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Influence of Fe addition on the formation of a quasicrystalline phase in bulk Al-rich Al—Mn base alloys

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ABSTRACT

This work evaluates the effect of Fe addition on the formation of a quasicrystalline phase in the 94Al—6Mn base alloys obtained by wedge casting. Different Mn to Fe ratios (1, 1.4, 2, 5) were used to estimate Fe content that leads to formation of a quasicrystalline icosahedral phase (I-phase) at the selected Al concentration (94 at.%). Based on the obtained results, Fe enhances I-phase nucleation in the studied ternary alloys compared to the non-modified binary composition. Although the maximum thickness of the cast wedge at which the I-phase was formed in the prepared alloys varied with Fe content, its nucleation was always limited to a certain range of cooling rates above 10^3 K/s. Hardness increased significantly for areas composed of Al matrix and I-phase particles. The highest HV values were obtained for parts of castings where the microstructure consisted of very fine I-phase/Al eutectic.

1. Introduction

The quasicrystalline phase has been reported to be an effective strengthening agent in various aluminium alloys modified by transition metal additions. This includes primary metastable icosahedral particles (I-phase) formed by rapid solidification [1–5], metastable precipitates formed after aging [6–7], and composite materials where stable quasicrystals are mixed with pure Al or an alloy powder and subsequently consolidated [8–10]. The first approach results in high strength (above 600 MPa) [1–5] due to a relatively high fraction of fine I-phase particles in the microstructure and a coherent particle/matrix interface confirmed by the presence of crystallographic relations between the two phases [11–13]. The disadvantage are high cooling rates 10^4–10^9 K/s necessary for I-phase formation which limit at least one size of samples to tens of microns and lead to additional processing steps in order to obtain bulk forms. Quasicrystal-strengthened Al—Mn alloys have been obtained at considerable lower cooling rates using casting into thin copper molds [14–16]. This was possible due to Be addition which was found to strongly enhance I-phase nucleation in the alloy [17,18]. However, beryllium is known for being carcinogenic and causing other serious diseases as berylliosis [19]. Thus, other elements that could enhance the I-phase formation during conventional casting processes are desirable. Cerium was reported to be an effective addition improving the I-phase formation in Al—Mn alloys under different solidification conditions [20,21]; nonetheless, its effectiveness was recently disputed by Coury et al. [22].

Previously, we have reported the enhanced I-phase formability in the 94Al—6Mn thin rods modified by Fe addition (2 at.%) obtained by suction casting into cylindrical copper molds with different diameters [23]. Formation of the two-phase (I-phase/Al) microstructure in samples cast at slower cooling rates could lead to application of these materials to industrial processes such as die casting, a technique which is currently applied to various aluminium alloys including Al—Cu, Al—Si and Al—Mg base materials. This technique allows for obtaining thin-walled products with a wall thickness of 1–1.5 mm. Since the cooling rates achievable in this method are between 50 and 500 K/s [24] the desired microstructure needs to be formed at the required range.

This work evaluates the effect of Fe addition on the formation of a quasicrystalline icosahedral phase in the Al—Mn alloy cast at intermediate cooling rates (10^3–10 K/s) obtained by simple gravity casting into a wedge-shaped copper mold. Different Mn:Fe ratios were used to determine the concentration range in which a dual I-phase/fcc-Al matrix microstructure can be formed at given solidification conditions.

2. Material and methods

Alloys of the following nominal compositions (at.%): 94Al—6Mn, 94Al—5Mn—1Fe, 94Al—4Mn—2Fe, 94Al—3.5Mn—2.5Fe, 94Al—3Mn—3Fe were prepared by melting a mixture of pure elements (Al 99.99, Mn 99.99, Fe 99.99 at.%) in a resistant furnace under argon atmosphere. The samples were subsequently cast into a steel mold whose cavity had the size of

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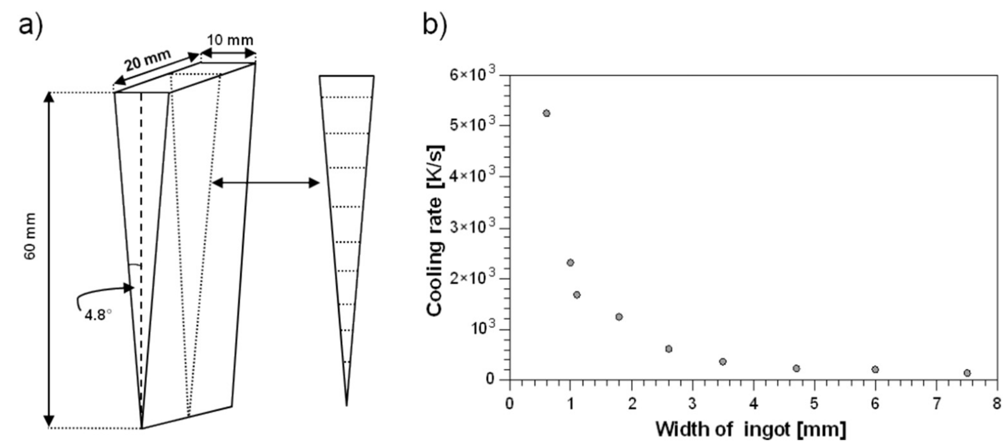

Fig. 1. a) Schematic of the mold cavity used in the experiment and the part of the sample cut to perform microstructure analysis at different thicknesses. b) Width of the wedge-shaped ingot along its longitudinal cross-section over cooling rates calculated for particular region based on cell spacing in the reference alloy.

20 × 30 × 60 mm. The resulting chemical composition was confirmed by energy dispersive X-ray spectroscopy (EDS) in a scanning electron microscope (SEM). The prepared ingots were cut into pieces, re-melted (at 1300 K) and obtained by gravity casting into a wedge-shaped copper mold. Calibration of the cooling rates was done by analyzing the microstructure of a reference 98Al−2Cu at.% alloy. The microstructures of castings were examined using FEI scanning electron microscope E-SEM XL30, and FEI transmission electron microscope Tecnai SuperTWIN G2 (TEM) operating at 200 keV equipped with a field-emission gun (FEG) and high-angle annular dark field scanning transmission electron microscopy detector (HAADF-STEM). Preparation of the thin foils was done using Tenupol-5 (Struers) double jet electropolisher and electrolyte containing nitric acid and methanol (1:3) at the temperature of 243 K and voltage of 15 V. Hardness was measured using Vickers indenter under a load of 10 N.

3. Results and discussion

Wedge casting enables to analyze the continuous change of cooling rate with the change of sample thickness, and thus, continuous microstructure development within the available cooling conditions. Those provided by the mold selected for the experiment (Fig. 1) were estimated by measurement of dendrite arm spacing in the 98Al−2Cu (at.%) reference alloy. Resulting cooling rates are plotted in Fig. 1 against thickness of the obtained wedge ingot.

The microstructures observed in the reference binary 94Al−6Mn wedge sample were similar to that obtained in rods prepared by the suction casting reported previously [23]. Three main intermetallic phases were formed in fcc-Al matrix depending on cooling rates present at different parts of the resulting casting: hexagonal λ-Al_4Mn in thicker part, orthorhombic Al_6Mn, and orthorhombic L-phase (nomenclature of the last phase taken after Singh et al. [25]) in the thinnest parts. The schematic shown in Fig. 2 reveals the mentioned change in phase composition and refinement of microstructure elements as the thickness of ingot decreases.

Interestingly the orthorhombic L-phase with lattice parameters a = 12.4 Å, b = 12.6 Å, c = 30.5 Å has not been previously reported in Al−Mn binary alloys. This is the second experiment in which we confirmed formation of this orthorhombic structure in the Al−Mn system at intermediate cooling rate ~ 10^3 K/s.

In the second part the same casting technique was used to prepare a ternary alloy containing 2 at.% of Fe substituted for Mn. In contrast to the binary composition, a quasicrystalline icosahedral phase (I-phase)

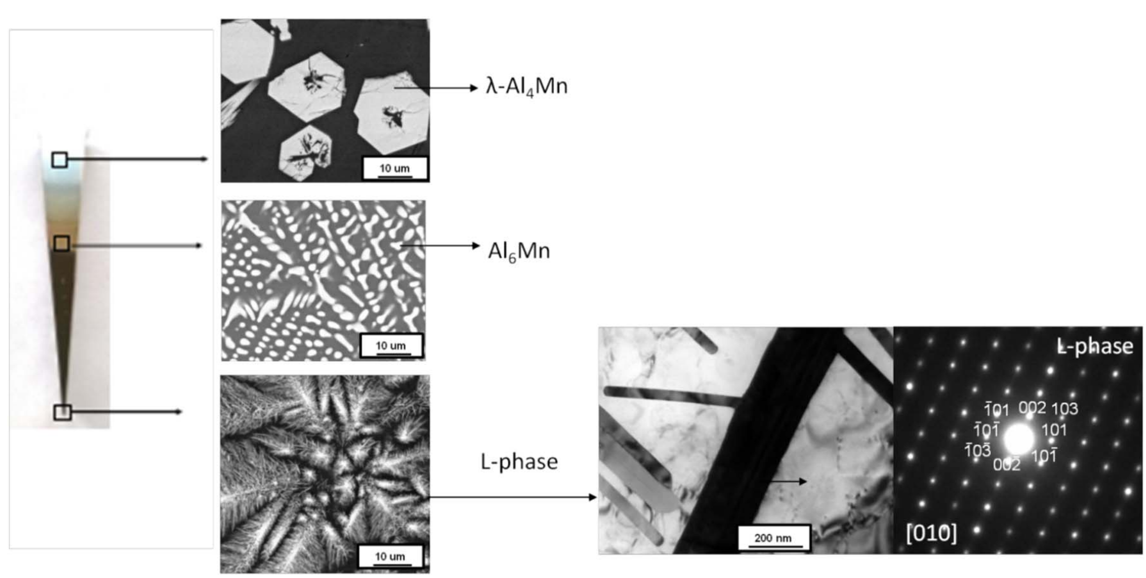

Fig. 2. Longitudinal cross-section of the 94Al−6Mn wedge showing the microstructure formed at different sample areas (SEM/BSE insets), additionally TEM bright-field and selected area electron diffraction pattern (SADP) corresponding to the L-phase observed at the thinnest part of sample is added.