The Structure Analysis of Secondary (Recycled) AlSi9Cu3 Cast Alloy with and without Heat Treatment

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Al-Si alloys are very universal materials, comprising of from 85% to 90% of the aluminium cast parts produced for the automotive industry (e.g. various motor mounts, engine parts, cylinder heads, pistons, valve retainer, compressor parts, etc.). Production of primary Al-alloys belong to heavy source fouling of life environs. Care of environment of aluminium is connected to the decreasing consumption of resource as energy, materials, water, and soil, and with an increase in recycling and extension life of products in industry. Recycled (secondary) aluminium alloys are made out of Al-scrap and workable Al-garbage by recycling. The automotive casts from aluminium alloys are heat treated for achieving better properties. Al-Si alloys contain more addition elements, that form various intermetallic phases in the structure. They usually contain a certain amount of Fe, Mn, Mg, and Zn that are present either unintentionally, or they are added deliberately to provide special material properties. These elements partly go into the solid solution in the matrix and partly form intermetallic particles during solidification which affect the mechanical properties. Controlling the microstructure of secondary aluminium cast alloy is therefore very important.

Key words: secondary Al alloy, intermetallic phases, structural analysis, solution treatment, mechanical properties.

1. Introduction

The characteristic properties of aluminium, good formability, good corrosion resistance, high strength stiffness to weight ratio, and recycling possibilities make it as the ideal material to replace heavier materials (steel, cast iron or copper) in the car [1]. More than half aluminium on the present produce in European Union comes from recycled raw material. The primary aluminium production needs a lot of energy and constraints decision mining of bauxite. The European Union has big interest of share recycling aluminium and therefore increases interest about secondary aluminium alloys and cast stock from them [2].
The replacement of primary aluminium with recycled has in recent years increasing tendency. The recycled metal is a positive trend, because secondary aluminium produced from recycled metal requires only about 2.8 kWh/kg of metal produced while primary aluminium production requires about 45 kWh/kg produced. The remelting of recycled metal saves almost 95% of the energy needed to produce primary aluminium from ore, and, thus, triggers associated reductions in pollution and greenhouse emissions from mining, ore refining, and melting. Increasing the use of recycled metal is also quite important from an ecological standpoint, since producing Al by recycling creates only about 5% as much CO$_2$ as by primary production [3].

Due to the increasing utilization of recycled aluminium cast alloys, the quality of recycled Al-Si casting alloys is considered to be a key factor in selecting an alloy casting for a particular engineering application. The mechanical properties will be radically increasing by implementing adaptable alloying- and process technology, leading to larger application fields of complex cast aluminium components such as safety details. Generally, the mechanical and microstructural properties of aluminium cast alloys are dependent on the composition; melt treatment conditions, solidification rate, casting process and the applied thermal treatment [4, 5]. The mechanical properties of Al-Si alloys depend, besides the Si, Cu, Mg and Fe-content, more on the distribution and the shape of the silicon particles [6]. The presence of additional elements in the Al-Si alloys allows many complex intermetallic phases to form, such as binary phases (e.g. Mg$_2$Si, Al$_2$Cu), ternary phases (e.g. $\beta$-Al$_5$FeSi, Al$_2$CuMg, AlFeMn, A$_{17}$Cu$_4$Ni and AlFeNi) and quaternary phases (e.g. cubic $\delta$-Al$_{15}$($\text{FeMn}$)$_3$Si$_2$ and Al$_{15}$Cu$_2$Mg$_8$Si$_6$) [5, 6–10], all of which may have some solubility for additional elements.

In AlSiCu cast alloy can form these intermetallic phases:

- **Fe-rich intermetallic phases** – Al$_5$FeSi and Al$_{15}$($\text{FeMn}$)$_3$Si$_2$. The dominant phase is phase known as beta- or $\beta$-needles phase Al$_5$FeSi. This needle-shape phase is more unwanted; because can bring high stress concentrations, thereby increase crack imitation and decreasing the ductility [11, 12]. The deleterious effect of Al$_5$FeSi can be reduced by increasing the cooling rate or superheating the molten metal. Another way that might by use to suppress the formation this monoclinic phase is converting the morphology by the addition of a suitable “neutralizer” like Mn, Co, Cr, Ni, V, Mo and Be. The most common addition has been Mn. Excess Mn may reduce Al$_5$FeSi phase and promote formation Fe-rich phases Al$_{15}$($\text{FeMn}$)$_3$Si$_2$ (know as alpha- or $\alpha$-phase) in form “skeleton like” or in form “Chinese script”. This phase has according to some author’s cubic or hexagonal structure. If Mg is also present with Si can phase called as pi- or $\pi$-phase form – Al$_5$Si$_6$Mg$_8$Fe$_2$. Al$_5$Si$_6$Mg$_8$Fe$_2$ has script-like morphology [11–13].
• Cu-rich intermetallic phases – Al$_2$Cu, Al-Al$_2$Cu-Si and Al$_5$Mg$_8$Cu$_2$Si$_6$ [11–14]. In unmodified alloys copper is present primarily as Al$_2$Cu or Al-Al$_2$Cu-Si phase, in modified alloys as Al$_5$Mg$_8$Cu$_2$Si$_6$. The average size of the copper phase decreases upon Sr modification. The Al$_2$Cu phase is often observed to precipitate both in a small blocky shape with microhardness 185 HV 0.01. Al-Al$_2$Cu-Si phase is observed in very fine multi-phase eutectic-like deposits with microhardness 280 HV 0.01 [5, 11, 14, 15].

Influence of intermetallic phases to mechanical and fatigue properties depends on size, volume and morphology these phases [16]. The formation of these phases should correspond to successive reactions during solidification with an increasing number of phases involved at decreasing temperature. In practice, Bäckerud et al. [17] identified five reactions in Al-Si-Cu alloy:

609°C: α-dendritic network;
590°C: Liq. → α-phase + Al$_{15}$Mn$_3$Si$_2$ + Al$_5$FeSi;
575°C: Liq. → α-phase + Si + Al$_5$FeSi;
525°C: Liq. → α-phase + Al$_2$Cu + Al$_5$FeSi + Si;
507°C: Liq. → α-phase + Al$_2$Cu + Si + Al$_5$Mg$_8$Si$_6$Cu$_2$.

The quality and the tolerances of compositional secondary alloys are very important, therefore are still under investigation of many academicals and industrial projects. The purpose of the present article is to investigate microstructure of cast Al-Si alloy (without and with heat treatment) prepared by recycling with combination different analytical techniques (light microscopy upon black-white, scanning electron microscopy (SEM) upon deep etching and energy dispersive X-ray analysis (EDX)). As well as changes of the mechanical properties, which are depending on the microstructure changes.

2. Experimental work

For investigation a microstructure was used the AlSi9Cu3 cast alloy with chemical composition 9.4% Si, 2.4% Cu, 0.9% Fe, 0.28% Mg, 0.24% Mn, 1.0% Zn, 0.03% Sn, 0.06% Pb, 0.04% Ti, 0.05% Ni, 0.04% Cr (wt. %). The chemical analysis of cast alloy was carried out using an arc spark spectroscopy. The experimental alloy (prepared by recycling of aluminium scrap) was received in the form of 12.5 kg ingots. Experimental material was molten into the chill mould (chill casting) (Fig. 1). The melting temperature was maintained at 760°C ±5°C. Molten metal was purified with salt AlCu4B6 before casting and was not modified or grain refined.
AlSi9Cu3 cast alloy has lower corrosion resistance and is suitable for high temperature applications (dynamic exposed casts, where are not so big requirements on mechanical properties) – it means to max. 250°C. The AlSi9Cu3 alloy has these technological properties: tensile strength ($R_m = 240–310$ MPa), offset 0.2% yield stress ($R_{p0.2} = 140–240$ MPa), however the low ductility limits ($A_5 = 0.5–3\%$) and hardness HB 80–120 [18, 19].
Microstructural characterization was performed using light microscope Neo-phot 32 and SEM observation with EDX analysis using scanning electron microscope VEGA LMU II, while phase microanalysis was performed using energy dispersive X ray spectroscopy EDX (EDX analyzer Brucker Quantax). The samples for metallographic observations (1.5 × 1.5 cm) were prepared by standards metallographic procedures (wet ground, polished with diamond pastes, finally polished with commercial fine silica slurry – STRUERS OP-U and etched by standard reagent (Dix-Keller, 0.5% HF). Some samples were also deep-etched in order to reveal the three-dimensional morphology of the silicon phase and intermetallic phases for 30 s in HCl solution. The specimen preparation procedure for deep-etching consists of dissolving the aluminium matrix in a reagent that will not attack the eutectic components or intermetallic phases. The residuals of the etching products should be removed by intensive rinsing in alcohol. The preliminary preparation of the specimen is not necessary, but removing the superficial deformed or contaminated layer can shorten the process. Same pictures were made with using backscattered electrons. The backscattered electrons are beam electrons that are reflected from the sample by elastic scattering. BSE are often used in analytical SEM, because the intensity of the BSE signal is strongly related to the atomic number of the specimen, BSE images can provide information about the distribution of different elements in the sample [16].

For better mechanical properties of Al-Si alloys is good to use a heat treatment. Mechanical and fatigue properties of aluminium cast depends on size, volume and morphology of intermetallic phases and silicon particles [16]. During heat treatment the morphology of intermetallic phases and silicon particles was change and therefore was use solution treatment of AlSi9Cu3 alloy. Heat treatment consist of solution treatment at temperature 505, 515 and 525°C with holding time 2, 4, 8, 16 and 32 hours, than water quenching at 40°C and nature aging for 24 hours on air.

3. Results of experimental work

AlSi9Cu3 belongs to hypoeutectic aluminium cast alloys because contains 9.4% of Si. The Fig. 2 shows as-cast microstructure of the experimental secondary AlSi9Cu3 cast alloy. The analyzed microstructure contains primary aluminium dendrites (α-phase – light grey – 1), eutectic (mixture of α-matrix and spherical dark grey Si-particles – 2) and variously type’s Cu- and Fe-rich intermetallic phases (3, 4), that are concentrated mainly in the interdendritic spaces. In view of the grey scale values in the SEM secondary electron images are higher (brighter) for the higher atomic number of the elements, phases containing Cu and/or Fe are brightest (Fig. 2b).
3.1. Eutectic and eutectic silicon

The eutectic is mixture of $\alpha$-matrix and Si particles. The $\alpha$-matrix precipitates from the liquid as the primary phase in the form of dendrites and is comprised of Al and Si (Fig. 2, Fig. 3a). Silicon is an anisotropic phase.
Experimental material was not grain refined and not modified so eutectic Si grows in a faceted manner along preferred crystallographic directions according to the twin plane re-entrant edge mechanism (TPRE-mechanism) as platelets (Fig. 3b). Most likely Si-platelets grew epitaxial from the surrounding primary aluminium dendrites. This result is in accordance with reports for unmodified Al-Si alloys.
Fig. 4. Eutectic Si after heat treatment of AlSi9Cu3 cast alloy.
The mechanical properties of cast component are determined largely by the shape and distribution of Si particles and intermetallic phases in α-matrix. This plate-like type of morphology of eutectic Si is not good for mechanical properties, because Si platelets are hard but brittle and can crack exposing the soft α-matrix. Therefore are needs to affect this morphology with the appropriate manner. The kinetics of Si morphology transformation is influencing with the solution treatment (under certain conditions). Heat treatment affects the precipitates size, shape and distribution in a cast component too [20]. The spheroidization process of Si particles takes place in two stages with application of solution treatment: a) fragmentation or dissolution of the eutectic Si branches; b) spheroidization of the separated branches [21, 22]. Optimum tensile, impact and fatigue properties are obtained with small, spherical and evenly distributed particles. Silicon also imparts heat treating ability to the casting through the formation of compounds with Mg, Fe and Cu. For that reason the AlSi9Cu3 cast alloy was heat treated.

The effect of solution treatment on morphology of eutectic Si is demonstrated in Fig. 4. The changes in morphology of eutectic Si observed after heat treatments are documented for holding time of 4 hours. Eutectic Si without heat treatment (untreated state) occurs in platelets form (Fig. 3b). After solution treatment by temperature of 505°C we noted that the platelets were fragmented into smaller platelets with spherical edges (Fig. 4a). The spheroidized process dominated at 515°C. The smaller Si particles were spheroidized to rounded shape, see Fig. 4b. By solution treatment at 525°C the spheroidized particles gradually grew larger (coarsening) (Fig. 4c).

3.2. Fe-rich intermetallic phases

Iron is one of the most critical alloying elements, because Fe is the most common and usually detrimental impurity in cast Al-Si alloys. The Fe impurity in Al-Si cast alloys results mainly from the use of steel tools and scrap materials [10, 12, 23].

The solubility of iron is very low in aluminium alloys so most iron forms intermetallic phases. According to [24], the two main types of Fe-rich intermetallic phases occurring in this AlSi9Cu3 alloy are Al$_5$FeSi and Al$_{15}$(FeMn)$_3$Si$_2$. Significant levels of Fe (e.g. > 0.5%) can change the solidification characteristics of Al-Si alloys by forming pre- and post-eutectic Al$_5$FeSi phase [6, 11]. Al$_5$FeSi phases precipitate in the interdendritic and intergranular regions as platelets (appearing as needles in the metallographic microscope – Fig. 5a). Long and brittle Al$_5$FeSi platelets (more than 500 μm) can adversely affect mechanical properties and also lead to the formation of excessive shrinkage porosity de-
effects in castings [25]. TAYLOR [11] further suggested that the formation of large \( \beta \) platelets at high Fe-contents facilitates the nucleation of eutectic Si, therefore leading to a rapid deterioration of the interdendritic permeability. The \( \beta \) platelets appeared to be the main nucleation sites for the eutectic Si and Cu-rich
Fig. 6. Effect of heat treatment on Fe-rich phases of AlSi9Cu3 cast alloy: a) 505°C/4 h, etch. Dix-Keller, b) 515°C/4 h, etch. Dix-Keller, c) 525°C/4 h, etch. Dix-Keller, d) changes in average area of Fe-rich phases.
phase. Nucleation of Si and Al$_2$Cu may occur on large Al$_5$FeSi platelets. Phase with cubic crystal structure – Al$_{15}$($\text{FeMn}$)$_3$Si$_2$ is considered less harmful to the mechanical properties than $\beta$ phase [26, 27]. This phase (Fig. 5b) has a compact morphology “Chinese script” or skeleton-like, which does not initiate cracks in the cast material to the same extent as the Al$_5$FeSi (Fig. 5a).

The Fe-rich particles can be twice as large as the Si particles, and the cooling rate has a direct impact on the kinetics, quantities and size of Fe-rich intermetallic present in the microstructure. In experimental recycled AlSi9Cu3 cast alloy that contains less than 0.9% of Fe and 0.24% of Mn were observed Al$_5$FeSi needles (Fig. 5a) – on deep etcher samples plate-like form (Fig. 5a) and Al$_{15}$($\text{FeMn}$)$_3$Si$_2$ – skeleton-like form (Fig. 5b). In experimental material was satisfied condition Fe:Mn = 2:1, therefore intermetallic needles phases were observed in a few isolated cases.

Heat treatment was use for affecting the size of Fe-rich phases, because the shape and size of iron compounds is more influential than the quantity of those iron compounds. The evolution of the Fe-rich phases during solution treatment is described in Fig. 6. Al$_5$FeSi phase is dissolved into very small needles (difficult to observe). The Al$_{15}$($\text{MnFe}$)$_3$Si$_2$ phase was fragmented to smaller skeleton particles. In the untreated state Al$_{15}$($\text{FeMn}$)$_3$Si$_2$ phase has a compact skeleton-like form (Fig. 5b). Solution treatment of this skeleton-like phase at 505°C tends to fragmentation (Fig. 6a) and at 515 or 525°C to spheroidization and segmentation (Fig. 6b, c).

For the confirmation that solution treatment reduces Fe-rich phases area and affects its morphology was used the quantitative metallography. Quantitative metallography was carried out on an Image Analyzer NIS – Elements to quantify Fe-rich phases (average area) morphology changes, during solution treatment. Figure 6d shows the changes in the average area of Fe-rich phases during solution treatment. The maximum average area of Fe-rich phases was observed in as-cast samples (2495 $\mu$m$^2$). By increasing the solution temperature the average area of Fe-phases drop to (the increasing temperature of solution treatment causes dropping the average area of Fe-phases to 320 $\mu$m$^2$ by 515°C). With a prolonged solution treatment time more than 8 h, the extent of dissolution of Fe-rich phases changed little.

### 3.3. Cu-rich intermetallic phases

Half or more of the copper is found as a component of intermetallic compounds [28]. Cu intermetallic phases are in aluminium alloys forming such as Al$_2$Cu with tetragonal crystal structure, which solidified in two morphologies after Al-Si eutectic reaction. The first are as massive or blocky form (Al$_2$Cu
Fig. 7. Morphology of Cu-rich intermetallic phases in as-cast structure of AlSi9Cu3 cast alloy: a) 1-Al$_2$Cu, 2- Al$_2$Cu-Si, etch. Dix-Keller, SEM, b) Al$_2$Cu-Si, deep etch. HCl, SEM, c) detail of Al$_2$Cu phase, etch. Dix-Keller, d) detail of Al$_2$Cu-Si phase, etch. Dix-Keller.

The increasing level of Cu improves the strength of the aluminium alloy through the formation of Cu based precipitate during heat treatment. The effect of heat treatment on morphology of Cu-rich phases was followed by optical and
Fig. 8. Effect of heat treatment on Cu-rich phases of AlSi9Cu3 cast alloy: a) 505\(^\circ\)C/4 h, etch. Dix-Keller, b) 515\(^\circ\)C/4 h, etch. Dix-Keller, c) 525\(^\circ\)C/4 h, etch. Dix-Keller, d) changes in average area of Cu-rich phases.
electron microscopy. Morphology changes of Al-Al$_2$Cu-Si during heat treatment are demonstrated in Fig. 8. The changes of morphology of Al-Al$_2$Cu-Si observed after heat treatment are documented for holding time 4 hours.

Al-Al$_2$Cu-Si phase without heat treatment (as-cast state) occurs in form compact oval troops (Fig. 7). After solution treatment at temperature 505°C these phase disintegrated into smaller segments. The amount of Al-Al$_2$Cu-Si phase decreases. This phase is gradually dissolved into the surrounding Al-matrix with an increase in solution treatment time (Fig. 8a). By solution treatment 515°C was this phase observed in the form coarsened globular particles and these occurs along the black needles, probably Fe-rich Al$_5$FeSi phase (Fig. 8b). By solution treatment 525°C was this phase documented in the form molten particles with homogenous shape (Fig. 8c).

The changes of average area of Cu rich phases were confirmed by using the quantitative metallography, too. Figure 8d shows average area of Cu-rich phases obtained in solution heat treated samples. Maximum average area of Al-Al$_2$Cu-Si phase was observed by temperature solution treatment at 505°C with holding times 2 hours (357 µm$^2$). Minimum average area of Al-Al$_2$Cu-Si phase particle was observed by temperature solution treatment at 515°C (0.277 µm$^2$). It is evident that heating at temperatures below the final solidification temperature (505°C, 515°C and 525°C) results in dissolution of Al-Al$_2$Cu-Si phase [29–31]. Solution treatment at 525°C apparently causes a marked change (Fig. 8). This, however, is attributed to the melting of the Al-Al$_2$Cu-Si, rather than to its dissolution. Dissolution and melting of Al$_2$Cu phase in AlSi9Cu3 alloy has been studied in detail by Samuel [32]. When the AlSi9Cu3 alloy is solution treated at temperature about the melting point of the eutectic (Al+Al$_2$Cu) phase, e.g. 525–540°C, the Al-Al$_2$Cu-Si particles may undergo incipient melting even after periods as 4 hours [29–31].

### 3.4. SEM image and X-ray analysis

The SEM image and X-rays analysis were used for a complete structural analysis of experimental material. Figure 9 shows typical example: a SEM image and X-rays analysis of Al-Al$_2$Cu-Si.

Structural analysis identified of recycled (secondary) AlSi9Cu3 cast alloy as basic structural elements: α-phase, Si platelets, Fe-rich intermetallic phases: needles – Al$_5$FeSi (but in a small volume); skeleton-like Al$_{15}$(FeMn)$_3$Si$_2$ phase and Cu-rich intermetallic phase: Al$_2$Cu (but in a small volume); Al-Al$_2$Cu-Si ternary eutectic. The EDX analysis revealed that the identified Cu-rich and Fe-rich intermetallic phases by using light microscopy are really these intermetallic phases, because chemical composition of these phases was confirmed by EDX analysis.
[Fig. 9]
b)
3.5. Changes of mechanical properties cause with changes of structure

Heat treatment is one of the major factors used to enhance the mechanical properties of heat-treatable Al-Si alloys, through an optimization of both solution and aging heat treatments. The solution treatment homogenises the cast structure and minimizes segregation of alloying elements in the casting. Segregation of solute elements resulting from dendritic solidification may have an adverse effect on mechanical properties.

Changes of microstructural parameters cause changes in mechanical properties during solution treatment. Solution treatment performs three roles: homogenization of as-cast structure; dissolution of certain intermetallic phases such as Al$_2$Cu; changes the morphology of eutectic Si and intermetallic phases by fragmentation, spheroidization and coarsening, thereby improving mechanical properties.

After heat treatment were samples subjected for mechanical test (strength tensile and Brinell hardness). Influence of solution treatment and changes of microstructural parameters on mechanical properties are shown on Fig. 10 and
Fig. 10. Changes of strength tensile.

Fig. 11. After solution treatment tensile strength and hardness are remarkably improved, compared to the corresponding as-cast condition. Highest strength tensile was 273 MPa for 515°C/4 hours (Fig. 10). With further increase in solution temperature more than 515°C and solution time more than 8 hours, tensile strength gently decreases during the whole solution period.

Fig. 11. Changes of Brinell hardness.

Results of hardness (Fig. 11) are comparable with results of tensile strength. Highest hardness was 124 HBS for 515°C/2 hours. The hardness decreases during the temperature 525°C due to melting of the Al-Al$_2$Cu-Si phase by this temperature [29–31].

4. Conclusion

Understanding metal quality is of great importance for control and prediction of casting characteristics. The results of optical and SEM studies of recycled (secondary) AlSi9Cu3 cast alloy are summarized as follows:

- Structural analysis identified as basic structural elements: $\alpha$-phase, Si platelets, and intermetallic phases (Al$_{15}$(FeMn)$_3$Si$_2$ in the skeleton-like form, Al$_3$FeSi
in form needles and Cu-ternary eutectic Al-Al\textsubscript{2}Cu-Si, Al\textsubscript{2}Cu in a small block shape).

- In experimental material are dominant: Cu-rich phase Al-Al\textsubscript{2}Cu-Si and Fe-phases Al\textsubscript{15}(FeMn)\textsubscript{3}Si\textsubscript{2} (thanks to the presence of Mn). Chemical composition of all phases was confirmed by EDX analysis.
- During heat treatment Si particles spheroidized. As the optimum temperature of spheroidization the eutectic Si was specified the temperature 515\textdegree{}C.
- The morphology and size of iron phases are highly dependent on the solution treatment. Platelets Fe-rich phases (Al\textsubscript{5}FeSi) are dissolved into very small needle phases. Skeleton-like Fe-rich phases (Al\textsubscript{15}(FeMn)\textsubscript{3}Si\textsubscript{2}) are fragmented and dissolved (average area reduces from 2 495 to 320 \textmu{}m\textsuperscript{2}).
- Al-Al\textsubscript{2}Cu-Si phases are fragmented, dissolved and redistributed within \textalpha{}-matrix (average area of Cu-phases particle decreases from 9 995.5 \textmu{}m\textsuperscript{2} to 0.277 \textmu{}m\textsuperscript{2}) during heat treatment.
- Changes of microstructural parameters of AlSi\textsubscript{9}Cu\textsubscript{3} cause changes in mechanical properties. The highest strength tensile was at a temperature of 515\textdegree{}C with holding time 4 hours; the highest hardness at a temperature of 515\textdegree{}C with holding time 2 and 4 hours. For this was defined as optimum regime of solution treatment for experimental samples from AlSi\textsubscript{9}Cu\textsubscript{3} cast alloy using in automotive industries regime: 515\textdegree{}C with a holding time of 4 hours, water quenching at 40\textdegree{}C and nature aging for 24 hours on air. After heat treatment, casts for automotive industries have better mechanical properties as in an as-cast state.

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