Effect of Melting Process and Aluminium Content on the Microstructure and Mechanical Properties of Fe–Al Alloys

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This paper presents the effect of air induction melting with flux cover (AIMFC) versus vacuum induction melting (VIM) on the recovery of alloying element, reduction of impurities, workability and mechanical properties of Fe–(7–16 mass%)Al alloys. Three Fe–Al alloy ingots containing 7, 9 and 16 mass% Al were prepared by both AIMFC and VIM. All these ingots were hot-forged and hot-rolled at 1 373 K and were further characterized with respect to chemical composition, microstructure and mechanical properties. The recovery of aluminium as well as reduction of oxygen during both AIMFC and VIM is excellent. AIMFC ingots exhibit low level of sulphur and high concentration of hydrogen as compared to VIM ingots. VIM ingots of all the three alloys were successfully hot worked. However, AIMFC ingots of only those Fe–Al alloys containing lower concentration of aluminium could be hot worked. The tensile properties of hot-rolled Fe–7 mass%Al alloy produced by AIMFC and VIM are comparable. The present study clearly demonstrates that it is feasible to produce sound ingots of low carbon Fe–7 mass%Al alloy by AIMFC process with properties comparable to the alloy produced by VIM.

KEY WORDS: air inducting melting with flux cover; vacuum induction melting; Fe–Al alloy; microstructure; mechanical properties.

1. Introduction

Iron aluminides have been the most widely studied intermetallics due to their excellent resistance to oxidation and sulphidation at high temperatures, low density and low cost.1,2 Iron aluminides based on ordered Fe₃Al are potential low cost replacement for heat resistant austenitic and ferritic steels. However their potential use as structural material has been hindered by limited ductility at room temperature, low toughness and poor machinibility.3–5 The environmental effect associated with the formation of hydrogen from the reaction of aluminium in the alloy with moisture in the air has been identified as the primary cause for the poor ductility at room temperature.4–6 Recently it has been reported that the reduction in Al-content to 8.5 mass% resulted in enhanced room temperature ductility.6–8 The improvement in ductility has been mainly attributed to a reduced susceptibility to environmental embrittlement and presence of disordered structure.7–10 Iron aluminides are considered to be process sensitive and are being produced in small quantities employing techniques like arc melting, drop casting, rapid solidification followed by powder compaction.11–14 Though these techniques give high quality material, they are not cost effective and not amenable for bulk production. The air induction melting (AIM) of iron aluminides present difficulties in the form of interaction of aluminium in the alloy with the moisture in the atmosphere to form hydrogen-induced porosity. This problem can be avoided by vacuum induction melting but the material becomes expensive. It has been reported that use of flux cover during air induction melting of Fe–(9–16 mass%)Al–(0.05–1 mass%)C alloys results in elimination of hydrogen gas porosity. However, at low levels of carbon, ingots exhibit micro cracks due to the presence of higher concentration of residual hydrogen.13,14 Therefore, a minimum amount of carbon is essential to produce sound ingots of Fe–(9–16 mass%)Al alloys completely free from micro cracks. This paper describes the effect of melting process such as air induction melting with flux cover and vacuum induction melting on chemical composition, microstructure workability and tensile properties of low carbon Fe–Al alloys containing 7, 9 and 16 mass% Al.

2. Experimental Procedure

Thirty-kilogram melts of three Fe–Al alloys containing 7, 9 and 16 mass% Al (all compositions in mass% unless otherwise specified) were prepared in a 50 kg capacity, Inductotherm make, 125 kW, medium frequency air induction melting furnace with flux cover (AIMFC). A schematic diagram of air induction melting furnace with flux cover (AIMFC) is shown in Fig. 1. The furnace consists of an alumina lined crucible of approximately 150 mm diameter and 300 mm height. Soft iron scrap and commercial grade aluminium were used as raw materials. The chemical composition of the raw material is given in Table 1. The surface of the soft iron scrap was cleaned by pickling in dilute hydrochloric acid. The cleaned iron pieces
were melted in the crucible. After melting, the flux was skimmed off. A preheated alumina-based slag containing (11–17 mass%) CaF₂, (26–32 mass%) CaO, (6–8 mass%) MgO and (49–59 mass%) Al₂O₃ was added to provide a protective cover to the melt (Fig. 1). Approximately 400 g of slag was added to get a typical molten slag thickness of about 4 mm. The flux composition was carefully chosen to ensure that it does not attack the crucible lining of the melting crucible. During this melting the flux is kept molten to allow charging of aluminum. Pre-heated aluminium pieces were then plunged into the melt. The melt which was typically of 240 mm height was held for a very short time to minimize loss of aluminum. The flux does not take part in heat generation and is kept molten through heat transfer from the molten metal beneath and due to the heat release by the highly exothermic reaction between iron and aluminum. Therefore the flux quantity is limited to an amount which is just sufficient enough to provide adequate cover during charging and melting of aluminum. Due to the large density difference between molten metal (6 700 kg/m³) and molten slag (2 860 kg/m³), the molten slag always remains above the molten metal during melting and also during pouring. When the temperature of the melt reaches 1 893 K, the melt was top poured into 80 mm diameter split cast iron moulds.

Alloys with the same composition using the same raw material were melted in 50 kg capacity, 125 kW, medium frequency twin chamber vacuum induction furnace of ‘G.C.A make’ USA with vacuum level of 10⁻⁴ torr. The furnace consists of an alumina lined crucible of approximately 150 mm diameter and 300 mm height. A schematic diagram of vacuum induction melting furnace is shown in Fig. 2. The soft iron pieces were charged at the bottom of the crucible with aluminium pieces on top of these iron pieces in an alumina-lined crucible. The furnace was evacuated to a vacuum level of 10⁻⁴ torr and power was switched on. When the temperature of the melt reaches 1 893 K, the melt was poured into 80 mm diameter split cast iron moulds which was kept inside the vacuum chamber.

The chemical composition of AIMFC and VIM ingots was determined. Aluminum, silicon and manganese were determined by wet chemical methods. The analysis of carbon and sulphur was carried out by CC-444 Leco Carbon and Sulphur Determinator. Oxygen and nitrogen analysis was done by TC-136 Leco Oxygen and Nitrogen gas Analyser. Hydrogen analysis was done by RH-444 Leco Gas analyzer.

The cast ingots of AIMFC and VIM cast ingots were tested for their soundness in a radiography unit using 50 curie ⁶⁰Co γ-ray source. The AIMFC and VIM ingots were preheated in hearth furnace at 1 373 K for 1 h and hot-forged in one tonne pneumatic forge hammer with die platens at room temperature. The 24-mm thick sections of the forged billets were preheated at 1 373 K for 1 h and hot-rolled by 50% with 1-mm reduction in thickness per pass. The samples were reheated after every six rolling passes under similar conditions as in preheating. The final thickness of the rolled plate was 12-mm.

Longitudinal sections of the plates were cut using a high speed abrasive cut-off wheel and mechanically polished to 0.5-μm grid diamond powder finish and etched with an etchant comprising 33% HNO₃+33% CH₃COOH+33% H₂O+1% HF by volume for examination by optical microscope. The grain size measurement was carried out using the mean linear intercept method.

The bulk hardness measurements were made on the hot-rolled microscopy samples using a Vickers hardness machine with a 30 kg load. Longitudinal tensile specimens of 4.0-mm gauge diameter and 20-mm gauge length conforming to ASTM-E 8M were machined from hot-rolled AIMFC and VIM plates. These specimens were subsequently, polished using 600-grit. Tensile tests were carried out at room temperature on an Instron 5500R Universal Testing Machine at strain rate of 8.3×10⁻⁴/s.

### 3. Result and Discussion

Radiography showed that all the three alloy ingots produced by both AIM with flux cover (AIMFC) and VIM are free from gas porosity. Ingots of Fe–Al alloys containing higher aluminium produced by AIM are known to exhibit extensive hydrogen induced gas porosity. This has been attributed to the hydrogen liberated by the reaction between

| Raw material          | Composition (mass %) |
|-----------------------|-----------------------|
| Soft iron Scrap       | - 0.11 0.05 0.013 0.016 0.017 0.012 |
| Commercial grade Al   | 99.86 0.06 - - - - - |

Table 1. Chemical composition of raw materials.
aluminium in the melt with atmospheric moisture. Literature on processing of iron aluminide suggests that these alloys are generally melted under argon atmosphere or vacuum, which reduces chances of hydrogen pick-up, and hence gas porosity. The absence of gas porosity in the ingots produced with flux cover in the present work may therefore be attributed to the presence of protective flux cover which prevented the aluminium in the melt from coming into contact with the environmental moisture thereby suppressing formation of hydrogen.

The recovery of aluminium is excellent (95–98%) during both AIMFC and VIM. The recovery of aluminium (95–98%) during AIMFC is comparable to the recovery of aluminium (96–98%) during VIM. The excellent recovery of aluminium during VIM is well understood and the recovery of aluminium during AIMFC may be attributed to the flux cover and alumina present in the flux cover. AIMFC ingots exhibit low levels of sulphur as compared to VIM ingots and it may be attributed to desulphuring action of CaO present in the flux. The sulphur removal takes place by the following slag–metal reaction:

$$[S] + (CaO) = (CaS) + [O]$$

The equilibrium constant $K$ for this reaction is given by

$$K = \frac{a_{(S)}}{a_{(O)}} \times \frac{a_{(CaS)}}{a_{(CaO)}}$$

$$\frac{a_{(S)}}{a_{(O)}} = \frac{1}{K} \times \frac{a_{(CaS)}}{a_{(CaO)}}$$

where $a_{(S)}$ activity of sulphur in the metal, $a_{(O)}$ activity of oxygen in the metal, $a_{(CaS)}$ activity of sulphur in the slag, $a_{(CaO)}$ activity of oxygen in the slag.

It can be seen that the sulphur content of the metal is directly proportional to the concentration of the oxygen in the metal and inversely proportional to the basicity of the slag. The flux used in the present work does not have any acid oxide component and major component taking part in the desulphurization reaction are CaO and MgO. The total concentration of CaO and MgO put together is approximately 40 mass% while the remaining components in the slag (CaF₂ and Al₂O₃) are neutral. The very low level of sulphur in the AIMFC ingot may be attributed to (a) very low value of $a_{(O)}$ due to the presence of high concentration of aluminium in the alloy and (b) very high sulphide capacity of the slag due to the presence of CaO and MgO in the slag.

The oxygen level in the alloys produced by AIMFC is very low and comparable to VIM alloys. The presence of slag cover also prevents the melt from further oxidation. Consequently a very low level of oxygen was achieved by AIMFC. It can also be seen from the Table 2 that AIMFC ingots exhibited higher concentration of hydrogen compared to VIM ingots. Although the flux cover during AIM did prevent the formation of hydrogen gas porosity, it could not totally prevent the pick-up of hydrogen as the melt was exposed to the atmosphere during pouring and subsequent solidification. The reaction between highly reactive aluminium in the liquid metal with moisture in the air may be responsible for the formation of hydrogen as follows

$$3H₂O + 2Al = Al₂O₃ + 6[H]$$

The hydrogen gas thus liberated by this reaction gets dissolved in the liquid metal. The results presented in Table 2 indicate that alloys containing higher aluminium levels exhibited greater concentration of hydrogen. This may be attributed to the formation of higher concentration of hydrogen and its subsequent pick-up by the metal with increase in aluminium content as per the above reaction.

AIMFC ingots of Fe–7mass%Al alloy and VIM ingots of Fe–7mass%Al, Fe–9mass%Al and Fe–16mass%Al alloy were successfully hot forged and hot rolled whereas the hot working response of AIMFC ingots with Fe–9mass%Al and
16mass%Al was poor. Surface as well as internal cracks formed during hot forging and further hot rolling. These cracks may be attributed to the presence of higher concentration of residual hydrogen present in these as cast ingots.

Optical micrograph of all three hot rolled alloys produced by AIMFC and VIM process is shown in Fig. 3. All the alloys produced by both AIMFC and VIM process exhibit annealed microstructure with coarse grain size (Table 3). The grain size of all the alloys produced by AIMFC is comparable to the grain size of alloys produced by VIM process.

Table 3 summarises hardness and tensile properties of hot rolled AIMFC and VIM ingots of Fe–7mass%Al, 9mass%Al and 16mass%Al alloys. Each hardness data point reported here represents an average of 5 measurements and tensile data point represents an average of three tests. The hardness increases with increase in aluminium content and melting process has no influence on hardness. Alloys of same composition prepared by both AIMFC and VIM exhibited similar values of hardness (Table 3). No tensile test could be carried out on the rolled Fe–16mass%Al alloy produced by AIMFC because samples broke during fabrication where as samples from VIM ingot containing Fe–16mass%Al fractured at low tensile stress without exhibiting any significant plasticity. Though Fe–9mass%Al alloy produced by AIMFC is relatively free from environmental embrittlement effect, tensile samples failed at very low stress without exhibiting any significant elongation. The tensile properties of Fe–7mass%Al alloy produced by AIMFC are comparable to the alloy produced by VIM. This alloy also exhibited far superior room temperature ductility as compared to alloys containing higher aluminium.

The breaking up of AIMFC samples of Fe–16mass%Al alloy during fabrication and failure of AIMFC samples of Fe–9mass%Al alloy at very low tensile stress without exhibiting any tensile elongation may be attributed to the presence of micro cracks in the alloy. These micro cracks are as a result of hydrogen embrittlement due to the presence of high concentration of hydrogen (Table 2) present in the alloys. The poor tensile properties of Fe–16mass%Al alloys produced by VIM may be attributed to the environmental embrittlement (a) during fabrication of tensile samples by cutting and machining and (b) during tensile testing. The Fe–Al alloys containing greater than 8.5 mass%Al are known to have greater susceptibility to environmental embrittlement in the presence of atmospheric moisture.6,8) This embrittlement has been proposed to be due to the diffusion of nascent hydrogen liberated by the chemical reaction between aluminium present in the alloy and atmospheric moisture during tensile testing. The same reaction may also be responsible for the induced surface cracking during cutting and machining.

The hardness and room temperature strength of Fe–Al alloys produced by VIM increases with increase in aluminium content from 7–9 mass%. The increase in hardness and strength with the increase in aluminium may be attributed to solid solution strengthening. Further increase in aluminium content to 16 mass% results in increase in hardness and decrease in strength. Similar trend was observed in the case of carbon containing Fe–(9–16mass%)Al alloy.15)

It has been reported earlier that presence of carbon is essential to produce sound ingots of Fe–Al alloys by AIMFC process containing aluminium greater than about 9 mass%.8,16) The present study clearly demonstrates that AIMFC process can be successfully used to produce low carbon Fe–Al alloys when the aluminium is about 7 mass% with properties comparable to the alloys produced by VIM. The AIMFC process is less expensive and more universally available.
4. Conclusion

Effect of air induction melting with flux cover versus vacuum induction melting on recovery of alloying elements, reduction of impurities, workability and mechanical properties has been investigated.

(1) All the ingots of Fe–(7–16)mass%Al alloys produced by both AIMFC and VIM are sound and free from gas porosity.

(2) The recovery of aluminum during both AIMFC and VIM is excellent (95–98%).

(3) Oxygen level of all the three alloys produced by both AIMFC and VIM is very low (0.0021–0.0057 mass%).

(4) AIMFC ingots exhibited low levels of sulphur (0.0012–0.0016 mass%) and high concentration of hydrogen (0.00023–0.00068 mass%) as compared to the levels of sulphur (0.0026–0.0028) and hydrogen (0.00020–0.00028 mass%) in VIM ingots.

(5) AIMFC ingots of Fe–7mass%Al alloy and VIM ingots of all the three alloys were successfully hot worked. The hot working response of AIMFC ingots with Fe–9mass%Al and 16mass%Al was very poor.

(6) It is feasible to produce carbon free Fe–7mass%Al alloy by AIMFC process with properties comparable to the alloy produced by VIM.

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