Quasicrystal-strengthened cast Al-alloys

Aluminijeve livne zlitine, utrjene s kvazikristali

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Abstract: Modern engineering materials must be able to carry high load and simultaneously possess high reliability. Among convenient materials, aluminium alloys still play an important role, in spite of the rapid development of lighter magnesium alloys and different types of composites. In Al-Si casting alloys alloyed with different additions (e.g. AlSi12CuNiMg) tensile strengths up to 400 MPa can be attained, and the elongations around 5 %. In order to increase strength, dislocation mobility should be impeded or hindered. The effect of microstructure on fracture toughness is rather complex. Nonetheless, by the presence of only coherent precipitates strain localization can occur leading to rather low ductility and fracture toughness. Therefore, the presence of incoherent precipitates is desirable. Larger particles of secondary phases often impart ductility and toughness, especially when they grow in the form of needles and/or plates. They can serve as crack nucleation sites, and when they fracture, also as crack propagation paths.

The increase in strength normally results in decrease of ductility and toughness. A lot of work had been and still is directed to obtain appropriate combination of strength and ductility for particular application. This is possible by obtaining a hierarchical microstructure consisting of constituents of different sizes to produce various types of strengthening, but also to make the propagation of cracks more difficult. It was found that quasicrystals possess the ability

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to strengthen an aluminium matrix, and make more difficult crack formation and growth. Recently, some Al-alloys were found to have rather high quasicrystalline forming ability. This means that the icosahedral quasicrystalline phase (i-phase) can form during rather slow cooling, at cooling rates typical for solidification in metallic dies (cooling rates are estimated to be between 100 K/s and 1000 K/s). In some alloys, almost ideal two-phase microstructure consisting of α-Al and i-phase will form having convenient combination of strength and toughness. So in this contribution, some alloys of this type developed over the last few years will be presented.

Izvleček: Od sodobnih tehničnih materialov zahtevamo, da imajo veliko trdnost, hkrati pa morajo biti zanesljivi in varni. Med njimi imajo Al-zlitine še vedno velik pomen kljub hitremu razvoju še lažjih magnezijevih zlitin in različnih vrst kompozitnih materialov. V livnih zlitinah na osnovi Al-Si, ki vsebujejo različne zlitinske elemente (npr. AlSi12CuNiMg), se dosežejo natezne trdnosti do 400 MPa, medtem ko je razteznost le do 5-odstotna. Za povečanje trdnosti kovin je treba zmanjšati gibljivost dislokacij. Vpliv mikrostrukture na lomno žilavost je bolj zapleten. Ob navzočnosti koherentnih izločkov se lahko zmanjša žilavost zaradi lokalizacije deformacije, zato je navzočnost nekoherentnih izločkov koristna. Veliki delci sekundarnih faz (čisti elementi – Si, binarne spojine Al_3Fe ali kompleksne intermetalne faze) imajo negativen vpliv na žilavost in duktilnost, še posebej, če rastejo v obliki iglic ali ploščic. Na njih lahko nastanejo razpoke, lahko pa so tudi prednostna mesta za rast razpok.

Povečanje trdnosti je navadno povezano z zmanjšanjem žilavosti, zato je veliko raziskav usmerjenih k cilju, kako doseči primerno kombinacijo trdnosti in žilavosti za določeno aplikacijo. To je mogoče, če dosežemo mikrostrukturno hierarhijo, ki vsebuje mikrostrukturne sestavine različnih velikosti in lahko povzroči raznovrstno utrjanje, hkrati pa oteži napredovanje razpok. V zadnjem obdobju je bilo veliko raziskav namenjenih razvoju aluminijevih zlitin, utrjenih s kvazikristali. Za ikozaedrično kvazikristalno fazo (i-fazo) velja, da povzroči močno utrjanje aluminijeve osnove, hkrati pa oteži nastanek in napredovanje razpok. Šele pred kratkim so bile razvite zlitine, pri katerih nastane i-faza tudi pri sorazmerno počasnem ohlajanju, kot je npr. pri litju v kovinske kokile, kjer je hitrost ohlajanja 100−1000 K s^{-1}. Znani so primeri, da imajo skoraj idealno dvofazno mikrostrukturo ter ustrezno razmerje trdnosti in žilavosti. V tem prispevku predstavljamo nekaj zlitin, ki so bile razvite v zadnjih letih.

Key words: aluminium, quasicrystal, strengthening, mechanical properties
Ključne besede: aluminij, kvazikristal, utrjanje, mehanske lastnosti

Introduction

Aluminium alloys belong to the most important engineering materials. They can find their application in the most demanding areas, such as automotive and aerospace industries. Al-alloys with properties tailored for particular requirements can be utilized for the most critical constructional elements. This was made possible by exploiting the highly sophisticated casting technologies, such as SOPHIA and the latest methods of die-casting.[1] The building of aeroplanes normally requires the highest possible strengths, whereas car building demands the highest values of elongations, that can be attained at the lowest costs.[2] Very frequently, the convenient combination of strength and elongation (or ductility) is required. This can be expressed by the alloy index Q:

$$Q = R_m + d \cdot \lg(a)$$

where R_m stands for tensile strength, A is elongation at fracture, and d is an empirical constant. [3]

Table 1. Basic data regarding high-strength Al-cast alloys [4]

alloy	numerical designation	condition	yield strength $R_{p0,2}$/MPa	tensile strength R_m/MPa	elongation A/%
AlSi12CuNiMg	48 000	die cast / T6	320–390	350–400	0,5–2,5
AlSi5Cu3Mg	45 100	die cast / T6	310–380	420–450	2–7
AlSi9Mg1		die cast / F	240–310	320–400	2–4
AlCu4TiMg	21 000	die cast / T6	260–380	360–440	3–18
AlCu4MgAgTi		die cast / T6	410–460	460–510	5–8
AlCu4Ti	21100	die cast / T6	310–400	420–475	7–16

TENSI and HÖGERL[2] have shown that the strengthening of Al-alloys based on the Al-Si system can be considered as a combination of several hardening mechanisms:

- solid-solution strengthening (alloying with elements soluble in Al, having rather different atomic sizes than Al)
- grain boundary strengthening caused by the decreasing grain size of the matrix that can be caused through grain refinement and controlling the casting parameters
- texture hardening obtained by directional solidification
- particle hardening
- large particles, e.g. Si-phase in the eutectic (modification, casting parameters)
- dispersion hardening (uniformly distributed incoherent particles)
- precipitation hardening (coherent particles, heat-treatment).

In metals and alloys, the mobility of dislocations should be hindered in order to achieve hardening. Consequently, the stress for transition from elastic to plastic deformation is increased. On the other hand, elongation (ductility) can be increased by preventing the formation and propagation of cracks.

The highest yield strength, R_{p02}, that can be obtained in Al-Si alloys is slightly below 400 MPa, whereas the limiting value for the tensile strength, R_m, is approximately 450 MPa. However, this strength levels cause decrease in elongation at fracture to only few percents. Table 1 indicates that the highest strengths can be obtained in precipitation-hardened alloys. Nevertheless, the properties of these alloys are strongly influenced by the inherent properties of silicon. Silicon is a semimetal; it is brittle, having low fracture toughness and hardness around 1000 HV. It can be expected that the replacement of silicon with a harder, but at the same time tougher phase, may provide alloys with both higher strength and toughness. Many of the intermetallic phases found in the Al-alloys possess similar or even higher hardness than that of silicon, but, unfortunately, their fracture toughness is even lower. In addition, they are prone to grow in the form of needles or plates, which makes the resistance of an alloy against the crack propagation even inferior.

There is a strong interest in the automotive and aerospace industries for the application of alloys having better combinations of strength and elongation than alloys currently in use. In this respect, activities are carried to improve the properties of existing alloys, as well as to develop completely new alloys, such as those strengthened by quasicrystals.

Measures for attaining the highest strengths

Limiting strengths of Al-alloys can be calculated based on the aluminium modulus of elasticity, E = 70 GPa, and shear modulus, G = 26 GPa. Thus, the theoretical shear strength τ_t could be $G/15 \approx 1700$ MPa. This is a stress, at which elastic-to-plastic transition would take place in aluminium in the absence of dislocations. The cohesive strength of materials, σ_t, is approximately $E/15$. This is theoretical rupture strength of a material in the absence of cracks and/or other defects causing local stress peaks. It is known, that the real strengths can only approach to this theoretical values. They can be achieved most likely when the sample sizes decrease to micro- or even in the nanoregion (e.g. in whiskers), but they cannot be expected in castings for e.g. aerospace and automotive industries.

In »Inoue Superliquid Glass Project«, lead by Prof. Inoue on Tohoku University, Sendai, Japan, a range of novel aluminium alloys containing quasicrystals was developed.[5] They reported that by using powder metallurgy methods (gas atomisation of melt, densification with warm extrusion or pressing) it was possible to attain a wide range of properties of aluminium alloys that cannot be achieved by conventional methods (Figure 1). They have developed several alloys with combined high-strength and ductility (Al-Mn-M, Al-Cr-M and Al-Cr-Mn-M where M

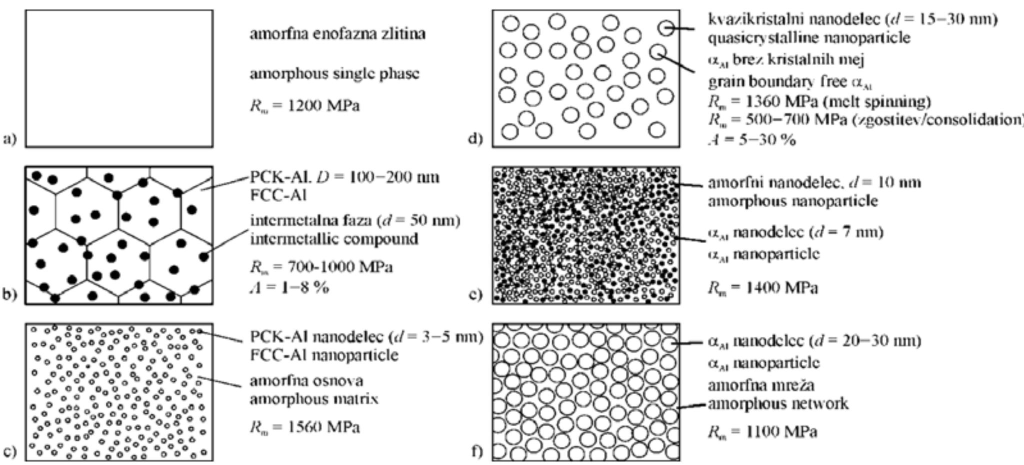


Figure 1. Microstructures formed during rapid solidification of special Al-alloys developed be Inoue and Kimura.[5] Using conventional casting technologies one can obtain a microstructure shown in d), however the particles would be much coarser.

stands for one or several alloying elements: Cu, Ni, Fe, Ti or Zr). Chromium and manganese were responsible for the formation of icosahedral quasicrystals on rapid solidification, whilst other elements provided either solid solution or precipitation hardening (Cu). Among the above-mentioned alloys, the alloys arising from the Al-Mn-Cu system possessed comparable strength to commercial high-strength aluminium alloys (500–600 MPa), but with much better ductility (elongation >20 %). The appropriate microstructure, consisting of aluminium solid solution and nanoscale i-phase particles, was achieved in the smallest particles only (Figure 2d). In these alloys, the i-phase did not form during slower cooling.

So far, there is no information regarding the practical applications of these alloys. Perhaps oxide layers formed during powder processing prevented adequate sintering, and thus achieving of designed – theoretically accessible – properties.

Casting alloys strengthened by quasicrystals

In order to increase the strength the dislocation mobility should be impeded. This can be achieved through interaction with atoms (solid solution strengthening); the effect is proportional to the differences in atomic sizes between aluminium and alloying elements. The effect is limited because only a small number of elements (Cu, Mg and Zn) have a considerable solubility in aluminium. In some aluminium alloys (Al-Cu, Al-Zn, Al-Mg-Si) coherent precipitates form during natural or artificial aging causing considerable hardening. In alloys containing chromium and manganese, complex incoherent precipitates form after faster cooling and additional aging. They do not contribute much to the hardening, but they prevent grain growth and softening due to recrystallization. Larger secondary phases that are often present in Al-alloys do not cause considerable hardening; their effect on properties can be calculated by using a simple rule of mixtures. The effect of microstructure on fracture toughness, on the other hand, is rather complex. Nonetheless, the presence of only coherent precipitates can cause strain localization leading to rather low ductility and fracture toughness. Therefore, the presence of incoherent precipitates is desirable. Larger particles of secondary phases (pure elements – Si, binary compounds – Al_3Fe or complex intermetallic phases) often have an adverse effect on ductility and toughness, especially when they grow in the form of needles and plates. They can serve as crack nucleation sites, and when they fracture, as crack propagation paths.

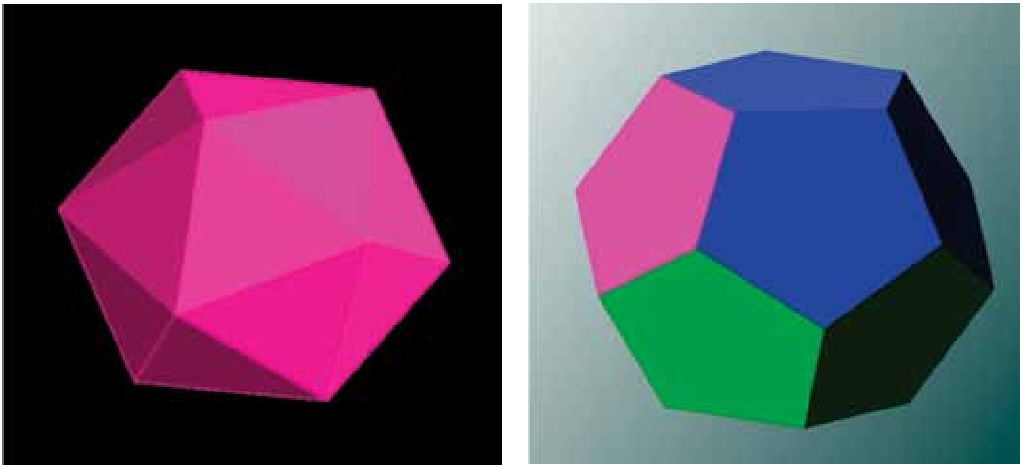


Figure 2. Possible shapes of quasicrystals with icosahedral symmetry: a) icosahedron, b) pentagonal dodecahedron.

The increase in strength normally results in decrease of ductility and toughness. A lot of work is directed to obtain appropriate combination of strength and ductility for particular application. The idea is to obtain hierarchical microstructure consisting of microstructural constituents of different sizes to produce various types of strengthening, but also to make the propagation of cracks more difficult. It would be of the utmost importance for particles to form in-situ during solidification or solid-state reactions because the interface strength is much higher in this case.

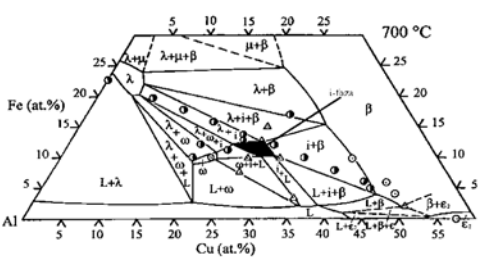
Icosahedral quasicrystalline phase (i-phase) belongs to the quasiperiodic crystals that were first discovered at the beginning of nineteen eighties by Shechtman et al. in Al-Mn system.[6] This phase has an orientational long-range order, but it lacks periodicity. That was rather revolutionary discovery at the time because of strong belief that the solids can only have periodic crystalline or glassy (amorphous) structure. Also, quasicrystals possess some unusual mechanical, magnetic and electrical properties, e.g. strain softening because of quasiperiodicity.

This can be shown in an icosahedron (Figure 2a), which has very high symmetry. Namely, it possesses six fivefold, ten threefold and fifteen twofold rotational axes.[7] Therefore, it can attain several different orientations with other phases. It is even possible to attain epitaxy on almost all interfaces. Small mismatches can be adapted by dislocations.[8] On this ground, it is rather safely to state that i-phase represents almost ideal strengthening phase for the Al-alloys. It was discovered that threefold directions present predominant growth directions, thus i-phase could in general grow in twenty different directions. During faceted growth, i-phase often adopts a shape of pentagonal dodecahedron (Figure 2b). It is of utmost importance that even during dendritic growth the spherical shape is attained, because of a strong tendency to equiaxed growth. In addition, this shape causes the smallest stress concentrations, thus making it more difficult for crack to form and grow.

In aluminium alloys stable and metastable icosahedral quasicrystal (i-phase) can be formed. The trouble with the stable i-phase is that it is not present in the equilibrium with α-Al phase (Al-rich solid solution, which is ductile and tough), which is the case in Mg-alloys from the system Mg-Zn-Y.[9] Contrary, the i-phase field of existence is surrounded by brittle intermetallic phases[10] (Figure 3a), making these materials inconvenient for structural applications. On the other hand metastable i-phase can be present in α-Al, however it can only be formed at higher cooling rates (during melt-spinning or melt atomization at cooling rates of 10^6 K/s) [6], because it is not a part of the equilibrium binary phase diagram (Figure 3b).[11]

SCHURACK et al.[12, 13] wanted to improve properties of aluminium alloys by applying strengthening with the quasicrystalline phases. Alloys were produced by melt spinning, mechanical alloying and conventional casting. They reported that mechanical alloying of stable AlCuFe-quasicrystals and aluminium powders did not give the required

a)



b)

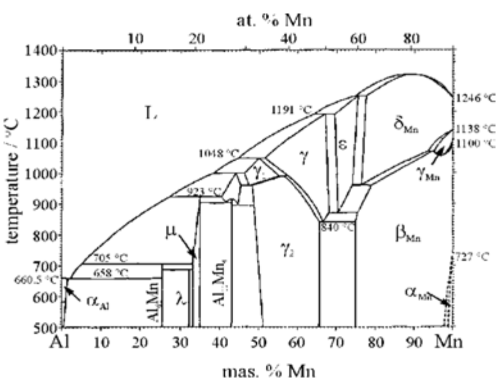


Figure 3. a) Isothermal section through the Al-Cu-Fe system at 700 °C. Near i-phase several intermetallic phases exist.[10] b) Phase diagram Al-Mn in Al-corner, where no i-phase region is. Thus, i-phase and other quasicrystalline phases can form by rapid solidification only.[11]

combination of strength and ductility. However, Ce-addition to Al-Mn-alloys improved the quasicrystalline forming ability, presumably due to stabilisation of the icosahedral structure in the melt. This allowed direct formation of icosahedral quasicrystals during continuous cooling of the melt. The milled and extruded melt-spun ribbons of the alloy $Al_{92}Mn_6Ce_2$ attained strength of approximately 800 MPa and elongation ≈25 %, but the conventionally cast rods had strength around 500 MPa and elongation ≈20 % only. It could be inferred that cerium represented an effective addition element to Al-Mn alloys, but it is unlikely, due it its high cost and reactivity to produce the Ce-containing alloys in an economic sound way.

Song et al.[14] found out that the addition of beryllium strongly reduced the critical cooling rate for the formation of quasicrystals and optimized the required Mn-content in Al-Mn alloys. They established that quasicrystals also formed using conventional casting methods, e.g. die-casting. However, in their alloys additional intermetallic phases (hexagonal approximants) were always present. Additional intermetallic phases besides i-phase were also discovered in other investigations (Table 2). Typical microstructures of alloy with several intermetallic phases and almost ideal two-phase (α-Al + i-phase) microstructure are shown in Figure 4.

Jun et al.[23] used »Mischmetal« instead of cerium. Mischmetal is much cheaper and commercially available. They attained almost two-phase microstructure α-Al + i-phase. Similar achievement was attained by Zupanic et al.[16] in a four-component Al-Mn-Be-Cu alloy (Figure 5a). Figure 5b shows microstructu-

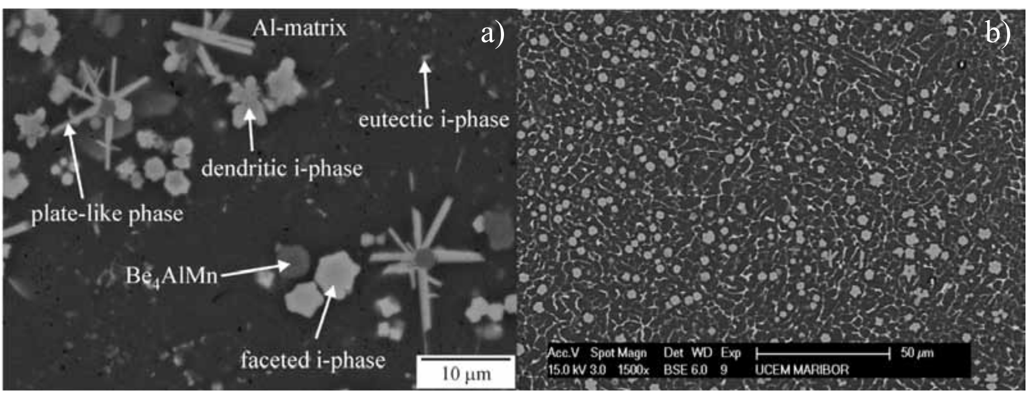


Figure 4. Microstructures of Al-alloys containing quasicrystalline phase: a) $Al_{84}Mn_5Be_{11}$ alloy[15] and b) $Al_{94}Mn_2Be_2Cu_2$ alloy[16]

Table 2. Phase composition of cast Al-alloys in which i-phase was observed

alloy	casting method	phase composition	reference
$Al_{91}Mn_7Fe_2$ $Al_{92}Mn_6Ce_2$ $Al_{80}Pd_{15}Mn_5$, $Al_{90}Pd_8Mn_2$	centrifugal casting	α-Al, i-phase, Al_6Mn $Al_{10}Mn_7Ce_2$, Al_6Mn, Al_4Ce, α-Al, i-phase α-Al, i-phase, Al_3Pd	[13]
$Al_{92}Fe_3Cr_2Mn_3$	wedge casting centrifugal casting	$Al_{75.7}Cr_{8.2}Mn_{8.4}Fe_{7.7}$ $Al_{75.7}Cr_{8.2}Mn_{8.4}Fe_{7.7}$ α-Al, Al_6Mn, $Al_{12}(Cr,Mn)$	[17]
$Al_{93}Fe_3Cr_2Ti_2$	suction casting	α-Al, $Al_{13}(Fe, Cr)$, Al_3Ti, i-phase	[18]
$Al_{90}Mn_{2.5}Be_{7.5}$, $Al_{79.9}Mn_{13.5}Be_{6.6}$, $Al_{93}Mn_{2.5}Be_{4.5}$, $Al_{80}Mn_{13.5}Be_{6.5}$ $Al_{83}Mn_6Be_{11}$	conventional casting injection moulding cone shape die	α-Al, i-phase, hexagonal approximants	[14, 15, 19-21]
$Al_{79.9}Mn_{13}SiBe_6$	conventional casting	α-Al, i-phase, α-Al-Mn-Si 1/1 cubic approximant	[22]
$Al_{94}Mn_2Be_2Cu_2$	conventional casting	α-Al + i-phase	[16]
$Al_{92}Mn_6Mm_2$ $Al_{90}Mn_6Mm_4$ $Al_{88}Mn_6Mm_6$	conventional casting	α-Al + i-phase	[23]

Table 3. Possible aluminium alloys containing quasicrystals

alloying elements required for formation of i-phase	alloying elements reducing critical cooling rate	alloying element enabling precipitation hardening
Mn, Cr, (V)	Ce, Be, Si, Cu, Fe, B, Mm (Mischmetall)	Cu, Zn, (Si + Mg)

re after compressive test, when the logarithmic deformation was 0.7. No cracks can be observed near i-phase particles, whereas the hexagonal phase has fractured. Also at Al_2Cu-particles, micropores have formed that can represents initiation sites for formation of microcracks.

Figure 6 shows characteristic tensile diagrams of the alloys Al-Mn-Mm [23]. It can be seen that very high strength is achieved in the as-cast condition, and a rather high elongation is retained. Addition of 6 % Mn is too high, because the alloy became too brittle.

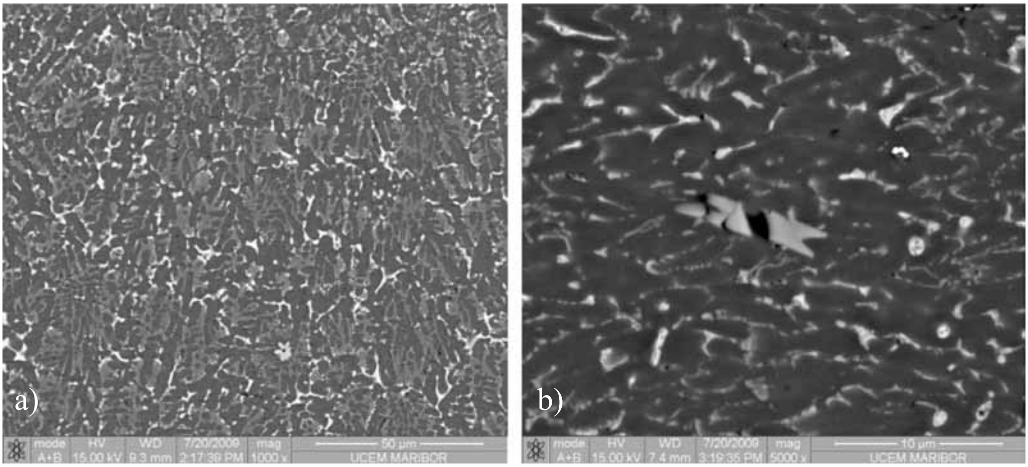


Figure 5. a) Microstructure of predominantly two-phase alloy i-phase + α-Al in the as-cast condition (mould diameter was 2 mm). i-phase has a shape of primary particles with a dendritic morphology, and it is present as a part of a binary eutectic (α-Al + i-phase). b) Microstructure after compressive test, logarithmic deformation was 0.7. No cracks can be observed near i-phase particles, whereas the hexagonal phase has fractured. At Al_2Cu-particles, micropores are formed that was found to form microcracks that can cause fracture.

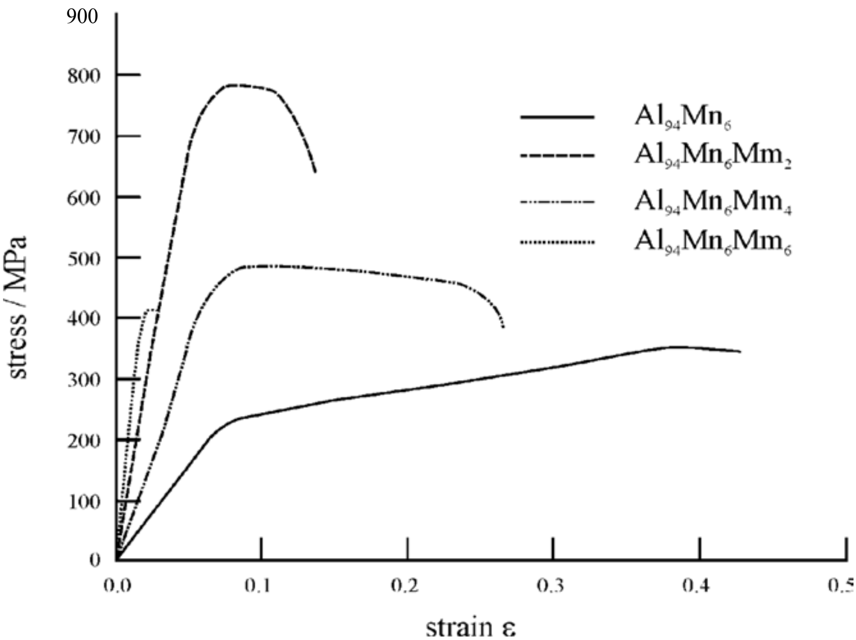


Figure 6. Tensile diagrams of alloys based on the Al-Mn-Mn system [23]

Further perspectives of quasicrystal-strenghtened Al-alloys

The main requirements for the quasicrystal-strengthened Al-alloys are:

- predominant two-phase microstructure consisting of α-Al + i-phase in the casting with higher wall thickness (the initial goal should be 10 mm)
- i-phase must be stable enough to survive required heat or thermomechanical treatment
- precipitation hardening is possible, in order to attain required combination of strength and elongation
- it is possible to attain microstructural hierarchy that allows several hardening mechanisms to be simultaneously active (using microstructural engineering)
- rupture strength should be higher than 400 MPa, and elongation at fracture larger than 10 %
- the liquidus temperature should be lower than 750 °C.

One of the main goals is the optimisation of chemical composition. The alloying elements can be divided according to their main effect (Table 3). It is necessary to take into account both positive and negative effects arising from interactions between different alloying elements. Most of them cannot be predicted because all of these alloys are or should be multicomponent. This means that fundamental researches will also be necessary, because not all ternary phase diagrams in question are available.

For further improvements, it is also necessary to stimulate development of new innovative casting technologies that will enable faster cooling rates. In this regard, it would be necessary to determine experimentally the critical cooling rates for the formation of the i-phase. By application of solidification simulations, it will then be possible to predict where in the casting will the required microstructure form itself.

Conclusions

According to the literature survey and our own experimental results, it could be stated that casting Al-alloys strengthened by quasicrystals possess a potential for becoming a new generation of alloys for special applications.

Nevertheless, for attaining this goal several obstacles should be overcome. Thus several fundamental investigations are required (determination of corresponding stable and metastable phase diagrams), as well as more applicative and development work (improvement of casting technologies, determination of critical cooling rates).

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