Microstructures and wear behavior of the rheo-squeeze casting high silicon aluminium alloys pipe with the gradient structure

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Abstract
In this article, the microstructural characteristics and the wear resistance performance of two kinds of high silicon aluminium alloys to produce gradient structure by rheological squeeze casting has been investigated. The mechanism effect of Fe-rich phase on microstructure homogeneity and wear characteristics was also studied. Experimental results present that liquid flow could be impeded because of being acicular Fe-rich phase, resulting in liquid segregation phenomenon and discontinuity gradient distribution of hard phase. Through adding 1.4 wt% Mn element into Al-22Si-2Fe alloys, the morphology of Fe-rich phase was improved, which acicular structure transformed to blocky structure. Because of higher hardness than Al matrix, blocky Fe-rich phase displayed softer hardness than Si particles, thereby buffering Hertz force and shear force, and bearing some load during wear process, which improved wear resistance of alloys.

1. Introduction
Hypereutectic Al–Si alloys have excellent characteristics of excellent wear resistance, high hardness, and low coefficients of thermal expansion [1, 2], which have been widely applied to make wear-resistant components in the automotive industry [3–6]. However, their low toughness and poor ductility, which are caused by harmful morphologies of primary Si particles (PSPs), such as coarse polygonal, star-like, and other irregular shapes, limit the wider application of these alloys [7].

Many researchers have focused on doping element into aluminium alloys to improve microstructure and mechanical properties. Kurt et al [8] performed research on the high Mg and high titanium additions on aluminium alloy and found that there appeared $\beta$-phase ($\text{Al}_3\text{Mg}_2$) $\alpha$-solid solution, $\text{Ti}_2\text{Mg}_3\text{Al}$ and $\text{TiAl}_3$ particles in the microstructure. The results of hardness and tensile strength showed that there was no accordance with the conclusion which previously regarded that smallest grain size contributed to highest mechanical properties. Kurt et al [9] also found that the average grain size of the alloys decreased with the addition of Ti. The tensile properties were worsened after the addition of higher concentration of Ti. Talamantes-Silva et al [10] studied the microstructure and solidification reaction of Al–Cu alloys by means of thermal analyses and found that solidification rate affected the microstructure, including dendrite arm spacing, grain size and porosity. A complex eutectic was detected in the regions which solidify last, that originated from Fe and Mg expelled from the material that solidified at an early stage. Bonneville et al [11] adopted hot isostatic pressing technique to produce Al and icosahedral Al-Cu–Fe powders. The final reinforcement displayed initial i-phase or tetragonal w phase. The compression test between Al/wo or Al/i exhibited different results as processing temperature increased. To satisfy the application requirement of hypereutectic Al high–Si alloys, the alloying element Fe is considered to be added. The addition of appropriate amounts of Fe may enhance the performance of these alloys [12–15]. On the contrary, too much Fe is detrimental to the properties of the Al–Si alloys. Investigations have
proved that the morphology of harmful acicular Fe-rich phases could be modified by adding Mn to hypereutectic Al–Si–Fe alloys [16, 17]. With Mn added, the morphologies of intermetallic compounds presented as: acicular, Chinese-script shape, fishbone shaped, and block-like. Differing Mn contents at 2% Fe content invoked changes in the crystallisation temperatures of the compounds [18]. Research demonstrates that morphology of PSPs can be refined and improved by preparing hypereutectic Al–Si alloys as semi-solid slurries adopting cooling slope (CS) casting [19–21].

Attributing to economical efficiency and working efficiency, squeeze-casting is especially suitable for semi-solid metal forming (SSM), which could manufacture components of good finished surface quality, high strength, high dimensional accuracy and high ductility [22, 23]. For hypereutectic Al–Si alloys, rheo-casting technology is more likely to cause segregation of the primary Si particles than which results in an impaired performance thixocasting [24–26]. In addition, SSM technology, which caused segregation, could be used as a method to produce a gradient structure [24–27].

This present paper is aimed at squeeze casting in fabricating gradient pipe structure of hypereutectic Al-22Si–Fe and Al-22Si-2Fe-1.4Mn alloys in radial direction (RD). The wear resistance properties of the two kinds of alloys inner walls and the effect and mechanism of Fe-rich phase on microstructure and properties were also investigated. Optical microscope was used for microstructure observation. The abrasion morphology and phase structure of two kinds of alloys were identified by means of scanning electron microscope, x-ray diffraction and transmission electron microscope, respectively.

2. Experimental work

Hypermorphic Al–22Si–2Fe (A1) and Al-22Si-2Fe-1.4Mn (A2) alloys were prepared by adding Si element, Fe agent and Mn agent into commercial pure aluminium in a 6.5 kw resistance furnace. Pure Al was heated to 1123 K and held for 10 min, when the melt was cooled down to 1073 K, then poured into a graphite crucible which has been preheated to 973 K by CS technology to produce a semi-solid slurry. Obtained the solidus and the liquidus temperatures of the alloy were approximately 843 K and 1050 K which were measured by differential scanning calorimetry, respectively [28]. Figure 1(a) shows the CS casting process using segmented cooling plates. Then, the slurry was transferred to a permanent mould, rapidly preheated to 473 to 523 K (i.e., within 5 to 10 s). The slurry in the lower die was squeezed under a pressure of 100 MPa for approximately 10 s.

The formed pipe was displayed in figure 1(b) and then split along axis direction into two halves. The microstructural specimens were prepared along the RD of the pipe wall. After grinding and polishing, the specimens were etched and examined to observe metallographic microstructure using an optical microscope. Five points along the radial direction were chosen to test the macro-hardness of the pipe wall with its gradient structure. The micro-hardness of the Si-and Fe-rich phases was measured using a Vickers hardness tester at 25 gf.

Figure 2 shows a schematic of sliding wear experiments, which were conducted at room temperature using a pin-on-disk tribometer. The tested pin was scanned along the RD of the wall. The objects tested were the inner surface of the pipe wall. The experiments were conducted at a sliding speed of 0.6 m s−1 for 1800 s, with 20 N normal load. The tested pin was used in an alloy against Cr13 steel disk configuration.

The longitudinal section of pipe was etched with 0.5% hydrofluoric acid. The polished and etched specimens were examined using an optical microscope (Leica). The scanning electron microscope (SEM) and energy spectrum (EDS) analysis system (Nova NanoSEM 450, FEI Co. VEGA-3SBH, Tescan Co.) were used to observe microstructure and abrasion morphology, respectively. X-ray diffraction (XRD, D/max-3B, R ricoh Co.) and transmission electron microscope (TEM, Tecnai G2 F30 S-TWIN, operated at an acceleration voltage of 300 kV.) were analyzed for phase structure of Fe-rich phases. The solidus and liquidus were evaluated using a simultaneous thermal analyser (NETZSCH STA 499F3).

3. Results and discussion

3.1. Microstructural characteristics and hardness of the pipe wall

Figure 3 shows the microstructures of semi-solid squeeze casting A1 alloy pipe wall, containing the formed solid structure of radical regions of pipe wall, and the different colored squares represent microstructure of different positions from the outer surface to inner surface. Though the overall trend of the distribution of the grey particles appears gradually increased, different sized regions with poor Si existed in the radial section of pipe wall, in which the distribution of primary Si is discontinuous. As shown in figures 3(a)–(e), the surface is divided into five regions to analyze microstructural characteristic. Figures 3(a)–(c) shows the microstructures of different region, which all contains PSPs, black acicular intermetallic compounds, α-Al and eutectic structure. It is worth noting that acicular Fe-rich phases do not present significant changes for their distribution along the
pipe wall: there was some liquid phase segregation on both sides of the acicular phases (as observed in figures 3(b)–(d)). The length of the acicular Fe-rich phases is approximately 150 to 200 μm. The equivalent diameters (ED) and volume fractions (VF) of PSPs in regions a to e are 22.5, 29, 31, 34.8, 35 ± 5 μm, and 12.5, 16.5, 13.8, 7.5, 12.7%, respectively (seen in figure 5(a)). The ED of PSPs gradually increased from regions a to e. According to the distribution of PSPs in the A1 alloy pipe wall, the VF of PSPs does not follow the rules of gradient distribution. In regions (c), (d), and (e), the VF of PSPs were irregularly distributed. Therefore, the effect of semi-solid squeeze casting, when applied to the production of a pipe wall with a gradient structure was unsatisfactory, while acicular Fe-rich phases still remained in the microstructure.

The formed solid structure of A2 alloy with addition of elemental Mn pipe wall are shown in figure 4. From the overall trend of the distribution of the grey particles, the area and number of poor Si regions are reduced compared with A1 alloy pipe wall. Meanwhile, the trend of continuous gradient distribution of the primary particles is more clearly. As well as analysis method for A1 alloy pipe wall, the radial pipe wall from the outer surface to the inner surface is divided into five regions (as shown in figures 4(a)–(e)). With addition of Mn, the
microstructures of A2 alloy all contain grey PSPs, the dark blocky Fe-rich phases, \( \alpha \)-Al and eutectic structure. In region a, the equivalent diameter of hard particles is 19.66 \( \pm \) 5 \( \mu \)m. The PSPs and the Fe-rich phases presented a regular structure. The micro-hardness of the Si particle and blocky Fe-rich phase were measured as 1279 and 276 HV, respectively. The two phases could be regarded as hard particles relative to the Al matrix for which the micro-hardness was 122 HV. The ED of hard particles in area b is approximately 23.24 \( \pm \) 5 \( \mu \)m. The Fe-rich phases present plate-like forms compared with those shown in region a. The VF of PSPs and Fe-rich phases in regions a to e were 12.68, 15.97, 16.84, 18.26 and 20.17\%, respectively. The EDs of hard particles in regions c to e are 27.53, 31.24, 35.56 \( \pm \) 5 \( \mu \)m, respectively (as shown in figure 5(a)).

According to these data stated above, the gradient distribution of the hard particles was regular in A2 alloy pipe wall. Figure 5(b) shows the macro-hardness of pipe wall from A1 and A2 alloy. The distribution of hard...
phases in the A1 alloy pipe wall caused the radial hardness distribution to deviate from a regular gradient pattern. The hardness of regions c to e was lower than that of region b. There was significant liquid segregation in the A1 alloy pipe wall and the segregation occurred on both sides of the acicular phase. It could be inferred that the acicular phase not only hindered the flow of liquid phase, but also hindered the flow of solid phase in the liquid phase during the filling process. That led to segregation occurring and the hard phase within the liquid phase cannot flow towards the lower pressure area as easily, without the liquid flowing.

The hardness of the A2 alloy pipe wall was increased from outer to inner surface, displaying a gradient distribution, which is the same as the distribution of the hard phase. The semi-solid slurry flowed towards the lower pressure region during the filling process, but the response of the solid-liquid two-phase region to pressure was different, leading the two phases unable to flow together. The liquidity difference of the two-phase region resulted in the eventual micro-structure gradient structure.

3.2. Analysis of phase and crystal lattice parameters of Fe-rich phases

Figure 6(a) shows the XRD patterns from the sample of the A1 alloy contains α-Al, δ-Al4FeSi2, PSP, and β-Al5FeSi phases and implies that the microstructure belonged to A1 alloy. The EDS analysis of acicular intermetallic compounds in the A1 alloy indicates that the molecular formula of the acicular intermetallic compounds was Al1.80FeSi2.47, which is close to the molecular formula of δ-Al4FeSi2 (figure 6(b)). Based on previous studies [18] which have showed the solidification sequence of an Al-25Si-5Fe alloy at a slow cooling rate was: L → β-Si → δ-Al4FeSi2 → β-Al5FeSi → ternary eutectic. According to related studies [29], the metastable δ-Al4FeSi2 phase can be transformed to stable β-Al5FeSi through a peritectic reaction below 883 K and at a lower cooling rate; but the transformation from δ-Al4FeSi2 to β-Al5FeSi below 883 K occurred in the metal mould. Due to the high thermal conductivity, the cooling rate of the melt was accelerated during the squeeze-casting process, the transformation also cannot be achieved, and the δ-Al4FeSi2 phase still remained in the solidified microstructure. Therefore, long acicular intermetallic compounds mainly include δ-Al4FeSi2 phases in squeeze-casting of this A1 alloy. Figure 6(c) shows the TEM bright-field images and selected area diffraction patterns of
the Fe-rich phase, which was in the form of tetragonal structure. Related research showed that the metastable \( \delta-Al_4FeSi_2 \) phase through peritectic reaction at a lower cooling rate transformed into a stable \( \beta-Al_5FeSi \) phase, however the strong cooling effect on the liquid metal inhibited the reaction during the inclined cooling plate treatment and the squeeze-casting process. Therefore, the \( \delta-Al_4FeSi_2 \) phase was the main Fe-rich phase present in the squeeze-cast A1 alloy.

The XRD patterns from the sample of the squeeze-cast A2 alloy pipe were showed in figure 7(a). When the ratio of Mn/Fe = 0.70, acicular Fe phases can not be observed in the microstructure of the A2 alloy (as seen in figure 4). Huang et al proved that some phases, such as \( \alpha-Al, \) PSP, \( \beta-Al_5(Fe, \) Mn)Si, \( \delta-Al_4(Fe, \) Mn)Si_2, and \( \alpha-Al_{15}(Fe, \) Mn)Si_3 were coexisted in the alloy with addition of Mn and Fe element when Mn/Fe = 0.625 in the solidification of hypereutectic Al–Si. In addition, they indicated that only \( \alpha-Al_{15}(Fe, \) Mn)Si_3 phase present when Mn/Fe = 1, as an intermetallic compound. Moreover, some researchers found that the formation of \( \alpha-Al_{15}(Fe, \) Mn)Si_3 can be improved by adding Mn. With increasing content of Mn, the crystallisation temperature range of \( \alpha-Al_{15}(Fe, \) Mn)Si_3 and \( \delta-Al_4(Fe, \) Mn)Si_2 decreased. The \( \alpha-Al_{15}(Fe, \) Mn)Si_3 phase may be primary since sufficient Fe and Mn were added to high-Si alloys.

During the squeeze-casting process of PSPs, a small amount of \( \delta-Al_4(Fe, \) Mn)Si_2 were discovered in the A2 alloy when Mn/Fe = 0.7. A large number of \( \alpha-Al_{15}(Fe, \) Mn)Si_2 was formed after being crystallised at higher temperatures between \( \delta-Al_4(Fe, \) Mn)Si_2 and liquid. Further, the \( \beta-Al_5(Fe, \) Mn)Si was transformed to the residual \( \delta-Al_4(Fe, \) Mn)Si_2 when the temperature was below 883 K. Although the diffraction peak of the acicular \( \delta-Al_4(Fe, \) Mn)Si_2 phase can be found in the A2 alloy, it proved to be hard to find this phase in the microstructure, due to the reduced nucleation site activity in the stage of initial solidification. Owing to \( \delta-Al_4(Fe, \) Mn)Si_2 phases involved in the peritectic reaction below 883 K, the acicular \( \beta-Al_5(Fe, \) Mn)Si phase was formed. Due to being a small quantity of \( \delta-Al_4(Fe, \) Mn)Si_2 in the reaction, almost no \( \beta-Al_5(Fe, \) Mn)Si phases were induced. In addition, the remaining liquid phase solidified, and lots of fishbone-shaped \( \alpha-Al_{15}(Fe, \) Mn)Si_2 phases were precipitated in the following quaternary eutectic reaction at its eutectic temperature. As stated above, it was concluded that there were no clear acicular Fe phases when Mn/Fe = 0.70, the intermetallic compounds which primarily formed blocky and fishbone-shaped or Chinese scripts \( \alpha-Al_{15}(Fe, \) Mn)Si_2 phases could be recognized in the microstructure.

The molecular formula of the polygonal block and that of the small Chinese characters in A2 alloy are \( Al_{12.63}(Fe, \) Mn)Si_3 and \( Al_{16.65}(Fe, \) Mn)Si_1.85 and were analysed by EDS, respectively (figure 7(b)), which are

Figure 6. Fe-rich phases in the rheo-squeeze casting A1 alloy pipe wall: XRD graph, (b) SEM image, (c) TEM bright field images and SAED.
close to Al$_{15}$(Fe, Mn)$_3$Si$_2$ phase. Figure 7(c) shows the TEM images and the chosen area diffraction patterns of Al$_{15}$(Fe, Mn)$_3$Si$_2$ phases in the bulk squeeze-casting process, which displays a body centred cubic structure.

3.3. Morphology of abrasive wear resistance

Figure 8(a) shows that the slender and acicular Fe-rich phase formed in the A1 alloy has a detrimental effect on the wear resistance. There appeared micro-cracks on the surface of the Fe-rich phase which were extended under compressive load. As a result, the Fe-rich phase was crushed and the sharp corners and edges of the phase prompted the appearance and extension of these cracks. Under the effect of shear stress, Fe-rich phase was lifted in plates on the worn layer and milled to trivial worn debris under the dual function of Hertz force and shear force. As shown in figure 8(a), the long strips of pores of different sizes are found on the worn sub-surfaces, and Si particles or the Fe-rich phase are broken to chunks, then ground to fine particles under the effect of the Hertz force. As these particles are brought out from the matrix during the wear process, cracks are more likely to appear beneath the worn surface, leading to the shedding of larger alloy plates from the specimens. All these indicate that the existence of the slender and acicular Fe-rich phase exerts a significant adverse influence on the wear resistance of the alloy.

The inner surface of the A2 alloy pipe presents the lowest mass loss and therefore the highest wear resistance among all experimental alloys tested (as shown in figure 8(b)). The Fe-rich phase plays an important role for improving morphologies and the physical properties in the wear process. A large amount of Si and blocky α-Al$_{15}$(Fe,Mn)$_3$Si$_2$ phases which are harder than the Al matrix are distributed on the inner surface of the A2 alloy pipe and bear the main loads and shear stress during the wear process. In the initial stage of the wear process, the harder Si particles bear the majority of the friction force after the Al matrix is rubbed out. As the experiment continuing, the loads on the worn surface constantly act on the Si particles. Since the Si particles contain cracks, there occurred cracked and crushed portions under the reciprocal action of the Hertz force. At the same time, the shear stress cuts the surface of the Si particles and causes fractured parts of the particles spalling from the main bodies of the particles. Under the rolling effect, the falling particles turn into granular or powdered abrasives, distributed on the ploughing edges or covering the worn surface. The cracks in the Si particles, or in the junctures between the Si particles and Al matrix, are extended in the long-term wear process. While the blocky α-Al$_{15}$(Fe,Mn)$_3$Si$_2$ phase was softer than the Si particles, they inhibited the extension of the cracks and stabilized
the Si particles tightly within the aluminum matrix, which avoided the tearing and falling of Si particles and reduced the mass loss during the wear process.

Figures 8(c) and (d) show the morphologies of the worn sub-surfaces of A1 and A2 alloy wall. It is known that the Fe-rich phase in the microstructure of A2 alloy is a blocky $\alpha$-Al$_{15}$(Fe,Mn)$_3$Si$_2$ phase. The Fe-rich phase with its average grain diameter of 30 $\mu$m surrounds the primary Si particles in the worn sub-surfaces, and some blocky $\alpha$-Al$_{15}$(Fe,Mn)$_3$Si$_2$ phase is crushed and rolled into worn particles, then grounds to powders that covers the worn surface. Under a load of 100 N, the sub-surfaces still show a certain plastic deformation and some Si particles are crushed. As mentioned above, the micro-hardness of blocky $\alpha$-Al$_{15}$(Fe,Mn)$_3$Si$_2$ phase is about 276
HV, which is lower than that of Si particles, showing a characteristic of greater plasticity. The softer blocky α-Al15(Fe,Mn)3Si2 phase can buffer certain Hertz forces and shear stress, thereby impairing the extension of cracks along the Si particles. Moreover, as harder than the aluminum matrix, that phase can bear some load during the wear process and thus bestows a certain wear-resistance.

4. Conclusions

(1) Squeeze casting hypereutectic Al–Si alloy semi-solid slurry with or without addition of Mn element to produce gradient structure distribution, showing different microstructure along the radial direction from outer surface to inner surface of alloy pipe wall. However, the exist of the acicular Fe-phases could hinder the liquid phases flow which caused gradient distribution discontinuity of blocky hard phases.

(2) The aluminum matrix was split seriously by the acicular Fe-rich phases, resulting to mass loss from the worn surface, deteriorating wear resistance of Al1 alloy. When added Mn element into Al1 alloy, the morphology of Fe-rich phase, the continuity of the liquid phase flow and the wear resistance of alloy were all improved by the Fe-rich phases modified that transformed into blocky structure.

(3) Al15(Fe,Mn)3Si2 phase with a body centred cubic structure and Si particles were harder than aluminium matrix, leading to bear shear stress and Hertz stress during wear process, inhabiting the extension of the cracks and reducing the mass loss of worn surface, resulting to be tightly stabilized within the aluminum matrix of Si particles, avoiding falling and tearing phenomenons.

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