Microstructural Modifications Produced by Nano-Metal-Phosphate Additions to Aluminum-Silicon Alloy by new System Projects

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Abstract. Al-13Si alloy microstructure affected by separate additions of Nano-Metal-Phosphate, prepared by die-casting, to synchronize refine eutectic Si and modify eutectic Al is studied. The nucleation and growth mechanisms of the Si phase are discussed via morphology and kinetics analysis. The effects point out that the measurement of eutectic Si is refined to 0.37-2.46µm and the morphology of the large platelet eutectic Si particles had been modified into the fine fibrous shape. The technique synchronizing consequences of Nano-Metal-Phosphate is related to the AIP and Al₂O₃ formation. These consist of the following, the impact of Al₂O₃ at the interfaced between the Al segment and Si particles for obstacle the growth and enhance the refinement of the eutectic Si. The impact of AIP acts to locate of heterogeneous nucleation for eutectic Si, consisting of three aspects: interaction of dopant elements with molten Al-Si alloy to shape the nucleation site with reducing the interface energy between liquid and Si phase and increase the activation of diffusion energy throughout the liquid-solid interface. The results improve the mechanical properties of modified Al-13Si alloy and thus attributed to reducing silicone partials dimension and fibrous structure of eutectic silicone, presence of substantial AIP and Al₂O₃ in-situ precipitation in the eutectic location and the volume fraction increase of uniform distribution.

Keywords: Aluminum Silicon, Corrosion Resistance, Wear Resistance, Fracture Behavior.

1- Introduction

Al-Si alloys have often been used in manufacturing, aircraft, vehicle because of their high definition of strength, fluidity and relatively low cost. Microstructural studies have increasingly gained interest in technology advancements like the reduction of secondary-arm dendrite [1], the elimination of solidification failures [2], modifications and refining [2,4] to further increase mechanical efficiency [4]. The features of mechanical Al-Si cast alloys are known to be determined primarily by the shape and scale of the Si phase. There is a basic requirement to clarify the development of nanostructures and their interactions, such as twinning and Nano-particles, [5] to understand and control the development process of eutectic silicon.

The interactions between the twins and precipitates are important not only for a deep understanding of the evolution and development of the twins during solidification but also for increased strength in the course of deformation [6,7]. The interaction between twins and Nano-particles is not yet well defined due to the scientific difficulty in their synchronized introduction.

Eutectic Si twinning was well known when Nano-Metal Phosphate levels were added easily in inoculated Al-Si alloys [8]. The emphasis on impurities caused by twin [9] and contamination of the re-entrant borders of twin planes was scientifically strong [10]. Al-Si-P Nano-particles [11,12]. Nano-particles that rendered eutectic Si a good candidate for Nano-particles and twins interaction work in modified Al-Si alloys were observed at the intersection between Si twins. This is demonstrated by the discovery of Al-rich Nano-particles consistent with the Si. It should be noted that Nano-particles of Al-rich compatible with Si [13] were observed in the suppression of Si twinning[14], which may cause eutectic Si nucleation mechanisms to change. There have been contentious interaction between Nano-particles and twins, restricting the application and development of complex changes to Al-Si alloys. Al-
rich Nano-particles were observed in line with the Si. Al-rich Nano-particles in conjunction with Si [13] has been observed to suppress the Si-Twinning cycle[ 14] which can alter the eutectic Si-nucleation mechanism. Interactions of twins and Nano-particles appeared dispute restricting the application and creation of complex modifications to Al-Si alloys. Nanoparticles developed in eutectic Si [15,16] were documented inducing all Nano-metal-phosphate additives. A research on the evolution of eutectic Si and Nano-particles interactions, eutectic Si microstructures, mechanical properties and corrosion activity in the alloy Al-13Si. In order to demonstrate the mechanism of intermetallic phases during the Al-13Si alloy solidification process, the phase reconstruction and the construction phases were also studied.

2. Experimental Method

2.1 Preparation of Nano-dopant elements

The substantial refining of eutectic Si utilizing Nano-particles was used to improve the performance of refining and modification. Because no resource is possible to secure Nano-particles, they were produced in the lab. The chemical reactions of solid-liquid, using powdered elements with an aqueous solution of H3PO4, synthesize Nano-particles way. Table 1 displays the phosphoric acid chemical reaction formulae with powder components.

| Material | Formulas |
|----------|----------|
| Fe       | Fe + 2 H₃PO₄ = Fe(PO₄)₂ + 3 H₂ |
| Mg       | 2 Mg + 2 H₃PO₄ = 2 MgPO₄ + 3 H₂ |
| Zn       | 3 Zn + 2 H₃PO₄ = Zn₃(PO₄)₂ + 3 H₂ |

2.2 Prepare Nano-Metal-Phosphate

Prepare of compact pellet consist of pure aluminium powder commercial aluminium, that used 0.3 g and mixing with dopant element and compaction powders under 150 bar to enhance oxidation resistance and flotation, as shown in Table 2. The particles are compressed together (Nano-particles and aluminium power) elasticity, then the pressure action leads to permanent bonding at the contact points. The result of compact powder is pellets, which makes it easy to handle.

| Material | Wt.% added | Wt.% added | Al-Pure | Compact pressure |
|----------|------------|------------|---------|------------------|
| Fe(PO₄)₂ | 0.01 g     | 0.03 g     | 0.3 g   | 150 bar          |
| Mg(PO₄)₂ | 0.01 g     | 0.03 g     | 0.3 g   | 150 bar          |
| Zn₃(PO₄)₂| 0.01 g     | 0.03 g     | 0.3 g   | 150 bar          |

2.3 Casting Procedures

The Al-13Si was made by liquid metallurgy. The chemical composition of the aluminium alloy is shown in Table 3. The Al-13Si in the electric furnace (Carbolite), the alloy charge is heated up to 780°C. Mechanical agitation with 500rpm and degassing by using an inert gas (argon) as shown in figure 1. Dopant elements were preheated at 350°C to minimize all humidity and added to the molten alloy. In the molten alloy was added a small amount (0.01-0.03%) of Nano-metal-phosphate with particle size (200 nm). The molten agitation took approximately ten minutes; to promote uniform dispersion of nucleates wetting and a uniform reinforcement dispersion. Then the molten alloy was poured into a 1.5 cm × 15 cm preheated steel mould.

Table 3. Al-Si alloy Chemical composition (wt. %) used in this study.
| Composition wt% |   |   |   |   |   |   |   |
|----------------|---|---|---|---|---|---|---|
| Al             | Zn | Ni | Mg | Mn | Cu | Fe | Si |
| Bal.           | 0.004 | 0.008 | 0.02 | 0.01 | 0.01 | 0.14 | 12.86 |

**Figure 1.** The process of stir casting

2.4. Microstructural Examination

The specimens of SEM microscopes for Al-13Si alloy were produced by grinding papers started: 220, 320, 600, 800, 1000 and 1200 respectively. The samples were finished with diamond paste and then washed and etched. Keller's reagent is a mix of NHO3 5ml, HCl 3ml and HF 2ml and H2O 190 ml.

2.5. Hardness Test

Vickers hardness tests (type ZWICK Z313 – Germany) were carried out on the Al-13Si alloy. The load was (0.5Kg) for (10 seconds). Obtaining the average diameter value was taken by three readings for each sample. The equation used to estimate Vickers hardness is:

\[ H_V = 1.8544 \times \frac{P}{(D_{AV})^2} \]  

where

- \( H_V \): Vickers hardness number,
- \( P \): applied load (N),
- \( D_{AV} \): diagonal length (mm)

2.6. X-Ray Diffraction Analysis

The phases of the modified Al-13Si samples were analyzed with the X-ray diffraction Shimadzu (XRD-6000) diffractometer, which is available from the University of Technology, Nano Technology Center. Radius scanning: 185 mm, Radio X-rays of C, NF (154060 nm), Radius leakage: under 2.5 μSv / h at maximum output.

2.7 Corrosion Tests

The Al-Si alloy electrochemical examination was approved with cyclic polarization. A controlled unit (PCI4/750, Warminster, PA, GAMRY, Inc) disbursed all tests in 3.5 per cent NaCl sol. In 25C. Ag / AgCl as the pole of reference and the auxiliary pole and working pole of noble metal (Pt) used as the specimen keeper. The specimen was exposed to NaCl solution of 3.5%. The region of exposure had been 1 mm2. Before each test, the samples got a metallographic polishing. Samples were immersed in the solution until a stable open circuit potential (OCP) was obtained. The test specimens exposed area
was approximately 10 mm\(^2\). All data is neutralized according to the area of the surface. It is calculated using the Echem analyser [10] to identify the test results identically.

2.8. Wear rate measurement
Dry slip wear tests were performed at room temperature using a laboratory pin on disc wear test with ASTM F732-82 [11]. Nails, which were modified Al-Si alloy o 10 mm and a length 20 mm, were polished by various sheets of grain carbide (220, 320, 500, 800 and 1000), and cleaned with acetone and then dried. The worn equipment consists of a motor with constant rotation speed (510 rpm). The disc is made of tool steel with a hardness of 65 HRC. Relative mass changes were tested by weighing the samples before and after wear using a digital scale according to the sensitive scale classification (DENVER Tool, Japan) (Max-210gm) at 0.0001 mg. The used loads were 500 kN. The disc is cleaned after each test.

The following equation is to calculate the wear rate:

\[
\text{Wear rate} = \frac{\Delta W}{S} \tag{2}
\]

\[
S = V \times t \tag{3}
\]

\[
V = \frac{\pi DN}{1000 \times 60} \tag{4}
\]

where \(D\): diameter (m) =0.14, \(W\): The different in weight of sample before and after the test (gm), \(S\): sliding distance (m), \(t\): time (Sec), \(N\): r.p.m=510, \(V\): velocity (m/sec)

3. Results and Discussion

3.1 Microstructure analysis
Originally, without addition, Nano-metal phosphates made up of larger eutectic grains Si and platelet eutectic Si with an average grain size from 37.82\(\mu\)m. Figure 2a indicates that SEM microstructures Al-13Si have been moulded as cast without modifiers. After additive Nano-iron phosphate Fe(PO\(_4\))\(_2\), figure 2b show Al-13Si's SEM microstructures. The Nano-iron phosphate Fe(PO\(_4\))\(_2\) may act as a promoter of grain refining and eutectic modifier composed of the fines grains (2.46\(\mu\)m) by adding 0.03 percent concentrations. Nano-iron phosphate can change the phase morphology or increase the precipitation of Fe-rich particles that are less harmful to needles by adding Fe(PO\(_4\))\(_2\) particle. Observing the simple \(\alpha\)-phase dendrites, the (mechanical \(\alpha\)-phase mixture), and the different intermetallic phases of all the experimental materials have shown

After an additive of 0.03% Nano-magnesium phosphate, the optical microstructures of Al-13Si can be identified in Figure.2(c). The average size of the grain is 1.64 \(\mu\)m. It is formed of fine grains. The \(\alpha\)-matrix takes the liquid in the form of dendrites as a eutectic phase and consists nominally of Al and MgP in AlMg, alp and Al\(_2\)O\(_3\) cast. For every state of our experimental materials, dendrites (finer) are similar in size, but the secondary dendrite arms space (SDAS) is somewhat different. Particles AlP and Al\(_2\)O\(_3\) are like grains rounded. On the periphery of \(\alpha\)-phase dendrites, however, thickened cells were observed.

After an additive of Nano-Zn phosphates, figure 2d indicates Al-13Si optical microstructures. Fine-grain residues of the average size of grain are 0.37\(\mu\)m. Al-ZnP cast alloy microstructure contains \(\alpha\)-Al phase, AlP and Al\(_2\)O\(_3\) [ 7,12] particulates. In the form of dendrites, the \(\alpha\)-matrix precipitates from the liquid, which is nominally Al and Zn. For both states of experimental material, the size of dendrites (fineness) is identical. On the periphery of \(\alpha\)-Al phase dendrites, small, rounded grains were observed. The \(\alpha\)-dendrites are mainly composed of Zn-containing, such as the AlZn phases. Studies have confirmed that the length of dendrites and the fine arms rise in industrial pure aluminium as the amount of phosphate metals increases. This alloy is a traditional alloy because it has a similar structure, given its zinc content.
3.2 Mechanical Properties
The results of mechanical characteristics show improved or comparable properties with the content of experimental material Nano-metal phosphate forms Figure. 3. Vicker's hardness Hv = 73.56 is the first sample without an additive. Vickers hardness Hv = 83.06 and 91.12 are respectively in specimens of the content of 0.01-0.03% of Nano-iron phosphate. Vickers hardness Hv = 79.13 and 88.64 respectively are specimens containing 0.01-0.03% of Nano-magnesium phosphate. The Vickers durability Hv = 80.36 and 86.64 for specimens with the content of 0.01-0.03% of Nano-zinc phosphate. The difference in hardness is small, because the Nano-metal phosphate is substituted, which means that the Al-matrix is very similar.

Figure 3. Show comparable hardness with the content of Nano-metal phosphate types.
3.3 X-ray diffraction and crystallographic texture

The liquid becomes saturated towards a solid phase and eutectic solidification of the phase $\alpha$-Al occurs according to the phase diagram of Al-Si alloy composition. The liquid solution will be enriched by silicone by the end of the eutectic solidification. The solidification of the $\alpha$-Al phase finishes in the alloy and the second state, the solidification of the eutectic composition melt, begins. All the liquid solidifies and the alloy at the eutectic temperature consists of eutectic $\alpha$-phase crystals and the $\alpha$-Al and Si-based eutectic mixture, distributed between $\alpha$-Al matrix grains. Then, phase transformations occur in the solid-state on cooling. The silicon crystals (a solid aluminium solution of the variable composition in silicone) precipitate from $\alpha$-Al crystals when the temperature drops.

Effect of the bulk alloy is superimposed upon the diffraction pattern of the interactive intermetallics. Therefore, after the removal of the Al matrix, there were still some peaks potentially correlated with phases (Fe(PO$_4$)$_2$ and AlP's, Al$_2$O$_3$, AlFe and AlFeSi). Phase fractions measured using a card for calibration requirements as shown in Figure 4(a). A local XRD analysis of bulk material has been performed to determine phase transformations. In-site measurements for 2$\theta$ volumes ranging from 38.64 to 65.36° were performed to determine the interaction (FePO$_4$) during casting. Effect of the bulk alloy is superimposed upon the diffraction pattern of the interactive intermetallic. It is obvious that the phases of AlFe and AlFeSi existed in the system which implies no breakdown that will correspond in AlP. Similarly, AlP-related peaks stayed unchanged, suggesting their higher thermal stability. Overall, the findings provided suggest that the collection of intermetallic phases mainly depends on the application of (FePO$_4$) grain refiner in Al-Si melts, and reduction in grain size or alteration in eutectic Si, main Si and grain morphology $\alpha$-Al.

![Figure 4. XRD Pattern for Al-23Si Samples processed by different Nano-Metal Phosphate.](image_url)
The XRD pattern for Al-Si samples treated by the master alloy (Mg$_3$(PO$_4$)) as shown in figure 4b. Shows the characteristic (111), (200), (220) and (311) peaks of face-centred-cubic Al, in addition to the diamond-like Si (111) point, which indicates a two-phase content and that Si is not soluble in the Al matrix. As the solubility of Si in α-Al is reached during solidification, the precipitation of Mg$_2$Si crystals begins. Mg$_2$Si peaks have been observed but the alloy needs to be able to precipitate them. A textured substance is one in which the crystallites have a preferred orientation, whereas a textureless condition is where the orientations are usually random.

The XRD pattern of Al-Si samples processed by (Zn$_3$(PO$_4$)$_2$) master alloy is illustrated in figure 4c. The large peaks produced from the XRD test are primarily aluminium and silicone, whereas the minor peaks are AlP and Al$_2$O$_3$. ZnSiO$_3$ contents of Al-Si alloys resulting from the interplay of molten Al and Zn$_3$(PO$_4$)$_2$ have effects on dendritic structure microstructures and eutectic silicon as ZnSiO$_3$ successively enters into a sound solution during solidification, and only the liquid area is therefore required to be altered. Zinc’s position is not well understood. However, the changes in Al–Si alloys to eutectic silicone result from AlP and Al$_2$O$_3$ alter the morphology and the fibrous, flake form of the Si particles rather than acicular. Contains optical micrographs with a solidified alloy of 0.01 and 0.03% ZnP showing the eutectic Si morphologies.

3.4. Wear Behaviour
The relationship between addition Nano-metal-Phosphate and wear rate of experiments alloys are presented in figure 8. It is clear the wear rate decreases with increase in the percentage of Nano-Metal-Phosphate particles. Furthermore, the relationship is linear. This means, decreases of grain size and good distribution of eutectic Si, eutectic and intermetallic phases, wear rate decreases regardless of the reinforcement Nano Al$_2$O$_3$ size and percentage. Also, wear and tear rate of processed composites decreases with increasing modifier elements (Nano-Metal-Phosphate). The improved wear resistance to casting procedure is referred to the distribution of particles within the base Al and refinement of grain.

Figure 5. The variation of wear rate with sliding time at different Nano-metal phosphate.

Figure 6 shows optical micrograph of worn surface of stir processed composites. The micrograph reveals that the worn track breadth decreases in modified alloy compared to unmodified alloy. It’s proof that the extent of wear and tear in composite is below that of unmodified alloy. The depth and breadth of the grooves generally imply that associate in foster quantity of fabric off from the specimen surface [2]. It is expected that uniform distribution of particles and modification of Si grains can cause a high wear resistance.
3.5 Corrosion Behaviour
The cyclic polarisation curve was determined and shown in figure 7 to evaluate the electrochemical stability of prepared samples in 3.5% NaCl. Corrosion variations were evident in the alloys tested. Cyclic polarization experiments have been carried out to assess the polarization domains for the analyzed samples. All three possible domains, namely: catholic domain and passive domain, were shown by cyclic polarization curves. The large passive dominance and low-density values show higher resistance to corrosion in the analyzed sample in the trans passive domain [3].

**Figure 6** optical micrographs of the worn track: (a) as cast; (b) additive of Nano-Fe phosphate (c) additive of Nano-Magnesium phosphate (d) additive of Nano-Zn phosphate.

**Figure 7.** Cyclic polarization curves of the experimental materials in 3.5 % NaCl solution. (a) additive 0.01% of Nano-Metal-phosphate (b) additive 0.03% of Nano-Metal-phosphate.
Figure 8. Microstructure characteristics of the tested alloys after the corrosion test. (a) as cast; (b) additive of Nano-Iron phosphate (c) additive of Nano-Magnesium phosphate (d) additive of Nano-zinc phosphate.

Indeed, despite the use of the Tafel extrapolation method for the determination of corrosion rates for Al-based alloys [17,18], the Tafel tables and their respective calculated uniform corrosion rates for metal covered with a semi-conductive passive film raised strong. Active dissolution near $E_{\text{corr}}$ occurred in the polarization curve of pure Al. This was followed by a major improvement of the current, with the potential added due to dilution and weakening of the passive layer, due to the attacks by Cl-anions shown in Figure 8a.

The cyclic curves of polarisation also demonstrate the action of the board in figure 8. This is obvious for the divisions of $E_{\text{corr}}$ Al with the inclusion of phosphate elements, which displayed a certain curvature over the entire range of applications.

As a result of micro galvanic corrosion processes, the effect of FeP on the pitting of alloys starts on the interface between the α-Al matrix and Fe-rich intermetallic. General irregular corrosion affected specimens, with grey corrosion products covering their surface.

In contrast with Al-13Si alloy corrosion, the microscopic observations demonstrated the strong activity of Al-FeP. Separation at the limits of grain contributes to galvanic and intergranular corrosion effects. A microscopic examination of Al-FeP surfaces following exposure analysis has shown that identified corrosion pit sites are found at the surface Figure 8(b).

Figure. 8(c,d) microscope observations indicate that Al ZnP and al-MgP have been extremely sensitive to corrosion relative to Al-13Si alloy. Furthermore, Al-ZnP and Al-MgP alloys continue to passively cover a broad spectrum of potential at a very low current. The results reflect the strong passively and a higher propensity to resist this solution, which refers to broad corrosion resistance in the anodic polarization curve of the traditional passive film. In the very small passive zone, remarkable changes took place over $E_{\text{pit}}$. The passivity of the alloys studied persisted until the pit potentials were reached $E_{\text{pit}}$. These did not increase the current density of the corrosion and the formation on the reverse potential examination of a small or no hysteresis loop. This was a clear sign that passivity in the layer resisted, pitting corrosion began to spread and spread[ 19,20,21].

3.6 Fracture Analysis

A cross-sectional view of the fracture surfaces shown in figure 9 may further explain the failure mechanism. As a cast material, the crack extends along the crossroads between the center and the limit as the softer areas, which was consistent along the surfaces of the fracture. De-cohesion [17] at the Si particles, the voids coalesce and spread until a complete separation occurs and the sample fails under tensile loading.
Fe(PO₄) applied to the base alloy resulted in a higher ultimate tensile strength (UTS) than Fe precipitate to the base alloy. The effect of Al₂O₃ and AlP on Fe is because of its neutralizing effect. The high-iron intermetallic phase has been transformed to AlFe by Chinese script morphology. This chapter describes the morphological structure of words in Mandarin Chinese. Word is defined as a syntactically free form, that is, a form that can stand independently in a syntactic slot. Most words in Chinese consist of one or two morphemes. Single-morpheme words are termed simple, while words consisting of two or more morphemes are termed complex. Morphemes in Chinese undergo virtually no morphophonemic alternation, that is, they retain their phonological shapes when they appear together with other morphemes in a word. Chinese has four morpheme types: content word, function word, bound root, and affix. The four morpheme types combine to yield the following four complex word types: compound, bound root word, derived word and inflected word, which presumably has lower harmful effects on mechanical properties. In comparison, Al₃Fe has adverse effects on the hardness properties of aluminium-silicon alloys because it is usually found as Fe-bearing metallic compounds, which increase the hardness but decrease many other mechanical properties.

The (UTS) increases with the addition of Mg(PO₄) to an Al-Si alloy for 0.03% but the elongation decreases. Due to the solid solution durability and precipitation of the solidification of Mg₅Si equilibrium in intermetallic phases the amount of these AlPs and Al₂O₃ phases increases at the content of Mg(PO₄) and explains that the hardness increase is relatively proportional. The hardness and ultimate tensile strength were significantly increased.

The absolute level of tensile strength is probably because Si eutectic particles are fully spheroidized and Al₂O₃ hardening phases precipitate. The addition of Zn(PO₄) can be shown to delay the complete process of spheroidization of the eutectic Si particles. Zn(PO₄) is applied to improve the alloy elongation. The ultimate stress and elongation between Al-13Si alloys are very stable. Zn(PO₄) adding undoubtedly reduces the number of micro-porosities in the matrix. Throughout tensile processing, dislocations such as intermetallic phases, Al₂O₃ or silicone particles can not move through hard phases.

Figure 9. Fracture analysis of modified-Al-13Si alloys (a) as cast; (b) additive of Nano-Fe phosphate (c) additive of Nano-Mg phosphate (d) additive of Nano-Zn phosphate.
4. Conclusions

Based on the present work, one can summarize the following remarks:

1. The added 0.01-0.03wt of Nano-Metal-Phosphate to Al-13Si alloy favored intermetallic compound formation (AlFe, AlMg, Al$_2$O$_3$ and AlP). Besides, the morphology, distribution, and grain size changes.

2. In samples modify with additional Nano-Metal-Phosphate a complete modification was observed and a fine fibrous structure of eutectic silicon, mainly in the addition MgPO$_4$.

3. Increase of hardness and wear values of modifies samples processed by Nano-Metal-Phosphate additions, because of the formation of the more difficult intermetallic compound and higher density of the Al$_2$O$_3$.

4. This phase of work obtains a favourable combination of mechanical properties.

5. The XRD pattern reveals predominant aluminium and silicon peaks and slight AlP and Al$_2$O$_3$ peaks.

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