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E. Tillová, M. Chalupová: Solution treatment effect on microstructure and mechanical properties of automotive cast alloy

SOLUTION TREATMENT EFFECT ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF AUTOMOTIVE CAST ALLOY

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Resume

The contribution describes influence of the heat treatment (solution treatment at temperature 545°C and 565°C with different holding time 2, 4, 8, 16 and 32 hours; than water quenching at 40°C and natural aging at room temperature during 24 hours) on mechanical properties (tensile strength and Brinell hardness) and microstructure of the secondary AlSi12Cu1Fe automotive cast alloy. Mechanical properties were measured in line with EN ISO. A combination of different analytical techniques (light microscopy, scanning electron microscopy (SEM)) were therefore been used for study of microstructure. Solution treatment led to changes in microstructure includes the spheroidization and coarsening of eutectic silicon. The dissolution of precipitates and the precipitation of finer hardening phase further increase the hardness and tensile strength of the alloy. Optimal solution treatment (545°C/4 hours) most improves mechanical properties and there mechanical properties are comparable with mechanical properties of primary AlSi12Cu1Fe alloy. Solution treatment at 565 °C caused testing samples distortion, local melting process and is not applicable for this secondary alloy with 12.5 % Si.

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1. Introduction

The use of aluminium components in the automotive industry has increased considerably during the past ten years due to their lightweight and reduced fuel energy consumption advantages. Another advantage, which is equally important from an environmental point of view, is the fact that aluminium components may be recycled at relatively low energy costs. Secondary aluminium produced from recycled Al-metal requires only about 2.8 kWh/kg of metal produced. Primary aluminium production is highly energy-intensive and requires about 45 kWh/kg of Al-metal produced. Please note, however, that the metal's melting point (approx. 660 °C) is so low that remelting requires only about five per cent of the original energy input and creates only about 5 % as much CO_2 as by primary production [1]. This

means that efficient aluminium recycling is profitable.

Among aluminium alloys, aluminiumsilicon (Al-Si) alloys are known for their good castability and mechanical properties. The addition of Mg, Cu, and Zn makes the alloys heat-treatable [2-3], providing the means to enhance their properties with the use of appropriate heat treatments. The mechanical properties of an Al-Si cast alloy are mainly determined by its cast structure and the microstructural characteristics such as the grain size, dendrite arm spacing (DAS), the size, shape and distribution of the eutectic silicon particles, as well as the morphologies and amounts of present intermetallic phases [2-7]. These parameters are completely changed after heat treatment, which, in turn, influences the

resultant mechanical properties [2-3, 8-12].

The present study is a part of larger research project, which was conducted to investigate and to provide a better understanding of secondary (recycled) cast alloys after heat treatment. Present work is focused on study of the effect of solution heat treatment parameters on mechanical properties and on microstructure (changes in morphology of eutectic Si and intermetallic phases) of AlSi12Cu1Fe cast alloy. In the automotive industry this recycled alloy is used in the form of various motor mounts or pistons, cylinder blocks and so on.

2. Experimental part

As an experimental material was used secondary (recycled) eutectic AlSi12Cu1Fe cast alloy (in the form of 12.5 kg ingots). The chemical composition is presented in the table 1. The alloy was molten into the sand form. Sand casting is the simplest and most widely used casting method. A pattern, of the final casting (Fig. 1), was formed from metal. The melting temperature was maintained at 760 °C \pm 5 °C. Melting was before casting refined with salt AlCu4B6. The melt was not modified or grain refined. The chemical analysis of AlSi12Cu1Fe cast alloy was carried out using arc spark spectroscopy.

AlSi12Cu1Fe as eutectic cast alloy has very good castability, lower corrosion resistance and is suitable for high temperature applications (dynamic exposed casts, where are not so high requirements on mechanical properties) - it means to 250 °C. Such high silicon contents assure the dimensional stability of the casting upon heating e.g. for new Audi V6 and V8 cylinder block.

Chemical composition of AlSi12Cu1Fe cast alloy							
Mass concentration of the element, %							
Si	Mg	Cu	Fe	Mn	Zn	Ni	Ti
12.5	0.347	0.85	0.70	0.245	0.43	0.044	0.024

Table 1



Fig. 1. Experimental castings

The sand casting produced from primary AlSi12Cu1Fe cast alloy achieves high values for tensile strength (R_m = 240 MPa), offset 0.2 % yield stress (R_p 0.2 = 140 MPa), however the low ductility limits (1 - 3 %) and Brinell hardness 70 HB.

Experimental samples (standard tensile test specimens) were treated with T4 heat treatment - solution treatment for 2, 4, 8, 16 or 32 hours at two temperatures (545 °C and 565 °C); water quenching at 40 °C and natural aging for 24 hours at room temperature. After heat treatment were samples subjected for mechanical test. For as cast state, each solution temperature and each aging time, a minimum of five specimens were tested.

Metallographic samples were prepared from selected tensile specimens (after testing) and the microstructures were examined by optical electron microscopy and (SEM). Samples were prepared by standards metallographic procedures (mounting in bakelite, wet ground, DP polished with diamond pastes, finally polished with commercial fine silica slurry (STRUERS OP-U) and etched by Dix-Keller. Some samples were also deepetched for 15-30 s in HCl solution in order to reveal the three-dimensional morphology of the eutectic silicon [4]. The specimen preparation for deep-etching procedure consists of dissolving the aluminium matrix in a reagent that will not attack the eutectic components. 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.

Standard tensile test specimens with 6 mm diameter were measured in line STN EN 10002-1 at room temperature. Hardness measurement was preformed by a Brinell hardness tester with a load of 62.5 kp (1 kp = 9.807 N), 2.5 mm diameter ball and a dwell time of 15 s. The Brinell hardness value at each state was obtained by an average of at least six measurements.

3. Results

Heat treatment of Al-Si cast alloys is to some extent optional. High pressure die cast components tend to contain air-filled pores which expand during heat treatment, and are not heattreatable due to blistering, etc. Sand-cast or permanent mould cast components are more amenable to heat treatments. For experimental work was used T4 heat treatment consists of:

- solution treatment for two different temperatures 545 °C and 565 °C, that is necessary to produce a solid solution;
- rapid water quenching (40 °C) to retain the maximum concentration of hardening constituent in solid solution;
- natural ageing (24 hours at room temperature) to obtain the desired mechanical properties in the casting.

Solution heat treatment causes: homogenization of as-cast structure; rounds (spheroidization) of the Si-particles, and thus particularly improves the ductility; dissolves or transforms primary Mg- and Cu-intermetallic phases like Mg₂Si, π -AlMgFeSi, Al₂Cu and AlCuMgSi provided that the temperature is high; improves the potential for age hardening of Mgand Cu-bearing Al-Si alloys, especially if the temperature is high and the cooling is fast and removes internal stress, which can make it difficult to stay within the dimensional tolerances.

The heat treatment temperature should be high, but referable below the local melting

temperature. Local melting gives notches at the surface, greater tendency to sagging, and more oxidation during the heat treatment. The dangerous temperatures for local melting are the eutectic points of the Al-systems (e.g. pure Al-Si with Fe: 578 °C [13-14]; Al-Si with Cu and Fe: 525 °C [15]). Slow heating may remove eutectics and even out concentration gradients, thereby allowing heat treatment up to the equilibrium solidus temperature of the alloy. This temperature can in principle be found in the phase diagram. Solution heat treatment time depends on microstructure, section thickness, and furnace loading and can vary from less than a minute to 20 hours. Generally, the soak times for castings are longer than for wrought products due to coarser microstructures.

Although the morphology, the amount and the distribution of the precipitates during aging process significantly influence the mechanical properties, an appropriate solution treatment is a prerequisite for obtaining desirable aging effect. From this point of view, the solution heat treatment is critical in determining the final microstructure and mechanical properties of the alloys. Thus, it is very important to investigate the effects of solution heat treatment on the alloys, before moving on to aging issues.

Influence of solution treatment on mechanical properties for experimental alloy is shows on Fig. 2 and Fig. 4.



After solution heat treatment at optimal solution (545 °C) temperature, tensile strength and hardness are remarkably improved, compared to the corresponding as-cast condition.

Fig. 2 shows the results of tensile strength measurements. The as cast samples have a strength value approximately 200 MPa. For 2 hours the solution treatment strength value immediately increases. The increase of strength values is significant chiefly for holding times maximal 4 hours. Highest tensile strength was approximately 245 MPa. By holding time 8 hours begins decreasing of strength values and relates probably to gradual coarsening of eutectic Si by the holding time longer as 4 hours.



Fig. 3. Testing samples distortions (565 °C/32 hours)

With increase in solution temperature more than 545 °C tensile strength strong decreases. Probably by reason that the temperature 565 °C is nearly at eutectic point comes to decline of mechanical properties values from the reason of significant local melting process. Solution treatment at 565 °C / 32 hours led to massive testing samples distortion (Fig. 3) invoking by alloy melting and tensile strength values could not correct measured. This suggests that, to enhance the tensile strength, of this recycled eutectic alloy with 12.5 % Si, by increasing of solution temperature more than 545 °C does not seem possible.

Fig. 4 shows the evolution of Brinell hardness value. Results of hardness are comparable with results of tensile strength. The untreated (as cast samples) have hardness value approximately 83 HBS. For 2 hours the solution treatment, independently from temperature of solution treatment, hardness value immediately increases. The maximum was observed after 4 hours at 545 °C (approximately 108 HBS). However, up to 8 hours solution treatment at

545 °C the hardness values are continuously decrease as resulted from the coarsening of eutectic silicon, increasing of interparticle spacing and dissolution of the Al₂Cu phase. After prolonged solution treatment time up to 4 hours at 565 °C, it is clearly that the hardness values are strong decreasing probably due to local melting process of the alloy.



The mechanical properties of cast component are determined largely by the shape and distribution of Si particles in the matrix. Optimum tensile, impact and fatigue properties [16] are obtained with small, spherical and evenly distributed particles.

Structure of experimental eutectic AlSi12Cu1Fe cast alloy consists of eutectic (dark grey Si-particles in light grey α -phase) and intermetallic phases (Fig. 5). The formation of Fe- and Cu-rich intermetallic phases should successive reaction correspond to during solidification [17-18]. Si is the major alloying element in heat-treatable cast Al-Si-Cu alloys, and Si particles represent a large volume fraction of the eutectic alloy's microstructure.

The presence a minimum primary Si particles was observed too (Fig. 6). Numbers of hard coarse primary Si particles in the microstructure of eutectic Al-Si cast alloy are undesirable. Primary Si particles certainly increase locally the wear resistance of the alloy, but unfortunately Si is brittle and is easy to crack exposing the soft Al matrix to extreme wear resulting catastrophic for the automotive components.



Fig. 5. Microstructure of AlSi12Cu1Fe alloy (1-eutectic Si, 2-α-phase, 3-Fe-phase, 4-Cu-phase) etch. Dix-Keller



Fig. 6. Morphology of eutectic (1) and primary (2) silicon (as cast state), etch. HCl, SEM

The effect of solution treatment on morphology of eutectic Si is demonstrated in Fig. 6 and Fig. 7. The changes of eutectic Si morphology observed after heat treatments are documented for temperature 545 °C. Experimental material was not modified and so eutectic Si particles without heat treatment (untreated as cast state) are in form platelets (Fig. 6) [4], which on scratch pattern are in form needles (Fig. 5).

For 2 hours the solution treatment were noted that the Si-platelets were fragmentized into smaller platelets with spherical edges (on scratch pattern round particles and round needles (Fig. 7a)). The spheroidized process dominated after 4 hours. The smaller Si particles were spheroidized to rounded shape (Fig. 7b). Up to 8 hours solution treatment the spheroidized particles gradually grew larger (coarsening) (Fig. 7c, d). After solution treatment we can observed, that the primary Si particles rounded and reduced their size (refines) - Fig. 7a.

Fe-phases precipitate first of all as skeleton-like phase that are form by Fe, Si along with Mn [4, 17-21]. This Al₁₅(FeMn)₃Si₂ phase has a compact skeleton-like morphology, which does not initiate cracks in the cast material to the same extent as the needle-like phase Al₅FeSi (Fig. 5). The effect of solution treatment on the Fe-rich Al₁₅(FeMn)₃Si₂ phase for solution treatment is documented on Fig. 7 (marked with ••••) too. In untreated state is $Al_{15}(FeMn)_3Si_2$ phases in compact skeleton-like form (Fig. 5). Solution treatment of this skeleton-like phase tends to fragmentation, spheroidization and segmentation (Fig. 7a, 7b and 7c). Solution treatment reduces its area rather than change the morphology.

Presence of Cu improves the strength of the aluminium alloy through the formation of Cu based precipitate during heat treatment.

The Cu-rich intermetallic phase is formed with Al during solidification according to the reaction: $L \rightarrow (Al + Al_2Cu + \beta - Al_5FeSi + Si)$ at 525 °C. This reaction relates to the start of Al₂Cu precipitation towards the end of solidifications and consequently may be nucleated on other interdendritic particles (Si, Fe-rich phases).



a) 2 hours b) 4 hours c) 16 hours d) 32 hours Fig. 7. Effect of solution treatment at 545 °C on morphology of eutectic Si, Fe-rich (••••) and Cu-rich (----) intermetallic phases, etch. Dix-Keller

Effect solution of treatment on morphology of Al₂Cu correlated with works [6-7, 22-23]. In samples without heat treatment (untreated as cast state) is Al₂Cu phase observed in form of compact oval troops. After solution treatment these phase disintegrated into very fine smaller segments (marked in Fig. 7 with - - -) and the amount of Al₂Cu phase during heat treatment decreases (Figures 7a, 7b and 7c). This phase is gradually dissolved into the surrounding α -matrix with an increase of solution treatment time.

The alloy strengthening is mainly a result of the formation of Al_2Cu precipitates. The dissolution leaves vacancies and creates distortions in the Al matrix crystals. In a short time, the atoms will not have enough time to rearrange to fill and correct these vacancies and distortions, and these vacancies and distortions act as nuclei and facilitate the formation of large amount of fine Al_2Cu particle precipitates and thus strengthen the alloy. The eutectic Si particles also strengthen the alloy. Smaller and more uniformly distributed particles have a stronger effect. The fragmentation of Si particles reduces the particle size and increases the particle number and thus strengthens the alloy. The fragmentation of the eutectic Si takes place mostly at the early stage of solution treatment (Fig. 7a), and so, the strengthening effect caused by the morphology variation of Si particles occurs mostly in that time. Most strengthening effects from Al₂Cu precipitation, crystal distortion, and Si particle fragmentation occur at short solution treatment time (2-4 hours), and thus, the alloy's strength increases rapidly during this period. Because the dissolution of Si is complete at short solution treatment time, a longer solution treatment time does not add more vacancies and/or distortions.

Instead, the earlier formed vacancies and distortions are reduced and smoothened and become less and less active as nuclei, resulting in fewer Al_2Cu precipitates and slowing the increase in strength. The reduction in vacancies and distortion itself also reduces the strengthening effect.

Thereafter (i. e., after 4 hours), the strength variation will be determined mainly by changes in Si particle size and morphology. Si fragmentation still may be occurring, but slowly, and at the same time, Si particles are spheroidized and coarsened. In that time, the strengthening effect caused by Si fragmentation is counteracted by the weakening effects of coarsening and spheroidization, resulting in small variations in strength. Generally, solution treatment homogenizes the microstructure by dissolving the as cast Al₂Cu precipitates as well as intermetallics (such as Fe-rich compounds in aluminium), it reduces segregation, and spheroidizes intermetallics, and inclusions, which all improve mechanical properties.

4. Conclusions

In the present study, the effects of temperature and holding time on solution heat treatment of secondary eutectic AlSi12Cu1Fe cast alloy for automotive applications in the light of metallographic parameters of silicon particles and mechanical properties (tensile strength and Brinell hardness) was investigated. From an analysis of the results, the following conclusions can be drawn:

- It has, so far, been shown that the mechanical properties are highly influenced by the microstructure of the material, by the coarse silicon plates or the small hardening Al₂Cu precipitates. Consequently, the mechanical properties can be related to the duration of the solution heat treatment.
- Optimal condition of heat solution treatment (545 °C/4 hours) most improves mechanical properties thanks to spheroidization of eutectic Si to rounded shape and the values of tensile strength (245 MPa) or Brinell hardness (cca 108 HB) are comparable with mechanical properties heat treated primary

cast alloy ($R_m = 240$ MPa; 70 HB). Further increases solution time (above 4 hours), leads to gradual coarsening of eutectic Si and the mechanical properties continuously drop get to. The primary Si particles rounded and reduced their size (refines).

- Al₂Cu phases during solution treatment are fragmented, dissolved and redistributed within α-matrix. Skeleton - like Al₁₅(FeMn)₃Si₂ phases are fragmented, dissolved a spheroidized.
- Solution treatment at 565 °C caused testing samples distortion, local melting process and is not applicable for this secondary alloy with 12.5 % Si.

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