Preparation and Crystal Structure of the Pseudo-Decagonal Approximant \( \text{Mn}_3\text{Ga}_5 \)

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The crystal structure of tetragonal \( \text{Mn}_3\text{Ga}_5 \) has been determined from single-crystal X-ray diffraction data. The space group is \( P4_2/nmc \) with \( a = 12.659(2) \) Å and \( b = 24.616(6) \) Å. \( R_w = 1.50\% \). The structure is mainly made up of 38 atoms clusters isotypic to those found in \( \gamma \)-brass, Cu\(_5\)Zn\(_4\).

Key Words: quasicrystal; approximant; Mn–Ga; alloys; intermetallic compounds; \( \gamma \)-brass; crystal structure; synthesis.

INTRODUCTION

The binary Mn–Ga alloy system is rich in phases, several with large unit cells and unknown structures. It has won renewed interest due to its close chemical relationship to the Mn–Al system where quasicrystals were first discovered (1). A decagonal quasicrystal has also been identified in the Mn–Ga system (2). The large unit cell \( \text{Mn}_3\text{Ga}_5 \) phase was found by Meissner (3) and co-workers during an investigation of the Mn–Ga binary phase diagram. Wu and Kuo (2) studied this pseudo-decagonal \( \text{Mn}_3\text{Ga}_5 \) phase with electron diffraction and deduced the space group and cell parameters. Here an X-ray single-crystal structure determination and refinement of \( \text{Mn}_3\text{Ga}_5 \) is reported.

EXPERIMENTAL

Single crystals of the title compound with sizes up to 0.5 mm were produced using a liquid–solid state synthesis route. Slow steady crystal growth was achieved by the slow dissolution of manganese into liquid gallium. The reaction mixture contained 0.25 g of approximately \( 1 \times 1 \times 4 \) mm large manganese chips (99.9\%, ABCR) and 1.75 g of gallium (99.99999\%, ABCR). The reaction mixture was put into a quartz glass tube with a quartz wool filter mounted below. A support for pieces of quartz glass, as shown in Fig. 1. The tube was evacuated and sealed. It was then kept at 585°C for 40 hours inside a stainless steel cylinder, upright with the reaction mixture on the bottom and the filter and glass support in the top of the tube. The steel cylinder delayed the cooling of the sample by storing heat. Immediately after it had been taken out from the furnace, the steel cylinder with the quartz glass sample tube inside was turned upside down and centrifuged, whereby the gallium-rich melt was forced through the filter. Crystals of \( \text{Mn}_3\text{Ga}_5 \) were found, together with remaining unreacted pieces of manganese chips, on top of the quartz wool filter. Several freely lying platelike \( \text{Mn}_3\text{Ga}_5 \) crystals with well-exhibited tetragonal morphology were found. The crystals had a metallic luster.

Guiner–Hägg X-ray powder diffraction data were recorded on a film with monochromatic CrK\(_\alpha\) radiation in order to estimate the homogeneity of the sample and to refine the cell parameters. LaB\(_6\) (NIST, SRM 660) was used as an internal standard. Diffraction angles and intensities were measured by a film scanner (LS-20, KEJ Instruments). The cell parameters were refined with PIRUM (4). Besides the title compound the sample contained two impurity phases—cubic \( \text{MnGa}_3 \) (5) and tetragonal \( \text{Mn}_2\text{Ga}_5 \) (2). The impurity fractions were estimated with Rietveld methods to be 30\% and 1\% of the whole sample, respectively.

A rectangular crystal was selected and cut in smaller pieces for single-crystal X-ray diffraction data collection at ambient temperature with graphite-monochromated MoK\(_\alpha\) radiation on a STOE image plate diffractometer. A small crystal was used for the measurement in order to minimize extinction. The data were corrected for Lorentz, polarization, and absorption effects using equivalent reflections with the STOE X-RED (6) program.

The chemical composition of the crystal used for the X-ray experiment was analyzed with EDX on fresh-cut surfaces in a scanning electron microscope. According to this analysis the crystal contained 41 ± 3 atom-% Mn and 59 ± 3 atom-% Ga.

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Structure Determination and Refinement

The unit cell parameters refined from Guiner–Hägg data are $a = 12.659(2)$ Å, $c = 24.616(6)$ Å. The space group $P4_2/mmc$ was uniquely determined from the systematically absent reflections, consistent with the results of Wu and Kuo (3). The structure was solved by direct methods using SHELXS (7) and refined with JANA98 (8) in the space group $P4_2/mmc$ with the origin on the fourfold screw axis. The refined parameters were the scale factor, atomic coordinates, anisotropic temperature factors, mixed occupancy, and extinction. Due to the rather small difference in the scattering power of gallium and manganese, special care was taken to determine atom types. The metal–metal distances were distributed smoothly, and give no decisive information about the type of atoms involved in the bond. However, when the structure was refined with all atoms assigned to be Ga, two close but not overlapping distributions of temperature factors resulted. All atoms with temperature factors in the distribution of lower temperature factors were then changed to be Mn. With the exception of Mn29 this assignment turned out to give the lowest $R$-value in the refinement. Finally the partial occupancy of Ga and Mn was refined for all 29 sites with the restriction that the total occupancy for each site equals 1. Seven sites were found to have mixed composition, see Table 2. Mn29 had the largest fraction of the complementary element. Mn29 was also the only atom with an atomic displacement ellipsoid appreciably deviating from spherical shape.

Final atom coordinates are listed in Table 1. The structure contained two unique clusters of 38 atoms each, which were very similar, but not symmetry related. Evaluation of the similarity between these was done with the program ROTPLOT (Norrestam, Unpublished, Stockholm University, Department of Structural Chemistry) which minimized the root-mean-square deviation (rmsd) between the 38 atoms in these clusters.

**RESULTS AND DISCUSSION**

The unit cell contains 264 atoms on 29 unique positions. According to the refinement 61.4 atom-% of the cell content

<table>
<thead>
<tr>
<th>TABLE 1 Crystal Data and Intensity Collection for Mn$_3$Ga$_5$</th>
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<tbody>
<tr>
<td><strong>Crystal Data</strong></td>
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<td>Chemical formula</td>
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<td>Formula weight (g/mol)</td>
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<tr>
<td>Crystal system</td>
</tr>
<tr>
<td>Space group</td>
</tr>
<tr>
<td>$a$ (Å)</td>
</tr>
<tr>
<td>$c$ (Å)</td>
</tr>
<tr>
<td>$V$ (Å$^3$), Z</td>
</tr>
<tr>
<td>Density (calculated) (g/cm$^3$)</td>
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<tr>
<td>Crystal form</td>
</tr>
<tr>
<td>Crystal size (mm)</td>
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<tr>
<td>Color</td>
</tr>
<tr>
<td>Absorption coefficient (mm$^{-1}$)</td>
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<td>$T_{	ext{min}}$, $T_{	ext{max}}$</td>
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<td>$R_{	ext{int. all}}$</td>
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<tr>
<td>$-14 \rightarrow k \rightarrow 14$</td>
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<tr>
<td>$-27 \rightarrow l \rightarrow 28$</td>
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<tr>
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</tr>
<tr>
<td><strong>Refinement</strong></td>
</tr>
<tr>
<td>No. of reflections used in refinement</td>
</tr>
<tr>
<td>No. of parameters refined</td>
</tr>
<tr>
<td>Weighting scheme</td>
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<tr>
<td>$w = 1/\sigma^2(F)$</td>
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<tr>
<td>$S_{\text{all}}$, $S_{\text{all}}$</td>
</tr>
<tr>
<td>0.00283, 0.0150</td>
</tr>
<tr>
<td>$\Delta_{	ext{max}}^\alpha$, $\Delta_{	ext{max}}^\beta$</td>
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<td>7.94, -2.53</td>
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### Table 2
Positional and Thermal Parameters for Mn$_3$Ga$_5$

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<th>Atom</th>
<th>Wyckoff position</th>
<th>Occ.</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>$U_{eq}$ (Å$^2$)</th>
<th>Type*</th>
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<td>Ga1</td>
<td>8g</td>
<td>0.13(3)</td>
<td>0</td>
<td>0.808(1)</td>
<td>0.06714(9)</td>
<td>0.072(7)</td>
<td>r</td>
</tr>
<tr>
<td>Mn1</td>
<td>8g</td>
<td>0.87(3)</td>
<td>0</td>
<td>0.808322</td>
<td>0.067138</td>
<td>0.0727(7)</td>
<td>r</td>
</tr>
<tr>
<td>Ga2</td>
<td>8g</td>
<td>1</td>
<td>0</td>
<td>0.8150(1)</td>
<td>0.329536</td>
<td>0.0113(4)</td>
<td>f, m</td>
</tr>
<tr>
<td>Mn3</td>
<td>8g</td>
<td>1</td>
<td>0.8071(2)</td>
<td>0</td>
<td>0.20522(8)</td>
<td>0.0661(6)</td>
<td>f</td>
</tr>
<tr>
<td>Ga4</td>
<td>8f</td>
<td>1</td>
<td>0.87539(9)</td>
<td>0.87539(9)</td>
<td>0</td>
<td>0.0166(4)</td>
<td>r</td>
</tr>
<tr>
<td>Ga5</td>
<td>4c</td>
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<td>0</td>
<td>0.19700(9)</td>
<td>0.0130(7)</td>
<td>s</td>
<td></td>
</tr>
<tr>
<td>Ga6</td>
<td>16h</td>
<td>0.85(3)</td>
<td>0.69274(9)</td>
<td>0.86677(8)</td>
<td>0.29599(5)</td>
<td>0.0136(4)</td>
<td>f</td>
</tr>
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<td>Mn6</td>
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<td>0.15(3)</td>
<td>0.69274</td>
<td>0.86672</td>
<td>0.295988</td>
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<td>f</td>
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<tr>
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<td>4c</td>
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<td>0</td>
<td>0.07293(9)</td>
<td>0.0116(6)</td>
<td>r</td>
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<tr>
<td>Ga8</td>
<td>8g</td>
<td>1</td>
<td>0.6110(1)</td>
<td>0.22274(6)</td>
<td>0.0088(4)</td>
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<tr>
<td>Ga9</td>
<td>8g</td>
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<td>0.8710(1)</td>
<td>0.30026(6)</td>
<td>0.0106(5)</td>
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<td>Ga10</td>
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<td>0.82338(8)</td>
<td>0.12254(4)</td>
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<tr>
<td>Ga11</td>
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<td>1</td>
<td>0.64486(7)</td>
<td>0.82019(8)</td>
<td>0.07077(5)</td>
<td>0.0126(3)</td>
<td>f, m</td>
</tr>
<tr>
<td>Ga12</td>
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<td>1</td>
<td>0.6551(1)</td>
<td>0</td>
<td>0.37824(7)</td>
<td>0.0105(5)</td>
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<tr>
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<td>8g</td>
<td>1</td>
<td>0.6561(1)</td>
<td>0</td>
<td>0.3735(1)</td>
<td>0.071(7)</td>
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<td>0.13(5)</td>
<td>0</td>
<td>0</td>
<td>0.373487</td>
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<tr>
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<td>0.87608(8)</td>
<td>0.82254(8)</td>
<td>0.23295(4)</td>
<td>0.0129(3)</td>
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<tr>
<td>Mn16</td>
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<td>1</td>
<td>0.6732(2)</td>
<td>0</td>
<td>0.119669</td>
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<tr>
<td>Ga17</td>
<td>8g</td>
<td>0.81(3)</td>
<td>0</td>
<td>0.6086(1)</td>
<td>0.355806</td>
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<td>f</td>
</tr>
<tr>
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<td>8g</td>
<td>0.19(3)</td>
<td>0</td>
<td>0.608607</td>
<td>0.355801</td>
<td>0.0055(6)</td>
<td>f</td>
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<td>Mn18</td>
<td>8g</td>
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<td>0.6732(2)</td>
<td>0</td>
<td>0.25197(9)</td>
<td>0.0059(6)</td>
<td>f</td>
</tr>
<tr>
<td>Mn19</td>
<td>8f</td>
<td>1</td>
<td>0.6753(1)</td>
<td>0.6753(1)</td>
<td>0</td>
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<td>m</td>
</tr>
<tr>
<td>Ga20</td>
<td>8g</td>
<td>1</td>
<td>0.8555(1)</td>
<td>0</td>
<td>0.458866</td>
<td>0.0094(4)</td>
<td>m</td>
</tr>
<tr>
<td>Mn21</td>
<td>4d</td>
<td>1</td>
<td>0.5</td>
<td>0</td>
<td>0.3123(1)</td>
<td>0.0063(9)</td>
<td>f, r</td>
</tr>
<tr>
<td>Ga22</td>
<td>16h</td>
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<td>0.83959(7)</td>
<td>0.69455(8)</td>
<td>0.06083(5)</td>
<td>0.0159(3)</td>
<td>r</td>
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<tr>
<td>Ga23</td>
<td>8g</td>
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<td>0.5</td>
<td>0.6693(1)</td>
<td>0.062529</td>
<td>0.0073(7)</td>
<td>m</td>
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<tr>
<td>Mn23</td>
<td>8g</td>
<td>0.79(3)</td>
<td>0.5</td>
<td>0.69294</td>
<td>0.062519</td>
<td>0.0073(7)</td>
<td>m</td>
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<td>Ga24</td>
<td>16h</td>
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<td>0.873119</td>
<td>0.383649</td>
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<td>Ga25</td>
<td>8g</td>
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<td>0</td>
<td>0.6941(2)</td>
<td>0.01627(7)</td>
<td>0.0237(6)</td>
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<tr>
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<td>16h</td>
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<td>0.68271(1)</td>
<td>0.81441(1)</td>
<td>0.18264(6)</td>
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<td>f</td>
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<td>Mn27</td>
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<td>1</td>
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<td>0</td>
<td>0.484188(8)</td>
<td>0.0092(6)</td>
<td>m, r</td>
</tr>
<tr>
<td>Mn28</td>
<td>4d</td>
<td>1</td>
<td>0.5</td>
<td>0.448001</td>
<td>0.0122(9)</td>
<td>f, r</td>
<td></td>
</tr>
<tr>
<td>Ga29</td>
<td>4d</td>
<td>0.39(5)</td>
<td>0</td>
<td>0</td>
<td>0.446115</td>
<td>0.033(1)</td>
<td>r</td>
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<tr>
<td>Mn29</td>
<td>4d</td>
<td>0.61(5)</td>
<td>0.5</td>
<td>0</td>
<td>0.446115</td>
<td>0.033(1)</td>
<td>r</td>
</tr>
</tbody>
</table>

* m = atoms in male clusters, f = atoms in female clusters, r = atoms in rods, s = single atoms.

is gallium. This is within experimental errors consistent with the EDX analysis. Due to possible uncertainties in the assignment of the elements in the refinement, we have chosen to continue to refer to this compound as Mn$_3$Ga$_5$, corresponding to 62.5 atom-% Ga.

Of the 264 atoms in the unit cell 252 belong to two types of structural elements. The largest of these structural units is isostructural with the 38 atom cluster found in c-brass, Cu$_5$Zn$_8$ (9). The c-brass cluster has the shape of a tetrahedron stellated with icosahedra, see Fig. 2. There are atoms in the middle of the icosahedra but the central tetrahedron is empty. Complete c-brass clusters account for 74% of the Mn$_3$Ga$_5$ unit cell content. The other structural element has the shape of a rod as shown in Fig. 3. The rod consists of one cube, two Archimedean antiprisms, and an elongated semicapped cube octahedron, which are all filled. A similar structural element is found in Cr$_{23}$C$_6$ (10, 11), see Fig. 3, consisting of one empty cube, two filled Archimedean antiprisms, and a filled cube octahedron in the same sequence as in Mn$_3$Ga$_5$.

In Cu$_5$Zn$_8$ the whole structure is made up of c-brass clusters located at the lattice points of a body-centered cubic cell. Every cluster in that structure is connected by sharing three atoms with adjacent clusters in two different ways. To discern between these two different ways of attachment, we here introduce the terms male connection and female connection. A c-brass cluster has a male connection when the three shared atoms belong to the same icosahedron. A cluster has conversely a female connection if the three common atoms belong to three different icosahedra of that cluster.
The \( \gamma \)-brass-type clusters in \( \text{Mn}_3\text{Ga}_5 \) appear at two different symmetry-unrelated positions. The two symmetry-unrelated clusters are highly similar. Between the 38 atoms in these clusters the rmsd is only 0.25 Å, and they are thus only slightly distorted variants of each other. The more symmetrical cluster at (1/2, 1/2, 0) contains eight unique atoms. It has only male connections and will therefore here be referred to as the *male cluster*. The less symmetrical cluster at (0, 1/2, 0.184) contains 14 unique atoms. It has only female connections and will therefore here be referred to as the *female cluster*. There are two male and four female clusters in the unit cell. Every male cluster is connected to four adjacent female clusters. The female clusters are each connected to two adjacent male clusters. No atoms are shared between symmetry-related clusters. In \( \text{Mn}_3\text{Ga}_5 \) this way of linkage results in a slab of a two-dimensional infinite network of \( \gamma \)-brass clusters. Perpendicular to the \( c \) axis, this infinite network is of diamond type. There are two slabs of this kind in the unit cell; at \( c = 1/4 \) and at \( c = 3/4 \), as shown in Fig. 4. The two slabs share no atoms and are rotated 90° with respect to each other. On the elucidation of the structural relationship with a true three-dimensional diamond network, it is found that even if the \( \gamma \)-brass slabs in the unit cell of \( \text{Mn}_3\text{Ga}_5 \) were not rotated with respect to each other, they would still not build up a three-dimensional diamond network. This is because the female clusters would still be 4 Å too far from each other to superimpose along the \( c \) axis.

All links between the \( \gamma \)-brass-type clusters in \( \text{Mn}_3\text{Ga}_5 \) are parallel to the (100) and (010) planes, as illustrated in Fig. 5.

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**FIG. 2.** (a) Arrangement of \( \gamma \)-brass-type clusters in \( \text{Mn}_3\text{Ga}_5 \). The central male (unhatched) cluster is connected to four female clusters (hatched in the figure). Black spheres are Mn. White spheres are Ga. (b) One unit cell of \( \text{Cu}_5\text{Zn}_8 \) with half of the clusters removed. The locations of the removed clusters are indicated by tetrahedra. This part of the \( \gamma \)-brass structure is isostructural to the part of \( \text{Mn}_3\text{Ga}_5 \) shown in Fig. 2a. Black spheres are Zn. White spheres are Cu.

**FIG. 3.** (a) Rod in \( \text{Mn}_3\text{Ga}_5 \) built up by one cube, two Archimedean antiprisms, and one elongated capped cube. The length of the cell axis is indicated. The capping atoms are shown as free atoms. Black spheres are Mn. White spheres are Ga. (b) Rod in \( \text{Cr}_2\text{S}_3 \). The free capping atoms are C; all others are Cr.
FIG. 4. Locations of the \( \gamma \)-brass-type clusters in the unit cell of Mn\(_3\)Ga\(_5\). Hatched clusters are female and unhatched are male. All male clusters have four adjacent female clusters, and all female clusters have two adjacent male clusters.

This linking is isostructural with the diagonal links parallel to \((110)\) in the \( \gamma \)-brass structure shown in Fig. 2b. This isotypism is expressed in the only 1% difference of the lengths of the surface diagonal of the cubic unit cell in Cu\(_5\)Zn\(_8\) and the \( a \) axis of Mn\(_3\)Ga\(_5\).

The second structural unit, the rod, is built up in the same way as a structural element found in Cr\(_{23}\)C\(_6\) (10). In Cr\(_{23}\)C\(_6\) the perpendicular rods are linked by a cube octahedron. In Mn\(_3\)Ga\(_5\) two perpendicular rods share a face of the cube, so that one rod is below the other. The rods run parallel to the \( a \) and \( b \) axes in Mn\(_3\)Ga\(_5\), as shown in Fig. 5, and build up an infinite two-dimensional network. These networks at \( c = 0 \) and \( c = 1/2 \) fill up the space within the diamond-type layers and the slightly expanded space between them. The rods contain 42% of all the atoms in the unit cell. Half of these atoms are shared with the \( \gamma \)-brass-type clusters. The two rod slabs and the two \( \gamma \)-brass cluster slabs in the unit cell are sandwiched parallel in the order rod\(-\gamma \)-brass\(-\text{rod}\)-\(\gamma \)-brass.

The 12 atoms not yet accounted for are Ga5 on a 4\( c \) position and Ga10 on an 8g position. Ga10 can be considered as the sole atoms in two uncompleted \( \gamma \)-brass clusters adjacent to the male clusters. Ga5 caps the cube in the rod, above the surface opposite to the surface shared by two perpendicular rods.

Wu and Kuo (2) classified Mn\(_3\)Ga\(_5\) as an approximant to the decagonal quasicrystal (12) based on the pseudo-decagonal \([100]\) diffraction pattern and the presence of a decagonal quasicrystal with a similar composition. The crystal structure of Mn\(_3\)Ga\(_5\) reported here confirms this classification with its abundance of icosahedral local order, which is considered to be fundamental in the structures of quasicrystals.

An approximant should normally have a point group symmetry which is a crystallographic subgroup of the point group of the corresponding quasicrystal. Mn\(_3\)Ga\(_5\) has the point group \( 4/mmm \), but this is not a subgroup of \( 10/mmm \), which is the diffraction pattern symmetry of the decagonal quasicrystal. However, this does not have to disqualify the

FIG. 5. Location of the rods in the unit cell of Mn\(_3\)Ga\(_5\). The \( \gamma \)-brass clusters are removed for clarity, but their positions are indicated by tetrahedra and the links between them by solid and dashed lines. Dashed lines are behind the rods. Tetrahedra indicating female clusters are hatched.
classification of Mn₃Ga₅ as an approximant, since half of the cell with \( c' = 1/2c \) has only orthorhombic symmetry, with the lower point group symmetry \( mmm \), which is a subgroup of 10/\( mmm \). Thus the point group symmetry relation between Mn₃Ga₅ and the decagonal quasicrystal involves both a decrease and an increase in symmetry.

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