PROPERTIES OF AL-BASED ALLOYS PREPARED BY CENTRIFUGAL ATOMISATION AND HOT EXTRUSION

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Abstract

Aluminium alloys are prospective materials that can be used in wide range of applications requiring good weight-to-strength ratio, convenient corrosion resistance and others. However, low thermal stability is a problem that limits their use in applications with temperatures typically exceeding 300 °C. Solution to this problem can be found in addition of appropriate amounts of transition elements (Fe, Ni) that are characterized by low diffusion coefficients in solid aluminium. However, their solubility in solid aluminium is limited only to a few percents. Centrifugal atomisation, as one of many rapid solidification techniques, can solve this problem. In this work, two PM alloys with nominal composition of Al-12Fe and Al-7Fe-5Ni (in wt %) were prepared by a centrifugal atomisation. Prepared powders were compacted via hot extrusion at an extrusion ratio of 10:1 and a deformation rate of 2.5 mm/min into rods of 6 mm in diameter. Microstructure of tested materials was examined by light microscopy and by electron scanning microscopy. Chemical composition as well as phase composition was confirmed by XRF and XRD analysis. An Al-12Si-1Cu-1Mg-1Ni alloy that is commonly considered as thermally stable was used as a reference material. Thermal stability of tested materials was determined on the basis of Vickers hardness change during long-term annealing at 300 °C and 400 °C. To complete the thermal stability investigations, creep test were performed at a compressive stress of 120 MPa and temperature of 300 °C. All of prepared PM alloys exhibited better thermal stability compared to the reference material.

Keywords: Aluminium, centrifugal atomisation, extrusion, thermal stability.

1. INTRODUCTION

Aluminium and its alloys belong to prospective materials that are characterized by a good weight-to-strength ratio, convenient corrosion resistance and good thermal stability if appropriate amounts of transition elements (Fe or Ni) are added [1, 2]. However, solubility of these elements in solid aluminium is strongly limited and may result in excessive intermetallic phases deteriorating mechanical properties. This problem can be successfully solved e.g. by rapid solidification (RS), that eliminates dimension of those phases, increases the solubility of alloying element and may even produce materials containing metastable or amorphous phases. Generally, to consider a process to be in a rapid solidification regime, cooling rates must be greater of equal to 10^4 K s⁻¹ [3-5].

In the past, iron has been considered more as impurity rather than as an beneficial alloying element. Its ability to enhance thermal stability of Al-based alloys can be attributed to a much lower diffusion coefficient compared to more convenient alloying elements such as silicon, copper and magnesium. However, higher concentrations of iron are required to improve thermal stability. As it was already written a few rows above, higher concentrations of alloying elements produces large-scale brittle intermetallic phases such as Al₁₃Fe₄ that is commonly known to form in the slowly solidified Al-Fe alloys [6]. Centrifugal atomisation (CA), as one of many rapid solidification techniques, may eliminate dimensions of these excessive phases in terms of significant microstructural refinement. Several works reported that the CA technique can achieve cooling rates in the range of 10⁴-10⁷ K s⁻¹ [7-9]. However, these cooling rates strongly depend on the setting and arrangement of the experimental apparatus.
Therefore, this work focuses on Al-Fe and Al-Fe-Ni alloys prepared by PM. These alloys were prepared by the CA technique that belongs to a relatively simple and low-cost RS technique suitable for large-scale powder production.

2. EXPERIMENTAL DETAILS

The examined PM alloys with nominal chemical compositions of Al-12Fe and Al-7Fe-5Ni (wt.%) were prepared by vacuum induction melting of appropriate amounts of pure Al, Ni (purity at least 99.99 %) and master Al-12Fe alloy under argon protective atmosphere. After sufficient homogeneity was achieved, the melt was poured into a brass mould forming an slowly solidified cylindrical ingots that measured 20 mm in diameter and 150 mm in length. So prepared ingots were than remelted under argon protective atmosphere and injected throughout an graphite casting nozzle with 1 mm in diameter onto a high-speed rotating graphite disc (velocity speed of 30,000 rpm) to form RS powders (Fig. 1). The chemical composition of all the investigated alloys was confirmed by X-ray fluorescence spectrometry (XRF, ARL 9400 XP) and is listed in Table 1.

<table>
<thead>
<tr>
<th>Material (preparation)</th>
<th>Element (wt.%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Ca  Cr  Cu  Fe  Mg  Mn  Ni  Si  Ti  Al</td>
</tr>
<tr>
<td>Al-12Fe (PM)</td>
<td>-    -    -    11.9  -    0.1  -    -    -    Bal.</td>
</tr>
<tr>
<td>Al-7Fe-5Ni (PM)</td>
<td>-    -    -    7.2    -   4.7    0.2  -    -    Bal.</td>
</tr>
<tr>
<td>Al-12Si-1Cu-1Mg-1Ni (casting, T6 heat treatment)</td>
<td>0.1  0.1  1.2  0.2  1.0  0.2  0.9  11.8  0.1  Bal.</td>
</tr>
</tbody>
</table>

To compare the results obtained by our research, an Al-12Si-1Cu-1Mg-1Ni alloy, that is commonly considered as thermally stable and therefore used for engine part manufacturing, was used as an reference material. It was obtained in a form of an ingot from an external supplier and thermally treated by the T6 regime consisting of solution annealing (510 °C/5 h), water quenching and artificial aging (230 °C/6 h).

Prepared RS powders (Fig. 2) were sieved to obtain fraction with particles size of 0.1 - 2 mm that were used for further steps including pre-compaction and hot extrusion. Powders were pre-compacted by a pressing mould made from tool-steel into billets 19 mm in diameter and 25 mm in height by a pressure of 210 MPa providing sufficient cohesion. These billets were than preheated at 550 °C for 10 min and extruded at an extrusion ratio of 10 : 1 by a strain speed of 2.5 mm/min to form rods 6 mm in diameter.
The microstructures of prepared slowly solidified alloys, casting alloy and PM alloys were observed using a light microscope (LM, Olympus PME-3) and a scanning electron microscope (SEM, Tescan VEGA 3, 20 kV, SE+BSE) equipped with an energy dispersive X-ray spectrometer (EDS, Oxford Instruments INCA 350). Present phases were determined by X-ray diffraction analysis (XRD, PANalytical X’Pert Pro, Cu Kα1 λ=1.54059 Å).

Thermal stability of tested PM alloys was determined by hardness change during long-term annealing at temperatures ranging from 300 °C to 400 °C. In addition, compressive stress strain tests (LabTest 5.250SP1-VM machine) were performed at room temperature after annealing. To complete the thermal stability investigations, creep tests at 300 °C and a constant compressive stress of 120 MPa were carried out. Samples used for both tests were cylindrical in shape with 6 mm in diameter and 6 mm in height.

3. RESULTS

3.1 Microstructure and phase composition

The microstructures of slowly solidified Al-12Fe and Al-7Fe-5Ni alloys are shown in Figs. 3a and b, respectively. The Al-12Fe alloy consisted of primary intermetallic Al13Fe4 needle-like phases and of an α-Al + Al13Fe4 eutectic mixture. The Al-7Fe-5Ni alloy was similar to the previous one, consisting of sharp-edged primary intermetallic Al12FeNi phases and an α-Al + Al12FeNi eutectic mixture.

![Microstructures of slowly solidified: a) Al-12Fe; b) Al-7Fe-5Ni; c) Al-12Si-1Cu-1Mg-1Ni alloys.](image-url)
The microstructure of the T6 heat-treated Al-12Si-1Cu-1Mg-1Ni alloy, that was used as an reference material, composed of α-Al dendrites, an α-Al + Si eutectic mixture and Mg2Si, Al3Ni and Al6Cu3Ni intermetallic phases (Fig. 3c).

Present Al14Fe3 and Al9FeNi phases were confirmed by the XRD analysis (Al14Fe3 phase can be found in the JCPD database by card number 007-8608, C2/m space group; Al8FeNi by card number 014-7251, P21/c space group) as is shown in Figs. 4 and 5. Presence of the Al13Fe4 phase can be explained by relatively lower cooling rates that were achieved by the CA technique compared e.g. to melt spinning.

The microstructures of PM alloys are shown in Figs. 6a and b. One can see an significant microstructural refinement induced by the CA technique. Intermetallic phases changed its original dimensions reaching several micrometers to sub-micrometer size in booth of tested alloys. This refinement positively influenced the resulting mechanical properties as will be shown in following paragraph.

After long-term annealing at 400 °C for 100 hours, fragmentation of Al13Fe4 phases in the PM Al-12Fe alloy was observed (Fig. 7a and b). This fragmentation was probably caused by the different thermal expansion coefficient of these phases and the matrix. The needle-like intermetallic phases fragmented into small sharp-edged objects with dimension of approximately 1 - 2 µm (Fig. 7b). On the other hand, the Al8FeNi intermetallic phases in the PM Al-7Fe-5Ni alloy maintained their original size even after annealing (Fig. 7c and d). This was probably caused by the relatively low diffusion coefficients of iron and nickel in solid aluminium.
3.2 Mechanical properties

Mechanical properties of tested PM alloys as well as of the reference material are listed in Table 2. As is evident from these results, the highest initial hardness (121 ± 1 HV5) was observed in the case of the T6 heat-treated reference alloy. This high hardness was caused by the presence of a large fraction of fine silicon particles and the precipitation of Al₃CuMgNi with semi-coherent interfaces, which are the most pronounced hardening mechanism operating in this alloy. Both the as-prepared PM alloys showed almost identical hardness values of approximately 70 HV5. The main hardening mechanism can be attributed to the fine-grained structure and the fine dispersion of intermetallic phases.

Table 2: Mechanical properties of tested alloys at room temperature. *The CS of the PM alloys could not be determined.

<table>
<thead>
<tr>
<th>Alloy</th>
<th>As-prepared</th>
<th>Annealed at 300 °C/100 h</th>
<th>Annealed at 400 °C/100 h</th>
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<tbody>
<tr>
<td></td>
<td>HV5</td>
<td>CS</td>
<td>CYS</td>
</tr>
<tr>
<td>Al-12Fe (PM)*</td>
<td>68 ± 1</td>
<td>-</td>
<td>183</td>
</tr>
<tr>
<td>Al-7Fe-5Ni (PM)*</td>
<td>73 ± 1</td>
<td>-</td>
<td>226</td>
</tr>
<tr>
<td>Al-12Si-1Cu-1Mg-1Ni</td>
<td>121 ± 2</td>
<td>680</td>
<td>430</td>
</tr>
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</table>

After 100 hours of annealing at 300 °C and 400 °C, the reference Al-12Si-1Cu-1Mg-1Ni alloy reduces its hardness almost to half of its initial value. In contrast, the PM alloys maintained its hardness or lowered it by only a few points. These results indicate that both the PM alloys are thermally stable even at high temperatures.

To complete the thermal stability investigations, compressive stress-strain tests at room temperature (after annealing at 300 °C and 400 °C) as well as creep test at 300 °C were performed. As is evident from the Table 2, compressive yield strength (CYS) of the annealed PM alloys were the same as those of the as-prepared materials. On the other hand, the reference Al-12Si-1Cu-1Mg-1Ni alloy significantly reduced its CYS to approximately 50 % of its initial value. This poor stability can be directly connected with high diffusivities of silicon, copper and magnesium in solid aluminium. Fig. 8 shows the results from compressive creep tests. Generally, the lower the compressive strain the better the creep resistance. All tested materials exhibited only primary creep stage, characterized by a steep initial creep followed by a progressive decrease of the creep rate almost to zero. It is obvious, that the PM alloys exhibited better creep resistance than the reference material. The PM Al-7Fe-5Ni alloy was the most creep resistant material with a total compressive strain of approximately 15 %. Compared to the reference material that is considered as thermally stable the PM Al-7Fe-5Ni alloy showed more than three times better creep resistance.
4. CONCLUSION

This work demonstrates the ability of the CA technique to prepare PM Al-based materials with higher concentrations of alloying elements such as iron and nickel. Microstructure of the PM hot-extruded materials was refined, almost pore-free with sufficient diffusion bonding between the powder particles. It was further found that the PM Al-12Fe and Al-7Fe-5Ni alloys exhibited better thermal stability compared to the reference Al-12Si-1Cu-1Mg-1Ni alloy especially when annealed. They maintained their hardness and CYS almost on the identical values even when annealed at 400 °C for 100 h. Thus, the PM technology based on the CA technique and subsequent hot extrusion seems to be viable for the production of Al-based alloys.

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REFERENCES


