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Chemical composition of the Ni-containing intermetallic phases in the multicomponent Al alloys

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ABSTRACT

In this study an effect of the alloving elements content and cooling rate on the morphology and chemical composition of the Ni-containing intermetallic phases in the Al-Si piston alloys has been examined. Compiled procedure of the selection and classification of the point X-ray microanalysis results allowed to estimate homogeneity range of the particular classes of the Ni-containing intermetallic phases occurred in the AlSi12MCuNiMg and AlSi21CuNi alloys. Replacement range: $Cu \rightarrow Ni$ in the Al₃(CuNi)₂ phase, and Fe \rightarrow Ni or Cu \rightarrow Fe in the Al₉(FeNi)₂ phase has been estimated and compared to that reported previously. The obtained results showed that chemical composition of the Ni-containing microstructure constituents locally reflected the sequence of precipitation processes on the alloy solidification path. The process of the formation of the Ni-containing intermetallic phases in form of terminal solid solutions of alloying elements in the crystal structure of the base phase through series of both peritectic and eutectic reactions has been discussed.

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1. Introduction

The final effects of the microstructure formation process play an important role in determination of technological and mechanical properties of the cast piston alloy [1–6]. Volume fraction, morphology and dispersion degree of the intermetallic phase particles are important factors determining tensile [11], wear [4] and fatigue resistance [2,3] of alloy. Especially, material effects at elevated temperature are strongly influenced by characteristics of the Ni-containing intermetallic phases. The particles of the Al₃(CuNi)₂ phase of high hardness [7-9] as well as those of the Al₉(FeNi)₂ phase have the most important contribution to alloy strength at elevated temperature [2–4]. The thermal expansion anisotropy of these intermetallic phases has been considered as a cause of thermal stresses leading to cracks initiation [10].

The tendency to intermetallic phases formation in the Al-Si-X alloys is very strong. While the solubility limits of almost all components in the α -Al solid solution are very small, even trace amounts of the transition metals (TM) from I-st long period (group 3d, Fe, Mn, Ni, Cr, V, Ti) cause precipitation of structurally differentiated intermetallic phases [1]. Despite long range ordering in their crystal lattice, some of them remain stable in a wide range of chemical composition. It has been assumed that chemical

heterogeneity of the AITM intermetallic phases is caused by mutual replacing of atoms of the particular components in the sites belonging to the strictly defined sublattices. The atoms replacement process in the AITMSi intermetallic phases has been experimentally noticed in two separated sublattices $Al \leftrightarrow Si$ and TM1 \leftrightarrow TM2 [12–14]. Thus, the intermetallics, formed in the multicomponent piston alloys, can be considered as terminal solid solutions of all alloy components in the crystal lattice of one of the intermetallic phases from the equilibrium systems: Al-Cu-Ni [15,16], Al-Fe-Ni [17-20] or Al-Si-Mg-Ni-Cu-Fe-Mn [21-24]. However, range of homogeneity of ternary AlNi(Cu or Fe) intermetallics has not been systematically analyzed as affected by alloy chemical composition. Comparison of the experimental data published up to now [2,9-11] indicates that the estimated concentration limits of both $Cu \rightarrow Ni$ and $Fe \rightarrow Ni$ substitutions in these intermetallic phases present in the piston alloys have changed in a different range. The analysis of the data shows this $Cu \rightarrow Ni$ and $Fe \rightarrow Ni$ replacement range as influenced by Cu, Ni and Fe content in the examined alloy.

In this study the preliminary trials have been taken to estimate the chemical composition of the Ni-containing intermetallic phases, precipitated in technical piston alloys of different contents of transition metals Fe, Mn, Ni, and Cu. An influence of alloy chemical composition and solidification rate on the morphology and homogeneity range of these microstructure constituents has been discussed.









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2. Experimental

2.1. Material for examinations

Materials of examinations used in this study were technical cast Al–Si alloys of chemical composition present in Table 1. The specimens for microstructure examination have been cut from wedge-shape ingots, cast to either sand or metal molds. The alloy cooling rate of the examined zone in these ingots, estimated in the liquidus–solidus range was 2.5 K/s and 14.3 K/s, respectively.

2.2. Microstructure examinations

Microstructure examinations have been carried out by means of the light metallographic microscope AxioObserver OZm on the metallographic microsections prepared in the standard way. The microstructure constituents have been revealed by chemical etching. The reagents for selective etching of the Ni-containing intermetallics were used: 10 g NaOH + 100 ml H₂O dist. (4 m), 4 g KMnO₄ + 2 g Na₂CO₃ + 94 ml H₂O dist. (14 m), 0.5 ml HF + 100 ml H₂O dist. (15 m), 1 g (NH₄(MO₃) + 4 g NH₄Cl + 94 ml H₂O dist. (17 m) [25].

The chemical composition of the intermetallic phase particle has been estimated by means of the point X-ray microanalysis using Link ISIS 300 microanalyser EDS connected to the scanning electron microscope Stereoscan 420. The procedure of the standardless analysis (using standard spectra library) has been used with both FLS and ZAF corrections. The attribution rules (Table 2) adopted for the microanalysis results selection and classification procedure [26] have based on the literature data, taking into account some chemical coefficients, specific for the Ni-containing phases: Cu/Ni ratio, $\Sigma Cu + Ni$, Fe/Ni ratio, $\Sigma Fe + Ni$.

In a first step, the microregions for the point analysis have been chosen using "EDS size criterion" (I): $d < d_{crit} = 3 \mu m$). In the next steps, both chemical (II) and morphological (III) criteria have been used to obtain separated datasets of results (min. 10 measure points) established for representative particular class of the Ni-containing phases [26].

The particular class of the intermetallics contained other alloy components. They were considered as complex solid solutions, based on the binary and ternary equilibrium phases, reported previously in the technical alloys [3–5,21,22].

3. Results and discussion

3.1. Microstructure constituents in the examined alloys

The Ni-containing intermetallic phase particles, observed in the microstructure of the examined alloys represented four typical morphological forms:

- Diamonds - ε -Al₃Ni (Fig. 1a).

- Chinese script δ -Al₃NiCu (Fig. 1b).
- Irregular plates (branched) γ -Al₇Cu₄Ni (Fig. 1c) or T-Al₉(FeNi)₂ (Fig. 1d).
- Irregular "lakes" T-Al₉FeNi (Fig. 1e).

This four categories of shape have been used as an additional criterion of the intermetallic phase particle identification in the alloy microstructure (Table 2).

Final phase composition of the examined alloys was formed through several series of either invariant or polyvariant reactions, identified in Al–Cu–Ni–Si [21], Al–Fe–Ni–Si [21], and Al–Cu–Mg–Fe–Ni–Mn–Si [22–24] systems. Especially, Ni-containing phases crystallize through series of the peritectic reactions [13]:

− L + β -AlFeSi → (Al) + (Si) + Al₉(FeNi)₂ at 573 °C.

- L + Al₃Ni → (Al) + (Si) + Al₃(CuNi)₂ + Al₂Ni, at 540 °C.

- L + Al₃(CuNi)₂ \rightarrow (Al) + (Si) + Al₇Cu₄Ni, at 530 °C.

This series is followed by polyvariant eutectic reactions at lower temperature.

Since some of these reactions have not completed before alloy solidification end, non-equilibrium state of the microstructure has been observed, even in the specimens cooled slowly (poured into sand mold, Fig. 2a–d). When intermetallic phase is formed through peritectic reaction, the interface between liquid and properitectic precipitate formed at earlier stage of solidification became a support for peritectic phase nucleation. However, the interfaces observed in the alloy microstructure (Fig. 2a–d) do not reflect exactly the status expected on the basis of the mentioned sequence. The presence of the interface Al₉(FeNi)₂/Al₇Cu₄Ni or Al₉(FeNi)₂/Al₃(CuNi)₂ (Fig. 2a, p. A, 2d, p. A) observed in the microstructure indicates a preference for nucleation either Al₃(CuNi)₂ or Al₇Cu₄Ni phase at the surface of the Al₉(FeNi)₂ phase particles, precipitated earlier [27].

The Al₃(CuNi)₂/Al₇Cu₄Ni interface (Fig. 2b, p. B, 2d, p. A) could be formed **at** the intermediate stage of the polyvariant peritectic reaction: $L + Al_3(CuNi)_2 \rightarrow Al_7Cu_4Ni$, when peritectic phase nucleated and then grew through a direct exchange of components in the ternary point: $L/Al_3(CuNi)_2/Al_7Cu_4Ni$. Primary precipitation of

Table 1

Chemical composition of the examined alloys (Al-bal.).

Alloy designation	Alloy	Alloy components (wt.%)							
		Si	Cu	Mg	Mn	Ni	Fe	Zn	Ti
AlSi13Mg1CuNi	Standard alloy Examined alloy	11.5–13.0 11.54	0.8–1.5 1.12	0.8–1.5 1.22		0.8–1.5 1.46	0.6–0.8 0.82	0.2 0.12	0.1 0.05
AlSi21CuNi	Standard alloy Examined alloy	20–23.0 19.89	1.1–1.5 1.42	0.5–0.9 0.95	0.1–0.3 0.32	0.8–1.1 1.15	0.5–0.6 0.56	0.2 0.08	_ 0.04

Table 2

Attribution rules for sets of X-ray microanalysis results for Ni-containing intermetallic phases in the microstructure of the examined alloys [1,15–24].

Step	Criterion I "EDS size criterion": <i>d</i> < <i>d</i> _{crit}	Criterion II chemical composition Σ Me (at.%)	Criterion III morphology	Result of attribution
1. Selection	<i>d</i> < 3 μm	$\begin{array}{l} 15 \leqslant (Fe + Ni + Cu) \leqslant 60 \\ 6 \leqslant Ni \; Si \leqslant 5 \end{array}$	-	Preliminary-TM-containing phases class Preliminary-Ni-containing phases class
2. Classification	-	Ni ≤ 25 Cu < 1 Si < 0.5	Diamond	AlNi class – ε
		$35 \leqslant (Ni + Cu + Fe) \leqslant 45$ $Cu \leqslant Ni$	Chinese script	AlCuNi class – δ
		$20 \leqslant (Ni + Cu + Fe) \leqslant 60$ Cu > Ni	Irregular plate (branched)	AlCuNi class – γ
		$2\leqslant F~e\leqslant 10~8\leqslant Ni\leqslant 16$	Irregular plate (branched), Lake	AlFeNi class – T

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