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Effect of iron and copper on the microstructure and mechanical properties of gravity Die-cast Al-Si-Ni-Mg alloys

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Abstract. The microstructure and mechanical properties of gravity die-cast experimental Al-Si-Ni-Mg alloys with varying addition of recycled Cu wire and Fe pick-up from mild steel crucible and tools was investigated. Addition of Cu and Fe in the as-cast experimental alloys, improves the tensile strength by 6.11%. Yield strength increases marginally by 3.43% up to 1.07 wt.% of iron and 2.98 wt.% of copper and then decreases. Relative percentage elongation drops significantly by 89.38%. A very small drop in impact strength was observed. The positive influence of copper and iron addition in the Al-Si-Ni-Mg alloy system due to fine grain strengthening and delay in the formation of hard plat like β-Al5FeSi phase, iron bearing inter-metallic phase (β-Al5FeSi) found only when iron content reaches 1.07 wt.%. X-ray Diffraction, optical microscope (OM) were used to characterize the material.

1. Introduction

Aluminium alloys are used as a soft foil to rigid machinery parts. Aluminium is next to steel in the application area of building and construction; containers and packaging; transportation; machinery and equipment [1]. Light weight, high strength Al alloy is highly recommended for all kinds of vehicles [2]. Especially, Al-Si-Mg system is widely used as structural parts due to its excellent combination of cast-ability and mechanical properties.

Cast aluminium alloy system such as Al-Si-Mg (A356.0) and Al-Si-Cu (319.0), iron is considered as the natural unavoidable impurity. Fe level is controlled to a very low level, less than 0.2 wt.% in A356.0. Thus, the foundry process requires primary raw material, high cooling rate [3] which are direct impact on the cost of raw material; additional melt treatment such as iron neutralization and gravity segregation process are required to maintain the iron level; well protected steel tools and/or furnace equipment is in demand to manufacture iron free Al alloys. This iron controlling foundry practice increases the overall cost of aluminium alloy [2,4].

Paradoxically, deliberate addition of iron to a certain system of aluminium alloys has many advantages: improvement in hot-tear resistance, increases strength, better creep characteristics, stabilizes and reduce the grain size by the finely dispersed Fe rich second phase, modifies the silicon phase. Refractory aluminides of iron improves the thermal stability [5], reduce sticking between mould and the casting called die soldering [4,6].

Addition of iron and copper increase wear resistance, by increasing the volume fraction of complex Si bearing intermetallic phases [2]. A trace addition of Cu in Al-Fe-Si ternary, refine and disperse the iron bearing intermetallic phases [4]. Iron can be added up to 2.0 wt.% in Al-Si-Ni-Mg-Cu system of
alloys to increase strength at elevated temperatures. Example, in commercial grade piston alloy with Si wt.% in the range of 11-23 can accommodate up to 1.3 wt.% Fe [7]. In this work, addition of copper and iron in the hypo-eutectic Al-Si-Ni-Mg alloy was investigated.

2. Experimental procedure

Table 1. Composition of the experimental alloys in wt.%

| Samples | Si       | Cu     | Fe     | Ni      | Mg     | Mn     | Cr    | Zn     | Ti     | Pb     | Sn     | Al     |
|---------|----------|--------|--------|---------|--------|--------|-------|--------|--------|--------|--------|--------|
| S1      | 4.04     | 0.503  | 0.577  | 0.613   | 0.371  | 0.019  | 0.004 | 0.048  | 0.009  | 0.022  | <0.002 | Rem.   |
| S2      | 3.53     | 2.98   | 1.07   | 0.539   | 0.236  | 0.021  | 0.007 | 0.044  | 0.009  | 0.020  | <0.004 | Rem.   |
| S3      | 3.49     | 6.77   | 1.34   | 0.532   | 0.212  | 0.023  | 0.006 | 0.041  | 0.009  | 0.022  | <0.008 | Rem.   |

Al-Si-Ni-Mg alloy with varying Cu and Fe percentage were prepared by melting commercially pure (99.7%) Al, laboratory recycled or re-melted Al-Si-Mg alloy containing 0.105 wt.% iron, nickel and recycled copper wire. Iron was not added externally through iron bearing master alloys. Instead, iron picked up from the mild steel crucible and the tools used during melting were the source of iron. An electric resistance furnace with 1000°C capacity was used for melting. The alloying elements were heated to 760°C in a 2.5 litre capacity mild steel crucible and maintained for 25 minutes, this homogenize the alloying elements and also iron pick-up from the mild steel crucible and other steel tool used during melting and melt treatment process. Meanwhile, 0.1 wt.% of Al-Ti-B master alloy was added to the melt for grain refinement and stirred for 30 seconds. The melt was degassed with the commercial grade dry, pure argon gas for 2 minutes to remove dissolved hydrogen and inclusion. Then, 0.1 wt.% of dry NaCl was added to the melt for modification. Molten metal was stirred and cleaned for dross manually. The melt was covered with a commercial cover flux during melting.

Finally, molten metal taken out from the furnace and a part of the molten metal was poured into a preheated mild steel permanent mould for a pouring temperature of 730°C. The mild steel crucible with remaining molten metal was maintained at 760°C in the furnace. Recycled copper in the form of wire was pre-heated and added to increase the copper content. Melt was maintained for 20 minutes in the subsequent two pouring with required melt treatment. Thus, three different compositions of alloys were prepared. Three test specimens were made from each alloy for tensile and impact test. Tensile specimens were machined to ASTM B557M-02a standard (figure 1). Charpy V-notch impact test specimens were made with ASTM E23-16b standard. The average chemical composition of the
Experimental alloys are listed in the table. X-ray diffraction (XRD with Cu anode, $\lambda_1 = 1.54060$ nm, $\lambda_2 = 1.54439$ nm), optical microscope (OM) were used to characterize the material.

**Figure 2.** OM images a,b; c,d; and e,f for specimen S1, S2 and S3 respectively.
Figure 3. XRD report for the sample S1, S2 and S3.

Figure 4. Tensile properties of as-cast experimental alloys.
3. Results and discussion

3.1. Microstructures

Optical micro photograph of as-cast hypo-eutectic experimental alloy (sample S1) shown in the figure 2(a,b) revealed coarse fish-bone and star like morphology of primary aluminium solid solution, which is surrounded by eutectic silicon and Fe, Cu & Ni bearing intermetallic phases.

Increase in Cu to 2.98 wt.% and Fe to 1.07 wt.% in sample S2 increases $\pi$-$\text{Al}_5\text{FeMg}_5\text{Si}_6$, $\text{Al}_3\text{Cu}_4\text{Ni}$ and introduces new XRD peak of $\text{Al}_3\text{Cu}_5\text{Fe}$, eutectic $\text{Al}_2\text{Cu}$ and plate like $\beta$-$\text{Al}_3\text{FeSi}$ phases. Subsequently, course Chinese script like $\pi$-$\text{Al}_5\text{FeMg}_5\text{Si}_6$ phase marked in the micrograph shown in figure 2(d) increases relatively by 0.5%. The intensity of $\alpha$-$\text{Al}_5\text{Fe}_3\text{Si}$ phase start decreases relatively by 12.2% which is evidenced in the XRD plot (figure 3) and the new XRD maximum peak with 2.6 relative percentage of $\beta$-$\text{Al}_3\text{FeSi}$ phase confirms the start of transformation of hexagonal $\alpha$-$\text{Al}_5\text{Fe}_3\text{Si}$ to monoclinic/orthorhombic $\beta$-$\text{Al}_3\text{FeSi}$ phase in the sample S2. From this observation, Fe-rich phases have strong influence in modifying and refining the dendritic morphology of the $\alpha$-aluminium, which is clearly visible in the metallographic images shown in the figure 2(c,d). This supports fine grain strengthening mechanism in the system.

Further, increasing Cu to 6.77 wt.% and Fe to 1.34 wt.% in sample S3 shows a remarkable increase in the network of eutectic $\text{Al}_3\text{Cu}$ in the grain boundary. Also, rod shaped (in two dimension) $\beta$-$\text{Al}_3\text{FeSi}$ phase increases in inter dendritic region as showed in the micrograph (figure 2(f)). The maximum grain size of the sample S3 is 288 µm. It is 25% lower than the grain size of S2 and 45% lower than grain size of the sample S1. This grain refinement is due to the transformation of course $\pi$-$\text{Al}_5\text{FeMg}_5\text{Si}_6$ phase to Si and $\text{Al}_3\text{Cu}_4\text{Mg}$ phases, which is confirmed by XRD (figure 3). Course Chinese script like morphology is not found in the micrograph (figure 2(e,f)) of sample S3.

3.2. Tensile properties

The tensile property of gravity die cast experimental alloys has been shown in the figure 4. As the percentage of Cu and Fe content increases, the ultimate tensile strength increases from sample S1 to S3 by 6.11%. Yield strength increases marginally by 3.43 % up to 1.07 wt.% of iron and 2.98 wt.% of copper (S2) and then decreases. Relative percentage elongation drops significantly by 89.38% (S1 to S3). At high solidification rate in the metallic mould gravity die casting, Al-4Si-Ni-Mg alloy containing 0.5 wt.% Cu and 0.5 wt.% Fe (Sample S1) promotes the non-equilibrium phase reaction (Al + Si + $\text{Al}_3\text{Cu}$ + $\alpha$-$\text{Al}_5\text{Fe}_3\text{Si}$) in the as cast alloy. This is confirmed with the XRD of sample S1 shown in the figure 3(S1), shows a trace of Si, $\text{Al}_3\text{Cu}$ and peaks of $\alpha$-$\text{Al}_5\text{Fe}_3\text{Si}$ phase. There is no trace of equilibrium $\beta$-$\text{Al}_3\text{FeSi}$ phase. The Chinese script like morphology of $\alpha$-$\text{Al}_5\text{Fe}_3\text{Si}$ phase exerts positive impact on the mechanical properties of the alloy [5]. The multi-component solute atoms of Cu, Mg, Ni and Fe with similar atomic radii difference with aluminium exert a substitutional solid solution strengthening mechanism in the alloy. These are the reason behind 212.8 MPa of tensile strength, 165.7 MPa yield strength and 1.6 elongation percentage in the gravity die cast sample S1 in as-cast condition.

On increasing Cu and Fe content in sample S2, increases the tensile strength by 3.8% and yield strength by 3.43 % with 64.38 % loss of ductility. Increasing copper in the sample S2 in the presence of 1.07 wt.% iron content, modify and disperse the iron bearing intermetallic phase in the solid solution [4,8]. This restricts dislocation motion and thus improves the tensile strength [8,9] of the material by solid solution strengthening mechanism. Decrease in the percentage elongation to fracture clearly states the loss of ductility of the alloy as Cu and Fe increases. Because, eutectic Cu containing phase exert negative influence on plasticity [5].

With higher iron (1.34 wt.%) and copper (6.77 wt.%) content in the sample S3 introduces new peaks of AlFeNi and increase $\text{Al}_3\text{Cu}_5\text{Fe}$ and $\text{Al}_3\text{Cu}_4\text{Ni}$ phases relatively, which marginally improves the tensile strength and a small drop in yield strength of the sample S3 compared to S2, even though copper content increases two fold. Drop in yield strength of the sample S3 may be due to increase in
the β-Al₅FeSi phase, which impedes the flow of molten aluminium in interdendritic regions and leads to shrinkage porosity. The micro porosity counteracts the mechanical performance gain from the grain refinement by iron addition and eutectic network of copper bearing intermetallic phases.

In aluminium alloy systems, detrimental effects of iron are due to the formation of β-Al₅FeSi phase, which acts as a point of stress riser. This deteriorates the mechanical properties of the alloys. Copper reduces the temperature of iron bearing eutectic phase reactions. This decreases the amount of iron in solid solution [5]. This supports the formation of relatively ductile α-Al₈Fe₂Si phase. Also, Cu and Ni in the alloy system forms iron containing intermetallic phase (Al₇Cu₂Fe and Al₉FeNi). Similarly, Mg in the Al-Si-Fe system consumes iron through the formation of a relatively large iron and silicon containing π-Al₈FeMg₃Si₆ phase [10]. Thus, the iron in the solid solution decreases; this suppresses the transformation of α-Al₈Fe₂Si to β-Al₅FeSi phase or delay the formation of hard plate like β-Al₅FeSi in the alloy.

The critical level of iron for 4 wt.% Si in the aluminium alloy system is only 0.25 wt.% with reference to John A. Taylor [4]. But, in this experimental alloys, the formation of β-Al₅FeSi is visible with relative intensity of 2.6% only at 1.07 wt.% of iron (Sample S2). There is no evidence of presence of β-Al₅FeSi phase in the sample S1 with reference to the XRD report shown in the figure 3(S1).

![Impact Strength of as-cast experimental alloys](image)

**Figure 5.** Impact strength of as-cast experimental alloys.
3.3. Impact strength

Notched-bar impact test used to evaluate energy absorbed during fracture, which is termed as toughness. Toughness is directly related to the ductility. Ductile material absorbs more energy and thus high toughness. The impact strength of the experimental alloys is shown in the figure 5. Increase in Cu and Fe in the Al-Si-Ni-Mg system of alloys marginally (2.7%) decreases the impact strength alloy, in spite of Cu-Ni combination in the aluminium alloy have the tendency to increase the tensile and impact strength. The following reasons may be behind the loss of toughness; while copper content in the experimental alloy increases; Al$_2$Cu inter-metallic phase increases in the grain boundary. This increases the hardness with expense ductility and impact strength. Similarly, iron in the experimental alloys, increases the complex iron bearing hard inter-metallic phases, this may reduce the toughness of alloys. Especially, at higher iron level in the alloy (Sample S3), higher volume fraction of plate like β-Al$_5$FeSi phase was observed and it conformed with reference to micrograph (figure 2(e,f)) and XRD (figure 3), this increases the shrinkage porosity in the alloy, which has strong negative influence on ductility of the alloy, consequently there is a loss in impact toughness.

3.4. Practical guide to iron pick-up from crucible and tooling

\[ Fe_t \approx 0.019 (Tp - Tm) + Fe_i \]  

Where
- \( Tm \): Time to reach melting point in Minute
- \( Tp \): Cumulative time to pour the melt in Minute
- \( Fe_i \): wt.% of iron from the raw material
- \( Fe_t \): Total expected iron pick-up in wt.%

A mathematical model was proposed (equation (1)) to find the expected iron pick-up from mild steel crucible and tooling (in wt. %) with respect to melting rate, holding time (cumulatively) and initial Fe content (wt.%) from raw materials.

4. Conclusions

Effect of combined addition of Cu and Fe on the microstructure and mechanical properties of gravity die-cast Al-Si-Ni-Mg alloy was investigated and the following conclusions were confirmed from the results.

- Fe-rich intermetallic phases have a strong influence in modifying and refining the dendritic morphology of α aluminium matrix.
- Addition of Cu and Fe in as cast Al-Si-Ni-Mg alloys increases ultimate tensile strength. Yield strength increases marginally then decreases. Relative percentage elongation and impact strength of the alloy decreases. Grain refinement was observed during the increase of Cu and Fe.
- Cu content from 2.98 wt.% to 6.77 wt.% and iron wt.% from 1.07 wt.% to 1.34 wt.% increases detrimental β-Al$_5$FeSi phase and subsequently more shrinkage porosity. This counteracts the mechanical performance gain from the grain refinement by iron addition and eutectic network of copper bearing intermetallic phases.
- The source of iron, from secondary alloying elements and iron pick-up from mild steel crucible and tools, is the natural reason behind the Fe content in Al alloys. This reduces the cost of raw materials, low cost tooling and thus overall cost of the Al product decreases. This makes the process environment friendly and fits for small scale aluminium foundry.
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