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Structural and mechanical characterization of rapidly solidified Al₉₅Ni₅ and Al₉₃Ni₅Mm₂ alloys prepared by centrifugal atomization

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ABSTRACT

Al₉₅Ni₅ and Al₉₃Ni₅Mm₂ (at.%) alloys were prepared by induction melting and centrifugal atomization. Both as-cast and rapidly solidified (RS) alloys were studied by various techniques, including light, scanning and transmission electron microscopy, energy dispersion spectrometry, differential scanning calorimetry and X-ray diffraction analysis. Room temperature hardness was measured for the rapidly solidified alloys, as well as after their long-term annealing at 400 °C. It is observed that the RS alloys are crystalline. The binary Al–Ni alloy consists of Al and Al₃Ni phases, while in the ternary Al–Ni–Mm alloy, there are Al dendrites supersaturated with Ni, Al₃Ni and non-equilibrium α Al₁₁(Mm,Ni)₃ phases. Average Al dendrite arm thickness is about 100 nm. Both supersaturated Al and α Al₁₁(Mm,Ni)₃ decompose upon heating at 300–450 °C. Room temperature hardness of the RS Al₉₅Ni₅ and Al₉₃Ni₅Mm₂ alloys are 150 and 300 HV, respectively. These values are discussed in relation to various hardening mechanisms. Thermal stability of both alloys is low, due a rapid structural coarsening and hardness reduction during annealing at 400 °C.

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1. Introduction

Aluminum alloys are prospective materials for light-weight structural applications. Generally, tensile strength of commercial Al-based wrought alloys does not exceed about 700 MPa. This strength level can be achieved by an appropriate thermomechanical treatment combining cold working and precipitation hardening. It seems that 700 MPa is a limit of Al-based alloys in which classical hardening mechanisms, such as solid solution hardening, grain boundary hardening, precipitation hardening, operate. Another limitation of Al-based alloys is their poor thermal stability, meaning that mechanical characteristics rapidly reduce, as temperature increases above approx. 200 °C. One exception is a group of alloys containing small additions of scandium that show excellent stability at elevated temperatures. However, these alloys are still far from the mass commercial level, because of a very high price of scandium metal.

It was reported many times during the last two decades, that rapidly solidified (RS) Al–TM–RE alloys (TM = transition metals like Ni, Mn, Ti, Cr, Fe, V and other; RE = rare earth metals like La, Ce, Nd, etc.) can achieve strength levels much exceeding the limit of commercial Al alloys [1–6]. High strength of these materials is generally attributed to amorphous or nano-crystalline structure that

is extremely resistant to dislocation movement. There are a lot of studies devoted to RS Al–TM–RE alloys. However, among these alloys, only several systems can form amorphous phase during melt spinning or melt atomization. The glass forming ability (GFA) is generally enhanced with increasing number of alloying elements, large atomic size mismatch and negative heats of mixing of Al and additives [7]. When an alloy shows a low GFA, RS structure generally consists of nano-crystalline or nano-quasi-crystalline phases [8]. From the thermal stability point of view, positive role of TM is associated with their low solid solubility and slow diffusivity in Al. Al–Cr-based alloys appear as very prospective to replace heavier steels or Ti alloys in some thermally loaded applications [9].

In this study we focused our attention on RS Al–Ni–Mm alloys (Mm = mishmetal consisting of RE, such as Ce, La, Nd and Pr). RS Al–Ni–RE alloys have been extensively studied so far and it has been demonstrated that they fall into a group of alloys showing high GFA and, therefore, excellent mechanical properties [10–12]. In contrast to most of studies reported so far, we investigate an alloy containing a relatively low atomic percentage of Mm in this study, because high costs of RE metals are main barriers for mass production and application of Al–TM–RE alloys. Among rapid solid-ification techniques available at present, we selected centrifugal atomization in our experiment. In this technique a melt is atomized into powder by a fast rotating disc. In contrast to commonly used pressure gas atomization technique which produces almost spherical powder particles, centrifugal atomization produces flake-like

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 Table 1

 Chemical composition of investigated alloys (in at.%).

Alloy	Ni	Mm	Al
Al ₉₅ Ni ₅	5.2	-	Bal.
Al ₉₃ Ni ₅ Mm ₂	5.4	2.2	Bal.

particles showing a very low thickness. It is assumed that such particle shape will facilitate powder compaction.

2. Experimental

In this study we investigated two alloys with nominal compositions $Al_{95}Ni_5$ and $Al_{93}Ni_5Mm_2$ (hereafter, all concentrations are in at.%, unless otherwise stated). Symbol Mm denotes mishmetal, i.e. an alloy consisting of 45% Ce, 38% La, 12% Nd and 5% Pr. Both alloys were prepared by melting a pure Al (99.9% purity), nickel (99.8% purity) and Mm (99.5% purity) in an induction furnace under argon protective atmosphere. Obtained ingots were analyzed by X-ray fluorescence spectrometry (XRF) and results are summarized in Table 1.

Rapidly solidified powders of both alloys were prepared by centrifugal atomization. In this technique, a melt flows through a thin graphite nozzle onto the centre of a fast rotating (15 000 rpm) graphite disc. When the melt comes into contact with disc, it is atomized by a strong centrifugal force into droplets which are ejected towards a water cooled wall of atomizer where they rapidly solidify. As stated before, this type of atomization produces flake-like powder particles with thickness ranging from 10 to 50 μm and maximum size up to several mm. Morphology of prepared powders is presented in Fig. 1.

Prepared powders were sieved to obtain various granulometric fractions from <0.25 to >2.8 mm. For further study we selected granulometric fractions below 1.4 mm. Structural examinations of ingots and RS powders were carried out by using a light microscope (LM), scanning electron microscope (SEM, Hitachi S 4700, accelerating voltage 15 kV) equipped with energy dispersion spectrometer (EDS, Noran) and a transmission electron microscope (TEM, Jeol 3010, accelerating voltage 300 kV) equipped with EDS (Oxford Instruments). TEM foils were prepared from coarse particles by electropolishing in a mixture of ethanol and nitric acid (3:1 by volume) at 20 °C. Phase composition was determined by X-ray diffraction (XRD, Xipert Pro). Differential scanning calorimetry (DSC, Setaram, temperature range 25–500 °C, heating rate 10 °C/min) was used to monitor solid state transformations occurring in the powders on heating. Structural investigations of powders were completed by Vickers microhardness measurements (HV 0.005) of both rapidly solidified and annealed powders. Long-time annealing at 400 °C/300 h was used to estimate thermal stability of alloys.

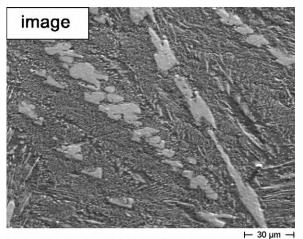
3. Results and discussion

3.1. Structure of the as-cast ingots

Fig. 2 presents SEM micrograph of the cast Al₉₅Ni₅ alloy ingot together with an X-ray map of Ni distribution. One can see that the structure consists of coarse elongated primary Al₃Ni phase and Al₃Ni + Al eutectic, see also XRD patterns in Fig. 4. According to the Al–Ni phase diagram [13], this type of structure corresponds to the hypereutectic composition of the alloy. SEM microstructure and elemental distribution of the as-cast ternary Al₉₃Ni₅Mm₂ ingot is shown in Fig. 3. Apparently, there are two types of pri-



Fig. 1. Morphology of powder prepared by centrifugal atomization.



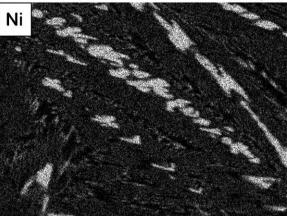


Fig. 2. SEM micrograph and Ni distribution map of the as-cast Al₉₅Ni₅ alloy.

mary phases, one enriched in Ni (gray) and another enriched in Mm (white). Point chemical analyses of these two phases are summarized in Table 2. According to this Table, to the Al–Ni, Al–Ce, Al–La and Al–Nd binary phase diagrams [13] and to the XRD analysis shown in Fig. 4, these phases are Al₃Ni and $\alpha Al_{11}Mm_3$. The latter phase can be regarded as a solid solution of isostructural orthorhombic $\alpha Al_{11}Ce_3$ and $\alpha Al_{11}La_3$. In Table 3, crystallographic parameters of present phases are summarized and it is seen that lattice parameters of $\alpha Al_{11}Ce_3$ and $\alpha Al_{11}La_3$ phases are very similar which supports their solid solubility [14]. One can also see in Table 2 that solubilities of Ni in $\alpha Al_{11}Mm_3$ and of Mm in Al₃Ni are negligible. In addition to the coarse primary phases, in the struc-

Table 2Local chemical compositions (in at.%) at points marked in Fig. 3.

Point no.	Element	Element				
	Al	Ni	Mm			
1	74.01	25.85	0.14			
2	75.22	0.04	24.74			

Table 3Crystallographic parameters of phases present in the Al–Ni–Mm alloy [14].

Phase	Crystal structure	Space group	Lattice parameters (Å)		
			а	b	с
Al	fcc	Fm3m	4.049	-	-
Al ₃ Ni	Orthorhombic	Pnma	6.598	7.352	4.802
Al ₁₁ Ce ₃ Al ₁₁ La ₃	Orthorhombic Orthorhombic	Immm Immm	4.395 4.431	13.025 13.142	10.092 10.132

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