

## SHAPES OF THE ICOSAHEDRAL QUASICRYSTALLINE PHASE IN MELT-SPUN RIBBONS

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Preliminary Note – Prethodno priopćenje

The shapes of icosahedral quasicrystalline (IQC) particles were determined in melt-spun ribbons of alloys based on the Al-Mn-Be alloy system. The sizes of the quasicrystalline particles ranged from a few tenths of nanometres up to 1  $\mu\text{m}$ . Therefore, different methods were employed for characterizing their shapes: projection of quasicrystalline particles using transmission electron microscopy (TEM), cross-sections of IQCs on metallographically polished surfaces, and observation of deep-etched samples and extracted particles using a scanning electron microscope (SEM). It was discovered that icosahedral quasicrystalline particles preferentially grow in three-fold directions and have a tendency for faceting and adopting the shape of a pentagonal dodecahedron. The evolution of quasicrystalline shapes is systematically presented.

*Key words:* Al-alloy, metallography, ribbon, icosahedral quasicrystalline phase, shape, melt-spinning

### INTRODUCTION

The point-group symmetry of a crystal dictates its morphology [1]. Quasicrystals possess non-crystallographic point-group symmetries. Thus different morphologies from those of periodic crystals may appear. The normal crystal should be bounded by flat surfaces at 0 K, satisfying the condition of the minimum total surface energy.

The temperature increases the disorder of the surface, and the sharp edges and corners of the crystal at 0 K start rounding. The transition from a faceted plane to a smoothly curved one takes place at the roughening transition temperature. The roughening transition temperature generally scales with the lattice parameter for a crystalline lattice. Thus, a crystal with a larger lattice parameter shows more faceting tendencies. The quasicrystal can be considered as a periodic crystal with infinite periodicity, therefore the roughening transition temperature should be infinite too. This means that it should be faceted up to the melting temperature. The experimental evidence ruled out this expectation [2], since the quasicrystals in the Al-Mn system often possessed the form of well-rounded dendrites. It could be inferred that the non-faceted growth of quasicrystals is due to dynamic roughening at high-melt undercooling. This leads to a continuous growth and yields a rounded growth form.

The morphology of quasicrystals was initially carried out for Al-Mn and Al-Mn-Si icosahedral quasicrystals. In most cases a dendritic structure was present with branches extending in the preferred threefold directions

[2], Chattopadhyay et al. [3] found a well-faceted morphology for quasicrystals. Thangaraj et al. [4] discovered that the pentagonal dodecahedron was the shape for the Al-Mn type of quasicrystal. Pentagonal dodecahedron was found to be the equilibrium shape for most stable quasicrystals [5]. In some cases, also quasicrystals with the icosidodecahedral morphology were also observed. A triacontahedral growth morphology of icosahedral quasicrystals was observed in several alloy system, such as:  $\text{Sc}_{12}\text{Zn}_{88}$  [6], Al-Li-Cu [7], Al-Mg-Zn [8] and Zn-Mg-Y face-centred icosahedral alloys [9]. The preferred growth direction was mainly along the threefold axes and occasionally also along the fivefold axes [2, 10].

The aim of this work was to determine the shape of quasicrystals within melt-spun Al-Mn-Be alloys with the addition of B.

### EXPERIMENTAL

The chemical compositions of melt-spun ribbons are given in Table 1.

Table 1 **Chemical compositions of the investigated alloys (ICP-AES) / wt.%**

alloy	Al	Mn	Be	B
Al-Mn-Be	90,6	5,4	4,0	-
Al-Mn-Be-B	92,33	3,93	0,77	2,97

The samples were melt-spun using a melt spinner (30M, Marko Inc). The wheel speed of the melt spinner varied between 19,6 m/s and 25,2 m/s.

Preparation of the samples for light-optical microscopy (LOM) and scanning electron microscopy (SEM)

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followed the standard mechanical metallographic procedures. In addition, considerable attention was given to the deep etching and particles' extraction techniques. The samples were observed under a Nikon Epiphot 300 light microscope and a FEI, Sirion 400 NC, scanning electron microscope, equipped with an INCA 350, Oxford Instruments EDS-analyser.

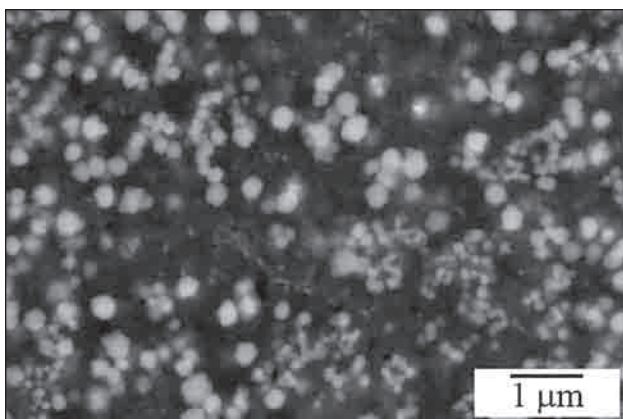
Transmission electron microscopy (TEM) was carried out in a FEI TITAN 80–300. The TEM specimens for the TITAN were cut out at specific sites using the focussed ion beam (FIB) in a FEI Nova 200 Nanolab.

## RESULTS AND DISCUSSION

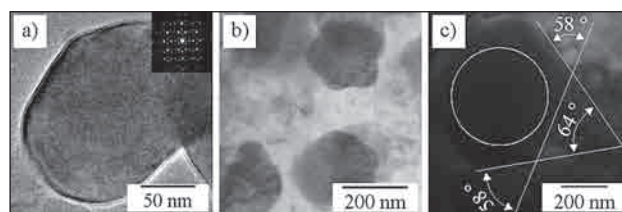
Melt spinning enables the study of equilibrium shape and the roughening behaviour of the quasicrystals. The rapid solidification of dilute Al-Mn-Be and Al-Mn-Be-B alloys makes possible substantial undercooling, thus promoting nucleation of the quasicrystal in the melt [11]. Later the Al nucleates and its very fast growth enables the trapping of small quasicrystallites.

Figure 1 shows a microstructure typical for 30–90  $\mu\text{m}$  thick ribbons. These ribbons possessed a rather uniform distribution of tiny spherical quasicrystalline particles with sizes between 50 nm and 500 nm in diameter, in an  $\alpha$ -Al matrix. Backscattered electron images indicated that some particles had spherical shapes, whilst some of them showed a tendency for faceting, however the resolution was normally too low to enable clear determination of shape.

Closer examination was possible using transmission electron microscopy. Typical transmission electron micrographs with the corresponding diffraction patterns are shown in Figure 2. The extracted particles from the Al-Mn melt-spun ribbons had rounded edges with sizes up to 100 nm (Figure 2a). On the other hand, particles can often exhibit equiaxed form (Figure 2b). Some of them had almost ideal spherical morphologies, whereas bumps could be seen on others. On the other hand, a particle in Figure 2.c possessed faceted morphology. In a TEM micrograph taken along a twofold axis the particle had a hexagonal shape. The angles between the edges were approximately  $58^\circ$  and  $64^\circ$ .



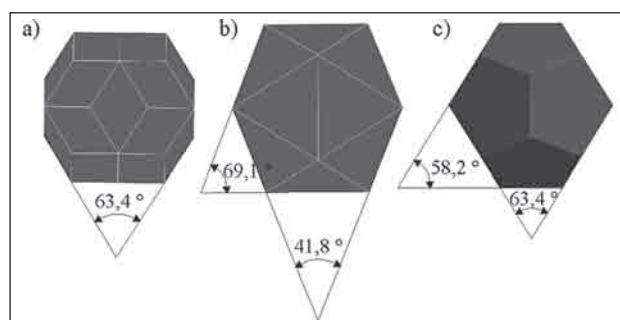
**Figure 1** Backscattered electron images of quasicrystalline particles in Al-Mn-Be alloy



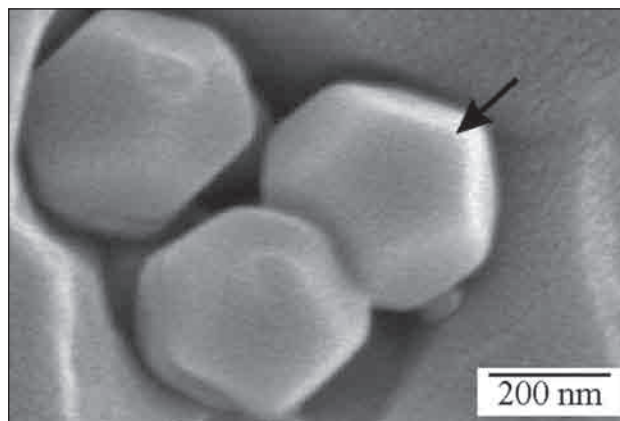
**Figure 2** I-phase in melt-spun ribbons. a) Extracted particles of i-phase in the alloy Al-Mn, the image was taken along twofold axis, b) bright-field micrograph of the alloy Al-Mn-Be and c) bright-field electron micrographs of the alloy Al-Mn-Be-B, the image was taken along a twofold axis.

The triacontahedron, icosahedron and pentagonal dodecahedron can only be distinguished when a TEM-micrograph is taken along a twofold direction (Figure 3). The angles of  $58^\circ$  and  $64^\circ$  are typical for the pentagonal dodecahedron [4]. Thus the addition of other elements (Be and B) did not change the shapes of the faceted particles. The results of the TEM study were confirmed by deep etching. This procedure revealed the 3D-shapes of the quasicrystalline particles. The pentagonal shape of the indicated particle's facet in Figure 4 can be clearly seen. It could be inferred that the particle had the shape of the pentagonal dodecahedron.

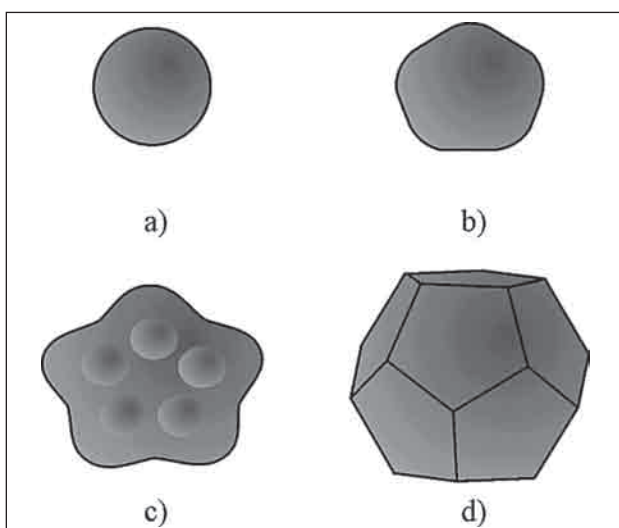
The results can be related to undercooling. Dynamic roughening is more pronounced at large undercooling, therefore almost spherical particles form.



**Figure 3** Shapes of icosahedral quasi-crystalline particles can be clearly distinguished in those projections along the twofold axis [4]. Projection of the a) triacontahedron, b) icosahedron and c) pentagonal dodecahedron along the twofold axis.



**Figure 4** Quasicrystalline particles with the shape of pentagonal dodecahedron in deep etched Al-Mn-Be-B alloy.



**Figure 5** Morphologies of IQC observed in melt-spun ribbons. a) sphere, b) particle with rounded edges, c) particle with bumps, d) pentagonal dodecahedron

With the decreasing cooling rate, the particles possess some flat faces with rounded corners. In some cases, constitutional undercooling can lead to morphological instability of the solid-liquid interface, resulting in the formation of bumps. Such development can lead to the formation of large dendrites with rounded arms [2]. At smaller undercooling, the effect of dynamic roughening diminishes, resulting in the formation of almost ideal pentagonal dodecahedrons. The schematic presentation of IQC-shapes found in melt-spun ribbons, is shown in Figure 5.

## CONCLUSIONS

The shapes of icosahedral quasicrystalline (IQC) particles were determined in melt-spun Al-Mn-Be-(B) alloys.

During melt spinning, the following shapes of IQC were observed: spheres, particles with rounded edges,

particles with bumps and particles with the shape of pentagonal dodecahedron. The sizes of these particles were up to 500 nm.

## Acknowledgements

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**Note:** Responsible for English language is Mr. George Yeoman, Maribor, Slovenia