

Structural analysis of heat treated automotive cast alloy

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Materials

ABSTRACT

Purpose: The present study is conducted to investigate and to provide a better understanding of the heat treatment T4 (solution treatment at 505, 515 and 525°C, holding time 2, 4, 8, 16 and 32 hours, then quenching in warm water in the range from 40°C and natural aging at room temperature during 24 hours) on the microstructure (morphology of eutectic Si, morphology of intermetallic Fe- and Cu-rich phases) and on mechanical properties (tensile strength and Brinell hardness) of recycled (secondary) AlSi9Cu3 cast alloy.

Design/methodology/approach: Metallographic samples were selected from tensile specimens (after testing) and prepared by standard metallographic procedures (wet ground, polished with diamond pastes, finally polished with commercial fine silica slurry (STRUERS OP-U) and etched by Dix-Keller, HNO₃ or H₂SO₄ (standard etching) or HCl (deep etching in order to reveal the three-dimensional morphology of phases). The microstructure 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). Hardness measurement was performed 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.

Findings: The results indicate that increasing solution treatment temperature results in spheroidization of eutectic Si, gradual disintegration of iron rich intermetallic phases on base Al₁₅(FeMn)₃Si₂, dissolution but also melting of intermetallic phases on base Al-Al₂Cu-Si. Optimal solution treatment (515°C/4 hours) most improves mechanical properties. Further increases of solution time, leads to alloy elongations, while both, the tensile strength continuously drop.

Practical implications: The present study is a part of larger research project, which was conducted to investigate and to provide a better understanding microstructure, heat treatment and mechanical properties of recycled (secondary) Al-Si cast alloy.

Originality/value: The paper contributes to better understanding effect of the T4 heat treatment on the microstructure (morphology of eutectic silicon and intermetallic phases) and mechanical properties (tensile strength and Brinell hardness) of recycled cast alloy.

Keywords: Aluminium; Microstructure; Natural aging; Eutectic silicon; Intermetallic phases

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1. Introduction

Aluminium alloys represent an important category of materials used especially in aerospace and automotive industries in heat treated state. The cast alloys of the Al-Si-Cu system have become increasingly important in recent years, mainly in the automotive industry that uses recycled aluminium in the form of various motor mounts, pistons, cylinder heads, heat exchangers, air conditioners, transmissions housings, wheels, fenders and so on [1]. The increase in recycled metal becoming available is a positive trend, as 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. Increasing the use of recycled metal is also quite important from an ecological standpoint, since producing aluminium by recycling creates only about 5% as much CO₂ as by primary production [2].

Al-Si-Cu systems usually contain a certain amount of Fe, Mn, Mg and Zn that are present either undeliberately, or they are added deliberately to provide special material properties. These elements partly go into solid solution in the matrix and partly form intermetallic particles during solidification [3-5]:

- *Fe-rich intermetallic phases.* The dominant phase is plate-shaped Al₃FeSi (known as beta- or β-needles phase). Al₃FeSi needles are more unwanted, because adversely affect mechanical properties, especially ductility. The deleterious effect of Al₃FeSi 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 Al₃FeSi phase and promote formation of Fe-rich phases Al₁₅(FeMn)₃Si₂ (known as alpha- or α-phase in form „skeleton like“ or in form „Chinese script“). If Mg is also present with Si, an alternative called prior π-phase can form, Al₅Si₆Mg₈Fe₂. Al₅Si₆Mg₈Fe₂ which has script-like morphology [6-11].
- *Cu-rich intermetallic phases.* In Al-Si-Cu alloys various types of copper containing phases were identified: Al₂Cu, Al-Al₂Cu-Si and Al₅Mg₈Cu₂Si₆ [1,3-4,12-21]. In unmodified alloys copper is present primarily as Al₂Cu or Al-Al₂Cu-Si phase, in modified alloys as Al₅Mg₈Cu₂Si₆. The average size of the copper phase decreases upon Sr modification. The Al₂Cu phase is often observed to precipitate both in a small blocky shape with microhardness 185HV0.01. Al-Al₂Cu-Si phase is observed in very fine multi-phase eutectic-like deposits with microhardness 280HV0.01 [11].

Influence of intermetallic phases on mechanical and fatigue properties depends on size, volume and morphology of these phases [6].

The present study is a part of larger research project, which was conducted to investigate and to provide a better understanding of secondary (recycled) AlSi9Cu3 cast alloy after heat treatment. Present work is focused on study of the effect of solution heat treatment on mechanical properties and on changes in morphology of eutectic Si and Cu- and Fe-rich intermetallic phases.

2. Material and experimental

As an experimental material secondary AlSi9Cu3 alloy (in the form of 12.5 kg ingots) was used. The alloy was molten into the

sand form (sand casting). The melting temperature was maintained at 760°C ±5°C. Molten metal was before casting purified with salt AlCu4B6. The melt was not modified or grain refined. The chemical analysis of AlSi9Cu3 cast alloy was carried out using arc spark spectroscopy. The chemical composition is given in the Table 1.

Table 1.
Chemical composition of secondary AlSi9Cu3 cast alloy

Mass concentration of the element, %							
Si	Cu	Fe	Mn	Mg	Ni	Zn	Ti
10.7	2.4	0.9	0.22	0.27	0.08	1.1	0.03

AlSi9Cu3 cast alloy has lower corrosion resistance and is suitable for high temperature applications (dynamic loaded casts, where are not so big requirements on mechanical properties) - it means to max. 250°C. The sand casting produced from primary aluminium achieves high values of tensile strength ($R_m = 160-250$ MPa), yield stress offset 0.2% ($R_{p0.2} = 100-230$ MPa), however the low ductility limits ($A_5 = 1-2\%$) and Brinell hardness 65-90 HB.

Experimental samples (standard tensile test specimens) were given a T4 heat treatment - solution treatment for 2, 4, 8, 16 or 32 hours at three temperatures (505°C, 515°C and 525°C); water quenching at 40°C and natural aging for 24 hours at room temperature (Fig. 1). After heat treatment samples were subjected to mechanical test. For as cast state, each solution temperature and each aging time, a minimum of five specimens were tested.

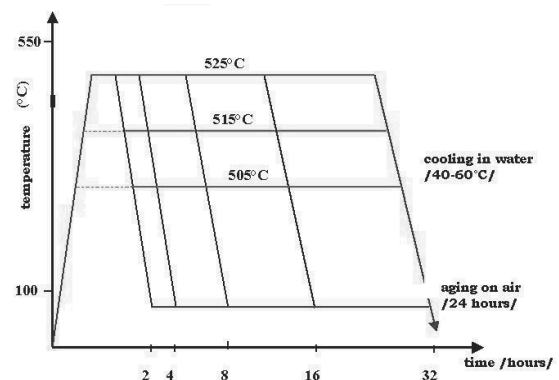


Fig. 1. The schematic diagram of solution treatment process

Metallographic samples were prepared from selected tensile specimens (after testing) and the microstructures were examined by optical and electron microscopy. Samples were prepared by standard 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, HNO₃ or H₂SO₄).

Some samples were also deep-etched for 30 s in HCl solution in order to reveal the three-dimensional morphology of the eutectic silicon and intermetallic phases [4]. 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.

To determine the chemical composition of the intermetallic phases was employed scanning electron microscopy (SEM) TESCAN VEGA LMU with EDX analyser BRUKER QUANTAX. The formation of these phases should correspond to successive reaction during solidification.

Hardness measurement was performed 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 15s. The Brinell hardness value at each state was obtained by an average of at least six measurements.

The phases Vickers micro-hardness was measured in HTW Dresden using a MHT-1 microhardness tester under a 1g load for 10 s (HV0.01). Twenty measurements were taken per sample and the median microhardness was determined.

3. Results and discussion

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 conditions.

Solution treatment performs three roles [22-26]: homogenization of as-cast structure; dissolution of certain intermetallic phases such as Al_2Cu ; changes the morphology of eutectic Si phase by fragmentation, spheroidization and coarsening, thereby improving mechanical properties, particularly ductility. Most of the recommended heat treatment of alloys that contain copper restricts the solution temperature below the final solidification point in order to avoid the melting of copper-rich phases. For experimental work T4 heat treatment consisted of:

- solution treatment for different temperatures: 505°C, 515°C and 525°C, that is necessary to produce a solid solution;
- rapid water quenching (40°C) to retain the maximum concentration of hardening constituent (for us - Al_2Cu) in solid solution;
- natural ageing (24 hours at room temperature) to obtain the desired mechanical properties in the casting, was used.

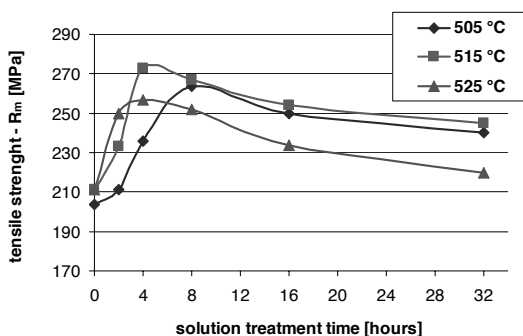


Fig. 2. Influence of solution treatment conditions on tensile strength

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 properties, before moving on to aging process [22].

Influence of solution treatment on mechanical properties (strength tensile - R_m and Brinell hardness - HBS) for experimental recycled AlSi9Cu3 cast alloy casted into the sand form is shown in Fig. 2 and Fig. 3.

After solution treatment, tensile strength, ductility 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 204 MPa. After 2 hours the solution treatment, independently of temperature of solution treatment, strength value immediately increases. By increasing the solution holding time from 2 to 4 hours, the tensile strength increased to 273 MPa for 515°C. With further increase in solution temperature more than 515°C and solution treatment time more than 4 hours, tensile strength decreases during the whole period as a result of gradual coarsening of eutectic Si, increase of inter particle spacing and dissolution of the Al_2Cu phase (at 525°C).

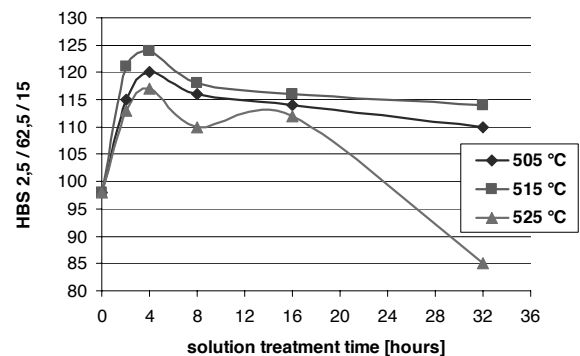


Fig. 3. Influence of solution treatment conditions on hardness

Fig. 3 shows the evolution of Brinell hardness value. Results of hardness are comparable with results of tensile strength. The as cast samples have a hardness value approximately 98 HB. After 2 hours the solution treatment, independently from temperature of solution treatment, hardness value immediately increases. The maximum was observed after 4 hours - approximately 124 HBS for 515°C. However, after 8 hours solution treatment, the HB values are continuously decreasing as results of the coarsening of eutectic silicon, increase of inter particle spacing and dissolution of the Al_2Cu phase. After prolonged solution treatment time up to 16 h at 525°C, it is clearly that the HB values strongly decrease probably due to melting of the Al- Al_2Cu -Si phase.

Obtained results (Fig. 2 and Fig. 3) suggests that to enhance the tensile strength or hardness of this recycled cast alloy by increasing of solution temperature more than 515°C and by extending the solution time more than 4 hours does not seem suitable.

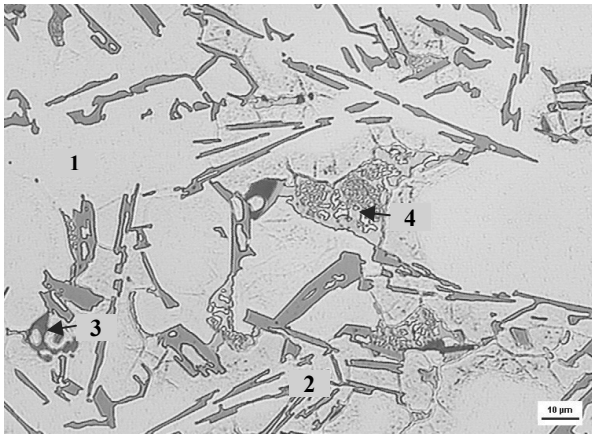
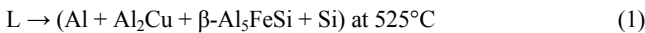


Fig. 4. Microstructure of secondary AlSi9Cu3 cast alloy (1 - α -phase, 2 - eutectic, 3 - Fe-rich phase, 4 - Cu-rich phase) etch. Dix-Keller

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. Microstructure of hypoeutectic AlSi9Cu3 cast alloy consists of dendrites α -phase (light grey), eutectic (dark grey) and intermetallic Fe- and Cu-rich phases (Fig. 4). The formation of these phases should correspond to successive reaction during solidification [3-5].

The effect of solution treatment on morphology of eutectic Si, for holding time 4 hour, is demonstrated in Figs. 5 and 6. Experimental material was not modified and so eutectic Si particles without heat treatment (untreated state) are in a form of platelets (Fig. 6a) [4,27], which on scratch pattern are in a form of needles (Fig. 5a). After solution treatment at the temperature of 505°C were noted that the platelets were fragmented into smaller platelets with spherical edges (Fig. 6b) (on scratch pattern round needles (Fig. 5b)). The spheroidized process dominated at 515°C. The smaller Si particles were spheroidized to rounded shape; see Figs. 5c and 6c. By solution treatment 525°C the spheroidized particles gradually grew larger (coarsening) (Figs. 5d and 6d).

Increasing the level of Cu improves the strength of the aluminium alloy through the formation of Cu based precipitate during heat treatment, the intermetallic phase that Cu form with Al during solidification according to the reaction:



This reaction (1) relates to the start of Al_2Cu precipitation towards the end of solidifications and consequently may be nucleated on other interdendritic particles (Si, Fe-rich phases). The phase Al_2Cu solidifies in two forms: one massive or blocky Al_2Cu particles (with microhardness 185HV0.01) and the other a fine ternary eutectic Al- Al_2Cu -Si (Fig. 7) with microhardness 280HV0.01. Al_2Cu -phases formed which depends on the level of Cu, Fe and Sr in the alloy [1,3-5,11,18-20,26].

Effect of solution treatment on morphology of Al- Al_2Cu -Si is demonstrated on Figure 8. The changes of morphology of Al- Al_2Cu -Si observed after heat treatment are documented for holding time 4 hours. Al- Al_2Cu -Si phase without heat treatment (untreated as cast state) occurs in form of compact oval formation (Fig. 8a).

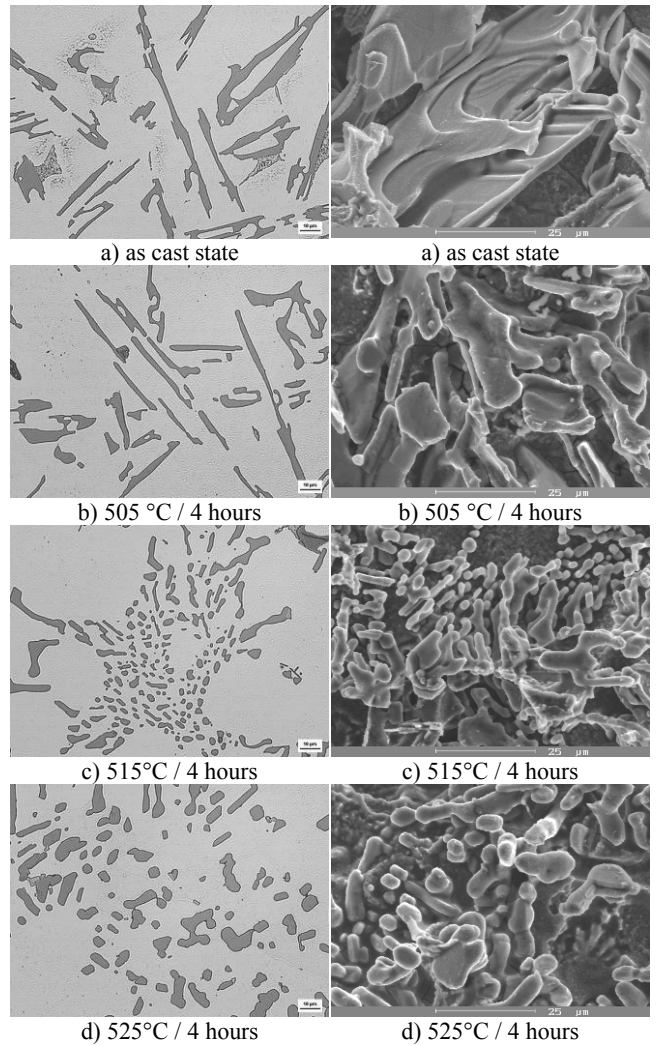


Fig. 5. Effect of solution treatment conditions on morphology of eutectic Si, etch. Dix-Keller

Fig. 6. Morphology of eutectic Si after deep-etching, etch. HCl, SEM

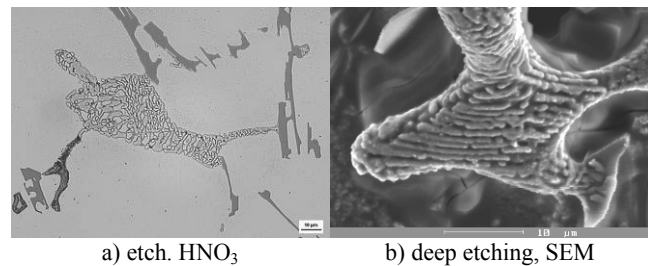


Fig. 7. Morphology of Cu-phase (Al- Al_2Cu -Si)

After solution treatment at temperature 515°C this phase disintegrated into fine smaller Al_2Cu segments and the amount of eutectic copper phase during heat treatment decreases. This phase is gradually dissolved into the surrounding Al-matrix with an

increase in solution treatment time (Fig. 8b). By solution treatment at 515°C is this phase observed in the form of coarsened globular particles and these occurs along the black needles, probably Fe-rich Al_5FeSi phase (Fig. 8c).

Solution treatment at 525°C apparently causes a marked change in morphology of copper eutectic phase. At this temperature, melting of the eutectic copper phase occurs and the molten particles transforming into a shiny structureless phase upon quenching (Fig. 8d).

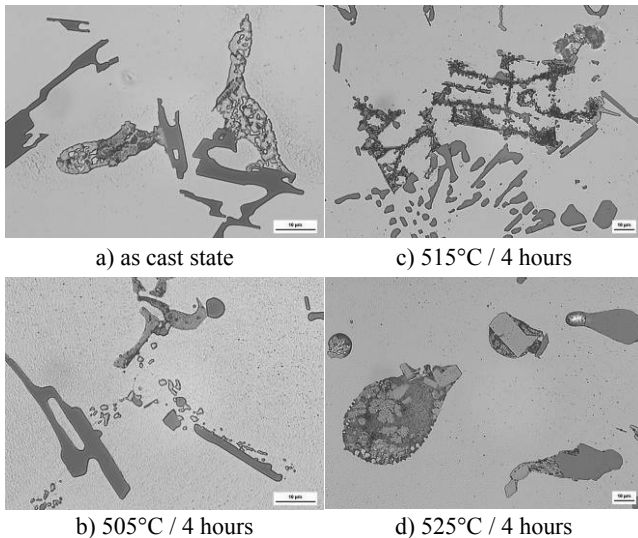


Fig. 8. Effect of solution treatment conditions on morphology of Cu-rich phases, etch. HNO_3

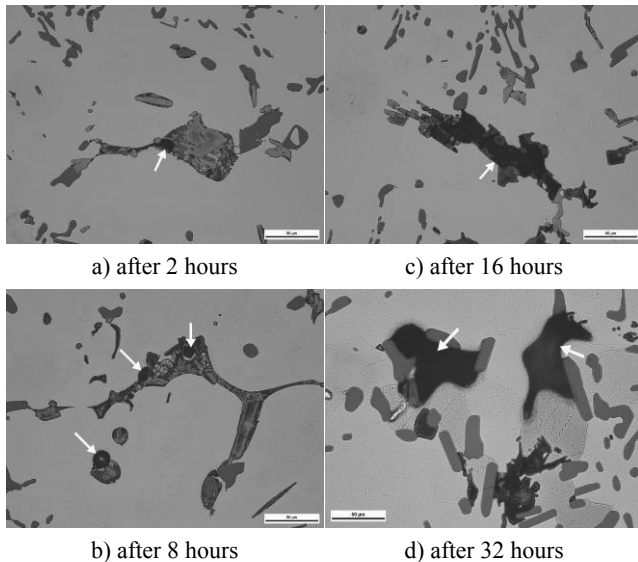


Fig. 9. Incipient melting process of Al- Al_2Cu -Si particles at 525°C, etch. Dix-Keller

Dissolution and melting of Al- Al_2Cu -Si phase in $AlSi9Cu3$ alloy has been studied in detail by Samuel too [14]. During

solution heat treatment, incipient melting of a binary alloy can occur when the alloy composition exceeds the critical composition and the alloy is annealed at a temperature higher than the eutectic temperature. In alloys with segregation of the alloying elements, the composition may locally exceed the critical values, even though that the mean composition is lower, incipient melting occurs in this case [14, 28].

When the $AlSi9Cu3$ alloy is solution treated at temperature about the melting point of the eutectic (Al + Al_2Cu) phase, e.g. 525°C-540°C, the Al- Al_2Cu -Si particles may undergo incipient melting even after periods of 4 hours as evidenced by the black spots observed within the Al- Al_2Cu -Si particles. Microstructure of test bars solution treated at 525°C is documented in Fig. 9. Melting process of Al- Al_2Cu -Si particles has 4 stages: after 2 hours (Fig. 9a) is observed initiation of melting (arrowed →); after 8 hours showing massive Al_2Cu coexisting with Si and Fe-phase (Fig. 9b); next showing structureless phase - note the presence of the previously existing Al_2Cu eutectic at the outer fringes (Fig. 9c); finally showing the presence of cavities and structureless phase particles (Fig. 9d).

In Al-Si cast alloy Fe-rich phases are encountered: $Al_{15}(FeMn)_3Si_2$ phase with cubic crystal structure and Al_5FeSi phase with monoclinic crystal structure. Al_5FeSi phase precipitates in the interdendritic and intergranular regions as platelets (needles). Fe-rich phases are as well as Si completely brittle [1,4]. 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.

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. J.A. Taylor [6] further suggested that the formation of large β platelets at high Fe-contents facilitates the nucleation of eutectic Si, therefore leading to a rapid deterioration of the interdendritic permeability [6-8]. The β platelets appeared to be the main nucleation sites for the eutectic Si, eutectic Al_2Cu and Cu-rich phase. Nucleation of Si and Al_2Cu may occur on large Al_5FeSi platelets.

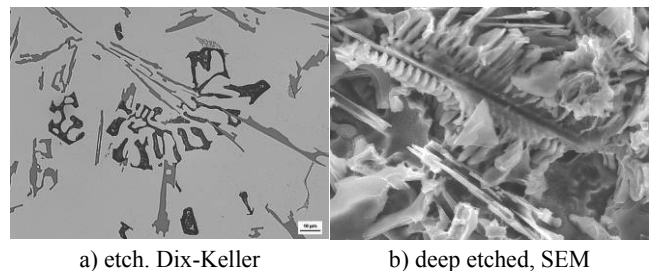


Fig. 10. Morphology of skeleton-like $Al_{15}(MnFe)_3Si_2$ phase

The effect of heat treatment on morphology of Fe-rich phases was followed by optical and electron microscopy. In experimental recycled $AlSi9Cu3$ cast alloy that contains less than 0.9% of Fe and 0.24% Mn only minimal amount of very short Al_5FeSi needles were present. This Fe-rich needles intermetallic phase was after solution treatment shortened and narrowed into very small needle-like phases.

Fe-phases precipitate first of all as skeleton-like phase that are form by Fe, Si along with Mn (Fig. 10). This $Al_{15}(FeMn)_3Si_2$ phase 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_3FeSi .

The effect of solution treatment conditions on the $Al_{15}(FeMn)_3Si_2$ phase for solution time 4 hours is described in Figure 11. In untreated state $Al_{15}(FeMn)_3Si_2$ phases is in form compact skeleton-like (Fig. 10a). Solution treatment of this skeleton-like phase by $505^\circ C$ tends to fragmentation (Fig. 10b) and at $515^\circ C$ or $525^\circ C$ to spheroidization and segmentation (Figs. 10c,d). Solution treatment reduces its area rather than change the morphology.

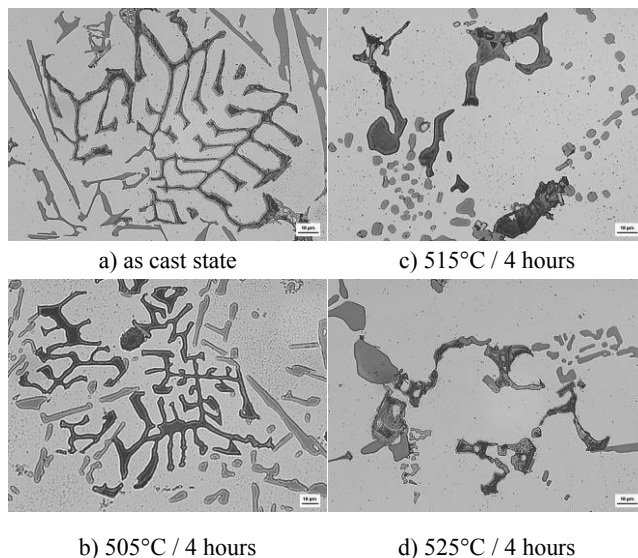


Fig. 11. Effect of solution treatment conditions on morphology of $Al_{15}(MnFe)_3Si_2$ phase, etch. H_2SO_4

4. Conclusions

In the present study, the effects of solution heat treatment on the microstructure (morphology of eutectic silicon, morphology of Cu- and Fe-rich phases) and on mechanical properties (tensile strength and Brinell hardness) of recycled $AlSi9Cu3$ cast alloy for automotive applications 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, be it the coarse silicon plates or the small hardening precipitates. Consequently, the mechanical properties can be related to the duration of the solution heat treatment.
- Optimal condition of solution treatment ($515^\circ C/4$ hours) most improves mechanical properties thanks to spheroidization of eutectic Si to rounded shape. With further increase in solution temperature more than $515^\circ C$ and solution treatment time more than 4 hours, mechanical properties decreases during the whole period as a result of gradual coarsening of eutectic Si, increase of inter particle spacing and dissolution of the Al_2Cu phase.

- $Al-Al_2Cu-Si$ phases are fragmented, dissolved and redistributed within α -matrix. Solution heat treatment at $525^\circ C$ can end in melting process of $Al-Al_2Cu-Si$ particles and finally caused showing the presence of cavities and structureless phase particles. Skeleton - like $Al_{15}(FeMn)_3Si_2$ phases are fragmented and spheroidized.
- 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.

Acknowledgements

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