedra having a common interface, atoms on which are shown by double circles, in the triacontahedron. The acute rhombohedron ($\alpha=63.43^\circ$) is a slightly distorted rhombohedral unit-cell of the fcc MgCu_2 structure ($\alpha=60^\circ$).

of mainly pentagons/12/ and that drawn by Mackay using two rhombi/14/, are equivalent if the icosahedral content is added to the skeleton of the two-dimensional quasi-lattice.

Turning now to the structure model of the three-dimensional icosahedral phase, Mackay/14/, following the suggestion of Robert Ammann, pointed out the possibility of filling the three dimensional space with the triacontahedral units consisting of 10 acute ($\alpha=63.43^\circ$) and 10 obtuse ($\alpha=116.57^\circ$) rhombohedra with icosahedral symmetry (Fig.12). The acute rhombohedron (Fig.12b) is in fact a somewhat distorted rhombohedral unit cell of the fcc $\text{Cl}_5 \text{MgCu}_2$ Laves phase ($\alpha=60^\circ$) and its projection along one of its edges yields the thick rhombus unit in the two-dimensional Penrose lattice (Fig.7). For a cubic MgCu_2 -type FK phase such as TaCo_2 , $a=0.673 \text{ nm}$, the rhombohedron edge length is $\sqrt{2}a/2 = 0.476 \text{ nm}$, in good agreement with the 0.46 nm obtained by Elser/18/ for the icosahedral Al_6Mn phase from its electron diffraction pattern. According to the structure of the $\text{Cl}_5 \text{MgCu}_2$ Laves phase, atoms are present not only at the lattice vertices of this rhombohedral unit cell but also at the midpoints of the cell edge and along the body diagonal. Therefore, the atom positions in the 63.43° acute rhombohedron can be fixed in a similar way, as those represented by the filled circles in Fig.12b. The structure of pentagonal FK phases can be considered as the result of applying such symmetry operations as twinning, rotation, inversion, etc. to the MgCu_2 unit, as suggested by Andersson/19/. After doing the same thing with the rhombus in Fig.7 or the acute rhombohedron in Fig.12b of the icosahedral phase, empty space will be created in these structures. In the two dimensional case, this is the thin rhombus of Penrose tiling (Fig.7). In the three dimensional case, obtuse rhombohedra have to be used to fill the empty space left by the acute rhombohedra. Consequently the acute and obtuse rhombohedra must have a face in common as shown in Fig.12 and therefore the atom positions in the obtuse rhombohedron are fixed automatically as shown by the open circles in Fig.12a if one only remembers that it is too thin to allow any atom located inside of it.

Since the MgCu_2 - type structure is known to consist of the Z16 Kasper polyhedron, also known as Friauf-Laves polyhedron, there must be a somewhat distorted one in this structure model. Assuming the rhombohedral cell edge being 0.476 nm , the distances of the centre atom of this polyhedron to its 12 closest atoms at 1 to 12 (Fig.12b) are 0.277 (1 to 3), 0.288 (4 to 6) and 0.293 nm (7 to 12), respectively. They are comparable to the interatomic distances of 0.291 and 0.279 nm , respectively, for Ta-Ta and Ta-Co pairs in the equilibrium TaCo_2 phase. Moreover, the atomic volumes of the acute and obtuse rhombohedra are 0.0136 and 0.0126 nm^3 , respectively, while the average atomic volume in TaCo_2 is 0.0127 nm^3 .

It has been shown recently that the icosahedral lattice can be recovered by a cut-and-projection method from a 6-dimensional simple cube projected onto an irrational 3-dimensional hyperplane/18/. In order to obtain the icosahedral structure, however, the 6-dimensional simple cube has to have a structure too. Analogous to the extra atoms in the MgCu_2 rhombohedron in Fig.12b, there are also atoms at $\langle \frac{1}{2}0000 \rangle$, $\langle \frac{3}{8}\frac{3}{8}\frac{3}{8}000 \rangle$ and $\langle \frac{5}{8}\frac{5}{8}\frac{5}{8}000 \rangle$ in addition to the atoms at the vertices. In case of incommensurable projection, the icosahedral structure shown in Fig.12 can be obtained, while in case of commensurable projection, a pentagonal FK phase will be resulted. Taking the monoclinic FK phase Mg_4Zn_7 as an example, the lattice parameters thus obtained ($a:b:c = 1:0.547:0.201$, $\alpha = \beta = 90^\circ$, $\gamma = 102.8^\circ$) compare favourably with the experimental data ($a:b:c = 1:0.550:0.202$, $\alpha = \beta = 90^\circ$, $\gamma = 102.5^\circ$). The positions of 110 atoms in one unit cell calculated by this cut-and-projection method have an average deviation of only 3.7% from those determined by X-ray structure analysis. Altogether 10 pentagonal FK phases have been calculated by this method/17/ and the good agreement with

the experimental data may serve to prove the correctness of our structure model (see Table 1).

Table 1 Calculated lattice parameters of FK phases by the cut-and-projection method from a 6-dimensional $MgCu_2$ cube compared with experimental data/17/.

FK Phases	Calculated Experimental		Lattice Parameters		
	b/a	c/a	α	β	γ
$MgCu_2$	1	1	90°	90°	90°
	1	1	90°	90°	90°
$MgZn_2$	1	1.633	90°	90°	120°
	1	1.647	90°	90°	120°
$MgNi_2$	1	3.266	90°	90°	120°
	1	3.282	90°	90°	120°
Zr_4Al_3	1	1.018	90°	90°	120°
	1	0.992	90°	90°	120°
μ	1	5.526	89.43°	89.43°	120°
	1	5.433	90°	90°	120°
W_2FeSi	0.8506	0.5164	90°	90°	90°
	0.8421	0.5122	90°	90°	90°
M	0.5878	0.3036	90°	90°	90°
	0.5721	0.3033	90°	90°	90°
$(Co_{0.57}Si_{0.43})_3V_2$	0.4425	0.2688	90°	90°	98.40°
	0.4397	0.2714	90°	90°	99.2°
X-phase	0.7987	0.2996	90°	90°	90°
	0.8032	0.3074	90°	90°	90°
Mg_4Zn_7	0.5470	0.2012	90°	90°	102.8°
	0.5500	0.2019	90°	90°	102.5°

IV - SUMMARY

From the above description of the discovery of a new icosahedral phase in NiTiV alloys and the derivation of structure model of two- and the three-dimensional quasicrystals, it becomes quite obvious how much we owe Sir Charles Frank for his ingenious and farsighted idea of the icosahedral clusters and also his pioneer work on the structure of FK phases. This resume touches only the latter point, but before ending this paper it is perhaps not out of place to say a few words about the former. Fig.13 is a high resolution image of amorphous silicon which has been heated up to $700^\circ C/20/$. Besides fivefold twins, (B in Fig.13), some icosahedral clusters have also been observed (marked with arrows, see the decagon of 10 bright spots surrounding an inner bright ring at A in Fig.13). This is quite similar in appearance to that found recently in the icosahedral phase of $Al_6Mn/21/$ and $Ti_2Ni/11/$ (compare with Fig.14), and to the calculated image of the icosahedral phase by Portier et al./21/. However, this configuration in Si, unlike the extended formation in alloys, occurred only in isolated cases. Therefore it is called icosahedral clusters. The occurrence of icosahedral clusters, like the amorphon anticipated by many investigations before, in an element such as Si will be of both theoretical as well as practical interest and this certainly is warranted of further studies. This proves that the transition structure between the amorphous and crystalline states is an exciting and fruitful field of research for solid state scientists.

Figure 13 may also serve to show that both multiple twins and the ico-

icosahedral phase may exist and they can clearly be identified by HREM. In other words, they can coexist and need not to dispel each other, as Pauling/22/ claimed in his recent article "Apparent icosahedral symmetry is due to directed multiple twinning of cubic crystals".

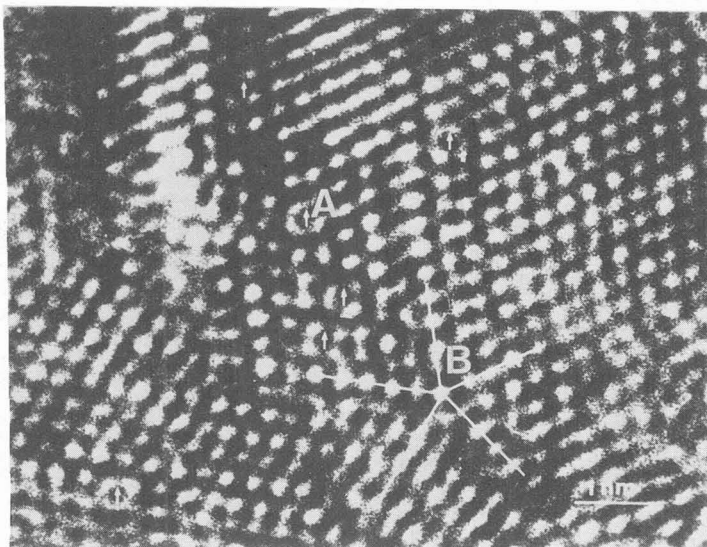


Fig.13 High resolution image of amorphous Si heated to 700°C showing both fivefold twins (B) and icosahedral clusters (A and others marked with arrows).

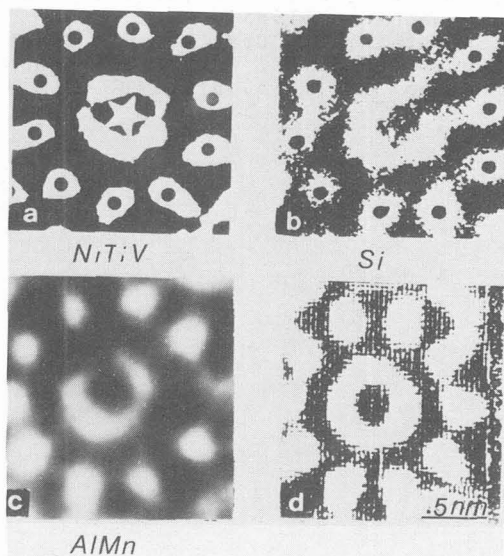


Fig.14 Comparison of the 10-member ring of bright spots surrounding an inner bright circle: (a) NiTiV /11/; (b) Si /20/; (c) Al₆Mn (kindly supplied by Mr. W. Liu); (d) simulated image /21/.

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