Grain refinement of Al-Si$_{9.8}$-Cu$_{3.4}$ alloy by novel Al-3.5FeNb-1.5C master alloy and its effect on mechanical properties

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Abstract. A novel Al-3.5FeNb-1.5C master alloy with uniform microstructure was prepared using a melt reaction process for this study. In the master alloy, basic intermetallic particles such as NbAl$_5$, NbC act as heterogeneous nucleation substrates during the solidification of aluminium. The grain refining performance of the novel master alloy on Al-Si$_{9.8}$Cu$_{3.4}$ alloy has also been investigated. It is observed that the addition of 0.1 wt.% of Al-3.5FeNb-1.5C master alloy can induce very effective grain refinement of the Al-Si$_{9.8}$-Cu$_{3.4}$ alloy. The average grain size of α-Al is reduced to 22.90 µm from about 61.22 µm and most importantly, the inoculation of Al-Si$_{9.8}$-Cu$_{3.4}$ alloy with FeNb-C is not characterised by any visible poisoning effect, which is the drawback of using commercial Al-Ti-B master alloys on aluminium cast alloys. Therefore, the mechanical properties of the Al-Si$_{9.8}$-Cu$_{3.4}$ alloy have been improved obviously by the addition of the 0.1 wt.% of Al-3.5FeNb-1.5C master alloy, including the yield strength and elongation.

1. Introduction
Some of the most important driving forces for the development of Al-Si cast alloys are the high strength to weight ratio, high fluidity, low shrinkage in casting, superior wear resistance, low coefficient of thermal expansion (CTE), high corrosion resistance and excellent castability which makes them potential materials for a number of applications in automobiles and other engineering sectors [1-3]. It is well known that the mechanical properties of aluminium alloys can be improved by means of grain refinement. Actually, this is quite well-established industrial practice for wrought aluminium alloys, where the refinement is done by means of Al-Ti-B master alloys [4-7]. The Al-Ti-B master alloy offers a remarkable performance in the continuous and semi-continuous casting of wrought alloys but fails to meet the expectations in the case of aluminium cast alloys, especially for the Al-Si alloys with a content of Si larger than 4% [8-10]. The reason for this drawback is that the Si in the melt will react with Ti to form titanium silicides which prevent the effective grain refinement of the alloy, a phenomenon known as poisoning [11-12]. By now, various attempts have been done to solve this problem [13-15]. One effective method is to reduce the Ti content in the mater alloys. Some new master alloys, such as Al-3Ti-3B, Al-1Ti-3B, Al-3B and Al-3Ti-1B-0.2C have been reported to provide better grain refinement performances than the Al-5Ti-1B master alloy [16-17]. However, these methods have not been adopted and grain refinement problems of Al-Si alloys are still unresolved in Al cast industry. To overcome this poisoning effect, recently Nb-B grain refiner can efficiently refine binary Al-xSi alloys due to the formation of niobium borides which are more stable than titanium.
borides, therefore, appreciably limiting the so called poisoning effect [18]. From an industrial point of view, availability and cost of material are very important. Due to adequate availability and less cast of Ferro niobium (FeNb) and Carbon powder (C), in this study Al-FeNb- C has been taken as grain refiner for grain refinement of α-Al of the commercial Al-Si9.8-Cu3.4 alloy by means of heterogeneous nucleation.

2. Materials and Methods
The raw materials employed in this study are: Pure Aluminium (99.5%), Ferroniobium metal (60% of Niobium), Carbon powder (99.5%) and Al-Si alloy (Al-Si9.8-Cu3.4). The required amounts of Aluminium, FerroNiobium and Carbon powder were prepared in order to fabricate an Al-3.5FeNb-1.5C master alloy. Aluminium was melted at 790 °C for 1 hour and, subsequently, the carbon powder was added to the melt and the temperature was increased to 850 °C. After 1 hour at 850 °C, FerroNiobium metal was added and left to dissolve for 2 hours with intermediate stirring. The microstructure of the Al-3.5FeNb-1.5C master alloy was analysed using Scanning Electron Microscopy (SEM)

The present study was performed on Al-Si9.8-Cu3.4 alloy with a chemical composition given in Table 1. A standard 230-tons cold chamber High Pressure Die Casting (HPDC) machine was used to fabricate the cast samples at 720 °C molten temperature. Prior to casting, the melt was treated with different weight percentages of the grain refiner such as 0.1 and 1.0 wt.% of Al–3.5FeNb–1.5C master alloy were added to the mix and then poured into a pouring hole of the HPDC machine to perform casting. After the Die casting experimentation process, the casted part has been taken from the die. Once cast, the samples were cut and their cross-sections prepared for metallographic analysis by using the standardized route of grinding with 120-1200 SiC papers plus polishing with OPS solution was employed. Macrotetched cross-section samples were etched by means of Tucker’s solution (15ml HF + 15ml HNO3 + 45ml HCl + 25ml H2O). The microstructural analysis was carried out with a Carl Zeiss Axioscope A1 optical microscope. Also, the microstructure and the extracted particles of the Al-3.5FeNb-1.5C master alloy were analyzed by X-ray diffraction (XRD) and Field Emission Scanning Electron Microscope (FESEM).

| Table 1: Chemical composition of Al-Si9.8-Cu3.4 alloy (wt.%). |
|-----------------|-------|-------|-------|-------|-------|-------|-------|
| Si              | Cu    | Fe    | Ti    | Pb    | Sn    | Al    |       |
| 9.839           | 3.474 | 0.189 | 0.184 | 0.043 | 0.013 | Balance |       |

3. Results and discussion

3.1 Microstructure and microanalysis of the Al-3.5FeNb-1.5C master alloy
Figure 1 shows the XRD results of the Al-3.5FeNb-1.5C master alloy. It has been seen that the master alloy mainly contains four kinds of phases: α-Al, NbAl3, NbC and FeC. These intermetallic particles (NbAl3 and NbC) act as a heterogeneous nucleation substrates during the solidification of Aluminium. Also NbC particles control the agglomeration of FeC particles. The microstructure of the Al-3.5FeNb-1.5C master alloy is shown in the Figure 2. It may be seen that the second phase particles are dispersed in the aluminium matrix homogeneously. As shown in Figure 2, the particles are not connected with each other and the size is various. Most of the particles are large such as NbAl3, NbC surrounded by lots of smaller particles.

3.2 Effect of FeNb-C inoculation in the binary Al-Si9.8-Cu3.4 alloy.
The microstructures of the Al-Si9.8-Cu3.4 alloy before and after the addition of Al-3.5FeNb-1.5C master alloys are shown in Figure 3. It may be seen from Figure 3(a) that the grain structure of the unrefined binary Al-Si9.8-Cu3.4 alloy is coarse with an average grain size of approximately 62µm. These coarse grains are obviously refined to small equiaxed ones by adding the Al-3.5FeNb-1.5C master alloy.
Notably, when inoculated with 0.1wt.% of Al-3.5FeNb-1.5C master alloy, the grains of the Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloys are very fine as shown in Figure 3(b) and uniform for the entire range of holding times, implying remarkable grain refining efficiency. After the addition of 0.1wt.% of Al-3.5FeNb-1.5C master alloy, the average grain size of α-Al is 22µm which is much finer than those refined by the 1.0wt.% of grain refiner. The microstructures of Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloy with the addition of 1.0 wt.% of Al–3.5FeNb–1.5C grain refiner are shown in Figure 3(c). From this microstructure, it could be understood that the grain sizes are again increased because of the formation of β-AlFeSi platelets which are cause to the formation of coarse grains. These platelets (or needles as they appear in the microstructure) often result in the formation of large shrinkage cavities due to the inability of the liquid metal to fill the spaces between the branched platelets. From these results, it can be concluded that the Al-3.5FeNb-1.5C master alloy has a much better grain refining performance particularly at 0.1 wt.% of grain refiner on Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloy; also it shows a strong anti-fading effect in Al-Si casting alloys.

**Figure 1. XRD results of the Al-3.5FeNb-1.5C.**  **Figure 2. SEM microstructure of Al-3.5FeNb-1.5C.**

### 3.3 Mechanical properties of the Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloy

The grain refinement effect on the mechanical properties of Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloy is investigated in this work, including the yield strength (YS) and elongation. The YS and elongation of the Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloys before and after addition of grain refinement are shown in Table 2 and Figure 4. As expected, the refined microstructure could improve the tensile properties of Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloy. Before the addition of grain refiner, the YS and elongation values are 186.706 MPa and 2.76% as shown in the Table 2. After applying 0.1 wt.% of Al-3.5FeNb-1.5C master alloy, the YS and elongation values are 204.73MPa and 4.48% for Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloy. From these results, it was cleared that the YS and elongation of the Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloys are significantly improved by the addition of grain refiner, particularly at 0.1 wt.% of Al-3.5FeNb-1.5C. After the addition of 0.1 wt. % of Al-3.5FeNb-1.5C master alloy, that is increased from 186 MPa to 204 MPa. That is to say that the average value of the YS is increased by 9.7%. It is also worth noticing that the refinement using 0.1wt.% of Al-3.5FeNb-1.5C could double the elongation of Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloy than that of without refinement. But the YS and elongation of the Al-Si\textsubscript{9.8}-Cu\textsubscript{3.4} alloys are decreased instead of improved by the addition of grain refiner, particularly at 1.0 wt. % of Al-3.5FeNb-1.5C, because of the formation of β-AlFeSi platelets which causes to formation of the shrinkage cavities due to the inability of the liquid metal to fill the spaces between the branched platelets.
Table 2. Grain size and mechanical property data of Al-Si$_{9.8}$-Cu$_{3.4}$ cast alloy

| Wt.% of Grain refinement | Grain size ($\mu$m) | Mechanical property |
|--------------------------|---------------------|---------------------|
|                          |                     | Yield Strength (MPa) |
|                          |                     | Elongation (%)       |
| Without grain refiner    | 61.22               | 186.706              |
|                          |                     | 2.76                 |
| 0.1 wt.% of grain refiner| 22.9                | 204.73               |
|                          |                     | 4.48                 |
| 1.0 wt.% of grain refiner| 43.07               | 191.874              |
|                          |                     | 3.36                 |

Figure 3. Optical microphotographs of as-cast Al-Si$_{9.8}$-Cu$_{3.4}$ alloy at 100X magnification: (a) untreated (without grain refiner), (b) 0.1 wt. %: of Al–3.5FeNb–1.5C, (c) 0.3 wt. % of Al–3.5FeNb–1.5C.

Table 2 and Figure 4 also show the change of YS and elongation of the Al-Si$_{9.8}$-Cu$_{3.4}$ alloy after the addition of 1.0 wt. % of the Al-3.5FeNb-1.5C master alloy. Finally, it is worth noting that the sample with 0.1 wt. % of the Al-3.5FeNb-1.5C master alloy addition possesses the highest YS and % elongation. The result indicates that the grain refinement has resulted in a very evident improvement in the ductility of the Al-Si$_{9.8}$-Cu$_{3.4}$ alloy.
Figure 4. Yield strength and Elongation of Al-Si9.8-Cu3.4 at room temperature: (a) without addition of master alloy; (b) addition of 0.1 wt. % Al-3.5FeNb-1.5C; (c) addition of 1.0 wt. % Al-3.5FeNb-1.5C.

4. Conclusions
In the present work, a novel Al-3.5FeNb-1.5C master alloy with homogeneous microstructure was prepared and its effects on the microstructure and mechanical properties of Al-Si9.8-Cu3.4 alloy were studied. Based on the experimental results, the conclusions have been drawn as follows:

- In the novel Al-3.5FeNb-1.5C master alloy, basic intermetallic particles such as NbAl3, NbC act as heterogeneous nucleation substrates during the solidification of Aluminium. Also, NbC particles control the agglomeration of FeC particles.

- Significant grain refinement performance of novel Al-3.5FeNb-1.5C master alloy in Al-Si9.8-Cu3.4 alloy was observed, particularly at 0.1 wt. % of Al-3.5FeNb-1.5C master alloy. The average grain size of the α-Al in the Al-Si9.8-Cu3.4 alloy is reduced to 22.90 µm from 61.22 µm by the addition of 0.1 wt. % of Al-3.5FeNb-1.5C master alloy.

- The mechanical properties of the Al-Si9.8-Cu3.4 alloy have been obviously improved by the addition of the 0.1 wt.% of Al-3.5FeNb-1.5C master alloy. The yield strength of the Al-Si9.8-Cu3.4 alloy is increased by 9.7%, as well as the average value of the percentage elongation is increased from 2.76% to 4.48%.

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