3D Morphology of Al₅FeSi inclusions in high Fe-content Al-Si-Cu Alloys

I. Bacaicoa*, M. Luetje, M. Wicke, A. Geisert, F. Zeismann, M. Fehlbier, A. Brueckner-Foit
Institute for Materials Engineering, University of Kassel, Moenchebergstrasse 3, D-34109 Kassel, Germany

Abstract

The 3D morphology of the brittle platelet-like β-Al₅FeSi inclusions was analyzed in order to understand the effect of these particles on the degradation of the fracture-resistance of high Fe-content cast Al-Si-Cu alloys. The 3D data of the β-Al₅FeSi compounds was obtained with micro-computed tomography and revealed complex morphology of β-Al₅FeSi inclusions. Tensile tests were conducted and massive β-Al₅FeSi platelets were observed as the main crack initiation sites in the SEM fracture surface observations. The 3D geometry of the largest segmented β-Al₅FeSi phase was exported to a FE software and the maximum stress concentration factors were calculated. After heat treatment, a dissolved morphology of the β-Al₅FeSi inclusions was observed and ductility increased significantly.

Keywords: Al-Si-Cu cast alloys; Fe-rich phase; X-ray tomography; Morphology; Fracture

1. Introduction

The Al-Si-Cu system is one of the most important in the automotive industry due to its excellent castability, corrosion resistance, high specific strength, weldability, low thermal expansion and recycling possibilities [Lee et al. (2003), Moustafa et al. (2009)]

* Corresponding author. Tel.: +49 5618043505
E-mail address: i.bacaicoa@uni-kassel.de
The economical and ecological advantages of recycled aluminum has led to an increasing production trend as the recycling of aluminum alloys requires 95% less energy than primary aluminum, which implies a significant reduction of the CO₂ emission and production costs [Das et al. (2010)]. Nevertheless, the use of the recycled-grade as the base material for structural components is a major challenge, especially when fatigue life is critical. The accumulated iron can only be removed with a very costly process from the melt and high iron concentrations cause the formation of brittle iron rich compounds, from which cracks initiate and lead to premature failure. The most detrimental Fe-rich inclusions are the brittle plate-like β-Al₅FeSi, which have been reported as the main crack initiation sites in high Fe-content Al-Si-Cu alloys [Gao et al. (2004), Yi et al. (2004)].

Consequently, the fracture mechanics assessment of the high iron-content Al-Si-Cu requires the study of the morphology of the Al₅FeSi inclusions as it is essential for the determination of the fracture resistance degradation cause by these phases. High resolution micro-computed tomography (μ-CT) is the major tool in the analysis of the three-dimensional morphology of Fe-rich compounds in order to achieve a better understanding of their influence on the fracture mechanics.

Moreover, the data obtained from μ-CT can be imported into FE software for the analysis of crack initiation for different morphologies of Fe-rich phases and establish quality parameters of this alloy for high structural applications, where fatigue reliability has to be ensured.

The use of μ-CT for the analysis of the three-dimensional morphology of Fe-rich phases also enables to compare the morphology of the β-Al₅FeSi inclusions in the as-cast material and after specific heat treatments. Several studies have suggested non-equilibrium T6 heat treatments in order to avoid the detrimental effect of high iron concentration in the Al-Si-Cu alloys [Narayanan et al. (1995)]. In previous studies, fragmented and dissolved β-Al₅FeSi particles were observed after specific T6 heat treatments and tensile properties were significantly improved.

In this work, the 3D morphology of the β-Al₅FeSiFe has been analyzed with a view to determining the suitability of recycled Al-Si-Cu for fracture-critical components.

2. Materials and experiments

Table 1 shows the chemical composition of the Fe-rich near-to-eutectic Al-Si-Cu alloy used in the present work. Al–25%Fe master alloy was used in order to achieve 0.6 weight % of Fe content in the Al-Si-Cu system. The Mn:Fe relationship is 0.28, which can promote partial substitution from β-Al₅FeSi to the less detrimental α-Al₁₅(Fe,Mn)₃Si₂ [Ashtari et al. (2003)], although a significant amount of β-phase is formed.

<table>
<thead>
<tr>
<th>Si</th>
<th>Cu</th>
<th>Mg</th>
<th>Fe</th>
<th>Zn</th>
<th>Mn</th>
<th>Ni</th>
<th>Ti</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>12.96</td>
<td>1.52</td>
<td>0.68</td>
<td>0.6</td>
<td>0.48</td>
<td>0.17</td>
<td>0.05</td>
<td>0.04</td>
<td>rest</td>
</tr>
</tbody>
</table>

The material was sand casted at 760°C ± 5 °C in form of 4 mm thickness sheets in a sodium silicate mold at room temperature, from which normalized specimens for tensile tests and 20 mm long specimens with a square cross section of 4x4 mm for μ-CT analysis were machined out. Four tensile specimens were subjected to heat treatment at solution temperature of 525°C for 4 hours, quenched in water at temperature of 70°C and aged at temperature of 190°C for 11 hours in order to analyze the change of the morphology of β-Al₅FeSi inclusions after heat treatment.

The three-dimensional measurements were carried out with ZEISS Xradia Versa 520 X-ray microscope with an ultrahigh performance X-ray tube at a voltage of 60 KV and 5 W. 995 radiographs were captured with a pixel size of 1.0 µm and an exposure time of 20 s. After image correction filters, the acquired radiographs were reconstructed with “TXM Reconstructor” software. The image analysis was carried out with an advance rendering package Avizo, in which the Al₅FeSi inclusions were manually and automatically segmented with Multi-Thresholding after applying...
Median Filter corrections and data of volume, surface and Feret size of each inclusion was obtained with the Label Analysis option.

Tensile tests were conducted at room temperature in MTS E45 test machine at a strain rate of 1.5 \(10^{-4}\) s\(^{-1}\). A strain-gage extensometer was attached to the specimen for measuring the elongation. The fractured specimens were used for fracture surface analysis, which was carried out using a scanning electron microscope operating at 20 KV.

3. Results and discussion

The typical microstructure of Fe-rich Al-Si-Cu consists of the \(\alpha\)-matrix, eutectic Si particles, Fe-rich intermetallic phases and Cu-intermetallics. In the alloy used for experiments two main Fe-rich intermetallic phases have been observed: The \(\alpha\)-\(\text{Al}_{15}(\text{FeMn})_3\text{Si}_2\) with Chinese script and cubic crystal structure and the \(\beta\)-\(\text{Al}_3\text{FeSi}\) having needle-like appearance and monoclinic crystal structure (see chemical composition in Table 2). The \(\beta\)-phase is brittle and appears as highly faceted platelets, which causes the loss of strength and ductility in the cast alloy.

![Image](image.png)

**Fig. 1.** a) Main inclusions of high Fe-content Al-Si-Cu alloy: 1- \(\alpha\)-\(\text{Al}_{15}(\text{FeMn})_3\text{Si}_2\), 2- \(\beta\)-\(\text{Al}_3\text{FeSi}\), 3- Eutectic Silicon, 4- \(\text{CuAl}_2\); b) Long needle-like \(\beta\)-phases with Cu-rich intermetallics.

In the 2D micrographs very long needle-like \(\beta\)-phases were found with an average length of 150 \(\mu\)m and area fraction of 6%. The eutectic Si particles are present in form of long platelets (or needles on scratch pattern) and it can be observed that \(\text{CuAl}_2\) compounds and some Si particles lie along the \(\beta\)-\(\text{Al}_3\text{FeSi}\) inclusions (Fig. 1).

| Table 2 Chemical compositions of intermetallic phases. |
|---------------------------------|-----------------|-------------|-----|---|-----|
| Intermetallic phases            | Al   | Si  | Fe  | Cu | Mn  |
| \(\text{Al}_5\text{FeSi}\)      | 64.8 | 14.7| 15.5| 0.9| 0.9 |
| \(\text{Al}_{15}(\text{FeMn})_3\text{Si}_2\) | 60.8 | 8.5 | 16.6| 3.6| 8.4 |
| \(\text{CuAl}_2\)               | 75.8 | 0.58| 0.9 | 19 | -   |

3.1 3D Morphology of \(\beta\)-\(\text{Al}_3\text{FeSi}\) inclusions

The Fe-rich \(\beta\)-phases that in 2D micrographs appear as long-needle-like inclusions are seen in 3D as massive platelets. In the data from X-ray tomography 2171 \(\beta\)-\(\text{Al}_3\text{FeSi}\) inclusions were identified with an average volume of
0.0018 mm$^3$ and surface of 0.358 mm$^2$. The typical Feret size of the Fe-rich $\beta$-phases was 449 µm, which represents a considerably larger value than the average length obtained from image analysis of 2D micrographs. The largest Al$_3$FeSi compound presents a Feret size of 1016 µm and volume of 0.00589 mm$^3$.

Fig. 2 shows single $\beta$-Al$_5$FeSi inclusions segmented from µ-CT and Fig. 3 presents a SEM fractograph taken from the fracture surface of a tensile specimen. The brittle nature of the $\beta$-phases and their susceptibility to act as crack initiation sites can be noted as failure occurs by the cleavage fracture of a massive brittle $\beta$-Al$_5$FeSi platelet. Secondary cracks and Cu-rich inclusions are also visible in the vicinity of the $\beta$-phase platelets.

$\beta$-Al$_5$FeSi compounds present often certain curvature or branched morphology with protuberances and holes around their periphery. Fig. 4 shows the 3D geometry of two $\beta$-phases inclusions that cross each other, one of which is clearly curved around 39°, which has found to be approximately the average curvature (including also branched inclusions).

As it can be observed in Fig. 5, some $\beta$-Al$_5$FeSi platelets may even form complex structures with interconnected clusters of branched and crossed inclusions. In some cases, it is difficult to distinguish branching from crossing, as Fe-rich platelets may either slightly cross or smaller platelets may connect with larger platelets. No pattern in the orientation or distribution of the inclusions could be identified, although it can be observed that arrangement of $\beta$-Al$_5$FeSi compounds tend to form cavities in which micro-pores are found enclosed. This suggests that the $\beta$-phase platelets may have caused the formation of micro-porosity by restricting the flow of the liquid melt during the casting process. This is not the case of Cu-rich inclusions, which are seen to lay all around the periphery of the $\beta$-Al$_5$FeSi, as
a reason of which it becomes apparent that β-phase platelets favor the formation of Cu-rich inclusions after precipitating in a pre-eutectic reaction. Fig. 6 shows FE models of Fe-rich inclusions clusters with micro-pores retained in the cavities and Cu-rich compounds laying in contact with a massive β-Al$_5$FeSi phase.

Fig. 4. 3D microtomography of curved and crossing β-Al$_5$FeSi inclusions.

Fig. 5. 3D microtomography of complex β-Al$_5$FeSi inclusions clusters.

Fig. 6. 3D FEM of β-Al$_5$FeSi phases with: a) Micro-pores enclosed in the cavities formed by complex β-Al$_5$FeSi clusters; b) Cu-rich compounds laying around the periphery.
After heat treatment at a solution temperature of 525°C and 4 hours time, subsequent water quench at 70°C and ageing treatment at 190°C for 11 hours, the β-Al5FeSi are found with a dissolved and decomposed morphology, as it can be observed in the SEM fractograph of a heat-treated tensile specimen (see Fig. 7). A mixed fracture mode occurs in which both α-Al dendrites with dimple morphology and transcrystalline fracture of fibrous eutectic Si particles are present. The high magnification of the fractograph gives an idea of the small size of the Fe-rich β-phases and the decomposed form indicates the dissolution process that these phases have undergone. Elongation values increased from 0.48% in the as-cast alloy to 1.7% after heat treatment and a more ductile fracture mode can be noted.

3.2 Stress Distribution

The brittle nature of the at β-Al5FeSi inclusions and their platelet-like morphology generates high local stress/strain concentrations that lead to rapid crack initiation. Therefore, the failure mechanism of cast Al-Si-Cu alloys with high Fe-content are affected by the presence of these brittle β-Al5FeSi compounds and may be also influenced by neighbouring pores and free surfaces.

The largest identified β-Al5FeSi inclusion with a volume of 0.00589 mm³ and Feret size of 1016 µm was segmented and exported to a commercial software (ABAQUS) in order to calculate the local stress concentration factors (K_T). It was inserted in a finite element model of a cylindrical volume representing the alloy material and a quasi-static tensile test was simulated. The element type used for the meshing was the eight-node quadratical tetrahedral element (C3D10) of the ABAQUS code and the β-Al5FeSi particles were assumed to be stiff elastic solids. The Young’s modulus and Poisson’s ratio of the Fe-rich β-phases were 150 GPa and 0.28 [Yi et al. (2004)] respectively, whereas for the volume material a Young’s modulus of 70 GPa and Poisson’s ratio of 0.3 were considered. The stress concentration factor (K_T) was defined as the ratio between the peak von Mises stress and the nominal far-field stress. 512 configurations were analyzed in which the orientation of the β-Al5FeSi particle relative to the three axes of the volume material was gradually changed in order to simulate an arbitrary stress state.

The maximum K_T ranged between 1.74 and 3.46 for the different orientations, with an average value of 2.63. As it can be observed in Fig. 9, the peak stress concentrations were located on specific sites of the edge of the inclusion as the stress distribution varies all over the edge. For some orientations, the protuberances of the inclusions were found to be the sites with maximum stress concentrations (see Fig. 10), which indicates that the complex morphology of the Fe-rich β-phases has a significant influence on the local stress concentrations, and therefore, on the crack initiation process on β-Al5FeSi inclusions.

Conclusions

In this study, the 3D morphology of the β-Al5FeSi phases and their influence on the local stress concentration was analyzed. Bases on the results, the following conclusions may be drawn:

a) The β-Al5FeSi phases that in the 2D micrographs are seen as long needles present a platelet morphology in the 3D reconstructions and they were found to be the main crack initiation sites in the fracture surface observations.

b) The β-Al5FeSi inclusions present often complex morphology with crossing between phases, curved and branched shape, holes and protuberances. Complex interconnected clusters of inclusions with cavities were also found in which micro-pores were enclosed and Cu-rich phases were laying around the periphery.

c) After heat treatment, dissolved morphology of β-Al5FeSi phases and a more ductile fracture mode with a significant increase of elongation was observed in the SEM fractographs.

d) A maximum stress concentration factor (K_T) of 3.46 was obtained in the 3D FE analysis of the largest β-Al5FeSi phase segmented from µ-CT data. For most of the orientations, the peak K_T was located on the edge of the inclusion, although the protuberances of the phase were also high stress concentration sites.
After heat treatment at a solution temperature of 525°C and 4 hours time, subsequent water quench at 70°C and ageing treatment at 190°C for 11 hours, the ß-Al5FeSi are found with a dissolved and decomposed morphology, as it can be observed in the SEM fractograph of a heat-treated tensile specimen (see Fig. 7). A mixed fracture mode occurs in which both α-Al dendrites with dimple morphology and transcrystalline fracture of fibrous eutectic Si particles are present. The high magnification of the fractograph gives an idea of the small size of the Fe-rich ß-phases and the decomposed form indicates the dissolution process that these phases have undergone. Elongation values increased from 0.48% in the as-cast alloy to 1.7% after heat treatment and a more ductile fracture mode can be noted.

Fig. 7. SEM fractograph of a heat treated specimen (525°C-4 h) with dissolved Al5FeSi inclusions.

Fig. 8. Fracture surface of a heat treated tensile specimen with dissolved Al5FeSi inclusions and α-Al dendrites.

3.2 Stress Distribution

The brittle nature of the at ß-Al5FeSi inclusions and their platelike morphology generates high local stress/strain concentrations that lead to rapid crack initiation. Therefore, the failure mechanism of cast Al-Si-Cu alloys with high Fe-content are affected by the presence of these brittle ß-Al5FeSi compounds and may be also influenced by neighbouring pores and free surfaces.

The largest identified ß-Al5FeSi inclusion with a volume of 0.00589 mm³ and Feret size of 1016 µm was segmented and exported to a commercial software (ABAQUS) in order to calculate the local stress concentration factors (KT). It was inserted in a finite element model of a cylindrical volume representing the alloy material and a quasi-static tensile test was simulated. The element type used for the meshing was the eight-node quadratical tetrahedral element (C3D10) of the ABAQUS code and the ß-Al5FeSi particles were assumed to be stiff elastic solids. The Young’s modulus and Poisson’s ratio of the Fe-rich ß-phases were 150 GPa and 0.28 [Yi et al. (2004)] respectively, whereas for the volume material a Young’s modulus of 70 GPa and Poisson’s ratio of 0.3 were considered. The stress concentration factor (KT) was defined as the ratio between the peak von Mises stress and the nominal far-field stress. 512 configurations were analyzed in which the orientation of the ß-Al5FeSi particle relative to the three axis of the volume material was gradually changed in order to simulate an arbitrary stress state.

The maximum KT ranged between 1.74 and 3.46 for the different orientations, with an average value of 2.63. As it can be observed in Fig. 9, the peak stress concentrations were located on specific sites of the edge of the inclusion as the stress distribution varies all over the edge. For some orientations, the protuberances of the inclusions were found to be the sites with maximum stress concentrations (see Fig. 10), which indicates that the complex morphology of the Fe-rich ß-phases has a significant influence on the local stress concentrations, and therefore, on the crack initiation process on ß-Al5FeSi inclusions.

Fig. 9. Finite Element Model with the stress distribution on a ß-Al5FeSi inclusion.

Fig. 10. Finite Element Model with the location of the maximum KT for different orientations.

4. Conclusions

In this study, the 3D morphology of the ß-Al5FeSi phases and their influence on the local stress concentration was analyzed. Bases on the results, the following conclusions may be drawn:

a) The ß-Al5FeSi phases that in the 2D micrographs are seen as long needles present a platelet morphology in the 3D reconstructions and they were found to be the main crack initiation sites in the fracture surface observations.

b) The ß-Al5FeSi inclusions present often complex morphology with crossing between phases, curved and branched shape, holes and protuberances. Complex interconnected clusters of inclusions with cavities were also found in which micro-pores were enclosed and Cu-rich phases were laying around the periphery.

c) After heat treatment, dissolved morphology of ß-Al5FeSi phases and a more ductile fracture mode with a significant increase of elongation was observed in the SEM fractographs.

d) A maximum stress concentration factor (KT) of 3.46 was obtained in the 3D FE analysis of the largest ß-Al5FeSi phase segmented from µ-CT data. For most of the orientations, the peak KT was located on the edge of the inclusion, although the protuberances of the phase were also high stress concentration sites.
5. Acknowledgements

The authors would like to thank the Hessen State Ministry of Higher Education, Research and the Arts - Initiative for the Development of Scientific and Economic Excellence (LOEWE) - for financial support of the special research project “Safer Materials”.

6. References


