Electron diffraction study of octagonal-cubic phase transitions in Mn-Si-Al

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We report the observation of two kinds of octagonal-cubic transition to either the β-Mn-type structure or the Mn_3Si crystal following annealing Mn-Si-Al octagonal quasicrystal at different heating speeds. A theoretical analysis has been given and the simulated diffraction patterns match the experimental results well. It is confirmed that there exist two kinds of phason strain fields, which transform the octagonal phase into the two cubic crystalline phases, respectively. It is conjectured that the octagonal-cubic transitions feature the formation of local periodic structures.

Since the experimental discovery of icosahedral quasicrystals,[1] a number of different quasicrystals have been prepared and studied. Furthermore, many periodic structures closely related to quasicrystals have been discovered.[2] Because they have local atomic structures and chemical composition similar to the related quasicrystals, these kinds of materials are named the ''approximants'' of quasicrystals. In experiments, quasicrystalline phases often coexist with their approximant phases, and reversible transformations between quasicrystals and their approximants have been observed. For instance, it has been reported that the AlCuCoSi decagonal phase transforms to a microcrystalline state of a (5, 7) approximant at lower temperature, and vice versa.[3] In this case, there is not a smooth shift of diffraction peak positions between those of the approximant and those of the decagonal quasicrystal, but instead, there is a coexistence of the two sets of peaks with changes in relative intensities through the phase transition. On the other hand, in rapidly solidified AlMnSi and AlCrSi alloys intermediate structures during the transformation of a decagonal quasicrystal to tenfold twins of a crystalline structure have been observed.[4] Experimental electron diffraction patterns shows the continuous shift of spots from the quasicrystal to the *B*-centered orthorhombic phase through the intermediate stages.

Approximant structures can be described by the so-called ''strip-rotation'' projection scheme,[5] the projected strip in the higher-dimensional space is rotated away from the original one generating the quasiperiodic tiling so that the rotated strip is rational with respect to the higher-dimensional lattice. In general, the quasicrystal structure can be considered as the limit of sequence of periodic structures with the increasing unit cells of the approximants. Moreover, this kind of distortion of the system is induced by the phason strain field, the phason degrees of freedom associated with translations in the perpendicular space. Therefore the transformation from the quasicrystal to the approximant can be explained to be of phason-type.[6,7]

The two-dimensional octagonal quasicrystal is a metastable phase which was found in Cr-Ni-Si, V-Ni-Si, Mn-(Si, Al) alloy systems.[8–10] In a previous study, the symmetry of an octagonal quasicrystal was determined to be 8/mmm or 8/m.[11] Transmission electron microscopic (TEM) observations showed that 45° microtwin domains of β-Mn-type structure coexisted with the octagonal phase in Mn-Si-Al and Cr-Ni-Si alloys, indicating that these structures were closely related.[9] The process of the transition between the octagonal quasicrystal and the β-Mn-type structure crystal has been described by the linear phason strain field.[12,13] In the present article, the phase transformations in the octagonal ternary alloy of Mn_4(Si, Al) are studied in detail. The octagonal quasicrystals were heated by different heating speeds, which resulting two different kinds of octagonal-cubic phase transitions as observed by TEM.[14] Upon rapid heating, the octagonal phase transformed to the β-Mn-type structure, as reported in the Cr-Ni-Si octagonal alloy.[12,13] While slow annealing resulted in transforming the sample into the Mn_3Si crystalline phase. Our analysis confirmed that both of the octagonal-cubic phase transitions were of phason-type. In order to describe the process of the octagonal-approximant transition, distorted lattices in the higher-dimensional space are introduced. The advantage of this approach is that the reciprocal quasilattices of approximants can be given directly as well as the approximant tilings. From the tiling description, it is proposed that patches of areas formed with the identical local periodicity during the octagonal-cubic transition. By the increase of strength of the phason strain, the areas with this kind of local periodicity become so large to fill up the whole space, the final states are just the β-Mn-type structure and the Mn_3Si crystal, respectively.

The transition between quasiperiodic and periodic phases can be described by the one-parameter rotation in the higher-dimensional space.[15] Let Λ^4 denote a four-dimensional hypercubic lattice in the four-dimensional space E^4 with the basis $\mathbf{a}_i=(\mathbf{e},\mathbf{e}_i')$, $\mathbf{e}_i\in E$, $\mathbf{e}_i'\in E'$, $i=1,...,4$, and the reciprocal one $\mathbf{a}_i^*=(\mathbf{e}_i^*,\mathbf{e}_i'^*)$, $\mathbf{e}_i^*\in E$, $\mathbf{e}_i'^*\in E'$, $i=1,...,4$, respectively. Both the two-dimensional physical space E and the perpendicular space E' are the two unique invariant subspaces with respect to the point group $d_8=\langle\langle g_8,s\rangle\rangle$, where g_8 represents the eightfold rotational operator and s the reflection operator. Under an action of rotation on E^4, the subspaces E and E'

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are rotated to the subspaces $E(\phi)=R(\phi)E$ and $E'(\phi)=R(\phi)E'$, respectively, where $R(\phi)$ is the rotational operator with ϕ being the rotational angle. The absolute value of ϕ is related to the strength of the phason strain. The basis of Λ^4 can be rewritten as $\mathbf{a}_i=[\mathbf{e}_i(\phi),\mathbf{e}_i'(\phi)]$, $\mathbf{e}_i(\phi)\in E(\phi)$, $\mathbf{e}_i'(\phi)\in E'(\phi)$, $i=1,...,4$, and the reciprocal one $\mathbf{a}_i^*=[\mathbf{e}_i^*(\phi),\mathbf{e}_i'^*(\phi)]$, $\mathbf{e}_i^*(\phi)\in E(\phi)$, $\mathbf{e}_i'^*(\phi)\in E'(\phi)$, $i=1,...,4$. There are two families of rotations $R(\phi)$ in the four-dimensional space such that both of the rotated subspaces $E(\phi)$ and $E'(\phi)$ remain invariant with respect to either of the unique two subgroups of the octagonal point group with fourfold symmetry. One subgroup is $d_4'=\langle\langle g_4,s'\rangle\rangle$, where $g_4=g_8^2$ and $s'=g_8sg_8^{-1}$. The corresponding rotation $R(\phi)$ can be expressed by the matrix

$$R(\phi)=\begin{pmatrix} c & 0 & 0 & -s \\ 0 & c & s & 0 \\ 0 & -s & c & 0 \\ s & 0 & 0 & c \end{pmatrix}, \tag{1}$$

where $s=\sin\phi$ and $c=\cos\phi$. For another subgroup $d_4=\langle\langle g_4,s\rangle\rangle$, the rotational matrix $R(\phi)$ has the following form:[16]

$$R(\phi)=\begin{pmatrix} 0 & c & -s & 0 \\ c & 0 & 0 & -s \\ s & 0 & c & 0 \\ 0 & s & 0 & c \end{pmatrix}. \tag{2}$$

By analyzing the experiments illustrated below, it can be determined that the symmetry reduction from the point group d_8 into its subgroup d_4' corresponds to the transformation from the octagonal phase to the β-Mn-type structure. While the reduction to the subgroup d_4 refers to the octagonal-Mn_3Si crystal transition.

Instead of the strip-rotation projection scheme for the description of the quasicrystal-approximant transformation, an equivalent approach is proposed as follows. Let M^4 denote a distorted four-dimensional lattice in the four-dimensional space E^4 with the basis

$$\mathbf{b}_i=[\mathbf{g}_i,\mathbf{g}_i'(\phi)]=[\mathbf{e}_i,R^{-1}(\phi)\mathbf{e}_i'(\phi)/c], \quad i=1,...,4, \tag{3}$$

where $\mathbf{g}_i=\mathbf{e}_i\in E$ and $\mathbf{g}_i'(\phi)=R^{-1}(\phi)\mathbf{e}_i'(\phi)/c\in E'$. The reciprocal lattice M^* of M^4 can be given with the basis

$$\mathbf{b}_i^*=[\mathbf{g}_i^*(\phi),\mathbf{g}_i'^*]=[R^{-1}(\phi)\mathbf{e}_i^*(\phi)/c,\mathbf{e}_i'^*], \quad i=1,...,4, \tag{4}$$

where $\mathbf{g}_i^*(\phi)=R^{-1}(\phi)\mathbf{e}_i^*(\phi)/c\in E$ and $\mathbf{g}_i'^*=\mathbf{e}_i'^*\in E'$. It is obvious that the orthogonal relations $\mathbf{b}_i\cdot\mathbf{b}_j^*=\delta_{ij}$, $i,j=1,...,4$. are satisfied. In terms of distorted four-dimensional lattices M^4, a family of tilings of approximants with the change of ϕ can be obtained by the normal projection method, and furthermore the reciprocal quasilattices can be gained directly.

The projection of the basis of lattice M^4 onto the physical space $\mathbf{g}_i$, $i=1,...,4$, remains unchanged during the change of the angle ϕ, either in the case of the octagonal-β-Mn-type structure transition or the octagonal-Mn_3Si crystal transition. They are just the physical space components $\mathbf{e}_i$, $i=1,...,4$, of the basis of lattice Λ^4. Thus

$$\begin{gathered} \mathbf{g}_1=\alpha(1,0), \\ \mathbf{g}_2=\alpha(\alpha,\alpha), \\ \mathbf{g}_3=\alpha(0,1), \\ \mathbf{g}_4=\alpha(-\alpha,\alpha), \end{gathered} \tag{5}$$

where $\alpha=1/\sqrt{2}$. For the octagonal-β-Mn-type structure transition, the projection of the basis of lattice M^4 onto the perpendicular space $\mathbf{g}_i'(\phi)$, $i=1,...,4$, is written as follows:

$$\begin{gathered} \mathbf{g}_1'(\phi)=\alpha/c(c,s), \\ \mathbf{g}_2'(\phi)=\alpha/c[-\alpha(c+s),-\alpha(c-s)], \\ \mathbf{g}_3'(\phi)=\alpha/c(-s,c), \\ \mathbf{g}_4'(\phi)=\alpha/c[\alpha(c-s),-\alpha(c+s)]. \end{gathered} \tag{6}$$

In the case of the octagonal-Mn_3Si crystal transition, the perpendicular space components $\mathbf{g}_i'(\phi)$, $i=1,...,4$, of the basis of lattice M^4 are

$$\begin{gathered} \mathbf{g}_1'(\phi)=\alpha/c(c+s,0), \\ \mathbf{g}_2'(\phi)=\alpha/c[-\alpha(c-s),-\alpha(c-s)], \\ \mathbf{g}_3'(\phi)=\alpha/c(0,c+s), \\ \mathbf{g}_4'(\phi)=\alpha/c[\alpha(c-s),-\alpha(c-s)]. \end{gathered} \tag{7}$$

The projections of the basis of the reciprocal lattice M^* onto the physical and perpendicular space give both the vectors $\mathbf{g}_i^*(\phi)$ and $\mathbf{g}_i'^*$, $i=1,...,4$, respectively. The physical space components $\mathbf{g}_i^*(\phi)$, $i=1,...,4$, of the reciprocal lattice vectors constitute the basis of the reciprocal quasilattice of the approximant lattice.

For the octagonal-β-Mn-type structure transition, the physical space components $\mathbf{g}_i^*(\phi)$, $i=1,...,4$, of reciprocal lattice vectors is expressed as

$$\begin{gathered} \mathbf{g}_1^*(\phi)=\alpha/c(c,s), \\ \mathbf{g}_2^*(\phi)=\alpha/c[\alpha(c+s),\alpha(c-s)], \\ \mathbf{g}_3^*(\phi)=\alpha/c(-s,c), \\ \mathbf{g}_4^*(\Phi)=\alpha/c[-\alpha(c-s),\alpha(c+s)]. \end{gathered} \tag{8}$$

In the case of the octagonal-Mn_3Si crystal transition, the physical space components $\mathbf{g}_i^*(\phi)$, $i=1,...,4$, of reciprocal lattice vectors are given by

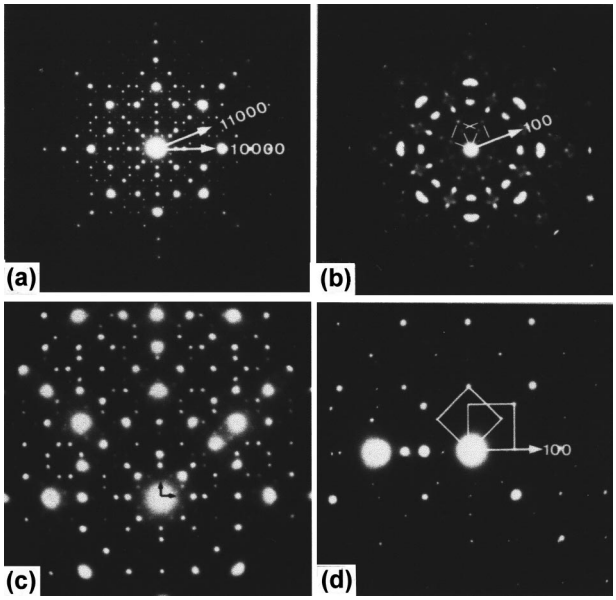

FIG. 1. Selected-area electron diffraction patterns illustrate (a) and (b) octagonal phase transforms to the 45° microtwins of β-Mn-type structure; (a)–(d) octagonal phase transforms to 45° microtwins of Mn_3Si crystal, and (c) the intermediate phase between the octagonal and Mn_3Si crystalline phases.

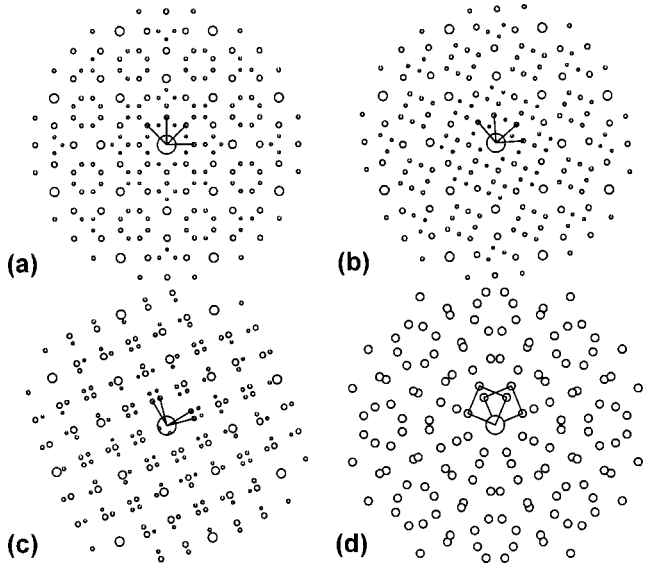

FIG. 2. Simulated diffraction patterns of the octagonal-β-Mn-type structure transition are given. (a) The octagonal phase is shown; (b) and (c) the intermediate phases with $\phi \approx 4.07°$ and $\phi \approx 14.4°$, respectively; (d) the superposition of two simulated diffraction patterns with $\phi = \pm 22.5°$, respectively, which specifies the diffraction pattern of 45° twins of β-Mn-type structure crystals.

$$\begin{aligned}
\mathbf{g}_1^*(\phi) &= \alpha/c(c-s,0), \\
\mathbf{g}_2^*(\phi) &= \alpha/c[\alpha(c+s),\alpha(c+s)], \\
\mathbf{g}_3^*(\phi) &= \alpha/c(0,c-s), \\
\mathbf{g}_4^*(\phi) &= \alpha/c[-\alpha(c+s),\alpha(c+s)].
\end{aligned} \tag{9}$$

The perpendicular space components $\mathbf{g}_i'^*$, $i=1,...,4$, are just the projection of the reciprocal basis of lattice Λ^4 onto the perpendicular space $\mathbf{e}_i'^*$, $i=1,...,4$, both in the case of the octagonal-β-Mn-type structure and the octagonal-Mn_3Si crystal transitions. Thus

$$\begin{aligned}
\mathbf{g}_1'^* &= \alpha(1,0), \\
\mathbf{g}_2'^* &= \alpha(-\alpha,-\alpha), \\
\mathbf{g}_3'^* &= \alpha(0,1), \\
\mathbf{g}_4'^* &= \alpha(\alpha,-\alpha).
\end{aligned} \tag{10}$$

Ternary alloy of the composition $Mn_{82}Si_{15}Al_3$ was prepared by melting of the high-purity elements into an Ar-arc furnace. Rapidly solidified $Mn_4(Si, Al)$ alloy films about 20 μm in thickness were prepared by the melt-spinning method in Ar atmosphere. The quenching speed was 10^5-10^6 °C/s. The films were thinned by Ar-ion milling in order to perform TEM studies. TEM observations were carried out in a Philips CM20 working at 200 kV.

Figure 1(a) is the selected-area electron diffraction (SAED) pattern taken from the $Mn_4(Si, Al)$ octagonal phase. It can be seen that this pattern shows the perfect eightfold symmetry. When the sample was heated rapidly, the 45° microtwins of β-Mn-type structure formed [Fig. 1(b)]. The octagonal-β-Mn-type structure transition has been observed in the Cr-Ni-Si octagonal alloy,[12,13] and some intermediate phases investigated [see Figs. 3(b) and 3(c) in Ref. 12]. If the sample was heated slowly, the octagonal phase transformed to 45° microtwins of Mn_3Si crystal [Fig. 1(d)]. The SAED diffraction pattern in Fig. 1(c) shows an intermediate phase between the octagonal and Mn_3Si crystalline phases. The β-Mn-type structure is a simple cubic phase with space group $P4_132$, and Mn_3Si is a body-center cubic structure with space group $Im3m$.[17]

To compare with the experimental results, some simulated diffraction patterns are plotted; Figs. 2(a)–2(d) illustrate the octagonal-β-Mn-type transition, while the octagonal-Mn_3Si crystal transition is shown in Figs. 3(a)–3(d). During the transformation from the octagonal phase to the β-Mn-type structure, the diffraction spots ''rotate'' around the eightfold axis [Figs. 2(b) and 2(c)]. Some groups of the diffraction spots get closed together. When the absolute value of rotational angle increases to $|\phi| = 22.5°$, the system is transformed into the β-Mn-type structure, the spots in each cluster are superposed upon a single point [Fig. 1(b) and Fig. 2(d)]. Depending on the orientation of the rotation $R(\phi)$, the [01] diffraction of the β-Mn-type structure has the orientation of 22.5° or −22.5° with respect to the [0001] diffraction of the octagonal phase, both of the two orientations of the β-Mn-type structure correspond to the opposite angles of rotation $R(\phi)$, that is, $\phi = 22.5°$ or $\phi = -22.5°$. It is obvious that the opposite angles correspond to the phason strains of equal magnitude and opposite sign.

The process of the octagonal-Mn_3Si crystal transition is shown in Fig. 3. One can see that the diffraction spots shift along the radial directions. One part of diffraction spots are pulled toward the center, at the same time another part of spots is scattered apart from the center. Figure 3(a) illustrates an approximant with $\phi \approx -2.5°$, which is consistent with the

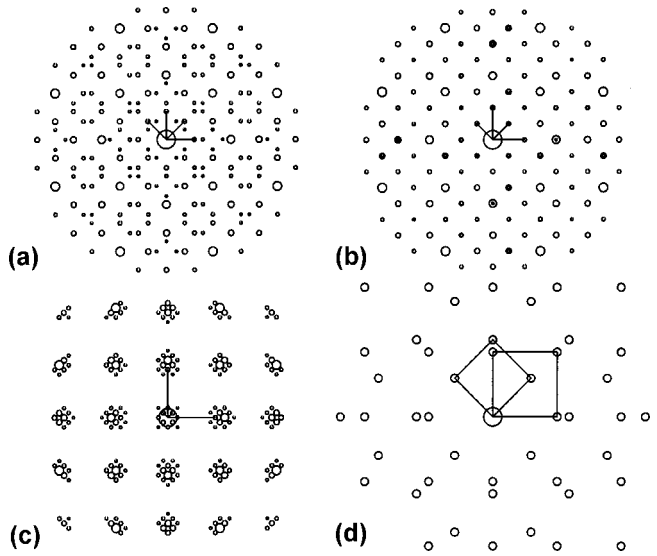

FIG. 3. Simulated diffraction patterns of the octagonal-Mn_3Si crystal transition are given. (a), (b), and (c) illustrate intermediate phases with $\phi \approx -2.5°$, $\phi \approx -9.74°$, and $\phi = -36.95°$, respectively; (d) the superposition of two simulated diffraction patterns with $\phi = \pm 45°$, respectively, specifying the diffraction pattern of 45° twinned Mn_3Si crystals.

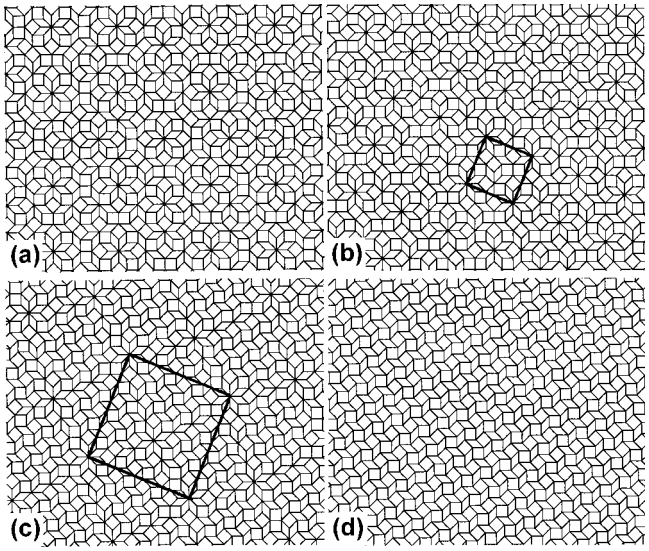

FIG. 4. The octagonal-β-Mn-type structure transition can be described by the tilings. (a) The quasiperiodic tiling represents the octagonal phase ($\phi = 0°$); (b) and (c) the intermediate phases with $\phi \approx 4.07°$ and $\phi \approx 14.4°$, respectively; the large squares denote unit cells of approximants; (d) the β-Mn-type structure is shown with $\phi = 22.5°$.

diffraction pattern of the intermediate phase shown in the Fig. 1(c). As well as the octagonal-β-Mn-type structure transition, the diffraction spots converge into clusters during the octagonal-Mn_3Si crystal transition. In the Mn_3Si crystalline phase with the absolute value of rotational angle $|\phi| = 45°$, the spots in each cluster are superposed upon a single point [Fig. 1(d) and Fig. 3(d)]. One can see that the [01] diffraction of the Mn_3Si crystal has either 0° or 45° orientation with respect to the [0001] diffraction of the octagonal phase, both of the two orientations correspond to the opposite rotational angles, either $\phi = 45°$ or $\phi = -45°$.

By superposing the two simulated diffraction patterns of the β-Mn-type structure with $\phi = 22.5°$ and $\phi = -22.5°$ together, a simulated diffraction pattern [Fig. 2(d)] is obtained which coincides with the diffraction pattern of 45° twins of the β-Mn-type structure [Fig. 1(b)]. In the same way, the superposition [Fig. 3(d)] of two simulated diffraction patterns of Mn_3Si crystal with $\phi = 45°$ and $\phi = -45°$ matches the diffraction pattern taken from 45° twins of Mn_3Si crystal. By symmetry, in the octagonal phase there is no physical difference between the phason strain fields with the positive and the negative signs (represented by $\pm\phi$, respectively). The octagonal phase could be distorted under the action of either of the two phason strains with the opposite sign, both of them appear equiprobably. So it is natural that the system transforms to 45° crystal twins.

It is obvious that the simulated diffraction patterns are consistent with the experimental ones, in the case of either the octagonal-β-Mn-type structure transition or the octagonal-Mn_3Si crystal transition. Therefore, it can be verified that the two octagonal-cubic phase transitions are of phason-type, and the reduction of the octagonal point symmetry corresponds to the unique two subgroups with fourfold symmetry, respectively.

By virtue of the distorted higher-dimensional lattice M^4, the tiling of approximant structure can be given by the normal projection method of quasicrystals. Projecting the lattice points of M^4 belonging to the strip, which is obtained by the translation of a unit cell of M^4 along the subspace E, the tilings representing the octagonal-cubic phase transition can be pictured. Figures 4(a)–4(d) is the tiling model for the structural transformation from the octagonal to the β-Mn-type structure, as well as Figs. 5(a)–5(d) describing the octagonal-Mn_3Si crystal transition. In the case of the octagonal-β-Mn-type structure transition, if the rotational angle ϕ satisfies the relations $\sin(45° + 2\phi) = p/q$ and $\cos(45° + 2\phi) = m/n$, where the two pairs of integers p, q

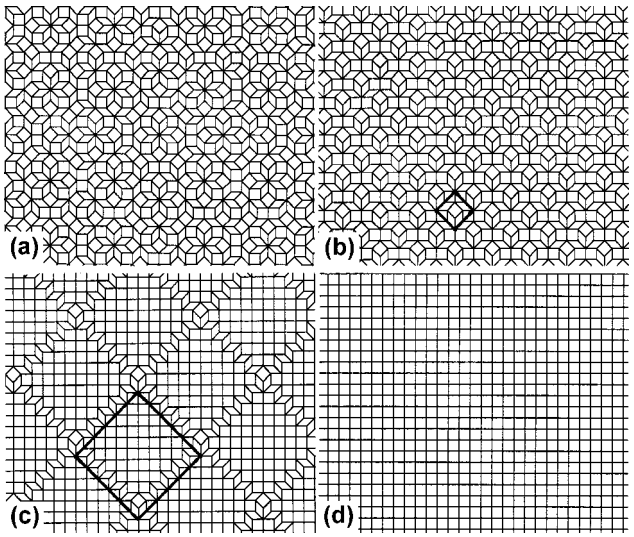

FIG. 5. The octagonal-Mn_3Si crystal transition can be described by the tilings. (a) The quasiperiodic tiling represents the octagonal phase ($\phi = 0°$); (b) and (c) the intermediate phases with $\phi \approx -9.74°$ and $\phi = -36.95°$, respectively; the large squares denote unit cells of approximants; (d) the Mn_3Si structure is obtained with $\phi = -45°$.

and m, n are coprime, respectively; the tiling obtained is periodic which represents a rational approximant structure of the octagonal phase. For the octagonal-Mn_3Si crystal transition, the approximant structure is obtained when the rotational angle ϕ satisfies the relation $\tan(45°-\phi)=\sqrt{2}(p/q)$, where the integers p and q are coprime.

In the case of the octagonal-β-Mn-type structure transition, the rotational angle ϕ can be chosen as $\phi=1/2[\arctan(4/3)-45°]\approx 4.07°$, the tiling shown in Fig. 4(b) represents an approximant structure with the square unit cell. During the octagonal-Mn_3Si crystal transition, an approximant structure with the square unit cell is illustrated in Fig. 5(b) when we choose the case $\phi=45°-\arctan(\sqrt{2})\approx -9.74°$. When the absolute value of the rotational angle ϕ continues to increase, within this tiling model it can be found that the approximant consists of domains separated by ''walls'' [see Fig. 4(c) or Fig. 5(c)]. All the domains have the same local periodic structure, which is just the structure of the β-Mn-type structure crystal (or the Mn_3Si crystal). The areas of local periodic structure become larger with the increase of the absolute value of ϕ. In the final states, either the β-Mn-type structure with $\phi=22.5°$ [Fig. 4(d)] or the Mn_3Si crystal with $\phi=-45°$ [Fig. 5(d)], the local periodic structure is not local any more, which extends to cover the whole space. It should be mentioned that the tiling model proposed above is two-dimensional, which describe only a transition from quasicrystal to an approximant with a square base.

The approximant structure with stronger phason strain has been investigated experimentally. For instance, the electron diffraction patterns illustrated in Figs. 3(b) and 3(c) of Ref. 12 showed intermediate phases during the octagonal-β-Mn-type structure transition, the correspondent rotational angles ϕ are about 5° and 12.5°, respectively, which are larger than the rotational angle $\phi\approx 4.07°$ of the approximant shown in Fig. 4(b).

In summary, by taking advantage of the distorted higher-dimensional lattice, the reciprocal quasilattice of the approximant is given directly, as well as the approximant tiling. Furthermore, It is presumable that by means of this kind of distorted lattice one can constitute the atomic surface in the higher-dimensional space, and then the process of the quasicrystal-approximant transition can be described by the cut method,[18,19] by which the locations of atoms are obtained by intersecting the atomic surface with the physical space.

Through our theoretical analysis, it can be concluded that the simulated diffraction patterns are in good agreement with the experimental results. This implies that there exist two kinds of phason strain fields in the octagonal phase, both of them cause the transformation of the octagonal phase into cubic crystalline phases. Different phason fields give rise to different transformations, one leading to the β-Mn-type structure phase upon the rapid heating of the sample and another to Mn_3Si crystalline phase upon slow heating. From our tiling model, it is conjectured that the state close to the cubic state consists of cubic phase domains separated by ''walls.'' The cubic phase may have different local atomic structure from that of the octagonal phase.

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