PROPERTIES OF NANOCRYSTALLINE POWDER METALURGY AL-SI-FE ALLOYS

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Abstract

Rapid solidification (RS) is perspective method which can be defined as rapid transfer of thermal energy including both superheat and latent heat during liquid-solid phase transformation. Materials prepared via RS exhibit extraordinary properties, compared to the conventional cast alloys, which can be attributed to increased content of alloying, improved homogeneity of alloy, formation of micro-, nano- or quasicrystalline microstructure and others. Transition metals (Fe, etc.) are known for their ability to increase thermal stability of aluminium alloys due to their low diffusivity in aluminium, however its concentration are strongly limited.

Centrifugal atomization with high-speed rotating graphite disc was used to prepare powders with different composition in this work. Prepared powders were sieved to obtain required powder fraction which was compacted with ultra-high pressure of 6 GPa at temperature 450 °C. Microstructure of prepared powders and compact samples was observed using light microscope and electron scanning microscope equipped with EDS analyzer. Thermal stability of pressed samples was evaluated like hardness change during annealing at various temperatures for 100 hours. Compressive mechanical tests were measured at room temperature for both materials in its initial states and after long-term annealing.

Keywords: aluminium, rapid solidification, transition metals

1. INTRODUCTION

Al-Si based alloys exhibit excellent weight-to-strength ratio, high thermal conductivity, great castability, and therefore are more often used in automotive or aerospace industry. Millions of parts are mostly prepared by gravity or die casting. Typically are these materials used for pistons, engine blocks and valves production and therefore there is always need to improve their properties e.g. by addition of another element. The addition can be strictly limited by the total solubility of element in aluminium after that formation of brittle and hard phases will deteriorate mechanical properties. This problem can be solved via rapid solidification (RS) technique like centrifugal atomization which was used in this work.

Rapid solidification is perspective process defined by rapid heat extraction including both superheat and latent heat during liquid-solid state transformation [1]. To consider a process to be in RS regime, cooling rates should be greater or equal to $10^4$ K.s$^{-1}$ although several papers reported that cooling rates $10^3$ K.s$^{-1}$ are sufficient to generate rapidly solidified microstructures [1, 2]. Aluminium alloys prepared via RS technique offers extraordinary properties due to increased content of alloying elements, improved homogeneity and creation of micro-, nano- or quasi-crystalline structures [3]. The addition of elements that are slow diffusers in aluminium can enhance the thermal stability, which is important for fabrication of thermally loaded parts such as pistons and turbochargers rotors. Figure 1 shows that transition metals are very effective in this [4].

Centrifugal atomization (CA) is simple low-cost RS method that can easily generate rapidly solidified materials. Typical products of CA processes are powders consisting of fine flake-like particles (Fig. 3) which can be more easily pressed into bulk samples [5]. Centrifugal atomizer with high speed rotating graphite disc ensure almost immediately transport of molten metal to water-cooled walls and therefore almost immediate liquid-
solid state phase transformation. In this work, centrifugal atomization was used to prepare rapidly solidified AlSi13Fe10. Composition of this alloy was deliberately chosen representing typical waste product of aluminium recycling process containing higher iron and silicon content.

2. EXPERIMENT

The alloy with nominal composition AlSi13Fe10 (in wt. %) prepared by CA was studied and compared in this work with the commercial AlSi12Cu1Mg1Ni1 alloy that is commonly considered to be highly thermally stable. The AlSi12Cu1Mg1Ni1 alloy was provided by external industrial supplier in a form of ingot with a thickness of 30 mm and length of 150 mm. The alloy was heat treated in T6 regime consisting of solution annealing (510 °C/5 h), water quenching and artificial aging (230 °C/6 h). In the following text, so treated alloy will be denoted as “as-cast”.

The AlSi13Fe10 alloy was prepared by melting of appropriate amount of pure aluminium and master alloys in a vacuum induction furnace under argon atmosphere. After a sufficient homogenization the melt was poured into a cast iron metal mould with 20 mm in diameter and 150 mm in height. So prepared alloy was consequently remelted under argon protective atmosphere and ejected onto high-speed rotating graphite disc with rotation speed of 30000 rpm (Fig. 2).

Prepared powder was sieved to obtain powder fractions with powder particle size smaller or equal to 2 mm with approximately thickness of 50 µm (Fig. 3). Afterwards, the powder was placed into a tungsten carbide mould and compacted by uni-axial pressing at 300 °C and ultra-high pressure of 6 GPa for 5 minutes. By this procedure, cylindrical samples 15 mm in diameter and 5 mm in height were prepared. It should be noted,
that pressure applied in our work was in one order of magnitude greater than the pressures applied in common uni-axial or isostatic pressing of powders. In the following text, so prepared alloy will be denoted as "as-compacted".

Microstructure of both materials was investigated using light microscopy (Olympus PME-3) and scanning electron microscopy (Tescan Vega 3) with EDS analyzer (Oxford Instruments Inca 350). The phase composition of materials was determined by XRD analysis (X Pert Pro). Thermal stability was evaluated like hardness change (Vickers hardness tests with 5 kg load) during long-term annealing at temperatures ranged between 300-500 °C. The compressive mechanical properties of both materials in its initial state and after long-term annealing were measured with Instron 5882 machine with deformation speed of 1 mm/min.

3. RESULTS AND DISCUSSION

3.1 Microstructure

The structure of as-compacted AlSi13Fe10 alloy (Fig. 4) was composed of primary α-Al dendrites, α-Al+Si interdendritic eutectic and needle-like intermetallic phases. Primary dendrites were very fine with average distance between dendritic branches approximately 5 μm. Eutectic silicon particles appear to be refined with size of 1 μm and smaller. Needle-like intermetallic phases have an average thickness of 2 μm and length of 30 μm.

Fig. 4: Optical (a) and detailed SEM (b) micrographs of as-compacted AlSi13Fe10

It is also important to notice, that as-compacted alloy shows almost no porosity and good contact between particles.

Fig. 5: Optical (a) and detailed SEM (b) micrographs of annealed (500 °C/100 h) AlSi13Fe10
Sufficient diffusion bonding between particles was achieved by ultra-high pressure of 6 GPa used in this experiment. A similar finding was also reported by Cieslak et. al. [6]. The XRD analysis proved presence of Al, Si and $\beta$-Al$_5$FeSi phases in the structure of the as-compacted AlFe13Si10 alloy.

After annealing, a significant structural refining was observed as is shown in Fig. 5. This structural change was characterized by coarsening of eutectic silicon particles and by fragmentation of the $\beta$-Al$_5$FeSi needle-like particles. The silicon particles changed their original sub-micrometer size to several micrometers. This coarsening can be explained by the tendency to reduce the $\alpha$-Al/Si interface area and by a relatively high diffusion coefficient of Si in solid Al. However, iron is characterized by a lower diffusion coefficient than silicon, and therefore, the coarsening and thickening of $\beta$-Al$_5$FeSi is slower and was not observed even after 100 hours of annealing at 500 °C. Instead, fragmentation of needle-like particles with an original length of 30 µm to shorter particles was observed. Both the coarsening of Si particles and the fragmentation of $\beta$-Al$_5$FeSi have a direct influence on the mechanical properties as will be shown.

![Fig. 6: Micrographs of AlSi12Cu1Mg1Ni1: a) as-cast + T6 thermal treated; b) annealed (500 °C/100 h)](image)

Fig. 6a demonstrate the relatively coarse microstructure of AlSi12Cu1Mg1Ni1 composed of primary $\alpha$-Al dendrites (light), $\alpha$-Al+Si eutectic and intermetallic phases containing mainly Ni and Al. The average distance between dendritic branches were 45 µm and 7 µm, respectively. After 100 hours of annealing at 500 °C (Fig. 6b), the microstructure contained large Si particles whose size grew from original 7 µm up to approximately 20 µm.

### 3.1 Mechanical properties

Figure 7 compares the development of the room temperature Vickers hardness of tested materials during annealing at 300-500 °C. Initial hardness was higher in the case of as-compacted AlSi13Fe10 than the as-cast and T6 heat treated AlSi12Cu1Ni1Mg1 alloy. This phenomenon was caused by much more smaller $\alpha$-Al grain size in the CA alloy due to its rapid solidification. Additionally, higher volume fraction of hard phases, namely Si and $\beta$-Al$_5$FeSi, positively increased the initial hardness and compressive strength (Fig. 8). The casting AlSi12Cu1Ni1Mg1 alloy contained large $\alpha$-Al grains (Fig. 6) whose contribution to the Hall-Petch strengthening mechanism is negligible.

Hardness and compressive strength decrease was observed during annealing, but this decrease occurred much more slowly in the case of AlSi13Fe10 alloy. This can be explained by the already above mentioned fact, that iron have lower diffusivity in solid aluminium than the elements in AlSi12Cu1Ni1Mg1 and therefore coarsening of $\beta$-Al$_5$FeSi needle-like particles occur much more slowly. Fragmentation of these needle-like particles (Fig. 5b) positively influenced hardness (Fig. 7), compressive strength and plasticity (Fig. 8) as well.


be shown in the following paragraphs. The casting AlSi12Cu1Ni1Mg1 alloy rapidly softens during annealing due to the fast growth of precipitates in the α-Al matrix (Fig. 6b).

Fig. 7: Vickers hardness change of AlSi13Fe10 and AlSi10Cu1Mg1Ni1 as a function of annealing time

The compressive strength of as-compacted AlSi13Fe10 alloy in its initial state reached the value of 720 MPa which is significantly higher than the recently reported compressive strength (~ 450 MPa) of the AlSi20Fe5 alloy [7]. However, the compaction conditions were significantly different from ours (400 °C, 250 MPa, 60 minutes). This suggests that the ultra-high pressure compaction at 300 °C/5 min did not induce excessive structural changes and softening.

The AlSi13Fe10 alloy shows certain plasticity, despite its high iron content. This can be attributed to the preparation procedure because the needle-like β-Al5FeSi particles always act as primary stress concentrators during compressive testing. The AlSi13Fe10 alloy prepared by common gravity casting containing large β-Al5FeSi particles would be thus extremely brittle. However, rapidly solidified materials with refined microstructure are able to reduce local stresses allowing the plastic α-Al phase to be deformed.

Fig. 8: Compressive stress-strain diagrams of tested alloys in various states

4. CONCLUSION

This work demonstrated that the AlSi13Fe10 alloy, when prepared by centrifugal atomization and high-pressure compaction, has a refined microstructure, which positively influenced strength, hardness and plasticity. Tested alloy also exhibited excellent thermal stability in comparison with the casting AlSi12Cu1Mg1Ni1 alloy which is generally considered to be thermal stable and therefore used for
combustion engine pistons production. Centrifugal atomization is the way how to prepare aluminium alloys with higher contents of iron eliminating creation of large needle-like $\beta$-$\text{Al}_5\text{FeSi}$ particles, which would result in an extreme brittleness of the as-cast alloy.

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