SEM and EDAX Evaluation of Al-Fe Alloy

Arthur Jebastine Sunderraj D, Arun Vasantha Geethan K, Ananthapadmanaban D

Abstract: ***The scanning electron microscopy (SEM) and energy dispersive X-Ray analysis of the Al-Fe alloy revealed the formation of varying ironaluminide intermetallic compounds. The Al-Fe alloys were produced by varying the composition of Fe. The pure 'Al' billets were heated up to 750^{o}c and in the molten condition of aluminium 'Fe' powder was added resulting in an exothermic reaction. The exothermic reaction raised the molten bath temperature to ~ 1400^{o}c which activated the formation of intermetallic compound.***

Keywords: Intermetallics, ironaluminide, microstructure.

I. INTRODUCTION

Aluminium is alloyed with elements like 'Cu', 'Si', 'Mg', 'Zn', 'Fe' and other elements. . Addition of excess 'Al' in 'Fe' results in the formation of intermetallic compounds such as FeAl, Fe_3Al, $FeAl_2$ and $FeAl_3$. Presence of intermetallic compounds and oxides reinforces the softer matrix aluminum and increases the mechanical properties. These intermetallic compounds exhibit neither metallic nor ceramic properties. Iron aluminides are compounds formed between 'Fe' and 'Al'. The phase diagram drawn between Al-Fe reveals the formation of intermetallic compounds like Fe_3Al, FeAl, $FeAl_2$, Fe_2 Al_5 and $FeAl_3$. On the other hand, Fe_3Al and FeAl, which have a high iron composition, are used as structural materials because of their good wear resistance, oxidation resistance, corrosion resistance and specific strength properties. The presence of a phase can impede grain boundary motion yielding better mechanical properties. The Fe_3Al-based iron aluminides were of interest because of their low cost, low density, ease of fabrication, good wear resistance, and resistance to oxidation and corrosion resistance properties [1]. Pithawala et al [2] reported the synthesis of intermetallic FeAl nanoparticles using the laser vaporization controlled condensation technique and observed that nanoparticles of iron aluminides enhanced the room temperature ductility and the high temperature strength. Chakraborty et al [3] performed reduction of oxides to obtain an alloy having a composition of Fe–15.2% Al–4.9% Cr–0.48% V–0.048% C which was very close to the targeted composition. The maximum overall yield of 99.4 wt % was achieved by using optimum reaction parameters and the alloys possessed reasonably good ductility and good oxidation resistance. The objective of the present study is to understand the effect of 'Fe' addition in commercially pure 'Al' on mechanical properties and matallurgical characteristics.

Revised Manuscript Received on October 15, 2019

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II. EXPERIMENTAL PROCEDURE

2.1 Material

The commercially pure wrought Aluminium rod of 20 mm in diameter having 99.5 % purity was purchased from the market. Small billets of 20 mm and 10 mm in length were cut in power saw. Iron powder having 99.9 % purity was purchased from the market. This powder was supplied in a vacuum sealed container.

2.2 Experimental setup

A muffle furnace of 380 mm in length and 180 mm in width is used in the present work. The temperature of the furnace can be raised upto 1000 ºC. The heating rate of this furnace can be varied. Small crucibles made with refractory clay were used for melting the aluminium metal. The experimental setup is shown in Figure 1

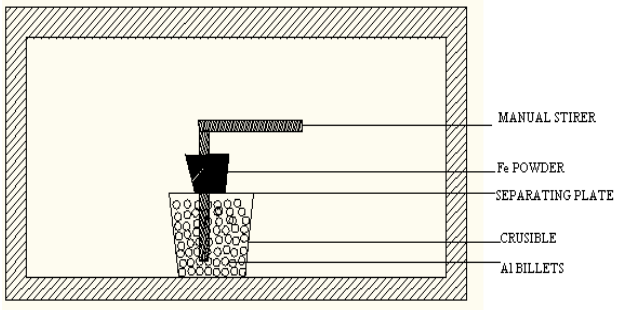

Figure 1 Experimental setup.

2.3 Experimental procedure

The billets were turned on the outer surface to remove oxide layer from the surface. After this chemical cleaning was performed using HCl and NaOH. The 'Al' billets were weighed according to weight percentage requirement and placed inside the crucibles and heated inside the furnace. The crucibles were heated upto 750 ºC and kept inside the furnace for 1 hour. In the molten 'Al', 'Fe' powder was added by removing the separating plate to initiate exothermic reaction. Because of this exothermic reaction the temperature inside the crucible was raised to 1400 ºC. After mixing with 'Fe' powder, the crucibles were kept inside the furnace at 750 ºC for two hours. Further, annealing heat-treatment was performed for 19 and 16 hours at 550 ºC. The order of experiments is shown in the Table 1.

Table 1 Composition of Al and Fe and annealing temperature.

S.No	Composition in wt. percentage	Melting temperature	Annealing temperature 600°C and time in h
1	80 % Al and 20% Fe	550°C and soaking at this temperature for 2h	19

2	80 % Al and 20% Fe	550 °C and soaking at this temperature for 2h and mechanically agitated	16
3	75 % Al and 25% Fe		16
4	65 % Al and 35% Fe		16
5	70 % Al and 30% Fe		16
6	60 % Al and 40% Fe		16

The specimens obtained from the furnace were machined in a lathe to obtain circular discs as shown in Figure 2 (a) and (b). Square metallographic specimens of 6 × 6 of 20 mm length and shear test specimens of 4 mm diameter cylinders of 20 mm in length were cut using wire-cut electric discharge machining technique. The WEDM specimens are shown in Figure 2 (c). The metallographic samples were mechanically polished using various grades of silicon carbide paper to obtain mirror finish. These samples were finished with $Al_2 O_3$ slurry to obtain mirror finish without any scratch marks. The polished specimens were etched with a chemical mixture containing 5 ml HCl + 10 ml HNO_3+3 ml HF+ 50ml H_2O for 1minute. The specimens were rinsed in water and dried using a drier.

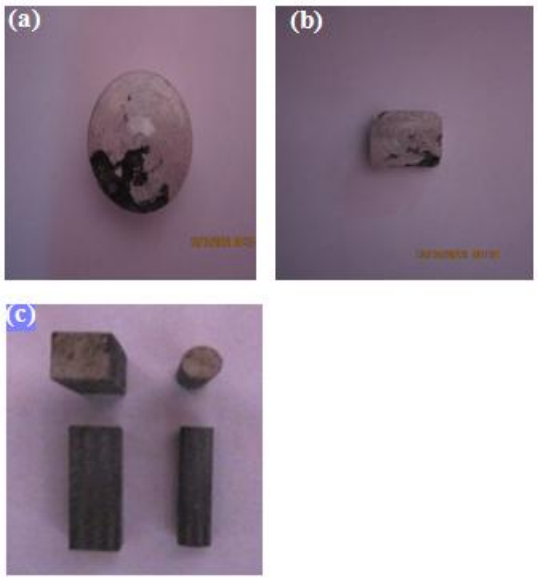

Figure 2 (a)and (b) Shows Al-Fe alloy samples turned in a lathe and (c) shows wire-cut EDM samples.

III. RESULT AND DISCUSION

3.1 Optical microscopy

The optical micrographs shown in Figure 3 (a)-(d) corresponds to the samples 1, 3, 5 and 6 as shown in Table 1. In Figure 3 (a) a combination of large Aluminium grains and fine grains are seen. The grain boundaries triple points are rich with 'Fe' and thick grain boundaries are observed. Adjacent to the grain boundaries on either side fine Aluminium grains are seen. The recrystallized zone adjacent tot the grain boundaries reveal diffusion of 'Fe' towards the grain boundaries and coarsening of the grain boundaries. At the grain boundary triple points presence of more amounts of dark particles is attributed to grain boundary diffusion mechanism playing a role in the formation of interemtallic compounds due to diffusion of more amounts of 'Fe' atoms.

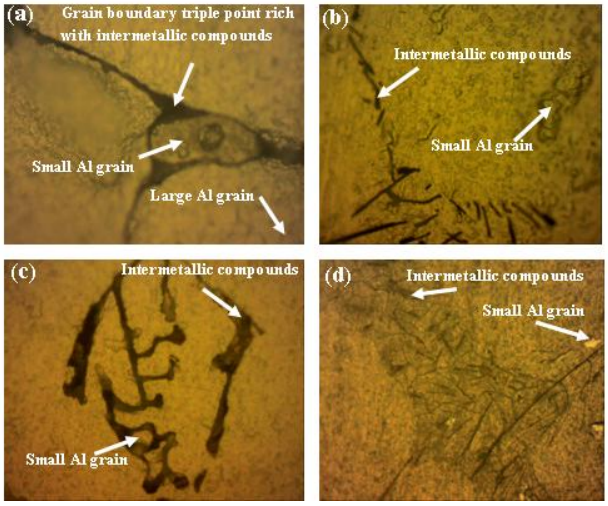

Figure 3(a) Optical micrograph for the sample1 at 1000X, (b) for the sample 3 at 500X, (c) for the sample 5 at 1000X and (d) for the sample 6 at 200X.

The optical micrograph shown in Figure 3 (b) discloses small Aluminium grains at few places and in the other regions the grain boundaries are not visible. The intermetallic compounds are present as elongated steaks in the Aluminium matrix. These compounds have grown as elongated particles and are segregated at few places. In the Figure 3 (C), rib cage pattern is noted which is rich with intermetallic compounds. This image is taken at 1000X revealing fine grains of aluminum embedded in the interemtallic compounds. This clearly reveals diffusion of 'Fe' atoms towards the grain boundaries and subsequent of growth of these atoms to intermetallic compounds as fine grains. This micrograph reveals presence of fine Aluminium grains close to the rib cage like structure in the micrograph. In Figure 3 (d) at few places bright fine Aluminium grains are seen. intermetallic compounds are dispersed as fine elongated needles in the Aluminium matrix. At the centre a band of grey region is observed which consists of fine grains of Aluminium and along the grain boundaries dark particles are present. These dark particles are seen as elongated needles rich with intermetallic compounds reinforcing the soft Aluminium matrix.

3.1 Scanning Electron Microscopy

The SEM micrograph for the sample 1 is shown in Figure 4 (a). The whit particles present in the micrograph are presumed to be rich with oxides and intermetallic compounds. Below this cloud of white particles presence of fine grains of Aluminium is noted. This structure resembles like a cellular structure. In the Figure 4 (b) deeply etched regions are rich with intermetallic compounds. The hydrofluoric acid etches oxides and intermetallic compounds readily than pure metals. Therefore, the elongated dark regions are rich with iron aluminide intermetallic compounds.

In these heavily etched elongated regions alternate layers of dark and white phases are noted. These phases reveal the presence of various intermetallic compounds such as Fe_2Al, Fe_3Al, FeAl, $FeAl_5$ and Fe_2Al_3. the islands are rich with fine Aluminium grains. The white lines are grain boundaries of Aluminium grains. These grains are 2-3 µm in size.

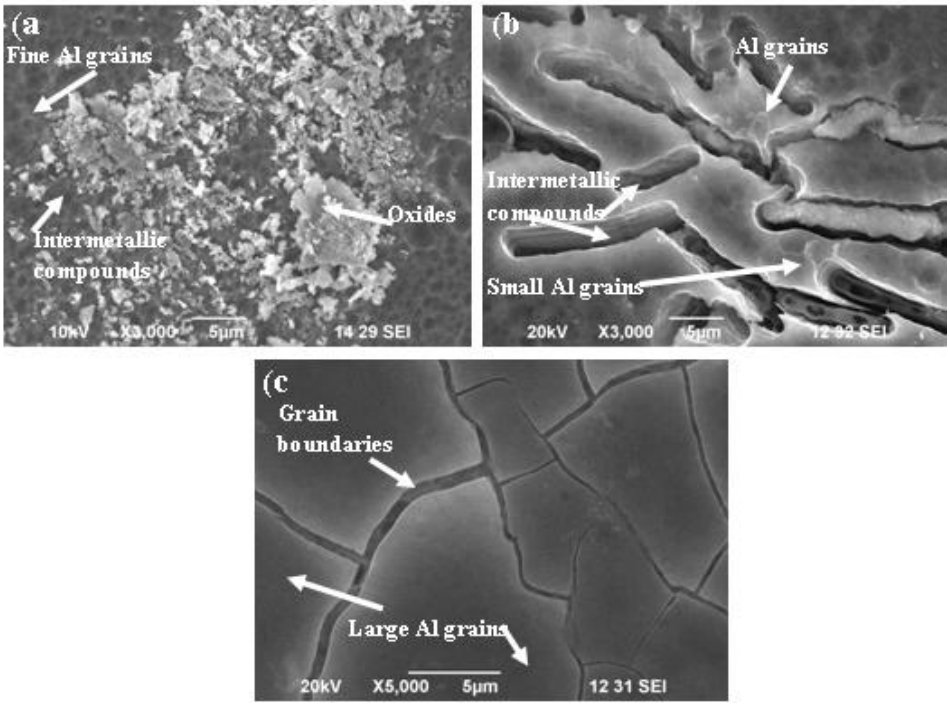

Figure 4(a) SEM image for the sample1, (b) for the sample 5 and (c) for the sample 6.

Large Aluminium grains are seen in the micrograph shown in Figure 4 (c). The very thick grain boundaries and the thin grain boundaries seen in this micrograph reveal the presence of varying amounts of 'Fe' diffusion into the grain boundaries. Also, the presences of 'Fe' based compounds are less compared to the compounds seen in the other two micrographs. At few places in the small grains dark spots are noted, these spots are presumed to be iron aluminides. Also, at few places presence of white compounds are observed in the SEM micrographs. These compounds are oxides and intermetallic compounds formed between Aluminium and iron.

3.2 Energy dispersive X-Ray analysis

The EDX analysis was carried out to determine the elemental composition of the various samples. The concentration profiles of various elements are shown in the graphical plot in Figure 6. Though, the alloys were produced with more amounts of 'Fe' (refer Table 1) but the concentration 'Fe' was less in all the samples. The concentration profiles shown in the Figure 5 of various alloys drawn using the original EDAX reports have confirmed this. The lesser amount of 'Fe' present at a point was attributed to the point scan taken at a point where dilution of 'Fe' has taken place in that region. The hard intermetallic compounds formed were not bonded to the molten Aluminium during the solid-liquid reaction phase. The maximum amount of 'Fe' present in an alloy is only 1.8 %. The element 'Fe' is dissolved in solid solution at the point where EDAX examination is carried out. The optical (see Figure 3 (a)-(d)) and SEM micrographs (see Figure (a) and (b)) reveal the presence of elongated needle like 'Fe'rich intermetallic compounds. The EDAX reports and the graph shown in the Figure 5 confirm the presence of elements like 'Mn', 'Si' and 'O'. The elements like 'Fe', 'Si', 'Mn' and 'O' had strengthened the soft Aluminium matrix by imparting solid solution strengthening and dispersion strengthening effect. The intermetallic compounds such as Fe_2Al, Fe_3Al, FeAl, $FeAl_5$ and Fe_2Al_3 and secondary phase particles like Al_2O_3 and FeO2 had imparted dispersion-strengthening effect.

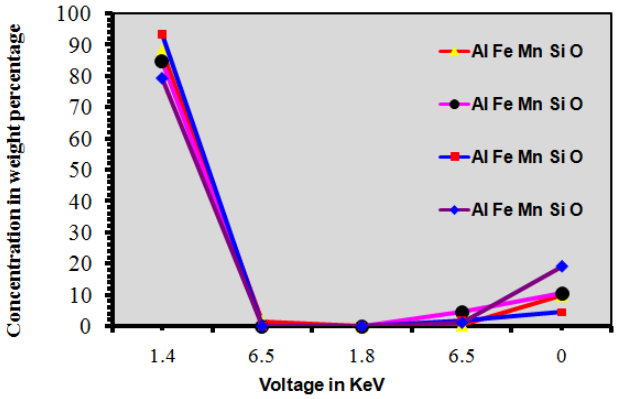

Figure 5 shows the EDAX graph of sample 1,3,4,5

IV. CONCLUSIONS

The EDAX and XRD analyses of the alloy samples confirmed the presence of compounds such as Fe_2Al, Fe_3Al, FeAl, $FeAl_5$, Fe_2Al_3, Al_2O_3 and FeO_2.

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