MECHANICAL PROPERTIES OF HEAT TREATED SECONDARY AlSi12Cu1Fe CAST ALLOY AT ROOM TEMPERATURE

The contribution describes changes of mechanical properties (strength tensile, absorbed energy and Brinell hardness) in secondary (recycled) eutectic aluminium-silicon cast alloy – AlSi12Cu1Fe during solution treatment. This work presents the influences of the solution treatment by 525 °C, 545 °C and 565 °C with the holding time 2, 4, 8, 16 and 32 hours, water quenching at 40 °C and natural aging with holding time 24 hours. Mechanical properties were measured in line with STN EN ISO at room temperature. Solution treatment has led to changes in mechanical properties, caused by the changes in microstructure including the spheroidization and coarsening of eutectic silicon, gradual disintegration, shortening and thinning of intermetallic phases.

Keywords: solution treatment, mechanical properties, cast Al-Si eutectic alloy

1. Introduction

Many mechanical components, especially those for cars and rail vehicles, are made of Al-Si alloy thanks to the great potential of these materials as replacements for heavier materials (steel, cast iron or copper) [1, 2]. Aluminium alloys are the ideal replacement materials in car due to good formability, good corrosion resistance, high strength stiffness to weight ratio and recycling possibilities [3, 4].

In recent years, however, plenty of aluminium alloys waste has increased, therefore secondary (recycled) aluminium alloys are used to replace primary alloys. The use of secondary aluminium alloys is important, because the production of primary aluminium alloys consumes about 45 kWh/kg of metal and the production of secondary only about 2.8 kWh/kg of metal. Aluminium industry has the advantage of maximizing the amount of recycled metal, taking into account energy-savings and the reduction of dependence upon overseas sources. The remelting of recycled metal saves almost 95 % of the energy needed to produce prime aluminium from ore, and reductions in pollution and greenhouse emissions from mining, ore refining, and melting. Increasing the use of recycled metal is quite important from an ecological standpoint, since producing aluminium by recycling creates only about 5 % as much CO2 as by primary production [5–7]. The utilization of secondary aluminium alloys has increased in recent years also due to its comparable properties with primary aluminium alloys [8].

Eutectic Al-Si alloys offer excellent fluidity, low density, high wear resistance and low expansivity and therefore are used for complex shape casting, thin-walled casting, pistons for rotors, compressor and so on [9, 10]. For example, eutectic Al-Si cast alloys used in the piston of petrol engines manufacturing, those the operating temperature at the top of pistons is 300 – 400 °C, must have good mechanical properties in order to fulfil the requirements for materials for pistons [9].

The mechanical properties of Al-Si alloys depend, besides Si, Cu, Mg and Fe-content, on the distribution and the shape of the silicon particles and changes in morphology of eutectic Si and intermetallic phases in secondary eutectic Al-Si cast alloy. Therefore it’s necessary to understand the effects of the main alloying elements on the microstructure and mechanical properties [11–14].

The morphology of structure parameters can be affected for example with using heat treatment, modifying and grain refining. T4 heat treatment was used in this work. It consists of [15–19]:
- solution treatment, that is necessary to produce a solid solution.
- Production of a solid solution consists of keeping the Aluminium alloy at a sufficiently high temperature and for such a time so as to attain an almost homogeneous solid solution. The holding time on temperature is required to achieve dissolution of the undissolved or precipitated soluble phase constituents and to attain a reasonable degree of homogeneity;
- rapid water quenching to retain the maximum concentration of hardening constituent in solid solution;
- natural aging to obtain the desired mechanical properties in the casting.

The alloy and its heat treatment presented in this work are the part of a larger research project which was conducted to investigate and to provide better understanding of the influence of heat treatment and structure parameters on mechanical properties in recycled (secondary) aluminium cast alloy.
2. Experimental material

Secondary AlSi12Cu1Fe cast alloy was used as an experimental material. The secondary alloy (prepared by recycling of aluminium scrap) was received in the form of 12.5 kg ingots. Experimental material was molten into the chill (chill casting). The melting temperature was maintained at 760 °C \(\pm\) 5 °C. Molten metal was purified with salt AlCu4B6 before casting and was not modified or grain refined. The chemical analysis of AlSi12Cu1Fe cast alloy was carried out using an arc spark spectroscopy and the chemical composition is: 12.3 % Si, 0.8 % Cu, 0.7 % Fe, 0.35 % Mg, 0.22 % Mn, 0.43 % Zn, 0.01 % Sn, 0.05 % Pb, 0.03 % Ti, 0.03 % Ni and 0.02 % Cr.

In that the eutectic reaction occurs at 577 °C and from 11.3 to 12.6 % Si content in the eutectic AlSi12Cu1Fe cast alloy can be regarded as an eutectic alloy. Metallographic samples for the study were cut from the selected tensile specimens (after testing) and hot mounted for metallographic preparation. The microstructures were studied using an optical microscope Neophot 32 and scanning electron microscope (SEM) VEGA LMU II upon deep etching. The samples were prepared by standard metallographic procedures (wet ground on SiC papers, DP polished with 3 μm diamond pastes followed by Struers Op-S and etched for study at an optical microscope by standard etcher Dix-Keller, HNO₃, H₂SO₄ or colour etcher Weck-Aluminium, MA). Colour etching was used to highlight the phases that were not visible very well on the samples which were etched by standard etcher (black-white etcher). By the colour etching the surface of metallographic samples reacts to colour etcher so that on the surface there is transparent film formation, function which interference coating is [20]. The thickness of this transparent film depends on chemical composition of material.

Some samples were also deep-etched for 30 s in HCl solution in order to reveal the three-dimensional morphology of the silicon phase and intermetallic phases [21, 22]. 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. Three-dimensional morphology was observed in such prepared samples, on a scanning electron microscope.

In the experimental cast alloy (AlSi12Cu1Fe), which has 12.3 % Si, the microstructure (Fig. 1) consists of eutectics (eutectic Si in \(\alpha\)-phase (1)), primary Si particles (2) and intermetallic phases (3). It is necessary to achieve maximum performance of casting to affect the morphology of eutectic silicon, which is excreted in the form of large needles [10]. Morphology, distribution and size of Si markedly affect mechanical properties. In that Si can be influenced by the heat treatment without modifying, therefore the experimental alloy was heat treated. The heat treatment of experimental cast samples consists of solution treatment by temperatures 525 °C, 545 °C and 565 °C with the holding time 2, 4, 8, 16 and 32 hours, water quenching at 40 °C and natural aging at room temperature with the holding time 24 hours.

3. Results and discussion

3.1 Mechanical properties

The samples were subjected for mechanical test (tensile test, impact test and Brinell hardness test) after solution treatment. Hardness measurement was performed by a Brinnell hardness tester with the load of 62.5 kp, 2.5 mm diameter ball and the dwell time of 15 s. The Brinell hardness value at each state was obtained by the average of at least six measurements. Fig. 2 shows the variation in hardness of samples with heat treatments time 2, 4, 8, 16 and 32 hours by different temperatures. It can be seen that Brinell hardness is increased during holding time up to 2 hours for all the used temperatures of solution treatment. After this first peak Brinell hardness is decreased for all the temperatures of solution treat-
ments. On the curve by temperature 545 °C of solution treatment two maximums can be seen. The second maximum can be seen after the holding time 16 hours.

For the samples which were heat treated by temperature 525 °C and 565 °C, the second maximum can not be seen (the Brinell hardness is only decreased). The highest Brinell hardness was 110 HBW for the temperature of solution treatment 545 °C with the holding time 2 hours.

The samples were subjected for tensile and impact tests in order to investigate all changes of mechanical properties during solution treatment. The results are shown in Figs. 3 and 5.

Fig. 3 shows the influence of heat treatment on strength tensile. It can be found out that by the temperature 525 °C strength tensile is increased for all holding times of solution treatment besides 32 hours. By the temperature 545 °C the first maximum can be seen after the holding time 2 hours, then strength tensile goes down a little. On this temperature the second maximum can be seen after the holding time 16 hours after that the strength tensile decreases a little.

After the solution treatment at the temperature 565 °C can be seen that strength tensile is lesser than strength tensile in as-cast. While at as-cast the strength tensile was 177 MPa, after the solution treatment at 565 °C with the holding time from 2 to 8 it ranged from 103 to 110 MPa. The decrease in strength tensile by this temperature is probably related to a high temperature of the solution treatment; because the temperature of eutectic reaction of Al-Si alloys is 577 °C. The results of strength tensile on the samples that were heat treated by the temperature 565 °C with the holding time 16 and 32 hours was immeasurable, because this temperature led to distortion of the testing samples (Fig. 4a).

In Fig. 3 it can be seen that strength tensile is increased during the temperatures 525 °C and 545 °C of the solution treatment. The highest strength tensile was 257 MPa for the temperature of solution treatment 545 °C with the holding time 16 hours.

By the temperature 565 °C can be seen that impact strength is increased only after the holding time from 2 hours, then the impact strength is decreased. The decrease of impact strength by this temperature is probably related to a high temperature of the solution treatment, which causes destruction of the testing samples (Fig. 4b) as by samples of strength tensile. The highest impact strength was 33 J for the temperature of solution treatment 545 °C with the holding time 32 hours.
The mechanical properties of cast components are determined largely by the shape and distribution of Si particles in α-matrix. The 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.

3.2 Microstructural control

The samples were subjected for metallographic study after the mechanical test. The microstructure evolution was carried out by using a light microscope and scanning electron microscope.

Eutectic and primary Si was studied on a light microscope, because mechanical properties of cast component are determined largely by the shape and distribution of Si particles in the matrix. Si particles represent a large volume fraction of the eutectic alloy’s microstructure and, therefore, are very important to affect their morphology. Small, spherical and evenly distributed particles provide the optimum tensile, impact, and fatigue properties of aluminium material [15, 17].

Eutectic Si and minimum primary Si particles were observed in the structure of experimental material (Fig. 6). Secondary AlSi12Cu1Fe cast alloy was not modified or grain refined and so eutectic Si particles without heat treatment (as-cast state) are in a form of large hexagonal platelets (Fig. 6b), which are in the form of needles on a scratch pattern (Fig. 6a). Primary Si particles without heat treatment are in the form of prism (Fig. 6c) that are in the form of polygon with sharp ends on a scratch pattern (Fig. 6a). This morphology of Si particles is not good because these particles are brittle and can crack exposing the soft Al matrix. Therefore, the experimental cast samples were heat treated.

The kinetics of Si morphology transformation is influenced by the solution treatment [23]. The effect of solution treatment on morphology of eutectic Si, for all the temperatures and holding times of solution treatment, is demonstrated in Fig. 7. After the solution treatment at the temperature of 525 °C it was noted that the platelets of eutectic Si were fragmentized into smaller round needles (Fig. 7a). The temperature 525 °C is low for Si-spheroidization.

The spheroidization process was dominated by the temperature 545 °C of solution treatment. Eutectic Si needles (in as-cast state)
are fragmented into smaller segments (at 545 °C) and these smaller Si particles were spheroidized to a rounded shape by the holding time 4 hours (Fig. 7b). After the holding time 16 and 32 hours, there is a change of morphology of eutectic Si particles, these spheroidized particles coarsen little by little (Fig. 7b). The solution treatment at the temperature 565 °C caused changes in morphology of eutectic Si particles, too. Eutectic Si particles gradually coarsen (Fig. 7c) and, therefore, this temperature of solution treatment (565 °C) is not convenient for heat treatment of experimental material. After the solution treatment we could observe that the primary Si particles rounded and reduced their size after all the temperatures and holding times of solution treatment – Figs. 7a, 7b and 7c.

3.3 Quantitative assesment of Si particles after solution treatment

The quantitative analysis was used to quantify the change of silicon morphology (size) in the microstructure during heat treatment. Quantitative analysis [24–26] was carried out on an Image Analyzer to quantify Si (average area Si particle and shape factor) by magnification 250 and 500 x. Fig. 8 shows the average area of Si particles obtained in the solution heat treated samples. This graphic relation is in line with the work of Paray and Gruzleski [23]. The average area of Si particles is decreased with increasing holding time of solution temperatures to 4 hours, after this holding time average area of Si particles is increased. The minimum value of average Si particles was observed by the temperature 545 °C with the holding time 4 hours (33 μm²). It’s probably caused by spheroidization of silicon on this temperature.

Fig. 9 shows the changes in the average area of primary Si particles during the solution treatment. The minimum average area of primary Si particles was observed after the solution treatment by the temperature 525 °C with the holding time 16 hours (147 μm²). By the increasing of the solution temperature the average area of primary Si particles was from 154 to 370 μm². After the solution treatment 565 °C the average area of primary Si was higher in comparison with other temperature of solution treatment because this temperature is not convenient for the heat treatment of experimental material.

4. Conclusions

In the present study, the effects of time and temperature of the solution heat treatment on mechanical properties in secondary eutectic AlSi12Cu1Fe cast alloy were investigated. These alloys are used for automotive applications and, therefore, their mechanical properties are very important. The results are summarized as follows:

- The mechanical properties are highly influenced by the temperature of solution treatment. Recycled AlSi12Cu1Fe cast alloy has in as-cast 83 HBW, $R_m = 177$ MPa and impact strength 3 J. Evaluation of mechanical properties has shown that the highest
mechanical properties are at the temperature 545 °C: Brinell hardness is after the holding time 2 hours 110 HBW, the strength tensile is after the holding time 16 hours 257 MPa and the impact strength is after the holding time 32 hours 33 J.

- Evaluation of microstructure, especially Si particles has shown that eutectic Si particles have spheroidized on the form of perfectly rounded grain at the temperature 545 °C with the holding time 4 hours. The temperature 525 °C was too low for spheroidization and temperature 565 °C was too high. Primary Si particles after the solution treatment, for all temperatures of treatment, rounded and reduced their size.
- Quantitative assessment has shown that minimum average area of Si particles was after the solution treatment by the temperature 545 °C with the holding time 4 hours. By this temperature the average area was from 33 to 127 μm².
- On the basis of the evaluation of mechanical properties and microstructure of AlSi12Cu1Fe cast alloy can say that the optimal mode of solution heat treatment for this secondary aluminium cast alloy is the solution treatment by the temperature 545 °C with the holding time up to 4 hours because it most improves mechanical properties thanks to spheroidization of eutectic Si to rounded shape and the values of tensile strength (250 MPa), Brinell hardness (cca 103 HB) and impact strength (24 J) are comparable with mechanical properties of heat treated primary cast alloy (Rm = 240 MPa; 70 HB).
- The solution treatment at 565 °C has caused distortion of the testing samples, local melting process and is not applicable for the secondary alloy with 12.3 % Si.

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References
[1] WANG, E. R., HUI, X. D., WANG, S. S., ZHAO, Y. F., CHEN, G. L.: Improved Mechanical Properties in cast Al-Si Alloys by Combined Alloying of Fe and Cu. Materials Science and Engineering A 527, 2010, pp. 7878–7884.
[2] MA, Z., SAMUEL, E., MOHAMED, A. M. A., SAMUEL, A. M., SAMUEL, F. H., DOTY, H. W.: Influence of Aging Treatments and Alloying Additives on the Hardness of Al-11Si-2.5Cu-Mg Alloys. Materials and Design 31, 2010, pp. 3791–3803.
[3] MILLER, W. S., ZHUANG, L., BOTTEMA, J., WITTEBROOD, A. J., SMET, P. DE, HASZLER, A., VIEREGGE, A.: Recent Development in Aluminium Alloys for the Automotive Industry. Materials Science and Engineering, A280, 2000, pp. 37–49.
[4] WITTHAYA, E., VIBOONPUN, N.: Effect of Solution Treatment Time on Microstructure and Hardness of Al-Si-Cu-Ni Alloy, from http://www2.mtec.or.th/th/seminar/Msativ/pdf/H01.pdf
[5] SENCAKOVA, L., VIRCIKOVA, E.: Life Cycle Assessment of Primary Aluminium Production. Acta Metallurgica Slovaca, 13, 3, 2007, pp. 412–419.
[6] DAS, K. S.: Designing Aluminium Alloys for a Recycling Friendly World. Materials Science Forum, vol. 519-521, 2006, pp. 1239–1244.
[7] DAS, K. S., GREEN, J. A. S.: Aluminum Industry and Climate Change-Assessment and Responses. Jom, 62, 2, 2010, pp. 27–31.
[8] HURTALOVA, L., TILLOVA, E., CHALUPOVA, M.: Optical and Electron Microscopy Study of the Mechanical Properties Improvement on Recycled AlSi9Cu3 Cast Alloy Along the Hardening. Intern. Virtual Journal for Science, Technique and Innovations for the Industry, MTM, vol. 7, 2011, pp. 48–51.
[9] WANG, E. R., HUI, X. D., CHEN, G. L.: Eutectic Al-Si-Cu-Fe-Mn Alloys with Enhanced Mechanical Properties at Room and Elevated Temperature. Materials and Design 32, 2011, pp. 4333–4340.
[10] TILLOVA, E., CHALUPOVA, M.: Strukturna analýza (Structural Analysis). Edis Zilina, 2009.
[11] TAVITAS-MEDRANO, J. F., MOHAMED, A. M., GRUZLESKI, E. J., SAMUEL, H. F., DOTY, W. H.: Precipitation - hardening in Cast Al-Si-Cu-Mg Alloys. J. of Materials Science, 2009, vol. 45, No. 3, pp. 641–651.
[12] WARMUZEK, M.: Aluminium/Silicon Alloys: Atlas of Microfractographs. Introduction to Aluminium – Silicon Casting Alloys, 2004.
[13] MOUSTAFA, M. A., SAMUEL, F. H., DOTY, W. H.: Effect of Solution Heat Treatment and Additives on the Microstructure of Al-Si (A413.1) Automotive Alloys. J. Materials Sci., (2003), 38 (22), pp. 4507–4522.
[14] SAMUEL, A. M., SAMUEL, F. H., DOTY, W. H.: Observations on the Formation of 13-AlFeSi Phase in 319 Type Al-Si Alloys. J. Materials Sci. 31, 1996, pp. 5529–5539.
[15] SJOLANDER, E., SEIFEDDINE, S.: The Heat Treatment of Al-Si-Cu-Mg Casting Alloys. J. of Materials Processing Technology, 2010, pp. 249–1259.
[16] TASH, M., SAMUEL, H. F., MUCCIARDI, F., DOTY, W. H.: Effect of Metallurgical Parameters on the Hardness and Microstructural Characterization of As-cast and Heat-Treated 356 and 319 Aluminium Alloys. Materials Science and Engineering, A443, 2007, pp. 185–201.
[17] ABDULWAHAB, M.: Studies of the Mechanical Properties of Age-hardened Al-Si-Fe-Mn Alloy. Australian J. of Basic and Applied Sciences 2 (4), 2008, pp. 839–843.
[18] JOHANSEN, H. G.: Structural Aluminium Materials. TALAT Lecture 2202 – Basic Level, 1994, pp. 2–28.
[19] TRENTA, G.: Solutions for Ductile Die Casting, from http://foundry-planet.com/fileadmin/redakteur/verwespartner/sag/SolutionsDuctileDieCasting.pdf, 07. 05. 2012.
[20] TILLOVA, E., CHALUPOVA, M., HURTALOVA, L., BONEK, M., DOBRZANSKI, L. A.: Structural Analysis of Heat Treated Automotive Cast Alloy. *J. of Achievements in Materials and Manufacturing Engineering*, 47/1, (2011), pp. 19–25.

[21] TILLOVA, E., CHALUPOVA, M., HURTALOVA, L.: Evolution of Phases in a Recycled Al-Si Cast Alloy During Solution Treatment. *The Scanning Electron Microscope*, 2011, pp. 411–438, INTECH.

[22] SKOCOVSKY, P.: *Colour Contrast in Metallographic Microscopy*. Slovmetal, Zilina 1993.

[23] PARAY, F., GRUZLESKY, J. E.: Microstructure-mechanical Property Relationships in 356 Alloy. *Cast Metals*, 7, No.1, 1994, pp. 29–40.

[24] VASKO, A., BELAN, J.: Applications of Methods of Quantitative Metallography in Materials Engineering. *Intern. J. of Applied Mechanics and Engineering*, vol. 15, No. 2, 2010, pp. 405–410.

[25] BELAN, J.: *Study of Advanced Materials for Aircraft Jet Engines Using Quantitative Metallography*. Recent Advances in Aircraft Technology, IN-TECH - Open Access publisher of Scientific Books and Journals, 2011, pp. 49–74.

[26] VASKO, A.: Influence of Transformation Temperature on Structure and Mechanical Properties of Austempered Ductile Iron. *Acta Metallurgica Slovaca*, vol. 17, No. 1, 2011, pp. 45–50.