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Hall-Petch equation in a hypoeutectic Al-Si cast alloy: grain size vs. secondary dendrite arm spacing

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Abstract

The Al-Si cast alloy family is widely used in the production of complex castings for various applications and known for its very good castability and high strength-to-weight ratio. However, early cracking under tensile loading is sometimes a limiting factor. Among other parameters, it is yet controversial whether grain boundaries are dominant strengthening factor in cast alloys, instead of dendrite/eutectic boundaries. This study presents the effect of secondary dendrite arm spacing (SDAS) and grain size on crack initiation and propagation of Al-Si cast alloys under tensile loading. The Al-10Si (wt.%) alloy with modified Si morphology was cast using inoculants (Al-5Ti-B master alloy) under different cooling rates to obtain a range of grain sizes (from below 138 µm to above 300 µm) and SDAS (6, 15 and 35 µm). Conventional tensile test as well as in-situ tensile test in a scanning electron microscope, equipped with an electron backscatter diffraction (EBSD) was carried out to understand the deformation mechanisms of the alloy. Observation of slip bands within the dendrites showed that in modified Si structure, the interdendritic (eutectic) area takes more portion of the strain during plastic deformation. Besides, only a few cracks were initiated at the grain boundaries; they were mostly initiated from dendrite/eutectic interface. All cracks propagated trans-granularly. Hall-Petch calculations also showed a strong relationship between SDAS and flow stress of the cast alloy. Although statistically correct, there was no physically meaningful relationship between the grain size and the flow stress. Nevertheless, formation of identical slip bands in each grain could be an evidence for the marginal effect of the grain size on the overall strength development of the alloy. Consequently, among other effects, the combinational dominant effect of SDAS and modest effect of grain size shall be considered for modification of the Hall-Petch equation for precise prediction of mechanical properties of cast alloys.

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1. Introduction

According to Hall-Petch equation, mechanical behavior of polycrystalline wrought metals mainly depends on the grain size, as well as their internal friction stress.

\[ \sigma = \sigma_0 + \frac{k}{\sqrt{d}} \]  

(1)

where \( \sigma_0 \) represents the grain interior resistant to deformation, and \( k \) is the strengthening coefficient [1]. According to Eq. 1, during plastic deformation, grain boundaries act as obstacles for dislocation movements and therefore a dislocation transformation (e.g. slip) must take place at the grain boundary to continue plastic deformation. By increasing the misorientation angle between the two adjacent grains, it would be more difficult for dislocations to pass through the grain boundary and reach to the next grain. Consequently, fine-grained metals, which contain higher density of grain boundaries, are generally stronger than coarse-grained metals [1].

Eq. (1) is mostly used for wrought alloys and only considers the effect of grain size, regardless of other microstructural parameters. To further modify the Hall-Petch equation for complex microstructures, Ghassemali et al. [2] have reported that other microstructural features such as subgrains (low-angle grain boundaries) can also affect the mechanical response of the material.

Compared to wrought materials, the microstructure of cast alloys is rather more complicated containing dendritic structure, secondary phases, intermetallics etc. The interactive effect of many of these microstructural features on the mechanical properties of cast alloys have been studied before [3, 4].

It has been proven that the secondary dendrite arm spacing (SDAS) influences the mechanical behavior of cast alloys [5, 6]. Specifically in Al-Si base alloys, besides the SDAS, the morphology of Si phase has an influence on the tensile properties of the material [5, 7]. Nevertheless, regardless of the morphology of the Si phase, grain refinement during solidification (by inoculants) affects the feeding during solidification as well as distribution of secondary phases and shrinkage porosities, which can indirectly affect the mechanical properties [8]. The effect of grain refinement on the overall mechanical properties of Al-Si alloys have been, however, a controversial debate. Birol [9] and Samuel et al. [10] reported that grain refinement during solidification has almost no effect on the tensile properties of as-cast Al-Si alloys, whereas Kori et al. [11] and Basavakumar et al. [12] showed the opposite. The reason for these contradictory reports could be related to different solidification history of the alloy (melt holding time, cooling rates, etc.). Nevertheless, the effect of grain size on the mechanical properties of cast alloys seem to require deeper investigations.

In this paper the effects of SDAS and grain size on crack initiation and propagation of the Al-10(wt.%)Si base alloy (without any alloying element) was investigated using conventional and in-situ tensile test. The final aim is to understand the importance of grain size and SDAS on the tensile behavior of cast alloys. To minimize the number of variables in this study, the morphology of Si was modified using Sr additions, in all investigated specimens.

2. Materials and methods

Pure Al and Si were used to prepare the hypoeutectic (near eutectic) Al-10(wt.%)Si alloy. Around 250 ppm Sr was added to the melt using Al-10Sr master alloy in a rod shape, which provides optimum eutectic (Si) modification [13]. The Al-5Ti-1B master alloy in a rod shape was added to act as the grain refiner, according to previous studies [14]. The melt was prepared in a 6 Kg Nabertherm K4/13 resistance furnace and then was cast into cylindrical bars, made by a copper die coated with graphite, preheated to 250 °C. Table 1 shows the chemical composition of the alloy.
Table 1. Average chemical composition of as-cast samples.

<table>
<thead>
<tr>
<th>Element</th>
<th>Composition</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si (wt.%)</td>
<td>9.05 ± 0.13</td>
</tr>
<tr>
<td>Fe (wt.%)</td>
<td>0.1 ± 0.01</td>
</tr>
<tr>
<td>Ti (ppm)</td>
<td>540 ± 20</td>
</tr>
<tr>
<td>B (ppm)</td>
<td>60 ± 4</td>
</tr>
<tr>
<td>Sr (ppm)</td>
<td>250 ± 9</td>
</tr>
<tr>
<td>Al</td>
<td>Balance</td>
</tr>
</tbody>
</table>

For the conventional tensile tests, cylindrical tensile testing samples were machined from the directionally solidified bars, as per the schematic in Fig. 1a. Conventional tensile tests were performed with a Zwick/Roell Z100 equipment at a 10⁻³ s⁻¹ strain rate. For in-situ tensile tests, the Kammrath & Weiss stage inside a TESCAN LYRA3 SEM was utilized using the EBSD module. The samples were machined into the flat-type down-sized tensile samples, as schematically shown in Fig. 1b. To be able to monitor cracking in the field of view, a notch was made in the middle of the gauge length. Grains were visualized using Inverse Pole Figure (IPF) maps in the EBSD analysis. The grain boundary threshold was considered as 15° misorientation. Grain sizes were measured by the intercept method per ASTM E112-96. The EBSD acquisition was done using the acceleration voltage of 15 KV and step sizes of 1 µm.

3. Results and discussion

3.1. Grain size

The EBSD technique revealed statistically-enough number of grains within a reasonable acquisition time (see a representative area in Fig. 2a-c). The grain size data are presented in Table 2. Previous studies shows that the finest achievable grain size of Al-Si alloys in the as-cast state, using Al-Ti-B master alloys is in the range of around 100 to 180 µm [14], which is in consistent with the presented results. Slower cooling rates beyond those reported here could result in a columnar grain structure or an un-modified Si morphology, which was excluded from this study. The reason for the latter could be due to settlement of inoculant during very slow solidification, which is under investigation.

Fig. 1. Schematics of the samples for a) conventional tensile, and b) in-situ tensile tests. All dimensions in mm.

Fig. 2. Inverse Pole Figure (IPF) map obtained from the EBSD mapping, showing grain structure of samples a) SDAS 36 µm, b) SDAS 15 µm, c) SDAS 6 µm. SD and TD are acronyms for solidification direction and transverse direction, respectively.

Table 2. Grain size and SDAS of the alloy solidified under 3 different cooling rates.

<table>
<thead>
<tr>
<th>Furnace pulling rate (mm. s⁻¹)</th>
<th>0.03</th>
<th>0.3</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>SDAS (µm)</td>
<td>36</td>
<td>15</td>
<td>6</td>
</tr>
<tr>
<td>Grain size (µm)</td>
<td>304 ± 48</td>
<td>200 ± 43</td>
<td>138 ± 25</td>
</tr>
</tbody>
</table>
3.2. Tensile properties

As depicted in Fig. 3, the smaller SDAS (i.e. faster cooling rate) would lead to a higher strength of Al-Si cast alloys, without much drop in the elongation. It is important to note that the Si morphology in all samples was modified. The higher elongation in the larger SDAS can be related to interactive effects of the dendrite size and the incompatibility of eutectic/dendrite, as previously reported by Wang and Cáceres [15].

To further analyze the effect of grain size and SDAS on the mechanical properties of the alloy, the tensile data for each sample was transferred into the Hall-Petch equation, as plotted in Fig. 4. The corresponding results from both SDAS and grain size measurements showed a linear relationship (with R² values of nearly equal to unity) between the flow stress and the inverse square root of the grain size and/or SDAS. Thus, it might be concluded that both SDAS and grain size fit well to the Hall-Petch equation. However, further calculations using these data together with Eq. (1), showed that it is only SDAS measurements that fit meaningfully within the Hall-Petch equation. In other words, using grain size measurements (Fig. 4a), the calculated value for the lattice frictional stress (σ₀) in Eq. (1) would be negative, as shown by typical equations in the curves in Fig. 4a and 4b, which physically is meaningless. This fact indicates that according to the Hall-Petch equation, in cast alloys, grain size may not play an important role in the overall mechanical behavior of the material.

Using SDAS measurements, however, a reasonable value for σ₀ (between of 65 to 79 MPa for the whole range of plastic deformation) and strengthening coefficient (k; between 390 to 420 MPa.µm⁻⁰.⁵) was obtained, which is in consistent with previous reports for Al alloys [16]. By increasing the strain, the σ₀ went up. This was because of the raise in dislocation density within the dendrite arms, that limited the dislocation mobility.

![Fig. 3. Engineering Stress-Strain curves obtained from conventional tensile test for all specimens solidified under three different cooling rates. For each condition three curves are shown for the sake of statistical validation.](image1)

![Fig. 4. Flow stress vs. inverse square root of a) grain size, noted as "d", and b) SDAS obtained from the conventional tensile test of Al-10Si cast alloy. Equations in the curve are shown as example from the trendlines to show typical equations for the plotted data.](image2)
3.3. In-situ tensile

In-situ tensile test was done on all specimens, but due to similarity in the cracking behavior, only the salient results from the SDAS-15 specimen is presented. Until after Yield stress of the material, only a few slip bands were observed throughout the microstructure. For this specimen, the first cracks started to appear around 155 MPa, which is in the plastic region before the necking point. Most of the cracks were initiated at the dendrite/eutectic boundary (see Fig. 5a, 5b). Only one out of 15 observed cracks were initiated near the grain boundary, as shown in Fig. 5c and 5d. This needs further investigation; however, it was obvious that grain boundary has a minor effect on the crack initiation.

As depicted in Fig. 6a and 6b, during plastic deformation, most of slip bands appeared inside the dendrite, which is due to the relatively softer nature of it. Besides, each grain activates an identical slip system as shown by the direction of slip bands for each grain (see Fig. 6a and 6b). This indicates the main effect of grains on the plastic deformation; i.e. finer grain leads to more variety of active slip system that improves the strength of the alloy [1].

After fracture, as illustrated in Fig. 6c-f, all cracks propagated trans-granularly throughout the microstructure for all specimens. Thus, in hypoeutectic Al-Si cast alloys with modified Si structure containing no intermetallics, the SDAS has a dominant effect on mechanical behavior and cracking of the material. Although the Hall-Petch equation did not show any meaningful effect of grain size on the flow stress of the material, in-situ observations revealed that refining the grain size can affect slip bands formation at the early stages of plastic deformation.
4. Conclusions

The effects of SDAS and grain size on mechanical behavior of the Al-10(wt.%)Si base alloy containing modified Si morphology was studied using conventional and in-situ tensile test. The main conclusions can be derived as below:

- SDAS measurements were in consistency with the Hall–Petch equation. Although statistically correct, the grain size measurements did not show a physically meaningful relationship to the flow stress of the alloy.
- In-situ tensile observations revealed no crack initiation until after Yield point of specimens. The first cracks started within the plastic deformation region, between the Yield point and Ultimate Tensile Strength of the material. This implied a good coherency between the microstructural constituents when the Si particles were modified in the alloy.
- Most of cracks were initiated at the dendrite/eutectic boundary, regardless of the grain boundary location. This could be due to higher elemental segregation at the dendrite/eutectic boundaries rather than grain boundaries in cast alloys, which can introduce a bit of stress concentration at the dendritic boundaries. All cracks propagated through the grains, as trans-granular.
- Due to the softer nature of the Al dendrites, plastic deformation (slip bands) appeared there first. This indicates the important role of the Al matrix in the overall mechanical properties of Al-Si alloys.
- Each deformed grain contained an identical slip band direction. Although grain boundaries were not the dominant factor in crack initiation and propagation, refining the grain structure increased the number of slip systems within the microstructure. This could slightly improve the strength of the alloy. Therefore, in such alloys, SDAS is reported as the dominant factor affecting the mechanical properties, while the modest effect of grain size should not be completely neglected for a precise modeling of flow stress in cast alloys.

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