# Characterisation of Quasicrystalline Particles in an Isothermally Aged Al-10Mg-0.5Ag (mass\%) Alloy 

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#### Abstract

The quasicrystalline structure found in the isothermally aged microstructure in an $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ ( $\mathrm{mass} \%$ ) alloy after solution treated, water quenched and then aged during the time between 20 and 40 min at $240^{\circ} \mathrm{C}$ has been characterised using transmission electron microscopy, electron microdiffraction and energy dispersive x-ray spectroscopy. The morphology of the quasicrystalline precipitate particles is rhombohedral in shape and those precipitate particles are homogeneously nucleated, and finely and uniformly dispersed in the matrix. The orientation relationship between the quasicrystalline phase and the $\alpha$-Al matrix is as follows; i5 $\|\langle 011\rangle_{\alpha}$ and i3 $\|\langle 111\rangle_{\alpha}$. The quasilattice constant $a_{\mathrm{R}}$ of the icosahedral quasicrystalline phase is estimated to be $0.505 \pm 0.01 \mathrm{~nm}$ from the present 5 -fold electron microdiffraction patterns. The lattice parameter $a_{\mathrm{c}}$ of the corresponding crystalline cubic approximant is thus calculated to be $1.390 \pm 0.028 \mathrm{~nm}$. This is in good agreement with the lattice parameter of the crystalline T phase $\left(\mathrm{Mg}_{32}(\mathrm{Al}, \mathrm{Ag})_{49}, a=1.416 \mathrm{~nm}\right)$. The morphology of the quasicrystalline precipitate particle is consistent with that predicted from the intersection point group $\overline{3}$, which was defined by symmetry elements common to the two lattices in the observed orientation relationship. The quasicrystalline particles contain elements of $\mathrm{Al}, \mathrm{Mg}$ and Ag . The quasicrystalline precipitate particles, which are the metastable phase, appear to be the primary strengthening phase in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ (mass\%) alloy aged at $240^{\circ} \mathrm{C}$.


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## 1. Introduction

Trace additions of Ag are known to stimulate the precipitation behaviour in all Al alloys containing Mg , with an acceleration in the kinetics of the age-hardening response and an increase in the maximum hardness achievable. ${ }^{1)}$ Polmear and Sargant first found that the age-hardening response in the $\mathrm{Al}-\mathrm{Mg}$ alloys was remarkably improved by small concentrations of Ag , and the precipitates were very finely distributed in the matrix. ${ }^{2)}$ Wheeler et al. ${ }^{3)}$ proposed that peak hardness in the ternary $\mathrm{Al}-\mathrm{Mg}-\mathrm{Ag}$ alloy can be associated with fine scale precipitates of the T phase, $\mathrm{Mg}_{32}(\mathrm{Al}, \mathrm{Ag})_{49}$ (space group $\operatorname{Im} \overline{3}$, body-centred cubic structure, $a=1.416 \mathrm{~nm}),{ }^{4}$ rather than precipitation of the $\beta$ phase, $\mathrm{Al}_{3} \mathrm{Mg}_{2}$ (space group $\mathrm{Fd} \overline{3} \mathrm{~m}$, face-centred cubic structure, $a=2.824 \mathrm{~nm}$ ). ${ }^{5,6)}$ Auld ${ }^{7}$ ) determined the orientation relationship between the matrix and precipitates of the T phase by X-ray diffraction technique; $(100)_{\mathrm{T}} \|(112)_{\alpha}$, $[001]_{\mathrm{T}} \|[110]_{\alpha}$. The aluminium-rich corner of the $\mathrm{Al}-\mathrm{Mg}-$ Ag ternary alloy phase diagram was investigated by X-ray diffraction, and it has been established that the possible phase presented is the T phase in the current experimental alloy composition when the alloys are aged at $200^{\circ} \mathrm{C} .{ }^{8)}$ However, Auld and Cousland analysed the ageing behaviour of the ternary $\mathrm{Al}-\mathrm{Mg}-\mathrm{Ag}$ alloy by using X-ray diffraction technique, and revealed that the T phase thought to be present at peak hardness was, in fact, the metastable $\mathrm{T}^{\prime}$ phase. ${ }^{9)}$ The $\mathrm{T}^{\prime}$ phase has a hexagonal structure with lattice parameters $a=1.4 \mathrm{~nm}$ and $c=2.8 \mathrm{~nm}$. The equilibrium T phase is supposed to form after long-term ageing. A more recent study using X-ray diffraction and single crystal alloy specimens also indicated that the precipitated $\mathrm{T}^{\prime}$ and T phases are observed in the $\mathrm{Al}-4.49 \mathrm{Mg}-0.54 \mathrm{Ag}(\mathrm{at} \%)$ alloy. ${ }^{10}$ ) Since the

[^0]1960s, only limited microscopic work and a few preliminary X-ray diffraction experiments have been completed and the alloys with microalloying additions of Ag are not well characterised in terms of microstructural analysis, such as the size, distribution and morphology of precipitate particles and the crystal structures of dispersed phases.

Since the first remarkable discovery of the icosahedral quasicrystalline phase in the rapidly solidified $\mathrm{Al}-14 \mathrm{at} \% \mathrm{Mn}$ alloy was made by Shechtman et al., ${ }^{11)}$ intensive investigations have been carried out to explore the alloy systems with compositions for the formation of icosahedral phases and to identify structures and their relationships with the crystalline phases. ${ }^{12)}$ Icosahedral quasicrystals have been found in several alloy systems, especially in Al-transition metals based alloys, which are generally produced by rapid quenching and mechanical alloying techniques. ${ }^{13)}$ Great progress on understanding of the structure of icosahedral quasicrystalline phases was made by recognising ${ }^{14,15)}$ that there are two major classes of icosahedral quasicrystals, i.e. Mackay icosahedral clusters in $\mathrm{Al}-\mathrm{Mn}$ and $\mathrm{Al}-\mathrm{Mn}-\mathrm{Si}^{16)}$ alloys and Pauling triacontahedral clusters in $\left.\mathrm{Mg}-\mathrm{Al}-\mathrm{Zn}^{4}\right)$ and $\mathrm{Al}-\mathrm{Cu}-\mathrm{Li}$ alloys.

The crystal structure of the stoichiometry phase $\mathrm{Mg}_{32}(\mathrm{Al}, \mathrm{Zn})_{49}$ was shown to be a cubic crystal structure based on a body centred cubic lattice with 162 atoms, which are a classic example of close-packed structure dominated by icosahedral coordination shells. ${ }^{4,17)}$ It has been suggested that equilibrium phases having a high proportion of icosahedral coordinated atoms can be rapidly solidified from the melt into the icosahedral quasicrystalline phases. ${ }^{18)}$ Based on the value of the quasilattice constant and the existence of $\mathrm{Mg}_{32}(\mathrm{Al}, \mathrm{Zn})_{49}$ type Frank-Kasper phase in the $\mathrm{Al}-\mathrm{Mg}-\mathrm{Zn}$ system, it has been suggested that the structure of the icosahedral phase belongs to the $\mathrm{Mg}-\mathrm{Al}-\mathrm{Zn}$ class of atomic arrangements of the quasicrystalline lattice. ${ }^{14,15)}$ Furthermore, a stable ternary compound in the $\mathrm{Al}-\mathrm{Cu}-\mathrm{Li}$ alloy, formerly designed as $\mathrm{T}_{2}\left(\mathrm{Al}_{6} \mathrm{CuLi}_{3}\right)$ by Hardy and Silcock, ${ }^{19)}$ has been found to exhibit an icosahedral quasicrystal structure when
the alloy was produced by either the conventional ingot casting (with low solidification rates) or by ageing treatment after supersaturated solid solution. ${ }^{20}$ However, the intrinsic strengthening effect of $\mathrm{T}_{2}$ phase is very low on an $\mathrm{Al}-$ $2.1 \mathrm{Li}-2.1 \mathrm{Cu}-1 \mathrm{Mg}$ (mass $\%$ ) alloy heated at $250^{\circ} \mathrm{C} .{ }^{21)}$

Preliminary identification work of the quasicrystalline particles formed in an $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ (mass\%) alloy during isothermal ageing process has been reported. ${ }^{22)}$ The purpose of this paper is to report results of a further detailed microstructures examination of the metastable quasicrystalline precipitates phase formed by isothermal ageing process but without employment of rapid solidification or other noneequilibrium methods in the process. Crystallography and morphology of the quasicrystalline phase in the $\mathrm{Al}-10 \mathrm{Mg}-$ 0.5 Ag (mass\%) alloy was studied using transmission electron microscopy with electron diffraction techniques. The results of analyses of the composition of the precipitate phase using energy dispersive X-ray spectroscopy (EDXS) are also reported and compared to similar analysis of the matrix phase in the same $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ (mass\%) alloy with the same ageing condition.

## 2. Experimental Procedures

The tested alloy with a nominal composition of $\mathrm{Al}-10 \mathrm{Mg}-$ 0.5 Ag (mass\%) was prepared by induction melting under air atmosphere from elemental components of high purity (i.e. $\mathrm{Al}(99.96 \%), \mathrm{Mg}(99.9 \%)$ and $\mathrm{Ag}(99.9 \%)) . \mathrm{Mg}$ was added just prior to pouring in metal mould in order to prevent any loss by oxidation. After these ingots were homogenised for 52 h at $300^{\circ} \mathrm{C}$, surfaces of ingot was scalped each side. These ingots then were hot rolled to approximately 0.3 mm for TEM specimens. Solution treatment for ageing was carried out in a salt bath at $500^{\circ} \mathrm{C}$ for 1 h , followed by water quenching and ageing in an oil bath at $240^{\circ} \mathrm{C}$.
The specimens for TEM observations were punched mechanically from the strips of 0.3 mm in thickness after appropriate heat treatments, then dry grounding to a thickness of $0.1-0.15 \mathrm{~mm}$. Thin foils were prepared by twin-jet electropolishing in a solution of $33 \mathrm{vol} \%$ nitric acid and $67 \mathrm{vol} \%$ ethanol at temperature range between $0^{\circ} \mathrm{C}$ and $-10^{\circ} \mathrm{C}$, using a Tenupol-3 jet polisher operating at 0.2 A and 12 V . After polishing, the thin foil specimens were washed and rinsed in a series of ethanol baths before drying on filter paper. The polished thin foils were stored in a vacuum desiccator to avoid the development of oxide films on specimen surface.
Microstructures were observed by a Philip CM20 and a JEM2010 transmission electron microscope (TEM) at 200 kV , equipped with facility of a LINK model energy dispersive X-ray spectrometer (EDXS). Microbeam electron diffraction patterns were recorded by conventional TEM mode using a $30 \mu \mathrm{~m}$ condenser lens aperture and an electron probe of $40-100 \mathrm{~nm}$.

## 3. Experimental Results

Bright-field (BF) images of the microstructure typical of the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ (mass\%) alloy aged for 0.5 h at $240^{\circ} \mathrm{C}$, together with the corresponding SAED patterns, are shown in

Fig. 1. The electron beam is approximately parallel to the (a) $[110]_{\alpha}$, (b) $[112]_{\alpha}$ and (c) $[111]_{\alpha}$ directions. Those transmission electron micrographs were recorded from a region within the centre of a matrix grain. A uniform distribution of finer-scale (typically 20 nm ), faceted precipitate particles was observed within the core of the grains suggesting that such precipitate particles were nucleated homogeneously from the supersaturated solid solution of the aluminium matrix during the isothermal ageing treatment. On the other hand, the regions close to the grain boundaries contained coarse-scale ( $60-80 \mathrm{~nm}$ ), faceted precipitate particles with evidence of the existence of precipitate-free zone. ${ }^{22)}$ The coarser distribution adjacent to the PFZ presumably reflects a lower nucleation rate in these regions, arising from a combination of depleted solute and reduced vacancy concentrations.

Detailed higher magnification images of the precipitate particles are presented in Fig. 2. The electron beam is approximately parallel to the $[110]_{\alpha}$ direction. The micrographs of Figs. 2(a) and (b) were taken exactly parallel to the zone axes in order to obtain the exact traces of the shape of the precipitate particles, while the micrograph of Fig. 2(c) was taken slightly off the zone axis in order to observe the projected image. Careful examinations of these microstructures in the $[1 \overline{1} 0]_{\alpha}$ orientation, Fig. 2(a), revealed that there were, in fact, two distinguishable shapes within the particle array. Figure 2(b) provides examples of these two forms of precipitate particles with distinguishable different angles defined by the edge traces. Particle labelled A has a regular well-faceted diamond shape with angles of $\sim 80^{\circ}$ between edge traces, while particle labelled B is irregular and has angles of $\sim 70^{\circ}$ between edge traces.

When these particles were observed in the $[010]_{\alpha}$ orientation, Figs. 3(a) and (b), there were also two apparent forms; one shape is almost rectangular or square in section and the other is not well-faceted square-shaped particle. From these observations of precipitate particles over a range of orientations, it is deduced that they have the form of rhombohedra.

A lattice fringe image of a precipitate particle in the $\alpha-\mathrm{Al}$ matrix recorded with the electron beam parallel to the $[110]_{\alpha}$ orientation is shown in Fig. 4. The inter planar spacing between each atomic plane in the Al matrix is approximately 1.6 nm along to the $\langle 111\rangle_{\alpha}$ directions, which is consistent with the d-spacing of $\{111\}_{\alpha}$ planes of the Al matrix phase. The edge angle of the traces of the faceted precipitate particle is $70.5^{\circ}$ which is corresponding to the angle between the $\{111\}_{\alpha}$ of the $\alpha$-Al matrix phase (i.e. Fig. 2(b)). The bright dots within the precipitate particle (quasi) are not clearly constructed with the five-fold rotational symmetry because this orientation is not perfectly aligned to perpendicular to the quasicrystalline structure (i.e. misorientation of few degree between $\langle 110\rangle_{\alpha}$ and i5 directions, Fig. 3(b) ${ }^{22)}$ ). However, very few bright dots array from the precipitate particle is showing periodic arrangement. It is worthwhile to note that the interface between the matrix and the precipitate particle phase seems to possess coherent boundary (arrows), and no any defects within the precipitate particle are observed.

A series of microbeam electron diffraction (MBED) patterns recorded from relatively large scale, diamondshaped particles in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ (mass\%) alloy aged


Fig. 1 TEM micrographs showing the morphologies and distribution of precipitate particles at core of grain in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ alloy aged for 0.5 h at $240^{\circ} \mathrm{C}$. The electron beam is approximately parallel to the (a) $[1 \overline{1} 0]_{\alpha}$, (b) $[112]_{\alpha}$ and (c) $[111]_{\alpha}$ directions.
for 0.5 h at $240^{\circ} \mathrm{C}$ has been previously reported. ${ }^{22)}$ In that paper, the precipitate pattern recorded near to the $[110]_{\alpha}$ orientation exhibited five-fold rotational symmetry, and the misorientation between the precipitate five-fold axis and the matrix $[110]_{\alpha}$ axis was approximately $2-4^{\circ}$. The three-fold rotational symmetry in the precipitate pattern was observed in the $[111]_{\alpha}$ orientation. The two-fold rotational symmetry was detectable in the precipitate diffraction pattern in the $[112]_{\alpha}$ orientation. In addition, a second variant of the fivefold rotational symmetry was observed in the $[011]_{\alpha}$ orientation. However, the precise form of the crystallographic relationship between the precipitate phase, tentatively identified as having icosahedral structure, and the matrix
was difficult to define. For example, Figs. 3(c) and (d) in the previously reported paper ${ }^{22)}$ suggests that there are three-fold and two-fold rotational axes of the icosahedral precipitate parallel to the $[111]_{\alpha}$ and $[112]_{\alpha}$ directions, respectively. However, the angle between the $\langle 111\rangle_{\alpha}$ and $\langle 112\rangle_{\alpha}$ axes is $19.5^{\circ}$, while that between the relevant axes of the icosahedral structure is $20.9^{\circ}$. This indicates that the two icosahedral axes cannot simultaneously be exactly parallel to the appropriate axes of the matrix structure, and thus the question whether either is an exact parallelism is raised.
In order to address this question and to confirm the icosahedral structure, further large angle tilting experiments were carefully carried out from the $[111]_{\alpha}$ zone axis through


Fig. 2 TEM micrographs showing two distinguishable profiles of diamond-shaped precipitate particles in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ alloy aged for 0.5 h at $240^{\circ} \mathrm{C}$. Micrographs (a) and (b) were taken exactly parallel to the $[1 \overline{1} 0]_{\alpha}$ zone axis, while micrograph (c) was taken after tilting off the on zone axis.
$[112]_{\alpha}$ (i.e. about $[110]_{\alpha}$ ) and to further $22^{\circ}$. For an icosahedral structure this would be expected to bring the electron beam at least approximately parallel to a second axis of three-fold rotational symmetry. Figure 5(a) shows a precipitate pattern of three-fold rotational symmetry superimposed on the $[111]_{\alpha}$ zone axis pattern. When the specimen was tilted systematically $1^{\circ}$ and $2^{\circ}$ away from the exact $[111]_{\alpha}$ zone axis towards $[110]_{\alpha}$, the precipitate pattern immediately lost three-fold symmetry in the diffracted intensity (Figs. 5(b) and (c), respectively), suggesting that within permitted accuracy, the three-fold axis of the precipitate phase is parallel to the corresponding three-fold axis of the matrix. The microbeam electron diffraction pattern in Fig. 5(d) was recorded in the $[112]_{\alpha}$ matrix orientation when the specimen was tilted from the $[111]_{\alpha}$ orientation. The two-fold rotational symmetry of the precipitate pattern is approximately superimposed on the [112] $]_{\alpha}$ zone axis pattern. When the specimen was tilted $\sim 2^{\circ}$ away from the $[112]_{\alpha}$ zone axis of the matrix towards the $[001]_{\alpha}$ direction, a symmetric pattern with two-fold rotational symmetry was observed from the precipitate phase, as shown
in Fig. 5(e). This confirms that the two-fold axis of the precipitate structure is not exactly parallel to the $[112]_{\alpha}$ axis. When the specimen was tilted a further $\sim 22^{\circ}$ away from the $[112]_{\alpha}$ zone axis towards $[001]_{\alpha}$, a second precipitate pattern with three-fold rotational symmetry was detected, Fig. 5(f), approximately parallel to the $[117]_{\alpha}$ direction. This series of microbeam electron diffraction patterns during the careful large angle tilting experiment agrees well with the tilt angles among major rotational symmetry axes in the result previously reported. ${ }^{11)}$

The distances between diffraction spots from the precipitate phase suggest a quasiperiodicity along systematic rows of the diffraction patterns. For example, the row from the transmit spot to the 220 matrix spot in five-fold and three-fold rotational symmetry patterns contained a sequence comprised of two segments that might be designated as $S$ (short) and $L$ (long). These segments were arranged with Fibonacci sequence, i.e. LSLLSLSL, where the ratio $(\mathrm{L}+\mathrm{S}) / \mathrm{L}$ is equal to approximately 1.7. This is entirely consistent with the golden ratio $\tau=\left(1+5^{1 / 2}\right) / 2=1.618$, and implies a quasiperiodic order in this direction.


Fig. 3 Bright-field images of the faceted precipitate particles recorded (a) on zone axis and (b) at slightly off zone axis in the Al-10Mg0.5 Ag alloy aged for 0.5 h at $240^{\circ} \mathrm{C}$. The electron beam is approximately parallel to the $[010]_{\alpha}$ direction.


Fig. 4 Lattice fringe image of the quasicrystalline particle, showing interface between matrix and precipitate phase and no defects within the particle, in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ alloy aged for 0.5 h at $240^{\circ} \mathrm{C}$ with electron beam parallel to the $[110]_{\alpha}$ direction.

Results of the systematic large angle tilting experiments, both Fig. 3 in the previously reported result ${ }^{22)}$ and Fig. 5 in this paper, suggest orientation relationships between the icosahedral quasicrystalline and matrix phases, namely the forms of i5 $\|\langle 110\rangle_{\alpha}$, i3 $\|\langle 111\rangle_{\alpha}$, i3 $\sim \|\langle 117\rangle_{\alpha}$, i2 $\sim \|$ $\langle 112\rangle_{\alpha}$. It is thus similar in form to that reported for the icosahedral precipitate phase formed during isothermal decomposition of strip-cast $\mathrm{Al}-\mathrm{Mn}-\mathrm{Fe}-\mathrm{Si}$ alloys. ${ }^{23)}$

In order to test whether measured angles were consistent with those previously reported, ${ }^{11)}$ further large angle tilting experiments were carried out from the $[00 \overline{1}]_{\alpha}$ direction to $[11 \overline{1}]_{\alpha}$, and further to the $[10 \overline{1}]_{\alpha}$ direction through the $[11 \overline{2}]_{\alpha}$ orientation. These systematic tilt directions were ob-
tained from the established orientation relationship between the quasicrystalline and matrix phases, as mentioned above. The resultant microbeam electron diffraction patterns are presented in Figs. 6(a)-(h). Figure 6(a) shows a precipitate pattern with some systematic symmetry superimposed on the $[00 \overline{1}]_{\alpha}$ zone axis pattern. When the specimen was tilted $\sim 1.5^{\circ}$ away from the $[00 \overline{1}]_{\alpha}$ orientation, the systematic reflections from the precipitate phase remained, as shown in Fig. 6(b). Figure 6(c) shows that a precipitate pattern with five-fold rotational symmetry was observed approximately parallel to the $[11 \overline{3}]_{\alpha}$ orientation when the specimen was tilted approximately $22^{\circ}$ away from the $[00 \overline{1}]_{\alpha}$ orientation. The misorientation between the five-fold rotational axis and the $[11 \overline{3}]_{\alpha}$ orientation was obtained experimentally to be approximately $3^{\circ}$. With further tilting, from close to the $[11 \overline{3}]_{\alpha}$ orientation towards the $[11 \overline{1}]_{\alpha}$ orientation, a precipitate pattern with two-fold rotational symmetry was observed approximately parallel to the $[11 \overline{1}]_{\alpha}$ orientation, Fig. 6(d). The regular array of reflection spots from the precipitate particle is observed in the $[10 \overline{1}]_{\alpha}$ direction, shown in Fig. 6(e), and two-fold rotational symmetry in the precipitate was observed approximately parallel to the $[10 \overline{1}]_{\alpha}$ orientation, as shown in Fig. 6(f). Figure 6 (g) shows an axis of three-fold rotational symmetry for the precipitate approximately parallel to the $[31 \overline{2}]_{\alpha}$ orientation. The tilting angle from the axis of two-fold symmetry to that of the three-fold symmetry was approximately $19.4^{\circ}$. A regular array of diffracted spots from the precipitate particle was observed in the $[11 \overline{2}]_{\alpha}$ orientation, when the specimen was tilted about $60^{\circ}$ from close to the $[10 \overline{1}]_{\alpha}$ to the $[11 \overline{2}]_{\alpha}$ matrix directions, Fig. 6(h). The five-fold rotational symmetry of the precipitate pattern which was observed in near the $[11 \overline{3}]_{\alpha}$ orientation was observed again when the sample was tilted $11.5^{\circ}$ from the $[11 \overline{2}]_{\alpha}$ to the $[11 \overline{3}]_{\alpha}$ orientations. The two-fold rotational symmetry that was observed near the $[11 \overline{1}]_{\alpha}$ orientation was obtained again when the specimen was tilted from the five-fold axis in close to the $[11 \overline{3}]_{\alpha}$ to


Fig. 5 A series of electron microdiffraction patterns recorded from a single large scale diamond-shaped particle in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ alloy aged for 0.5 h at $240^{\circ} \mathrm{C}$. (a) three-fold rotational symmetry recorded in the [111] $]_{\alpha}$ zone axis, (b), (c) tilted 1 and $2^{\circ}$ away from the exact $[111]_{\alpha}$ zone axis towards $[110]_{\alpha}$, (d) near two-fold rotational symmetry recorded in the $[112]_{\alpha}$ zone axis, (e) tilted $\sim 2^{\circ}$ away from the [112] $]_{\alpha}$ zone axis towards the $\langle 001\rangle_{\alpha}$ direction and (f) the other variant of the three-fold rotational symmetry (matrix: open symbol, precipitate: close symbol).
$[11 \overline{1}]_{\alpha}$ orientation by approximately $28.6^{\circ}$. These results obtained using microbeam electron diffraction and systematic tilting of the specimen are entirely consistent with the icosahedral structure for the precipitate phase, ${ }^{11)}$ and the orientation relationship between the icosahedral structure of the precipitate phase and the aluminium matrix is shown in Fig. 7, as a stereographic projection.

Typical energy dispersive X-ray spectra recorded from a diamond-shaped particle of precipitate phase and the aluminium matrix phase are compared in Fig. 8. A significant concentration of Mg , with a small but still detectable concentration of Ag , was associated with icosahedral particles, as shown in Fig. 8(a). However, similar analysis of the matrix phase, Fig. 8(b), revealed a relatively lower concentration of Mg , and no evidence of enhanced Ag concentration. Qualitative microanalysis thus suggested that the quasicrystalline phase was a ternary compound that contained all three elements $\mathrm{Al}, \mathrm{Mg}$ and Ag. Since the
nominal diameter of the electron probe ( $\sim 30 \mathrm{~nm}$ ) was slightly larger than the typical cross section of the precipitate particles, it was difficult to avoid some overlap of the electron beam to the aluminium matrix phase. Accurate quantitative chemical analysis of the particles thus might be impossible due to the difficulty of isolating individual particles from the matrix phase.

## 4. Discussion

### 4.1 Identification

A series of microbeam electron diffraction patterns recorded from relatively large scale, diamond-shaped particles established the detailed tilting angles among icosahedral symmetry together with the aluminium matrix orientations. As indicated in Fig. 7, there is a tilt angle of approximately $63^{\circ}$ between five-fold rotational symmetry. This angle is almost equal to that of $\langle 110\rangle_{\alpha}$ matrix axes. The


Fig. 6 A systematic series of electron microdiffraction patterns ((a)-(h)) recorded from a relatively large scale, diamond-shaped particle in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ alloy aged for 0.5 h at $240^{\circ} \mathrm{C}$. The patterns confirm the icosahedral structure of the precipitate phase and the orientation relationship (matrix: open symbol, precipitate: close symbol).
misorientation between the precipitate five-fold axis and the matrix $\langle 110\rangle_{\alpha}$ axis was experimentally obtained during a large angle tilting, and was approximately $2-4^{\circ}$ away from the $\langle 110\rangle_{\alpha}$ matrix zone axis towards the $\langle 002\rangle_{\alpha}$ direction. Three-fold rotational symmetry in the precipitate pattern is truly parallel to the $\langle 111\rangle_{\alpha}$ orientation, as confirmed in both Fig. 3(c) ${ }^{22}$ and Figs. 5(a)-(c). The angle of tilt required to move from the axis of five-fold symmetry to that of three-fold symmetry was approximately $37^{\circ}$. Two-fold rotational symmetry of the precipitate pattern was detectable in the $\langle 112\rangle_{\alpha}$ orientation, when the specimen was tilted approximately $21^{\circ}$ from the $\langle 111\rangle_{\alpha}$ to near $\langle 112\rangle_{\alpha}$ zone axis. These results from systematic tilting experiments entirely confirmed that tilt angles among major rotational symmetry axes are consistent with the result previously reported. ${ }^{11)}$

It is now well known that electron diffraction patterns recorded from icosahedral quasicrystalline phases display
three-fold and five-fold rotational symmetry with the golden ratio, which is a non-uniform scaling factor equal to the characteristic number of icosahedral quasiperiodicity $\tau=\left(1+5^{1 / 2}\right) / 2=2 \cos (\pi / 5) .{ }^{14,15)}$ To examine whether the observed microbeam electron diffraction patterns in the present research are consistent with this requirement, careful measurements of the electron diffraction patterns were carried out. The distances between the transmitted and the first, second or third (corresponding to the 220 spot) reflection spots from the precipitate phase are approximately $4.4 \pm 0.1 \mathrm{~mm}, 7.0 \pm 0.1 \mathrm{~mm}$ and $11.3 \pm 0.1 \mathrm{~mm}$, respectively in the three-fold rotational symmetry patterns (e.g. Fig. 3(c) ${ }^{22)}$ and Fig. 5(a)). These are thus closely corresponding to the golden ratio, $\tau=1.618$ (7.0/4.4 $=1.591$ ), and the square of the golden ratio, $\tau^{2}=2.618$ $(11.3 / 4.4=2.568)$. The values obtained from the measurements of the five-fold rotational symmetry patterns of the


Fig. 7 A stereographic projection showing the orientation relationship between the icosahedral structure of the precipitate phase and $\alpha$-Al matrix. The symmetry elements and zones with indices represent the superimposed zone axes for the icosahedral phase and Al matrix, respectively.


Fig. 8 Energy dispersive X-ray spectra (EDXS) comparing compositions of (a) a quasicrystalline precipitate particle and (b) the $\alpha-\mathrm{Al}$ matrix phase adjacent to the particle. Small concentrations of Mg and Ag are partitioned to the icosahedral phase.
precipitate phase (e.g. Fig. 3(b) ${ }^{22)}$ and Fig. 6(c)) are also equal to these ratios.

The microbeam electron diffraction patterns presented in Figs. 6(c), (d) and (g) clearly demonstrated five-fold, two-
fold and three-fold rotational symmetry with the golden ratio, respectively. It has been described that a quasicrystalline phase can be characterised by a quasilattice constant $a_{\mathrm{R}},{ }^{14,15)}$ which corresponds to the edge length of the rhombohedra (either prolate or oblate) that must be packed quasi-periodically to form three-dimensional Penrose tiling. Since the three-dimensional Penrose tiling structure can be generated by projections from a six-dimensional simple-cubic lattice, the cubic lattice constant $a_{\mathrm{c}}$ and the value of $a_{\mathrm{R}}$ are related ${ }^{15}$ ) in the form $a_{\mathrm{c}}=\left(4+8 / 5^{1 / 2}\right)^{1 / 2} a_{\mathrm{R}}$. From the observed microdiffraction patterns, the quasilattice constant $a_{\mathrm{R}}$ of icosahedral phase may be estimated ${ }^{12)}$ from an expression of the form $a_{\mathrm{R}}=\tau^{3} \mathrm{~d}_{\mathrm{i} 5} / 2$, where $\tau$ is the golden ratio, and $\mathrm{d}_{\mathrm{i} 5}$ represents the spacing of planes perpendicular to the five-fold axis giving rise to the most intense diffracted spots. Careful measurements for the $\mathrm{d}_{\mathrm{i}}$ value in the present 5 -fold electron microdiffraction patterns gave a value of $a_{\mathrm{R}}=0.505 \pm$ 0.01 nm . The lattice parameter $a_{c}$ of the corresponding crystalline cubic approximant was thus calculated to have a value of $1.390 \pm 0.028 \mathrm{~nm}$ from an equation of the form $a_{\mathrm{c}}=\left(4+8 / 5^{1 / 2}\right)^{1 / 2} a_{\mathrm{R}}$. This is in good agreement with the lattice parameter of the crystalline T phase $\left(\mathrm{Mg}_{32}(\mathrm{Al}, \mathrm{Ag})_{49}\right.$, $a=1.416 \mathrm{~nm}) .{ }^{4)}$

It is recognised that crystals with an apparent five-fold axis of symmetry also occur as a result of multiple twinning, not only as a result of true icosahedral symmetry. ${ }^{24)}$ The faces of a regular icosahedron may be generated by the $\{111\}_{\alpha}$ planes of an appropriate assembly of twenty twin-related crystals, which have a tetrahedral form defined by facets parallel to $\{111\}_{\alpha}$. If a cubic crystal is subjected to a slight rhombohedral distortion $\left(\alpha=92.88^{\circ}\right)$, then the angle between the $\{111\}_{\alpha}$ planes is equal to $72^{\circ}$ and an assembly of $\{111\}_{\alpha}$ tetrahedra may be assembled in twin-related orientations to construct an icosahedron. Therefore, it has been proposed that icosahedral and decahedral quasicrystals are the result of icosatwin and decatwin assemblies formed from cubic crystals. ${ }^{24,25)}$ In the present research, the electron microdiffraction patterns recorded from a large scale, faceted precipitate particle exhibited the uniformly sharp and strong intensity maxima as the evidence of the icosahedral structure. In addition, the very limited experimental HRTEM observation presented in Fig. 4 suggested no evidence of defect microstructures within the particles.
The combination of those considerations mentioned above and evidence from MBED patterns presented in Figs. $3,{ }^{22)} 5$ and 6 would suggest that the sharpness and sphericity of the electron diffraction spots in all three zone-axis diffraction patterns, combined with the observation that all main diffraction spots are perfectly aligned and angles between major row of reflections are as required for the icosahedral symmetry of zone axis, are an indication that the alloy is not a collection of multiple-twinned nanoscale crystallites. ${ }^{26)}$

### 4.2 Orientation relationship

Several orientation relationships observed in a range of different alloy systems and in materials produced by a range of different processing techniques have been reported between an icosahedral quasicrystalline phase and a crystalline phase with which it has contact. Most commonly, they have been estimated from samples in which the quasicrystal-
line phase has been produced by non-equilibrium processing (rapid solidification) and then surrounded by a crystalline phase during subsequent cooling. The reported orientation relationships can be classified into four major distinguishable groups.
In the group designated as type A , the orientation relationship is such that a two-fold axis of the icosahedral phase is parallel to a four-fold axis $\left(\langle 001\rangle_{\alpha}\right)$ of aluminium solid solution phase, while a three-fold axis of the icosahedral phase is simultaneously parallel to a three-fold axis $\langle 111\rangle_{\alpha}$. This relationship requires that a five-fold axis of the icosahedral phase is parallel to the $\langle 1 \tau 0\rangle_{\alpha}$ zone axis, where $\tau$ is the golden ratio. This orientation relationship has been observed in rapidly solidified $\mathrm{Al}-\mathrm{Mn}-\mathrm{Cr}-\mathrm{Si}$ alloys, ${ }^{27)}$ in rapidly solidified $\mathrm{Ti}-\mathrm{V}-\mathrm{Si}$ and $\mathrm{Ti}-\mathrm{Cr}-\mathrm{Si}$ alloys, ${ }^{28)}$ in rapidly solidified $\mathrm{Al}-\mathrm{Mn}-\mathrm{Si}$ alloys ${ }^{29}$ ) and in rapidly solidified $\mathrm{Al}-\mathrm{V}-$ Fe and $\mathrm{Al}-\mathrm{Mo}-\mathrm{Fe}$ alloys. ${ }^{30)}$ It can be summarised in the form: i2 $\|\langle 001\rangle_{\alpha}$, i3 $\|\langle 111\rangle_{\alpha}$ and i5 $\|\langle 1 \tau 0\rangle_{\alpha}$.

Orientation relationships of type B can be expressed as follows: i5 $\|\langle 305\rangle_{\alpha}$, i3 $\|\langle 013\rangle_{\alpha}$ and i2 $\|\langle 001\rangle_{\alpha}$. This orientation relationship has been observed in samples of Al-based alloys containing transition metals, which have been annealed at $400^{\circ} \mathrm{C}$ for 100 h after produced by melt spinning technique. ${ }^{31)}$ The type B requires different nucleation of icosahedral particles from that in the type A because the icosahedral particles of the type A are produced as liquidsolid transformation products, on the other hand, the icosahedral particles of the type $B$ are produced through the solid-solid phase transformation.

The third form of orientation relationship (type C) that has been observed between icosahedral and crystalline phases is that five-fold axes are parallel to the $[110]_{c},[101]_{c},[113]_{c}$ and $[311]_{c}$ axes of the adjacent cubic phase. A three-fold axis is parallel to $[111]_{\mathrm{c}}$, and a two-fold axis is parallel to one of the $\langle 001\rangle_{\mathrm{c}}$ cube axes. This relationship has been observed in the $\mathrm{Al}-\mathrm{Li}-\mathrm{Cu}-\mathrm{Mg}$ alloys produced by the conventional ingot metallurgy route, in which the alloys were solution treated at $540^{\circ} \mathrm{C}$, then water quenched and subsequently annealed at $400^{\circ} \mathrm{C} .{ }^{32)}$

The last form of orientation relationship (type D) that has been observed in strip-cast $\mathrm{Al}-\mathrm{Mn}-\mathrm{Si}$ alloys with cooling rates in the range of $300^{\circ} \mathrm{C} / \mathrm{s}$ to $700^{\circ} \mathrm{C} / \mathrm{s}$, and subsequently heated at $400^{\circ} \mathrm{C}$ for typically $\sim 18 \mathrm{~min}^{23)}$ is as follows;

$$
\begin{aligned}
& \text { i5 } \|\langle 110\rangle_{\alpha} \text { and }\langle 113\rangle_{\alpha} . \\
& \text { i3 } \|\langle 111\rangle_{\alpha},\langle 123\rangle_{\alpha},\langle 115\rangle_{\alpha},\langle 234\rangle_{\alpha} \text { and }\langle 113\rangle_{\alpha} \text {. } \\
& \text { i2 } \|\langle 110\rangle_{\alpha},\langle 111\rangle_{\alpha},\langle 112\rangle_{\alpha},\langle 114\rangle_{\alpha},\langle 115\rangle_{\alpha},\langle 125\rangle_{\alpha} \\
& \text { and }\langle 225\rangle_{\alpha} .
\end{aligned}
$$

In the present research, a newly identified icosahedral structure of a quasicrystalline phase, as a product of solidsolid phase transformation during the conventional isothermal ageing process, shared a certain orientation relationship with the aluminium matrix, and they were established from the series of recorded microbeam electron diffraction patterns. The orientation relationships between the icosahedral quasicrystalline and matrix phases were as follows; i5 || $[011]_{\alpha}$ and $\mathrm{i} 3 \|[111]_{\alpha}$. These two superimposed axes between the icosahedral and aluminium matrix phases are exactly parallel to each other. However, the following
orientation relationships between icosahedral and aluminium matrix phases have very small misorientation;

$$
\begin{aligned}
& \text { i5 } \sim \|[110]_{\alpha} \quad \text { and } \quad \text { i5 } \sim \|[11 \overline{3}]_{\alpha} . \\
& \text { i3 } \sim \|[117]_{\alpha} \quad \text { and } \quad \text { i3 } \sim \|[31 \overline{2}]_{\alpha} . \\
& \text { i2 } \sim \|[112]_{\alpha}, \text { i2 } \sim \|[11 \overline{1}]_{\alpha} \quad \text { and } \quad \text { i } 2 \sim \|[10 \overline{1}]_{\alpha} .
\end{aligned}
$$

In comparison with the orientation relationship of type A; i2 $\|\langle 001\rangle_{\alpha}$, i3 $\|\langle 111\rangle_{\alpha}$ and i5 $\|\langle 1 \tau 0\rangle_{\alpha}$, although a threefold axis of the icosahedral phase is simultaneously parallel to a three-fold axis $\langle 111\rangle_{\alpha}$, which is consistent with the currently established orientation relationship, a two-fold axis of the icosahedral phase is not parallel to a four-fold axis of aluminium in the present research. In addition, orientation relationships of type B are also not compatible with the newly established orientation relationship in the current research because a five-fold axis of the icosahedral phase is not simultaneously parallel to a $\langle 305\rangle_{\alpha}$ axis in this research. Furthermore, although the producing process of the quasicrystalline phase in the present research is almost equivalent to that for type C , orientation relationship of type C is not consistent with the present observation because the two-fold axis of the icosahedral phase is not parallel to one of the $\langle 001\rangle_{\alpha}$ cube axes in the present research. Thus the orientation relationships established in the present research are not compatible with these orientation relationships, described as types A, B and C. However, the similar type of the orientation relationship has been found to that of type D.

### 4.3 Relationship between morphology and orientation

The microstructure of quasicrystalline particles observed in this research, as shown in Figs. 1, 2 and 3, revealed that the core of the grains contained a uniform distribution of many finer-scale (typically 20 nm ), faceted precipitate particles, while that the regions close to the grain boundaries contained coarse-scale, faceted precipitate particles. ${ }^{22)}$ These unique features of microstructures imply that quasicrystalline particles nucleate from the supersaturated solid solution during the conventional isothermal aging treatment, and these are significantly different from quasicrystalline particles observed in samples produced by rapid solidification processing.
It is worthwhile to note that the series of detailed microstructural observations revealed clearly the morphology of precipitate particles. The dominant particles of the icosahedral quasicrystalline phase exhibited the regular diamond shape in the $[1 \overline{1} 0]_{\alpha}$ orientation and one of square outline in the $[010]_{\alpha}$ orientation. The traces of the projected edges of the precipitate particles in the $[010]_{\alpha}$ orientation were parallel to the traces of $\{110\}_{\alpha}$ planes of the aluminium matrix. Further careful examinations of a regular diamond shape in the $[1 \overline{1} 0]_{\alpha}$ orientation revealed that there were, in fact, two distinguishable shapes within the particle array defined by two different angles of approximately 70 and $80^{\circ}$. When these particles were observed in the $[010]_{\alpha}$ orientation, there were also two apparent forms; one shape is almost rectangular or square in section and the other is not wellfaceted square-shaped particle. From these microstructural observations of the precipitate particles over a range of different orientations, it was deduced that they have the form
of rhombohedra.
Certain orientation relationships may have a coherent interface with minimum interfacial energy. When the icosahedral quasicrystalline precipitate particles form from a supersaturated solid solution during the conventional isothermal ageing, it shares an orientation relationship with the aluminium matrix phase. This is clearly demonstrated by the observed microstructures and electron microdiffraction patterns, shown in Figs. 3, ${ }^{22)} 5$ and 6. The morphology of both the diamond- and square-shaped particles in sections in the $\langle 110\rangle_{\alpha}$ and $\langle 001\rangle_{\alpha}$ orientations may be accounted for reasonably by the symmetry analysis proposed by Chan and Kolonji. ${ }^{33)}$ The intersection point group based on symmetry elements common to the icosahedral quasicrystalline and the matrix phases were determined and the superimposed axes between them were shown in Fig. 7. The three-fold rotational symmetry of the icosahedral phase is parallel to the three-fold axis of the matrix $\langle 111\rangle_{\alpha}$ orientation and the five-fold rotational symmetry of the icosahedral phase is parallel to the two-fold axis of the matrix $\langle 011\rangle_{\alpha}$ orientation. Most prominently, common symmetry elements from these parallel axes are $\overline{3}$, thus the intersection point group symmetry is defined as $\overline{3}$. The crystal forms compatible with this point group include a rhombohedron, which is consistent with the observed precipitate shape. Since the point group of the matrix phase $m \overline{3} \mathrm{~m}$ contains 48 order symmetry elements and the intersection point group $\overline{3}$ contains 6 , the index of the intersection point group with respect to the point group of the matrix phase is $8(48 / 6)$. Therefore, symmetry permits eight crystallographic variants of the precipitate particles in a given matrix orientation.

## 5. Conclusions

The icosahedral quasicrystalline metastable phase has been firstly observed in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ (mass\%) alloy aged for 20,30 and 40 min at $240^{\circ} \mathrm{C}$. The uniformly dispersed icosahedral particles are formed from the supersaturated solid solution (the $\alpha-\mathrm{Al}$ phase) by solid-state reaction. The morphology of those particles is a rhombohedron in shape that was confirmed by both TEM observation and the intersection point group symmetry analysis. The orientation relationship between the icosahedral quasicrystalline phase and the matrix phases has been identified as i5 $\|\langle 011\rangle_{\alpha}$ and i3 $\|\langle 111\rangle_{\alpha}$. This newly found icosahedral phase appeared to be the same orientation relationship (type D) that has been observed in strip-cast $\mathrm{Al}-\mathrm{Mn}-\mathrm{Si}$ alloys with cooling rates in the range of $300^{\circ} \mathrm{C} / \mathrm{s}$ to $700^{\circ} \mathrm{C} / \mathrm{s}$, and subsequently heated at $400^{\circ} \mathrm{C}$ for typically $\sim 18 \mathrm{~min} .{ }^{23)}$ The icosahedral quasicrystalline phase identified in this research belongs to the Al-ZnMg group with the quasilattice constant $a_{\mathrm{R}}=0.505 \pm$ 0.01 nm . The cubic lattice constant $a_{\mathrm{c}}$ of the six-dimensional simple-cubic lattice has a value of $1.390 \pm 0.028 \mathrm{~nm}$. This is in the good agreement with the lattice parameter of the prototype crystalline T phase $\left(\mathrm{Mg}_{32}(\mathrm{Al}, \mathrm{Ag})_{49}, a=1.416\right.$ $\mathrm{nm}){ }^{4)}$ The icosahedral quasicrystalline precipitate phase contained all three principal elements in the alloy, with the Ag content apparently significantly lower than the Mg content. It is thus not so surprising that the icosahedral phase
that appears initially coherent with the aluminium matrix phase should have an opportunity to form as a precursor to the crystalline T phase in this alloy system. It is interesting to note that the newly found quasicrystalline metastable precipitate particles appear to be the primary strengthening phase in the $\mathrm{Al}-10 \mathrm{Mg}-0.5 \mathrm{Ag}$ (mass\%) alloy aged at $240^{\circ} \mathrm{C}$. This result is a quite important to design the microstructures for automobile and aerospace applications.

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