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Changes in Structural Characteristics of Hypoeutectic Al-Si Cast Alloy after Age Hardening

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The contribution describes influence of the age-hardening consist of solution treatment at 515 °C with holding time 4 hours, water quenching at 40 °C and artificial aging at different temperature 150 °C, 170 °C and 190 °C with different holding time 2, 4, 8, 16 and 32 hours on mechanical properties (tensile strength and Brinell hardness) and changes in morphology of eutectic Si, Fe-rich and Cu-rich intermetallic phases in secondary (recycled) AlSi9Cu3 cast alloy. A combination of different analytical techniques (light microscopy upon black-white and colour etching, scanning electron microscopy (SEM) upon deep etching and energy dispersive X-ray analysis (EDX)) were therefore been used for the identification of the various phases. Quantitative study of changes in morphology of eutectic Si, Cu-rich and Fe-rich phases was carried out using Image Analyzer software NIS-Elements. Mechanical properties were measured in line with EN ISO. Age-hardening led to changes in microstructure include the spheroidization and coarsening of eutectic silicon, gradual disintegration, shortening and thinning of Fe- rich intermetallic phases, the dissolution of precipitates and the precipitation of finer hardening phase (Al_2Cu) further increase in the hardness and tensile strength in the alloy.

Keywords: Al-Si-Cu cast alloys, solution treatment, age-hardening, Cu-rich phase.

1. INTRODUCTION

The Al-Si alloy usually has other coexisting elements such as copper, magnesium, manganese, zinc, and iron. Some elements are intentionally added into the Al-Si melt to a significant quantity so that the alloy's sensitivity to heat treatment and microstructure is changed. Thus alloying has become an effective method to improve the mechanical properties of Al-Si alloy. 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 [1, 2]. The addition of Mg and Cu alloying elements is mandatory for applications where high strength at high temperature is required and ductility is not of prime importance, e. g. cylinder heads, manifolds and engine blocks. The strengthening of these alloys is mainly due to the precipitation of hardening Mg- and Cu-rich phases, during age-hardening [3].

The presence of additional elements in the Al-Si alloys allows many complex intermetallic phases to form, such as binary phases (e.g. Mg_2Si, Al_2Cu), ternary phases (e.g. β-Al_5FeSi, Al_2CuMg, AlFeMn, $A_{17}Cu_4Ni$ and AlFeNi) and quaternary phases (e. g. cubic α-$Al_{15}(FeMn)_3Si_2$ and $A_{15}Cu_2Mg_8Si_6$), all of which may have some solubility for additional elements [1, 2, 4 – 9].

Today an increasing amount of the aluminium going into producing new aluminium alloy products is coming from recycled products. The increase in recycled metal becoming available is a positive trend, as secondary metal produced from recycled metal requires only about 2.8 kWh/kg of metal produced while primary aluminium production requires about 45 kWh/kg of metal produced.

It is to the aluminium industry's advantage to maximize the amount of recycled metal, for both the energy-savings and the reduction of dependence upon overseas sources. Increasing the use of recycled metal is also quite important from the ecological standpoint, since producing aluminium by recycling creates only about 4 % as much CO_2 as by primary production [10].

For increase strength or improve the ductility of the alloy was heat treatment used. The heat treatment of the Al-Si alloy normally involves the precipitation of the hard secondary particles from the matrix to produce precipitate strengthening of the matrix [11].

A typical heat treatment applied to sand and gravity die-cast Al-Si alloys is the T6 heat treatment – age-hardening. The T6 heat treatment involves the following stages:

1. Solution treatment at a relatively high temperature to dissolve Cu- and Mg-rich particles formed during solidification to achieve a high and homogeneous concentration of the alloying elements in solid solution.
2. Quenching, usually to room temperature, to obtain a supersaturated solid solution of solute atoms and vacancies.
3. Age-hardening, to cause precipitation from the supersaturated solid solution, either at room temperature (natural ageing) or at, an elevated temperature (artificial ageing) [11].

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 present study is a part of larger research project, which was conducted to investigate and to provide a better understanding microstructure quality control of secondary (recycled) Al-Si cast alloy.

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2. EXPERIMENTAL MATERIAL

As an experimental material was used secondary (scrap-based - recycled) hypoeutectic AlSi9Cu3 alloy, that contains 9.4 % Si, 2.4 % Cu, 0.9 % Fe, 0.28 % Mg, 0.24 % Mn, 1.0 % Zn, 0.03 % Sn, 0.09 % Pb, 0.04 % Ti, 0.05 % Ni, 0.04 % Cr. The melt was not modified or 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 cast alloy produced from primary aluminium 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 [12, 13].

The samples (1 cm × 1 cm) for metallographic observations were prepared by standards metallographic procedures (wet ground, polished with diamond pastes, finally polished with commercial fine silica slurry (STRUERS OP-U).

The microstructures of experimental material was studied using an optical microscope Neophot 32 and SEM observation with EDX analysis using scanning electron microscope VEGA LMU II linked to the energy dispersive X-ray spectroscopy (EDX analyser Brucker Quantax). Samples were etched by standard reagent (Dix-Keller and 0.5 % HF) [14 – 16].

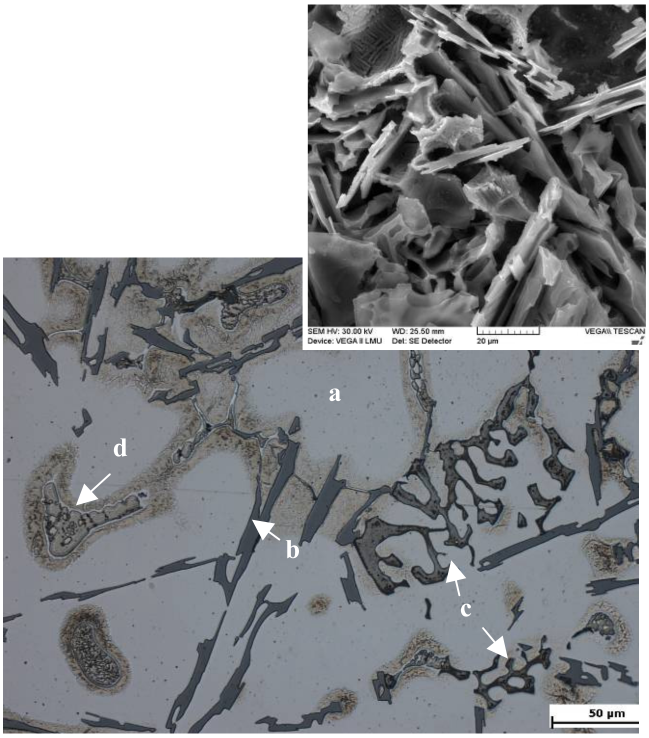


Fig. 1. Microstructure of AlSi9Cu3 cast alloys (as-cast), etch. Dix-Keller: a – α-phase, b – eutectic (mixture of α-matrix and Si-needles), c – Fe-rich intermetallic phases, d – Cu-rich intermetallic phases

Some samples were also deep-etched for 30 s in HCl solution in order to reveal the three-dimensional morphology of the silicon phase – Fig. 1 [14, 15]. 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. The various phases reported in this work were identified using scanning electron microscope VEGA LMU II linked to the energy dispersive X-ray spectroscopy (EDX analyser Brucker Quantax). All phases were analysed by EDX technique.

Experimental cast samples were heat treated by age-hardening. Age-hardening consists of solution treatment at 515 °C with holding time 4 hours, water quenching at 40 °C and artificial aging by different temperature 150 °C, 170 °C and 190 °C with different holding time 2, 4, 8, 16 and 32 hours. After age-hardening were samples subjected for mechanical test (tensile strength and Brinell hardness) and metallographic observations.

Hardness measurement was performed by a Brinell hardness tester with a load of 612.92 N, 2.5 mm diameter ball and a dwell time of 15 s. The hardness value at each state was obtained by an average of at least six measurements.

The phases Vickers microhardness was measured in HTW Dresden using a MHT-1 microhardness tester under a 1 g load for 10 s (HV 0.01). Twenty measurements were taken per sample and the median microhardness was determined.

Structure hypoeutectic AlSi9Cu3 cast alloy consists of dendrites α-phase (light grey), eutectic Si and intermetallic Fe- and Cu-rich (Fig. 1) phases [17].

3. RESULTS AND DISCUSSION

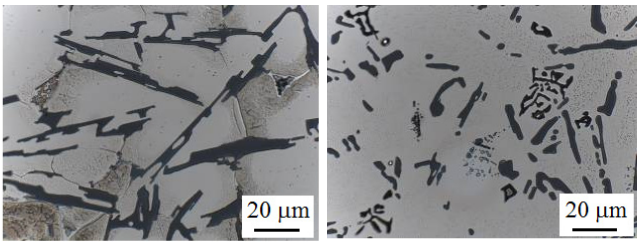
3.1. Effect of age-hardening to structural parameters

Structural parameters in Al-Si are more important for study, because mechanical properties are principally controlled by the cast structure. Therefore, for a given composition, it is the ability to optimize structural factors. A number of factors define the metallurgical structure in Al-Si castings and that determines the properties. Of primary importance are dendrite cell size or dendrite arm spacing, the form and distribution of microstructural phases (eutectic structure, the size and distribution of intermetallics) and grain size.

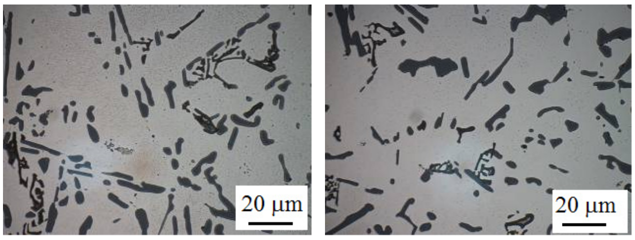
The mechanical properties of cast component are determined largely by the shape and distribution of Si particles in the matrix. Optimum tensile, impact and fatigue properties are obtained with small, spherical and evenly distributed particles.

The effect of heat treatment (age-hardening – T6) on morphology of eutectic Si is demonstrated in Fig. 2. The morphology changes of eutectic Si observed after age-hardening are documented for holding time 16 hours. Eutectic Si without heat treatment (untreated as-cast state) occurs in platelets form (Fig. 2, a). After heat treatment by all temperature of artificial aging were noted that the Si platelets were spheroidized to rounded shape (Fig. 2, b–d).

Quantitative metallography was carried out on an Image Analyzer NIS – Elements to quantify eutectic Si at magnification ×500.



a) untreated state b) T6 – 150 °C, 16 hours



c) T6 – 170 °C, 16 hours d) T6 – 190 °C, 16 hours

Fig. 2. Effect of heat treatment on morphology of eutectic Si, etch. Dix-Keller

Fig. 3 shows the average area of eutectic Si particles obtained in heat treated samples. This graphic relation is in line with work Paray and Gruzleski [18]. Average area of eutectic Si particles decreases with increasing of artificial aging, the most by 170 °C. Minimum value of average eutectic Si particles was observed by temperature of artificial aging 170 °C with holding time 4 hours (41.75 µm^2).

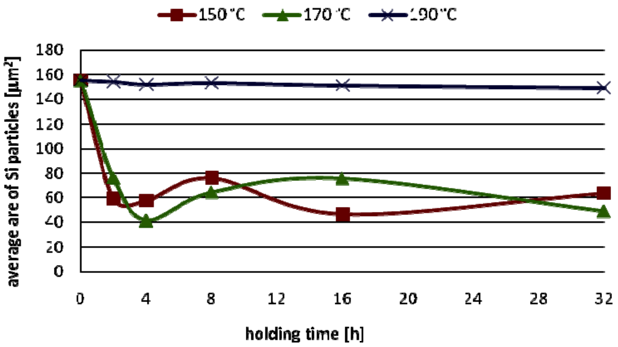
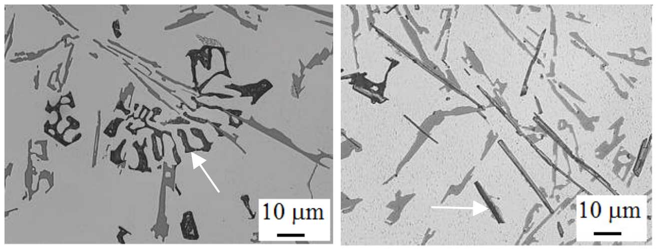


Fig. 3. Influence of age-hardening on average area of eutectic Si particles

After age-hardening and mechanical test were Fe-rich intermetallic phases studied. Fe-rich phases are like Si completely brittle [17]. It suggests that the intermetallic phases may act as stress raisers and crack initiation sites that reduce the strength and ductility of the recycled Al-Si-Cu alloys due to the lack of active slip systems in the intermetallic compounds.

The dominant Fe-rich phase is plate-shaped Al_5FeSi. Long Al_5FeSi platelets (more than 500 µm) can adversely affect mechanical properties, especially ductility, and also lead to the formation of excessive shrinkage porosity defects in castings. Taylor further suggested that the formation of large Al_5FeSi platelets at high Fe-contents facilitates the nucleation of eutectic Si, therefore leading to a rapid deterioration of the interdendritic permeability [6, 17, 19 – 22]. The deleterious effect of Al_5FeSi can be reduced by increasing the cooling rate, superheating the molten metal, or by the addition of a suitable "neutralizer" like Mn, Co, Cr, Ni, V, Mo and Be. The most common addition has been Mn.

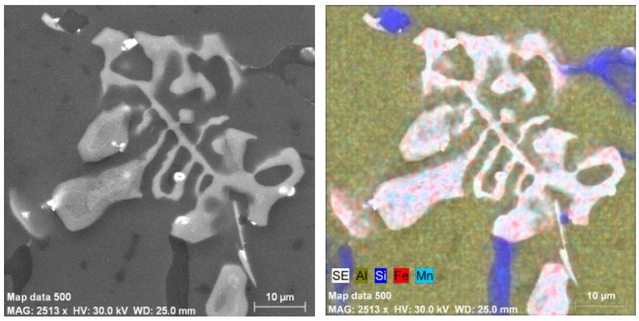
Excess Mn may reduce Al_5FeSi phase (Fig. 4, a) and promote formation Fe-rich phases $Al_{15}(FeMn)_3Si_2$ in form „skeleton-like" or in form „Chinese script" (Fig. 4, b). This compact morphology "Chinese script" (skeleton-like) does not initiate cracks in the cast material to the same extent as Al_5FeSi does.



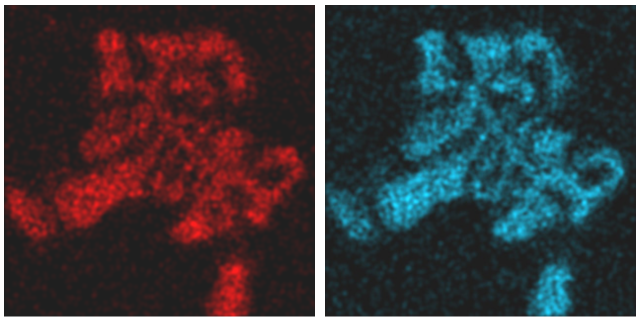
a) $Al_{15}(FeMn)_3Si_2$ b) Al_5FeSi

Fig. 4. Morphology of Fe-rich phases, etch. Dix-Keller

Optical microscopy and SEM observation with EDX analysis (combination of identification chemical data of each phase with mapping) have been combined to produce a simple method for phase identification.



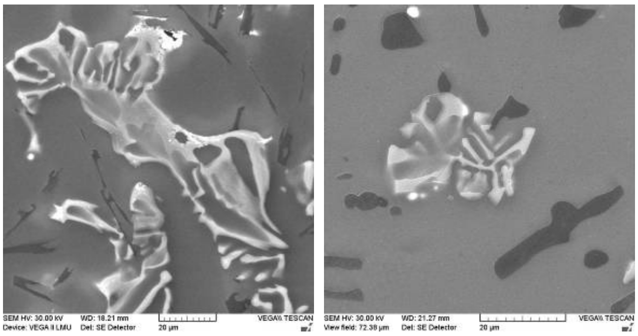
a) etch. 0.5 % HF, SEM b) mapping



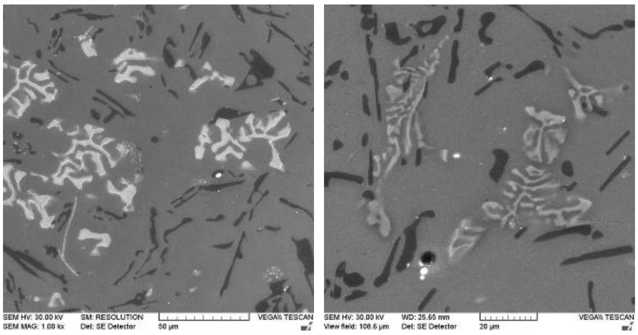
c) Fe – distribution d) Mn – distribution

Fig. 5. EDX phase analysis of $Al_{15}(FeMn)_3Si_2$ phase

X-ray microanalysis of the chemical composition with modern SEM systems is a very useful technique in microstructural analysis of selected microregions of the specimen. Fig. 5 shows typical example: a SEM image and X-ray mapping of the microstructure of skeleton-like Fe-rich phase in secondary AlSi9Cu3 alloy. Fe-phases in experimental secondary AlSi9Cu3 cast alloy precipitate first of all as $Al_{15}(FeMn)_3Si_2$ phase. $Al_{15}(MnFe)_3Si_2$ changes during experimental heat treatment demonstrates Fig. 6. In as-cast samples phase $Al_{15}(MnFe)_3Si_2$ has a compact skeleton-like morphology (Fig. 6, a). During age hardening compact phase dissolved and fragmented to smaller skeleton particles. Fig. 6, b, c, d, shows, that temperature of artificial aging markedly affected the Fe-rich phase's morphology. The effect of age hardening on morphology of Fe-rich phases was followed by optical and electron microscopy.



a) as-cast b) T6 – 150 °C, 16 hours



c) T6 – 170 °C, 16 hours d) T6 – 190 °C, 16 hours

Fig. 6. Morphology changes of skeleton-like Fe-rich phase $Al_{15}(MnFe)_3Si_2$, etch. 0.5 % HF, SEM

Fig. 7 shows the average area of $Al_{15}(FeMn)_3Si_2$ phases obtained in age hardened samples. A maximum value average area Fe-rich phase was observed in as-cast samples (250 μm^2) and by temperature 190 °C. Minimum average area was observed by temperatures 150 °C and 170 °C (51 μm^2).

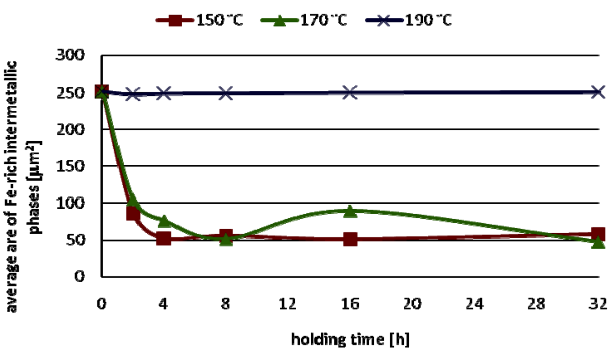
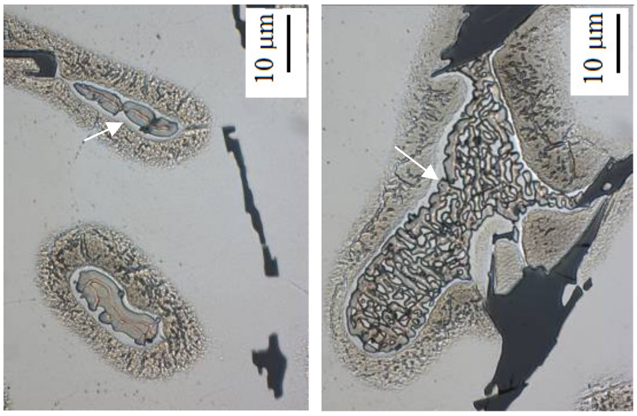


Fig. 7. Changes of average area of Fe-rich phases

Cu is in Al-Si-Cu cast alloys present primarily as phases: Al_2Cu, Al-Al_2Cu-Si or $Al_5Mg_8Cu_2Si_6$ [14, 15, 17]. The average size of the Cu-phase decreases upon Sr modification. The Al_2Cu phase is often observed to precipitate both in a small blocky shape with microhardness 185 HV 0.01. Al-Al_2Cu-Si phase is observed in very fine multi-phase eutectic-like deposits with microhardness 280 HV 0.01 [7, 17].

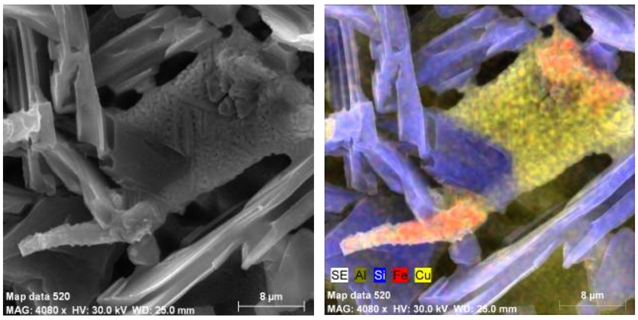
In secondary AlSi9Cu3 alloy was analysed two Cu-rich phases: Al_2Cu (Fig. 8, a) and Al-Al_2Cu-Si (Fig. 8, b).



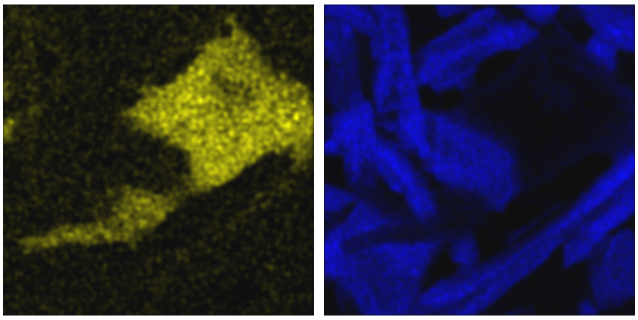
a) Al_2Cu b) Al-Al_2Cu-Si

Fig. 8. Morphology of Cu-rich phases, etch Dix-Keller

Fig. 9 shows typical example: a SEM image and X-ray mapping of the microstructure of Cu phases in deep etched AlSi9Cu3 cast alloy.



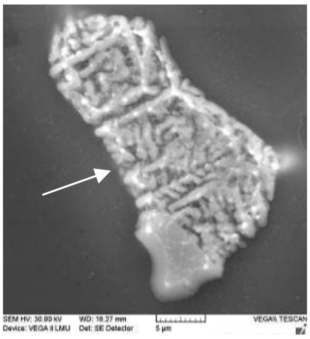
a) deep etch., HCl, SEM b) mapping



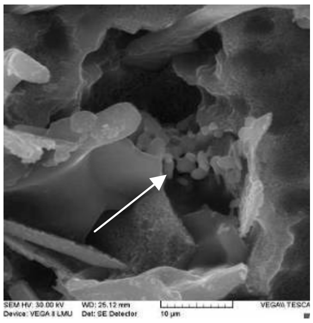
c) Cu – distribution d) Si – distribution

Fig. 9. EDX analysis of Cu-phases in AlSi9Cu3 cast alloy

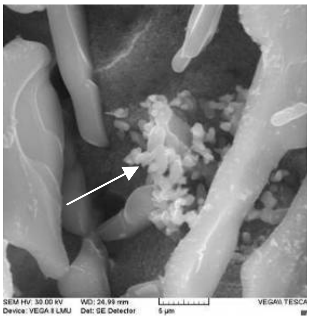
Al-Al_2Cu-Si phase without heat treatment (as-cast state) occurs in form compact oval troops (Fig. 10, a). After age-hardening compact Al-Al_2Cu-Si phase dissolved and disintegrates to separates Al_2Cu particles (Fig. 10, b – d). The amount of these phases was not visible on optical microscope. On SEM microscope we observed these phases in form very small particles for every temperatures of artificial aging (Fig. 10, b – d). By observation we had to use a big extension, because we did not see these elements. Small precipitates (Al_2Cu) incipient by age-hardening were invisible in the optical microscope and electron microscope so it is necessary observation using TEM microscopy.



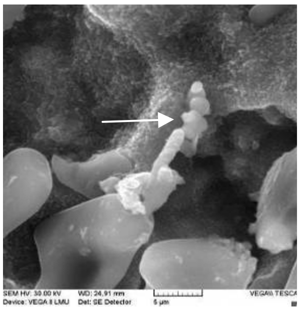
a) as-cast



b) T6 – 150 °C, 16 hours



c) T6 – 170 °C, 16 hours



d) T6 – 190 °C, 16 hours

Fig. 10. Changes of morphology Al-Al_2Cu-Si phase, deep etch. HCl, SEM

Fig. 11 shows the average area of Al-Al_2Cu-Si phases obtained in age-hardened samples. Maximum value average area Fe-rich phases was observed by untreated state and minimum average area was observed by temperature 170 °C with holding time 16 hours (1.53 μm^2).

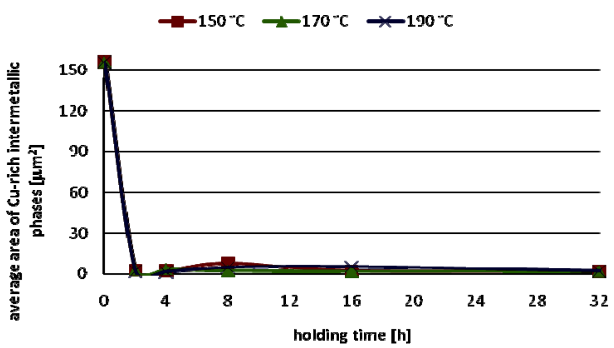


Fig. 11. Changes of average area of Cu-rich phases

3.2. Effect of age-hardening to mechanical properties

Mechanical properties of Al-Si alloys are largely dependent upon an appropriate heat treatment technology, prior to use T6 condition (solution and aging heat treatment), during which a series of changes in microstructure occur. These changes are in amount and the distribution of the precipitates during aging process, which significantly influence the mechanical properties. From this point of view, the age-hardening is critical in determining the final microstructure and mechanical properties of the alloys [23].

After age-hardening were samples subject for hardness measurement. Fig. 12 shows the variation in hardness with aging time 2, 4, 8, 16 and 32 hours at different temperatures. It can be seen that there is an obvious age-hardening phenomenon for each curve. At the early stage of aging for temperature 150 °C the hardness increases with aging time until reaches the first peak (after 4 hours). At intermediate stage of aging, after a little decrease the hardness increases again and reaches the potential second peak after 32 hours. The final stage of aging, when the hardness decreases as a result of over-aging, was not observed.

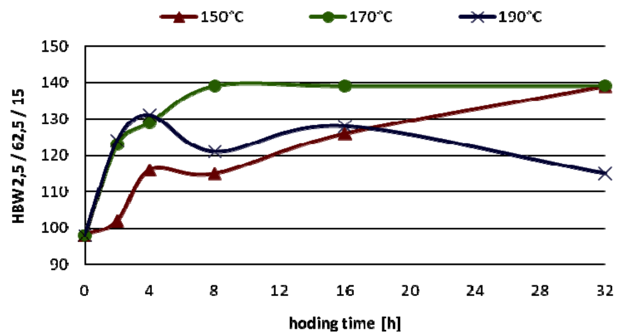


Fig. 12. Influence of age-hardening on Brinell hardness

For samples aged at temperature 170 °C a single aging peak after 8 hours and next a hardness high plateau from 8 to 32 hours was measured. The early stage of aging for temperature 190 °C by 4 hours was measured. The second aging peak for temperature 190 °C by 16 hours was observed. The age-hardening peaks are correlated to their precipitation sequence. The first hardness peak of age-hardening curve is attained depending on the high density GP zones (especially GP II zones), while the second one is acquired in terms of metastable particles. The aging plateau is corresponding to the continuous transition from GP zones to metastable phases.

Highest Brinell hardness was 140 HBS for 515°C/4 hours and artificial aging 170 °C/from 8 to 32 hours.

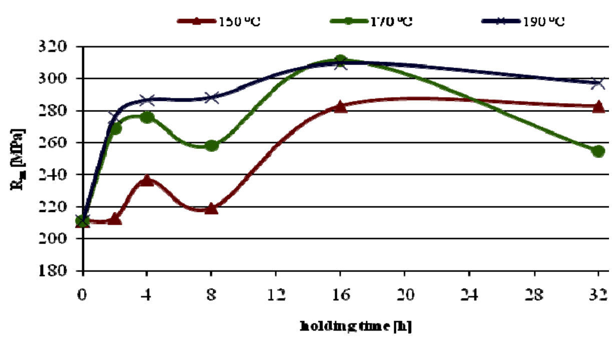


Fig. 13. Influence of age-hardening on strength tensile

In order to investigate the double-peak phenomenon of the AlSi9Cu3 alloy, tensile properties (STN EN 10002-1) of the alloy aged at 150, 170 and 190 °C for different times have been measured. The results are shown in Figure13.

It can be found that the double aging peaks were for temperature 150, 170 and 190 °C measured. The first aging peak we observed for all temperatures after holding time 4 hours. The second aging peak we observed after holding time 16 hours. Highest strength tensile was for artificial aging 170 °C from 16 hours (311 MPa) and lowest was 211 MPa by as-cast.

4. CONCLUSIONS

The present study describes improvement of mechanical properties by structure changes of recycled AlSi9Cu3 cast alloy. Structure changes depend on age-hardening of this experimental material.

The mechanical properties (Brinell hardness and tensile strength) increase after age-hardening for all artificial temperatures. The "optimum" schedule for mechanical properties is as follows: solution treatment: 4 hours at 515 °C; water quenching at 40 °C; artificial aging: 16 hours at 170 °C. This will produce the following properties: HB > 98 (cca 140 HB); R_m > 215 MPa (cca 311 MPa). They are comparable with properties of primary AlSi9Cu3 cast alloy.

The heat treatment caused changes of eutectic Si. The Si-platelets were spheroidized to rounded shape.

The age-hardening caused great changes of Fe-rich phases. Skeleton-like $Al_{15}(FeMn)_3Si_2$ phases are dissolved and fragmented. The value of average area was decreases from 250 μm^2 to 51 μm^2.

Al-Al_2Cu-Si phases are fragmented, dissolved and redistributed within α-matrix The dissolution of Cu-rich phases during hardening holding increases the concentration of Cu and other alloying elements (Mg, Si) in the Al-matrix. Cu also creates dispersed intermetallic precipitates and increases the overall matrix strength by a mechanism called the precipitation strengthening effect. Unfortunately, because these precipitates are very fine, their area fractions cannot be quantified using image analysis combined with light optical microscopy. The value average area of Cu-rich intermetallic phases in optimum age hardening was 1.53 μm^2.

Spheroidization of eutectic Si, fragmentation of Al-Al_2Cu-Si and $Al_{15}(FeMn)_3Si_2$ phases leads to better mechanical properties of recycled AlSi9Cu3 cast alloy.

Acknowledgments

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