MICROSTRUCTURE AND THERMAL STABILITY OF Al-Fe-X ALLOYS

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Abstract
In this work, Al-11Fe, Al-7Fe-4Ni and Al-7Fe-4Cr (in wt. %) alloys were prepared by combination of casting and hot extrusion. Microstructures of as-cast alloys were composed of aluminium matrix with large and coarse intermetallics such as $Al_{13}Fe_4$, $Al_{13}Cr_2$ and Al_5Cr. Subsequently, as-cast alloys were rapidly solidified by melt-spinning technique which led to the supersaturation of solid solution alloying elements. These rapidly solidified ribbons were milled and compacted by hot-extrusion method. Hot-extrusion caused that microstructures of all alloys were fine with uniform dispersed particles. Moreover, long-term thermal stability was tested at temperature 300 °C for as-cast and hot-extruded alloys and chromium was found to be the most suitable element for alloying to improve thermal stability.

Keywords: casting, melt-spinning, extrusion, scanning electron microscopy, hardness test, aluminium alloys

1 Introduction

Al-Fe alloys excel in mechanical properties at elevated temperatures. For this reason, thermally loaded parts - pistons or turbocharger rotors are made of these alloys [1]. However, iron is the most common impurity in aluminium alloys and influences mechanical properties negatively. Separation of iron from recycling aluminium components is expensive and inefficient economically because iron can be removed only by magnetic separation [2]. However, it is necessary to remove iron because this one forms large detrimental intermetallic phases in casting materials which causes mechanical properties very negatively. On the other hand, iron belongs to the group of very slow diffusers in aluminium as well as chromium, manganese or nickel [3, 4]. That means improvement of thermal stability, especially in temperatures higher than 250 °C [2, 5] which is critical for mechanical properties of Al-based alloys. These elements form thermally stable phases which prevent the grains growth.
Concentration of alloying elements is limited because hard and brittle intermetallic phases form when the concentration is exceeded. Solution is application of very high cooling rates during processing [6, 7, 8] e. g. melt-spinning or atomization. These methods are part of powder metallurgy which has attracted attention due to possibility of production aluminium alloys with high content of iron. Extremely high cooling rates cause better solubility of alloying elements, refinement of microstructure, increasing of concentration of element above the value of equilibrium solubility and allow to form non-equilibrium and metastable phases [9].

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Typical phases, which form in the presence of iron, are $Al_{13}Fe_4$ and Al_6Fe [1, 10]. Chromium as well as manganese modify the morphology of the needle-like intermetallic phases and decrease the amount of undesirable phases [11, 12]. Nickel forms with aluminium very thermal stable phase Al_3Ni which leads to the improvement of the high-temperature mechanical strength [1].
In this work hot-extruded alloys were prepared from rapidly solidified powders. The effect of addition of chromium and nickel on microstructure, phase composition and thermal stability during long-term exposure was observed and described. The rapidly solidified alloys were compared with the as-cast materials of the same compositions.

2 Experimental materials and methods

Master alloy Al-11Fe (in wt. %), pure chromium and nickel (over 99.5 % purity) were used for the studied as-cast alloys Al-11Fe, Al-7Fe-4Cr and Al-7Fe-4Ni (in wt. %). Casting was performed in electric furnace in graphite crucible which was followed by melt-spinning process. As-cast samples were melted in quartz glass nozzle at 1200 °C and injected onto copper wheel (CuCr1Zr0.1). Whole process was conducted under argon atmosphere with wheel speed 1420 rpm. Obtained ribbons were milled in stainless steel container with balls in planetary ball mill Retch PM 100 CM for 10 min. Ball-to-powder ratio was 15:1. At the beginning of milling, nitrogen was added for embrittlement of ribbons. Powders were afterwards uniaxially cold pressed using LabTest5.250SP1-VM universal loading machine with loading 70 kN into cylindrical tablets with diameter 19 mm and height 30 mm. First of all, furnace was preheated at 400 °C and the preform was inserted. Hot-extrusion proceeded at 500 °C. Height of resulted sample was 1 cm.
Microstructure was studied by scanning electron microscope TESCAN VEGA 3 equipped with OXFORD Instruments X-max EDS SDD 20 mm^2 detector and phase composition was determined by X-ray diffraction (PANalyticalX´Pert, CuKαradiation).
Hardness was measured by Vickers method with load of 5 kg (HV 5). Thermal stability was evaluated as hardness dependence on the duration of thermal exposure at 300 °C.

3 Results

3.1 Microstructure and phase composition

Figs, 1 a, b show microstructure of as-cast alloy and obtained microstructure after hot extrusion. It can be seen, that $Al_{13}Fe_4$ phase is present after both processing which was confirmed by XRD analysis (**Table 1**). This is a typical stable phase found in Al-based alloys with high amounts of iron [13, 14]. $Al_{13}Fe_4$ phase forms very large and coarse needle-like shape particles (**Fig. 1 a**) which act as a stress concentrator for initiation of microcracks. As a consequence, the alloys containing this phase are brittle. This phase is dispersed in solid solution α-Al (**Fig. 1 a**).
$Al_{13}Fe_4$ phase had round shape after hot-extrusion and its quantity also decreased according to the SEM micrograph (**Fig. 1. b**). The rapidly solidified ribbons usually contain supersaturated solid solutions and minimized amounts of intermetallics. Therefore, intermetallic phases precipitated from the solid solution of α-Al during hot-extrusion.
Microstructure of as-cast Al-Fe-Ni alloy was mainly formed by large $Al_{13}Fe_4$ phase (**Fig. 2 a**) in contrast with Al-Fe alloy (**Fig. 1 a**). Nickel was dissolved with iron in aluminium matrix. Al-Fe-Ni alloy had quite fine microstructure after hot extrusion (**Fig. 2 b**) where very small amount of undesirable $Al_{13}Fe_4$ was found and the grey places in aluminium matrix were identified as Al_4Ni_3 phase. So, nickel was found to be suitable alloying element for the prevention of grain

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coarsening. **Table 2** shows the results from XRD analysis which confirmed the presence of phases found during EDS analysis.

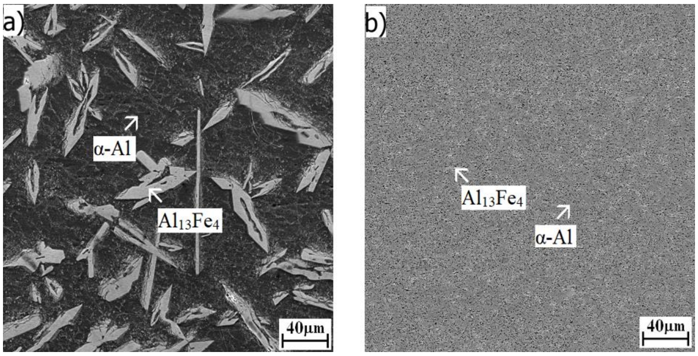

Fig. 1 Microstructure of a) as-cast, b) hot-extruded Al-11Fe alloys

Table 1 Phase composition of as-cast and hot-extruded Al-11Fe alloys

	Phase composition
As-cast alloy	Al, $Al_{13}Fe_4$
Hot-extruded alloy	Al, $Al_{13}Fe_4$

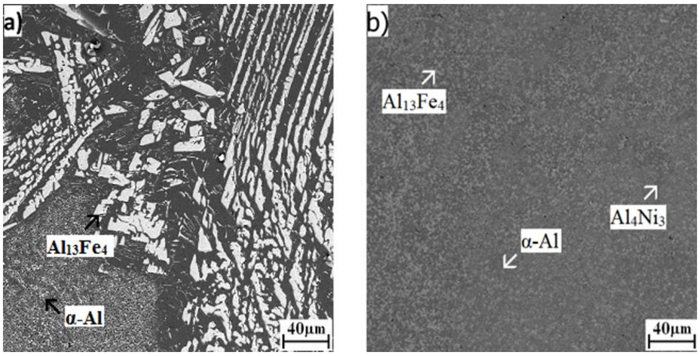

Fig. 2 Microstructure of a) as-cast, b) hot-extruded Al-7Fe-4Ni alloys

Table 2 Phase composition of as-cast and hot-extruded Al-7Fe-4Ni alloys

	Phase composition
As-cast alloy	Al, $Al_{13}Fe_4$
Hot-extruded alloy	Al, $Al_{13}Fe_4$, Al_4Ni_3

Microstructure of as-cast Al-Fe-Cr alloy was composed by solid solution of α-Al and by $Al_{13}Cr_2$ phase (**Fig. 3 a**). EDS analysis did not reveal the presence of $Al_{13}Fe_4$ phase with needle-like shape as in the case of previous alloys (**Figs. 1 a, 2 a**). Hot-extruded state of this alloy had the finest microstructure without coarse particles (**Fig. 3 b**). It can be seen that microstructure is composed by very fine intermetallic phases (light) in aluminium matrix (dark). According to presented results, it could be assumed that chromium stabilizes the material thermally and prevents the grain coarsening during compaction by hot-extrusion. XRD analysis confirmed

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other thermally stable phases (**Table 3**) including $Al_{13}Fe_4$ phase which was not detected during EDS analysis, due to a very low particle size.

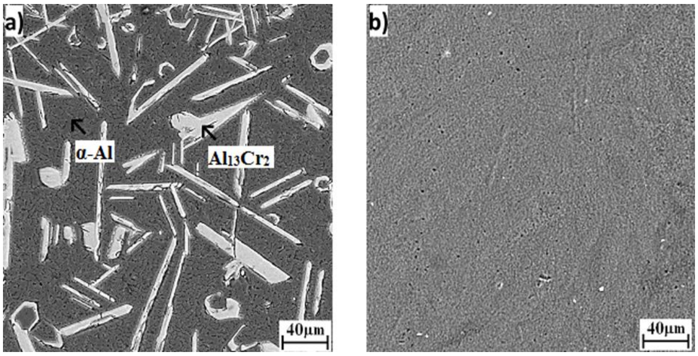

Fig. 3 Microstructure of a) as-cast, b) hot-extruded Al-7Fe-4Cr alloys

Table 3 Phase composition of as-cast and hot-extruded Al-7Fe-4Cr alloys

	Phase composition
As-cast alloy	Al, $Al_{13}Fe_4$, $Al_{13}Cr_2$, Al_5Cr
Hot-extruded alloy	Al, $Al_{13}Fe_4$, $Al_{13}Cr_2$

3.2 Thermal stability

The dependence of hardness of the alloys in as-cast state on the time of annealing at 300 °C is presented in **Fig. 4**. The hardness of Al-Fe alloy was almost constant due to the presence of thermally stable $Al_{13}Fe_4$ phase (**Fig. 1 a**) which keeps the thermal stability of this alloy. The curve belonging to Al-Fe-Cr alloy was very variable (**Fig. 4**). First, the hardness decreased slightly and then the hardness gradually increased. This phenomenon confirmed that chromium improves thermal stability. $Al_{13}Fe_4$, Al_5Cr and $Al_{13}Cr_2$ phases causing the high thermal stability were detected (**Table 3**). Al-Fe-Ni alloy also improved thermal stability of Al-Fe based alloy, however, the initial decrease of hardness was significant (**Fig. 4**). No phases with nickel were found after casting. Thus, chromium prevents the grain coarsening from the beginning of annealing and nickel stabilized the grain size after 150 h (63 HV 5). The similar effect was observed in study [15] where nickel improved thermal stability significantly and chromium kept this one satisfactory.

Hot-extruded alloys have higher hardness before and after annealing compared with as-cast alloy (**Figs. 4, 5**). At the beginning of measurement, the hardness of Al-Fe and Al-Fe-Cr decreased after first 150 h of annealing and then the hardness increased and has approximately the same values. Thermal stability is influenced mainly by chromium and its phases which caused the good thermal stability (**Table 3**). Hardness of Al-Fe-Ni increased in first 100 h, probably due to precipitation processes, and after that was constant. Variances in values of hardness are caused by the disappearance of metastable phases, precipitation of stable intermetallic phases from the supersaturated solution and their coarsening (**Table 2**). Lower decrease of hardness during annealing is due to the fact that the alloys were extruded at 500 °C before thermal exposure and the resulted materials were stabilized. Subsequent exposure at lower temperatures did not cause the changes in structures.

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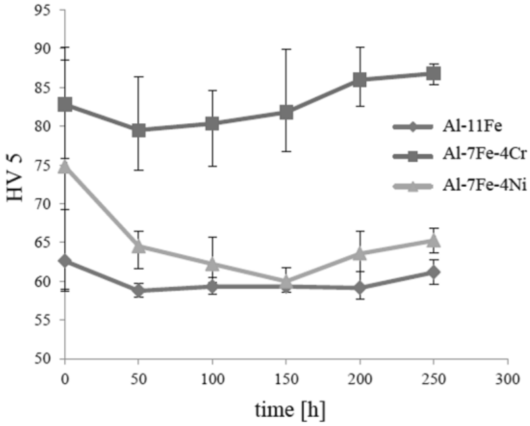

Fig. 4 Hardness (HV 5) of as-cast alloys vs. duration of annealing at 300 °C

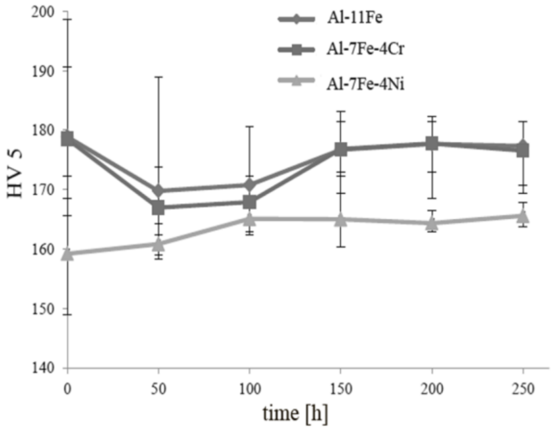

Fig. 5 Hardness (HV 5) of hot-extruded alloys vs. duration of annealing at 300 °C

4 Conclusion

This study was focused on the description of microstructure and thermal stability of Al-Fe-X alloys in as-cast state and after melt spinning and hot-extrusion. Microstructures were composed by thermally stable phases, which were coarse after casting. Nickel and chromium prevented the grain coarsening during hot extrusion. The results also show that the best thermal stability was found for the hot-extruded alloys and chromium was found to be the most suitable alloying element for Al-Fe based alloys. Nickel also improved thermal stability but less significantly than chromium.

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