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RESEARCH ARTICLE

Effect of heat treatment on fracture surfaces in recycled aluminium cast alloy

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Abstract

Recycled AlSi9Cu3 cast alloy was heat treated by solution treatment at temperature 515°C (4 hours), quenched and artificial aged at temperatures 130°C, 170°C and 210°C (2, 4, 8, 16 and 32 hours). Heat treatment led to spheroidization of eutectic Si, gradual disintegration, shortening and thinning of Fe-rich intermetallic phases, the dissolution of Cu-rich phases and the precipitation of finer hardening phase (Al_2Cu). The change of morphology of structural components significantly affects the fracture surface of Al-alloy. The fracture morphology changes gradually from dominant brittle transcrystalline cleavage fracture to ductile fracture with numerous small dimples (locally presence the cleavage fractures of Fe-rich phases).

Keywords

recycled aluminium alloys · fracture surfaces · structural parameters · mechanical properties

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1 Introduction

In recent years are aluminium alloys uses in applications in the industries of aerospace, automotive and even commercial products. In particular, the automotive industry demands both low weight and low cost materials in order to reduce fuel emissions and improve fuel economy at affordable prices (Grayson, G. N. *et al.* [1]). The need for aluminium alloys having a good toughness, high strength, adequate damage tolerance capability, good fatigue resistance and good corrosion resistance for use in industries led to study of the properties and the structure these materials (Srivatsan, T. S. *et al.* [2]). Today an increasing amount of the aluminium going into producing new aluminium alloy products is coming from recycled products (Fig. 1). Nowadays, recycled metal become more available what is a positive trend by all means. 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 (Das, S. K. [3]).

Fig. 1. The examples of the use of recycled AlSi9Cu3 cast alloy - aluminium engine block (www.shelmetcastings.com [4])

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 Al-Si-Cu cast alloy contains a certain amount of Fe, Mn and Mg that are present either accidentally, 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. The size, morphology and volume of intermetallic phases are functions of chemistry, solidification conditions and heat treatment (Rios, C. T. *et al.* [5]; Li, R. [6]; Paray, F. *et al.* [7]). Iron is a common impurity in Al- alloys. Fe-containing intermetallic are formed between the aluminium dendrites (α-matrix). The type of Fe-phase formed depends mainly on the cooling rate and the Fe to Si ratio of the alloy. Fe-rich intermetallic phases can adversely affect mechanical properties, especially ductility, and also lead to the formation of excessive shrinkage porosity defects in castings (Caceres, C. H. *et al.* [8]). Morphology of Fe-rich phases influences harmfully the fatigue properties (Wang, Q. G. *et al.* [9]). The dominant Fe-phase is plate/needle-like Al_5FeSi phase which is very hard and brittle and has relatively low bond strength with the matrix. Al_5FeSi needles are more unwanted, because adversely affect mechanical properties, especially ductility. 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 is Mn. Excess Mn may reduce Al_5FeSi phase and promote formation Fe-rich phase $Al_{15}(FeMn)_3Si_2$ in form of skeleton-like or in form of Chinese script (Rios, C. T. *et al.* [5]; Taylor, J. A., [10]; Seifeddine, S. *et al.*, [11]; Samuel, A. M. *et al.* [12]; Moustafa, M. A. *et al.* [13]). By reaction: Liq. + $Al_5FeSi \rightarrow Al + Si + Al_{15}(FeMn)_3Si_2$ the Al_5FeSi platelets are transformed into the $Al_{15}(FeMn)_3Si_2$ phase that does not appreciably reduce the ductility. In Al-Si-Cu alloys were detected various types of Cu-phases: Al_2Cu, Al-Al_2Cu-Si and $Al_5Mg_8Cu_2Si_6$ (Rios, C. T. *et al.* [5]; Taylor, J. A. [10]). Al_2Cu phase is often observed to precipitate both in a blocky shape and Al-Al_2Cu-Si in fine multi-phase eutectic-like deposits (Martinez D.E.J. *et al.* [14]). Samuel *et al.* [12] reported that these two shapes may be observed in castings cooled at very different rates, with apparently a lower proportion of blocky precipitates as the cooling rate are increased. In unmodified alloys copper is present primarily as Al_2Cu or Al-Al_2Cu-Si phase (Samuel *et al.* [12]; Martinez D. E. J. *et al.* [14]). The morphology of structure components indicates the mould of the fracture surface. The overall appearance of the fracture surface depends not only on the matrix (α-phase), but also on the shape and size of eutectic Si and intermetallic phases. The matrix is characterized by high plasticity while the crystals of eutectic silicon and intermetallic phases have higher values of hardness and almost zero values of plastic properties. Therefore, the fracture surface of Al-Si alloys is creating with ductile fracture of matrix and cleavage fracture of hard and brittle structural components (eutectic Si and intermetallic phases) (Tillová, E. *et al.* [15]).

The present study was conducted to investigate and to provide a better understanding of the influence of heat treatment to changes of morphology of structural parameters and influence these changes on the fracture surface in recycled AlSi9Cu3 cast alloy.

2 Experimental material and procedure

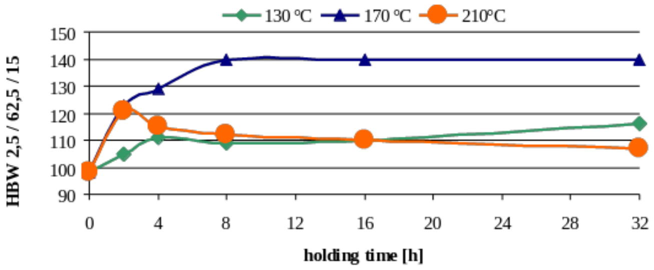
As an experimental material was used recycled (prepared from less clean, so-called metallurgical clean aluminium scrap) hypoeutectic AlSi9Cu3 cast alloy. The AlSi9Cu3 alloy was received in the form of 12.5 kg ingots. Experimental material was cast into the chill (chill casting). The melting temperature was maintained at 760°C ± 5°C. Molten metal was purified with salt $AlCu_4B_6$ before casting and was not modified or grain refined. There semi-products - bars were cast, which were then machined to produce tensile test samples (ø 14 × 20 mm). Chemical composition was carried out by using arc spark spectroscopy and was (wt.%): 10,7Si; 2,4Cu; 0,25Mn; 1Zn; 0,26Mg; 0,9Fe; 0,1Ni, 0,05Ti; 0,02Sn; balance Al. 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.

Experimental cast samples were heat treated in order to affect the phase's morphology. Heat treatment consisted of solution treatment at temperature 515°C; water quenching at 40°C and artificial aging at different temperature 130°C, 170°C and 210°C with different holding time 2, 4, 8, 16 and 32 hours. After heat treatment the samples were subjected to a mechanical test. Hardness measurement was performed by a Brinell hardness tester (HBW 2.5/62.5/15) with the load of 62.5 kp, 2.5 mm diameter ball and the dwell time of 15 s according to standard STN EN ISO 6506. The Brinell hardness value at each state was acquired as average of at least six measurements. The static tensile test was performed for experimental samples in as-cast state and after heat treatment on a tensile machine ZDM 30 at 21°C according to standard STN EN 10003-1. Values of ultimate tensile strength are determined by the average of value of three test bars.

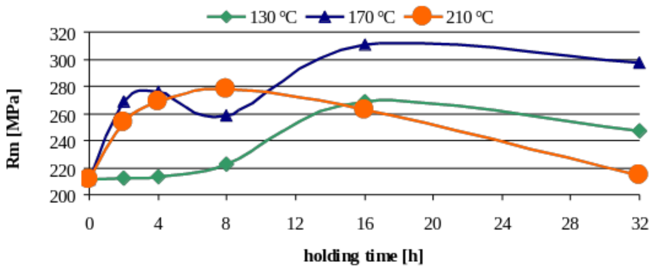
The metallographic samples were cut from the selected tensile specimens (after testing), prepared by standard metallographic procedures and etched by 0.5% HF. Some samples were also deep-etched for 30 s in HCl solution in order to reveal the three-dimensional morphology of the silicon phase or intermetallic phases (Tillová, E. *et al.* [16, 17]). The structures were studied using an optical (Neophot 32) and scanning electron microscope (SEM) VEGA LMU II.

3 Results

Heat treatment led to changes in mechanical properties, especially in hardness (Hurtalová, L. *et al.* [18]; Tillová, E. *et al.* [19]). Fig. 2 (a) shows the variation in hardness with aging time 2, 4, 8, 16 and 32 h. 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 130°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 (above 32 h). The final stage of aging, when the hardness decreases as a result of over-aging, was not observed. For samples aged at temperature 170°C a single aging peak after 8 h. and next a hardness high plateau from 8 to 32 h. was measured. After age-hardening at 210°C were observed effect of over-aging. Highest Brinell hardness was 140 HBW for 515°C/4 hours and artificial aging 170°C/from 8 to 32 hours.



(a) Brinell hardness



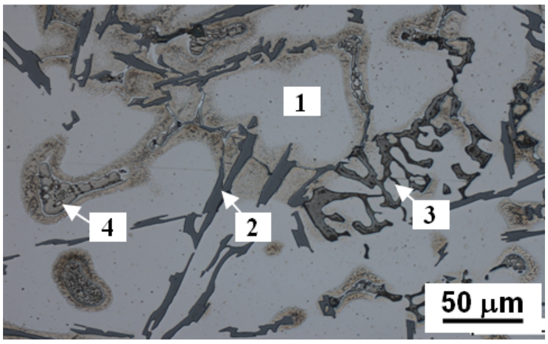
(b) Tensile strength

Fig. 2. The changes of mechanical properties during heat treatment

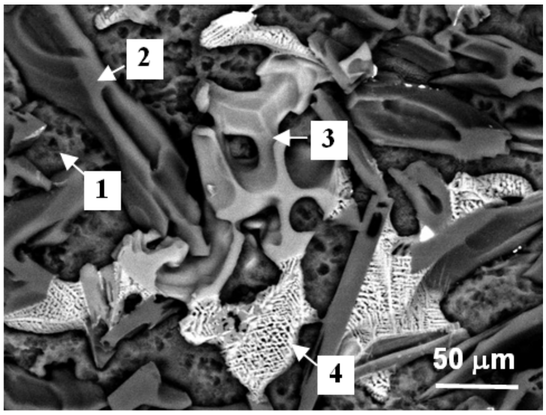
The results of tensile properties are shown in Fig. 2 (b). It can be found that the double aging peaks were only for temperature 170°C measured. The first aging peak after holding time 4 h. and the second after holding time 16 h. were observed. At this aging temperature was highest strength tensile 311 MPa measured. The results of mechanical test determined as optimum heat treatment for experimental material treatment consist of: solution treatment at 515°C with holding time 4 hours, artificial aging at 170°C with holding time 16 hours. The changes of morphology of structure parameters are therefore documented for holding time 16 hours.

The Al-Si eutectic and intermetallic phases form during the final stage of the solidification (Martinez, D. E. J. *et al.* [14]; Tillová, E. *et al.* [15]; Dobrzański, L. A. *et al.* [20]). Typical microstructures of the Al-Si-Cu as-cast alloys are shown in Fig. 3 (a) and 3 (b).

The microstructure (Fig. 3 (a)) of recycled Al-Si-Cu cast alloy consists of dendrites α-phase (1) (light grey), eutectic (2) (mixture of α-matrix and spherical dark grey Si-phases) and various types of intermetallic phases (3), (4). The α-matrix precipitates from the liquid as the primary phase in the form of dendrites and is nominally comprised of Al, Si and Cu. The different intermetallic phases are concentrated mainly in the interdendritic



(a) etch. 0.5% HF



(b) dep etch. HCl, BSE, SEM

Fig. 3. Microstructure of secondary AlSi9Cu3 alloy in as-cast state

spaces. Iron has a very low solid solubility in Al-alloy (maximum 0.05% at equilibrium $Fe_{krit.} \approx 0.075 \times (\%Si) - 0.05$), and most of Fe form a wide variety of Fe-containing intermetallic depending on the alloy composition and its solidification conditions (Taylor, J. A. [10]; Seifeddine, S. [21]). In accordance with Taylor [10], the two main types of Fe-rich intermetallic phases ($Al_{15}(FeMn)_3Si_2$ and Al_5FeSi) in experimental AlSi9Cu3 alloy were observed. The $Al_{15}(MnFe)_3Si_2$ has a compact skeleton-like morphology (Fig. 3 (b)). Al_5FeSi phases precipitates in the interdendritic regions as platelets (needle like form when observed via microscope). Because in experimental material was satisfied condition Fe : Mn = 2 : 1, intermetallic phases in needles form were observed very sporadically. In recycled AlSi9Cu3 alloy was analysed Al-Al_2Cu-Si phases (4) in very fine multi-phase eutectic-like deposits (with microhardness 280 HV 0.01 (Tillová, E. *et al.* [16,17]; Maniara, R. *et al.* [22]).

The effect of age-hardening on morphology of eutectic Si and intermetallic phases is documented in Figures 4 - 7. Eutectic Si ($\rightarrow$ 1) without heat treatment (untreated as-cast state) occurs in platelets form (Fig. 3 (a), 3 (b) and 4). After heat treatment at all temperatures of artificial aging were noted that the Si platelets were spheroidized to rounded shape (Figures 5 and 6).

The $Al_{15}(FeMn)_3Si_2$ phase ($\rightarrow$ 2) has in as-cast state a compact skeleton-like morphology (Fig. 3 (a) and 3 (b)). During heat treatment compact phase dissolved and fragmented to smaller skeleton particles (Fig. 7). The solution treatment and long aging time of artificial aging have significant affect on Fe-rich

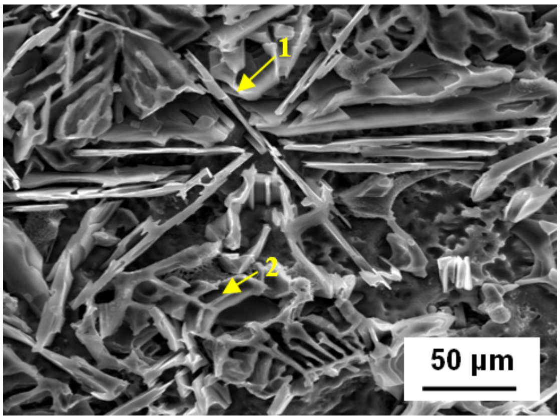


Fig. 4. 3D morphology of structure parameters in as-cast state (1 - Si-particles; 2 - Fe-rich phase)

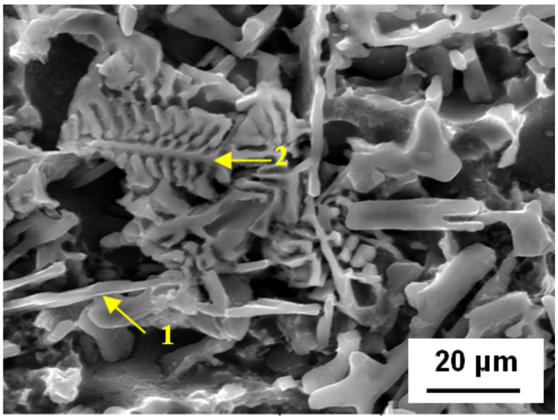


Fig. 5. 3D morphology of structure parameters after heat treatment 515°C / 4h + 130°C / 16h

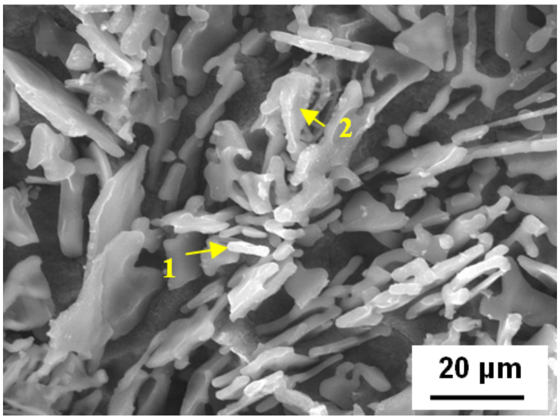


Fig. 6. 3D morphology of structure parameters after heat treatment 515°C / 4h + 170°C / 16h

phase's morphology. Al-Al_2Cu-Si phase without heat treatment (as-cast state) occurs in form of compact oval troops (Fig. 3 (a) and 3 (b)). After age-hardening compact Al-Al_2Cu-Si phase disintegrate to Al_2Cu particles. These phases were observed in form very small particles for every temperatures of artificial aging.

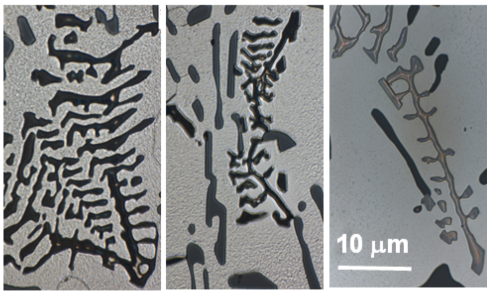
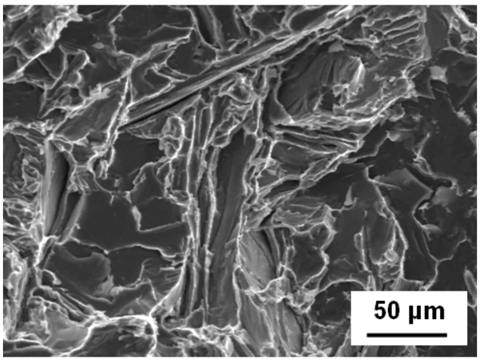


Fig. 7. Morphology of Fe-rich phases after heat treatment

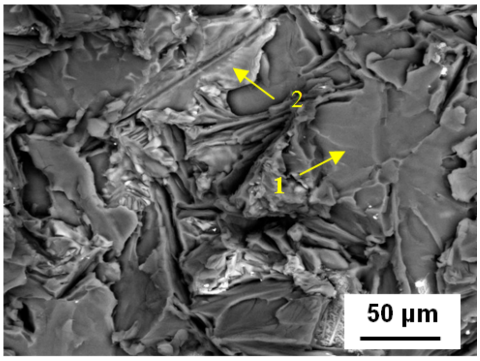
The morphology of fracture surfaces was evaluated by using a scanning electron microscope on the samples after tensile test. The fracture surface was influenced very significantly by structural components (eutectic silicon, intermetallic phases) and their distribution in the cross section. The fracture morphology of AlSi9Cu3 in as-cast state (Fig. 8 (a) and 8 (b)) is cellular. Cellular fracture is typical for material, where the microstructure components have different mechanical properties. On the cellular fracture surface, the features of both brittle and ductile fracture are present simultaneously. The fracture surface of experimental samples consists of transcrystalline cleavage and ductile fracture. Transcrystalline cleavage fracture is dominant in as-cast state. Cleavage fracture is related to the presence of large hexagonal plate-like Si particles and also hard and brittle iron intermetallic phases in the structure of experimental material. The transcrystalline ductile fracture of Al-matrix (α-phase) is observed in the smaller surface, despite the fact that the Al-Si alloys are breaking exclusively by transcrystalline ductile fracture. The brittle Si-particles (in form platelets) are surrounded with a relatively soft matrix and sometimes isolated. Due to the strong cohesion at the interfaces between α-matrix and silicon, the matrix is deformed under local active stress. The cells are formed around the silicon-cracked particles by plastic deformation of the matrix. The traces of these events are visible as the high tear ridges on the fracture surface.

For better identification the fracture of intermetallic phases on fracture surface is need to use the backscattered electrons (BSE) - Fig. 8 (b). 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. The fracture surface of heat treated samples is documented in Figures 9 (a), (b), 10 (a), (b).

The transcrystalline ductile mechanism is dominant for break-



(a) transcrystalline cleavage and ductile fracture



(b) application BSE to highlight the cleavage of eutectic Si and hard Fe-phases

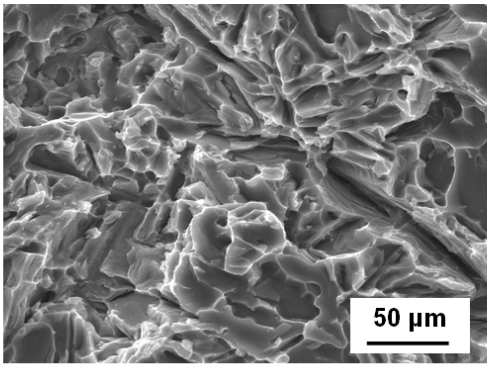
Fig. 8. Character of fracture surface in as-cast state, static tensile test, SEM

ing after age hardening. This mechanism dominates, because silicon particles (→ 1) were spheroidized and Fe- rich intermetallic phases (→ 2) were fragmented and dissolved after heat treatment. The shape and size of the dimples are determined by the size and distribution of eutectic silicon particles (Figures 9 (a), 10 (a)). On the fracture surface was observed the transcrystalline cleavage fracture in a few isolated cases in compare with as-cast state. This type of fracture is related with the appearance of Fe-intermetallic phases in the structure (Fig. 9 (b), 10 (b)). Copper intermetallic phases brakes by transcrystalline ductile mechanism at each state of samples (with and without treatment). Changes were only in size of pitting on fracture surface. The pitting of transcrystalline ductile fracture was grosser in samples in as-cast state as in samples after age-hardening. Age-hardening led to disintegrate of Cu-rich phases and so the pitting of transcrystalline ductile fracture was smaller in compare with as-cast state (without heat treatment).

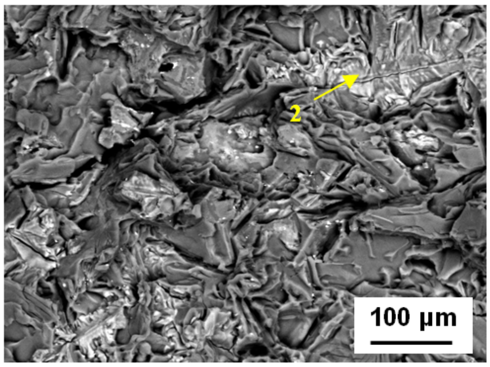
4 Conclusions

In the present study was studied the influence of heat treatment and phase's morphology on the fracture surfaces in recycled aluminium cast alloy with 9% Si and 3% Cu. From the analysis of the results the following conclusions can be drawn:

The age-hardening caused changes of eutectic Si, Fe-rich phases and Cu-rich intermetallic phases. Eutectic Si without heat treatment (in as-cast state) occurs in platelets form. After heat treatment the Si platelets spheroidized to rounded shape.

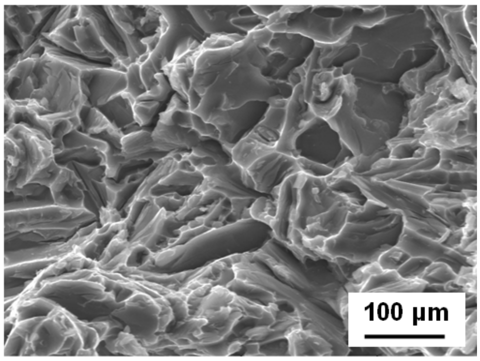


(a) transcrystalline ductile and cleavage fracture

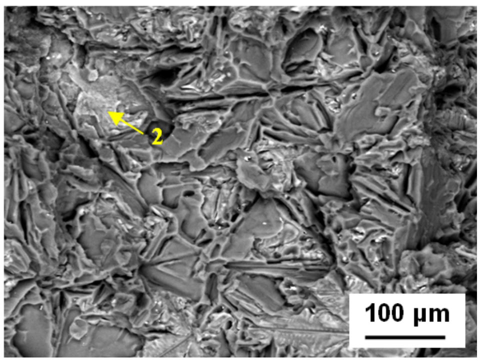


(b) application BSE to highlight the cleavage of eutectic Si and hard Fe-phases

Fig. 9. Character of fracture surface after heat treatment 515°C / 4h + 130°C / 16h, SEM



(a) transcrystalline ductile and cleavage fracture



(b) application BSE to highlight the cleavage of eutectic Si and hard Fe-phases

Fig. 10. Character of fracture surface after heat treatment 515°C / 4h + 170°C / 16h

Skeleton-like $Al_{15}(FeMn)_3Si_2$ phases are dissolved and fragmented. Al-Al_2Cu-Si phases are fragmented, dissolved and redistributed within α-matrix. The dissolution of Cu-rich phases led to increases concentration of Cu and other alloying elements (Mg, Si) in the aluminium matrix during hardening. Cu also creates dispersed intermetallic precipitates and increases the overall matrix strength by a mechanism called the precipitation strengthening effect The change of morphology of structural components significantly affects the fracture surface of Al-alloy. The fracture surface of as-cast state consists of transcrystalline cleavage and ductile fracture. Transcrystalline cleavage fracture is related to the presence of large hexagonal plate-Si particles in the structure and also brittle iron intermetallic phase's dominant. After age-hardening the transcrystalline ductile mechanism is observed. This mechanism dominated, because silicon particles were spheroidized and Fe-rich intermetallic phases were fragmented and dissolved. On the fracture surface was observed the transcrystalline cleavage fracture in a few isolated cases in compare with as-cast state. This type of fracture is related with the appearance of intermetallic phases based on iron in the structure. Copper intermetallic phases are brake by transcrystalline ductile mechanism at each state of samples (with and without treatment).

References

1 **Grayson GN, Schaffer GB, Griffiths JR**, *Observations of oxide films on fatigue fracture surfaces of a sintered 2xxx series Al-alloy*, Materials Science and Engineering: A, **454-455**, (2007), 99–103, DOI 10.1016/j.msea.2006.11.012.

2 **Srivatsan TS, Guruprasad G, Vijay Vesudevan K**, *The quasi static deformation and fracture behavior of aluminum alloy 7150*, Materials and Design, **29**(4), (2008), 742–751, DOI 10.1016/j.matdes.2007.01.006.

3 **Das SK**, *Designing Aluminum Alloys for a Recycling Friendly World*, Materials Science Forum, **519-521**, (2006), accessed 12/11/2012, 1239-1244.

4 `http://www.shelmetcastings.com/aluminum-engine-blocks.html`. (11-12-2012).

5 **Rios CT et al.**, *Intermetallic compounds in the Al-Si-Cu system*, Acta Microscopia, **12**, (2003), 77–82.

6 **Li R**, *Age-hardening behavior of cast Al-Si base alloy*, AFS Transaction, **26**, (1996), 777–783.

7 **Paray F, Gruzleski JE**, *Microstructure – mechanical property relationships in a 356 alloy*: *Part I. Microstructure*, Cast Metals, **7**, (1994), 29–40.

8 **Caceres CH, Svenson IL, Taylor JA**, *Strength-ductility behavior of Al-Si-Cu-Mg casting alloys in T6 Temper*, International Journal of Cast Metals Research, **15**, (2003), 531–543.

9 **Wang QG, Apelian D, Lados DA**, *Fatigue behavior of A356/357 aluminum cast alloys*: *Part II - Effect of microstructural constituents*, Journal of Light Metals, **1**, (2001), 85–97.

10 **Taylor JA**, *The effect of iron in Al-Si casting alloys*, 35th Australian Foundry Institute National Conference, (Adelaide, South Australia, 2004), In: Casting Concepts, Australian Foundry Institute (AFI), 2004, pp. 148–157.

11 **Seifeddine S, Johansson S, Svensson I**, *The influence of cooling rate and manganese content on the β–Al5FeSi phase formation and mechanical properties of Al-Si based alloys*, Materials Science and Engineering A, **490**, (2008), 385–390, DOI 10.1016/j.msea.2008.01.056.

12 **Samuel AM, Samuel FH, Doty HW**, *Observations on the formation of β-Al5FeSi phase in 319 type Al-Si alloys*, Journal of Materials Science, **31**(20), (1996), 5529–5539, DOI 10.1007/BF01159327.

13 **Moustafa MA**, *Effect of iron content on the formation of β-Al5FeSi and porosity in Al–Si eutectic alloys*, Journal of Materials Processing Technology, **209**(1), (2009), 605–610, DOI 10.1016/j.jmatprotec.2008.02.073.

14 **Martínez D. EJ, Cisneros G. MA, Valtierra S, Lacaze J**, *Effect of strontium and cooling rate upon eutectic temperatures of A319 aluminum alloy*, Scripta Materialia, **52**(6), (2005), 439–443, DOI 10.1016/j.scriptamat.2004.11.012.

15 **Tillová E, Panušková M**, *Effect of Solution Treatment on Intermetallic Phases Morphology in AlSi9Cu3 Cast Alloy*, Materials Engineering, **14**, (2007), 73–76.

16 **Tillová E, Chalupová M, Hurtalová L, Bonek M, Dobrzanski LA**, *Structural analysis of heat treated automotive cast alloy*, Journal of Achievements in Materials and Manufacturing Engineering/JAMME, **47**(1), (2011), 19–25.

17 **Tillová E, Chalupová M, Hurtalová L**, *Evolution of phases in a recycled Al-Si cast alloy during solution treatment*: **Kazmiruk V** (ed.), The Scaning Electron Microscope, INTECH, 2011, pp. 411–438.

18 **Hurtalová L, Belan J, Tillová E, Chalupová M**, *Changes in structural characteristics of Hypoeutectic Al-Si cast alloy after age hardening*, Medžiagotyra, **18**(3), (2012), 228–233.

19 **Tillová E, Chalupová M, Hurtalová L, Ďuriníková E**, *Quality control of microstructure in recycled Al-Si cast alloys*, Manufacturing Technology, **11**, (2011), 70–76.

20 **Dobrzański LA, Borek W, Maniara R**, *Influence of the crystallization condition on Al-Si-Cu casting alloys structure*, Journal of Achievements in Materials and Manufacturing Engineering, **18**(41276), (2006), 211–214.

21 **Seifeddine S**, *The influence of Fe on the microstructure and mechanical properties of cast Al-Si alloys*: *Literature review*, Vilmer project. Jönköping University, 2007.

22 **Maniara R, Dobrzański LA, Krupiński M, Sokołowski JH**, *The effect of Cu-concen-tration on the microstructure of Al-Si-Cu alloys*, Archiwes of Foundry engineering, **7**(2), (2007), 119–124.