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Processing of Fe-Al-C-Ce alloys through air induction melting with flux cover (AIMFC) and electroslag remelting (ESR)

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The effect of process parameters on recovery of reactive element, cerium, during air induction melting with flux cover (AIMFC) and electroslag remelting (ESR) of Fe-10.5 wt% Al-0.8 wt% C-(0.1 and 0.3) wt% Ce alloys and also the effect of melting techniques on hot workability, structure and tensile properties (at room temperature and at 873 K) of Fe-10.5 wt% Al-0.8 wt% C-0.3 wt% Ce alloy have been investigated. Good recovery of cerium was obtained by AIMFC. While conventional fluxes were found to be unsatisfactory, modified flux containing CeO_2 gave better recovery of cerium during ESR. The best recovery of cerium was achieved by using calcium as a deoxident during ESR. The combination of AIMFC and ESR yields a sound ingot of Fe-Al-C-Ce quaternary alloys free from gas and shrinkage porosity with very low oxygen, nitrogen and sulphur contents. Processing of AIMFC ingots through ESR has resulted in improved hot-workability. The ESR processed and hot-rolled alloy exhibited superior tensile elongation as compared to hot-rolled AIMFC alloy. This may be attributed to the comparatively sound, homogeneous and clean ingot, with a refined microstructure and fine uniform distribution of precipitates observed in hot-rolled ESR ingots. © *2002 Kluwer Academic Publishers*

1. Introduction

Fe_3Al based intermetallic alloys are being developed for elevated temperature structural application for temperatures up to 873 K [1–3]. They also provide potential replacement for the more expensive heat resistant alloys such as AISI 304, 310 and 316 stainless steels containing strategic elements like nickel and chromium. Although these binary Fe_3Al alloys can exhibit brittle behavior at room temperature, poor toughness, poor workability and sharp drop in strength above 873 K, improvement in these respects can be achieved by alloying addition and process control [3, 4]. The addition of Nb, Mo, to Fe_3Al alloy has significantly improved the strength. However, they resulted in poor room temperature ductility [5, 6]. Chromium was found to enhance the room temperature ductility with no effect on strength [7] and micro alloying of cerium has resulted in significant improvement in both yield strength and ductility [8–10]. Recently it has been shown that carbon may be an important alloying addition to iron-aluminium alloys containing 8–20 wt% Al [11–14]. The addition of carbon leads to improved strength, creep resistance, machinability and resistance to environmental embrittlement. These high carbon alloys also possess reasonable room temperature ductility. These improvements in properties were attributed to the formation of perovskite based $Fe_3AlC_{0.5}$ precipitates.

Successful commercialization of these alloys requires the application of processing techniques that can produce sound ingot without loosing the advantage of lower cost. Recently we have reported that the use of flux cover during air induction melting of Fe-Al alloys containing carbon using steel scrap as raw material results in the elimination of hydrogen gas porosity and significant reduction in impurity levels [15–17]. Subsequent processing of these ingots through ESR has resulted in further improvement in properties and hot workability [16]. This process route also exhibited excellent recovery of alloying elements like Al, Ti, Cr, Mn & Ni.

In this paper the effect of process parameter on recovery of reactive element, cerium, during air induction melting with flux cover (AIMFC) and electroslag remelting (ESR) of Fe-10.5 wt% Al-0.8 wt% C-(0.1 and 0.3) wt% Ce alloys and also the effect of melting techniques on hot working, structure and tensile properties of Fe-10.5 wt% Al-0.8 wt% C-0.3 wt% Ce alloy is reported.

2. Experimental procedure

Forty–kilogram melts of Fe-10.5Al-0.8C alloy containing 0.1 and 0.3% cerium were taken in an Inductotherm medium frequency air induction melting furnace of 50 kg capacity (all compositions are in wt% unless otherwise specified). Commercial purity aluminium, ferro-cerium and steel scrap were used as raw materials. The total amount of impurities (Mn, Si, P, Cu etc.)

present in the melt stock was about 0.7%. The surface of the steel scrap (mild steel) was cleaned by pickling in dilute hydrochloric acid. The mild steel scrap pieces were melted in an alumina-lined crucible by air induction melting. After melting, the slag was skimmed off. Graphite powder was then added to the melt. A preheated alumina-based flux containing CaF_2, CaO and MgO was added to provide a protective molten flux cover to the melt. Subsequently preheated aluminium pieces were plunged in to the melt. After melting aluminium, the ferro-cerium pieces covered in aluminium foil, were plunged into the melt. The melt was held for very short time to minimize aluminium and cerium losses and then top-poured into 55-mm diameter split cast iron moulds. Each melt has resulted in four ingots; each ingot weighed about 8–9 kg.

The electroslag remelting (ESR) was carried out in a 350 kVA ESR furnace. The flux was preheated and held at 1123 K for 2 h before use to remove moisture. The 55-mm diameter AIMFC ingots were remelted in 80-mm diameter water-cooled copper mould. At the end of the process, power supply was gradually reduced to impose a condition of hot topping. A majority of the ESR ingots weighed about 8–9 kg and had smooth surface finish. The chemical analysis of AIMFC and ESR ingots are shown in Table I. The experiments largely dealt with study of the following aspects in relation to the recovery of cerium during electroslag remelting of AIMFC ingots of Fe-10.5Al-0.8C alloy containing 0.1 and 0.3% cerium.

1. Effect of flux composition.
2. Effect of flux deoxidation.

The following two commercially used flux systems were chosen for ESR experiments

1. 70% $CaF_2 + 15\%$ $Al_2O_3 + 15\%$ CaO (70F/15/0/15)
2. 70% $CaF_2 + 30\%$ CaO (70F/30)

In addition to the above, the modified flux system (70% $CaF_2 + 30\%$ CaO) $+ 5\%$ CeO_2 was also used. This modified flux system was further deoxidized by continuous addition of Ca metal during ESR.

To study the effect of processing techniques on structure and properties, AIMFC 2 and ESR 2.4 ingots (Table I) were selected. The AIMFC 2 and ESR 2.4 ingots were tested for their soundness in a radiography unit using a 5 curie ^{60}Co γ-ray source.

The AIMFC 2 and ESR 2.4 ingots were held in the hearth furnace at 1373 K for 1 h and hot-forged in one tonne forge press with die platens at room temperature, to a forging reduction of 70%. The hot-forged 25-mm thick section were held in the furnace for 1 h at 1373 K and hot-rolled in a rolling mill to a rolling reduction of 50% with a reduction of 1-mm per pass. The samples were reheated after every six passes in the rolling mill. The final thickness of rolled plate was 14-mm.

Longitudinal sections of cast and hot-rolled AIMFC 2 and ESR 2.4 ingots were cut using bi-metallic band saw blade and high speed abrasive cut-off wheel. The cut-off sections were mechanically polished to 0.5 μm grade diamond powder finish and etched with an etchant comprising of 33% $HNO_3 + 33\%$ $CH_3COOH + 33\%$ $H_2O + 1\%$ HF by volume for microstructural examination by optical microscope. Scanning electron microscope (SEM) and electron probe micro analysis (EPMA) studies were carried out to determine the matrix and precipitate composition and to identify the phases present in the alloys. The microstructure of the samples were also studied in SEM. X-ray diffraction (XRD) studies were carried out in a Philips 3710 diffractometer on powder samples of cast and rolled ingots.

Longitudinal ASTM–E8M tensile specimens of 4-mm gauge diameter and 20-mm gauge length were machined from hot-rolled AIMFC 2 alloy and polished using 600-grit abrasive. Tensile tests were carried out at room temperature and 873 K in a 250 KN Instron 8500+ and 100 KN Instron 1185 Universal testing machine respectively at a strain rate of 0.05 mm^{-1}. The details of sample preparation and testing procedures are given elsewhere [18].

3. Results and discussion

Air induction melting (AIM) is the most economical melting route and is widely used for bulk production of alloys containing non-reactive elements. Recently it has been reported that recovery of various alloying elements during air induction melting of iron aluminides was excellent. The recovery of aluminium was 96–97% whereas the recovery of chromium varied from 91 to 99%. The recovery of niobium and zirconium was 100% [19]. The recovery of various alloying elements particularly reactive elements is attributed to the high concentration of aluminium present in the alloys, small quantity of which acts as an excellent deoxidizer as compared to other elements because of its high

TABLE I Chemical analysis of AIM and ESR ingots

Alloy	Expt. no.	Al	C	Ce	Mn	Si	S	O	N
Alloy 1	AIMFC1	10.9	0.8	0.1	0.36	0.05	0.003	0.08	<0.001
	ESR 1.1	10.7	0.8	0.01	0.36	0.04	<0.001	<0.001	<0.001
	ESR 1.2	10.6	0.8	0.02	0.35	0.04	<0.001	<0.001	<0.001
Alloy 2	AIMFC 2	10.6	0.8	0.3	0.4	0.05	0.004	0.06	0.002
	ESR 2.1	10.3	0.8	0.04	0.4	0.04	<0.001	0.0015	<0.001
	ESR 2.2	10.2	0.8	0.07	0.37	0.038	<0.001	<0.001	<0.001
	ESR 2.3	10.3	0.8	0.12	0.39	0.04	<0.001	<0.001	<0.001
	ESR 2.4	10.4	0.8	0.20	0.40	0.04	<0.001	0.0017	<0.001

TABLE II Recovery of aluminium and cerium during air induction melting with flux cover (AIMFC)

Alloy	Expt. no.	Element	Addition	Actual	Recovery
Alloy 1	AIMFC 1	Al	11.4	10.9	95%
		Ce	0.2	0.1	50%
Alloy 2	AIMFC 2	Al	11.0	10.6	96%
		Ce	0.8	0.3	38%

negative standard free energy of formation. It also forms a continuous Al_2O_3film (on top of the liquid metal) during melting which protects the melt from further oxidation from atmospheric air. However, this process has not been successful so far for bulk production of intermetallic alloys containing Fe and Al. Attempts to make bulk quantities by AIM resulted in extensive gas porosity [20]. Very recently it has been reported that air induction melting with flux cover (AIMFC) which protects the aluminium in the melt from atmosphere, results in elimination of hydrogen gas porosity, excellent recovery of Al, Ti, Cr, Ni and Mn [15, 17] and significant reduction in impurity levels [15, 16].

The results of chemical analysis and recovery of aluminium, cerium during AIMFC of the present work are presented in Tables I and II respectively. While aluminium recovery was greater than 95%, cerium recovery varied between 40 to 50%. The reasons for good recovery of aluminium during AIMFC is well understood [15, 16]. The reason for low cerium recovery is difficult to assess. The loss of cerium is probably due to the reduction of Al_2O_3in the flux and crucible material (alumina) by cerium. This reaction may be expressed as:

$$3[\mathrm{Ce}] + 2(\mathrm{Al_2O_3}) = 4[\mathrm{Al}] + 3(\mathrm{CeO_2}) \quad (1)$$

At standard conditions Al_2O_3 ($\Delta G^{o}_{1900\,\mathrm{K}} = -1065$ kJ/mol) is more stable than CeO_2 ($\Delta G^{o}_{1900\,\mathrm{K}} = -694$ kJ/mol) [21]. Though this indicates that cerium should not have been lost, a more rigorous analysis calls for solution thermodynamic data of cerium in the molten alloy phase and CeO_2 in the slag phase. In the absence of the data, the loss of cerium observed in this study indicates that activity coefficient of Ce may be high in Fe-Al-C melts and/or the activity coefficient of CeO_2 in the slag phase may be low.

Electroslag remelting (ESR) is an important secondary remelting process for commercial production of special steels and superalloys. The easily oxidisable elements such as Al, Ti, Ce and Zr are usually lost due to oxidation during ESR. The main sources of oxygen pick-up during ESR are [22, 23] (a) dissolved oxygen in the consumable ingot to be remelted, (b) ingot scale formed during operation (c) reducible oxides in the flux and (d) the remelting atmosphere. To minimize loss of reactive alloying elements during ESR, it is necessary to maintain the oxidation potential of the flux at a minimum. For this purpose, use of flux system free from reducible oxides, flux modified with the relevant oxide, flux deoxidation and inert gas shielding are some of the methods reported in the literature [24].

The ESR experimental condition and results of recovery of aluminium & cerium of the present work are shown in Table III. It is seen from the table, that the recovery of cerium during electroslag remelting of AIMFC-1 and 2 ingots containing 0.1 and 0.3% Ce with 70% $CaF_2 + 15\%$ $Al_2O_3 + 15\%$ CaO flux system is about 10–13%. Slight improvement in recovery of cerium (20–23%) has been achieved by using 70% $CaF_2 + 30\%$ CaO flux system without Al_2O_3. Further improvement in recovery of cerium (40%) was obtained by using modified flux system, 70% $CaF_2 + 30\%$ CaO containing about 5% CeO_2 during ESR of AIMFC 2 ingot containing 0.3% Ce. Thus qualitatively the absence of Al_2O_3 and presence of CeO_2 in the flux system is observed to increase the recovery of cerium. This is in consistent with the observation made in AIMFC. For the reaction (1) the activity of cerium is given by:

$$X^3_{[\mathrm{Ce}]} = K\frac{a^4_{[\mathrm{Al}]}a^3_{(\mathrm{CeO_2})}}{\gamma^3_{[\mathrm{Ce}]}a^2_{(\mathrm{Al_2O_3})}} \quad (2)$$

To retain the cerium content in the molten metal, the Al_2O_3 content should be decreased and CeO_2 content should be increased in the flux. Further, Al content in the melt should be increased. In addition to that, the activity coefficient of Ce in the melt should be high. This argument qualitatively explains the improvement in

TABLE III Effect of ESR process parameters on recovery of aluminium and cerium

Alloy	Expt. no.	Slag composition	Element	Before ESR (AIMFC) (wt%)	After ESR (wt%)	Recovery (%)
Alloy 1	ESR 1.1	70F/15/0/15	Al	10.9	10.7	98
			Ce	0.1	0.01	10
	ESR 1.2	70F/30	Al	10.9	10.6	97
			Ce	0.1	0.02	20
Alloy 2	ESR 2.1	70F/15/0/15	Al	10.6	10.3	97
			Ce	0.3	0.04	13
	ESR 2.2	70F/30	Al	10.6	10.2	96
			Ce	0.3	0.07	23
	ESR 2.3	70F/30 + 5% CeO_2	Al	10.6	10.3	97
			Ce	0.3	0.12	40
	ESR 2.4	70F/30 + 5% CeO_2 + Ca deoxident[a]	Al	10.6	10.4	98
			Ce	0.3	0.20	66

[a] 40 grams of Ca metal was added at equal intervals during ESR.

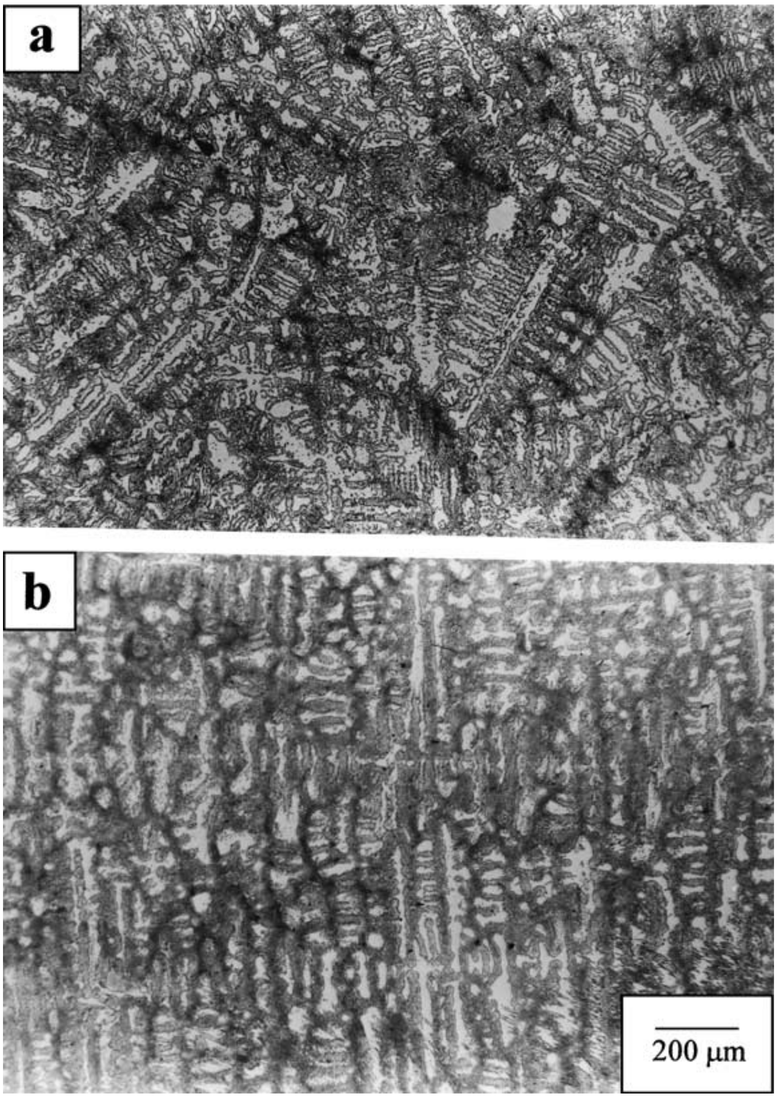

Figure 1 Optical micrographs showing (a) randomly oriented coarse dendritic structure in cast AIMFC 2 ingot and (b) axially aligned dendritic structure in the cast ESR 2.4 ingot.

recovery of cerium with flux containing low Al_2O_3 and high CeO_2 contents. It is also observed that the improvement in recovery of cerium to about 66% was achieved when the modified flux (70% $CaF_2 + 30\%$ CaO) containing 5% CeO_2 was deoxidized by metallic calcium during ESR. This indicates that the CeO_2 in the slag phase is reduced by calcium metal and cerium is transferred to the molten metal during ESR. The current experiments also indicate that increasing starting cerium content from 0.1 to 0.3% in AIMFC ingots did not result in any improvement in the recovery of Ce during ESR (Table III). This is probably due to high activity coefficient of Ce in the Fe-Al-C alloys. The recovery of Al was always greater than 95% during electroslag remelting with different slag compositions (Table III).

Gamma ray radiography showed that the AIMFC and ESR ingots were free from gas porosity. However microporosity and primary pipes observed in the AIMFC ingots were absent in the ESR ingots. The absence of gas porosity in the AIMFC ingots in the present work shows that the flux cover is also effective in preventing the formation of hydrogen gas porosity in high carbon Fe-10.5Al alloys containing cerium. The absence of microporosity and primary pipes in the ESR ingots as compared to AIMFC ingots may be attributed to the favorable directional solidification front in which dendrites are aligned along the ingot axis and are open to the melt (Fig. 1b) thus allowing the liquid metal to fill in the interdendritic region. In contrast, during conventional solidification (AIMFC) rapidly advancing solidification fronts from several directions results in more randomly oriented dendritic structure (Fig. 1a) and do not allow efficient filling of inter dendritic regions.

The ESR ingots exhibited comparatively excellent hot-workability. This improved hot workability of ESR ingots may be attributed to the axially oriented columnar grain structure (Fig. 1b) that is free from internal defects. The dendritic structure with large amount of precipitates in the inter dendritic regions observed in cast AIMFC 2 and ESR 2.4 ingots (Fig. 1) was absent in hot-forged and subsequently hot-rolled 14-mm thick sections (Fig. 2) indicating that the thermomechanical processing was successful in breaking down the cast structure.

The back-scattered electron micrographs revealed the presence of two types of precipitates in both cast

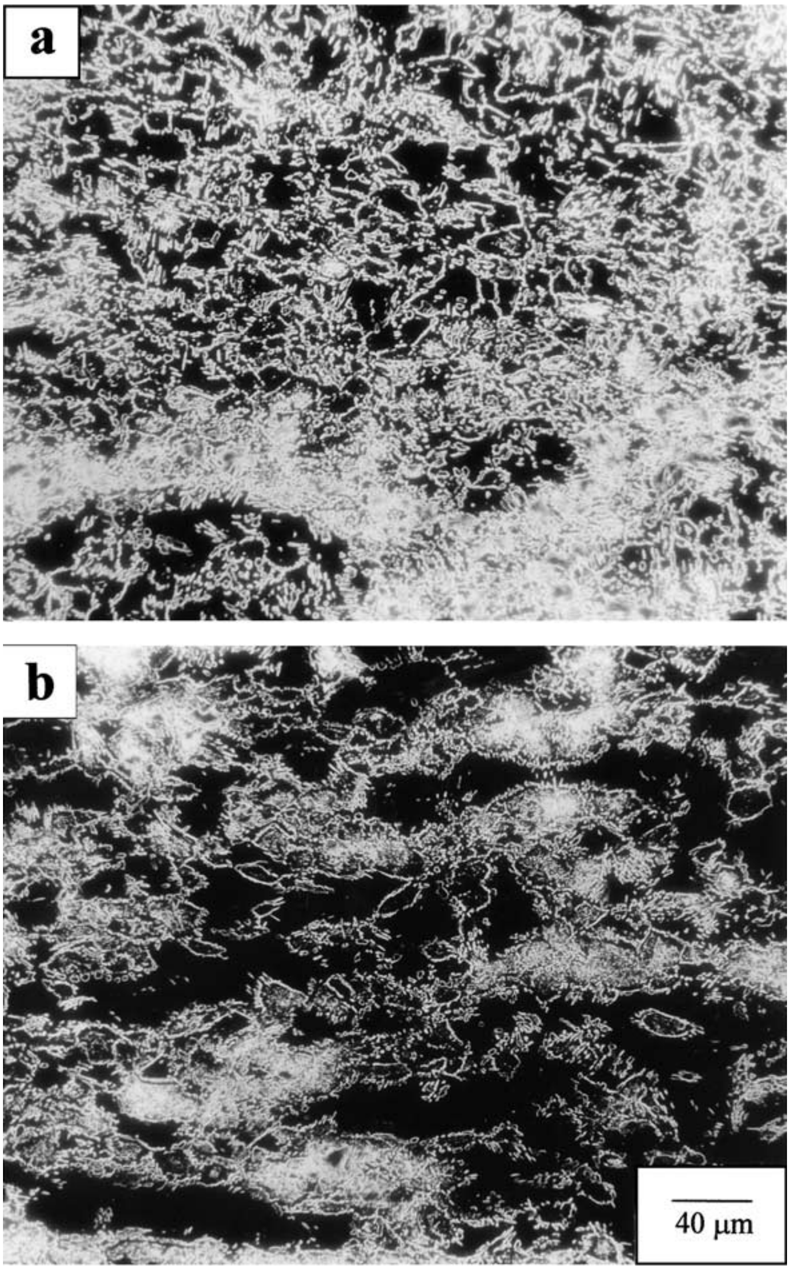

Figure 2 SEM secondary electron micrographs showing break down of cast structure after hot-rolling in (a) AIMFC 2 and (b) ESR 2.4 ingots.

and hot-rolled AIMFC 2 and ESR 2.4 ingots (Figs 3 and 4) The precipitate, which is in large quantity appearing dark in BSE micrograph, was confirmed to be $Fe_3AlC_{0.5}$ phase by X-ray diffraction analysis. Bright precipitates which constitute very small fraction, observed in BSE micrographs of SEM (Figs 3 and 4) and EPMA (Fig. 5) were identified as cerium oxy-carbide based on EDS images of EPMA (Fig. 5). The Figs 3b and 4b show that the $Fe_3AlC_{0.5}$ and cerium oxy-carbide precipitates are fine and more uniformly distributed in cast and hot-rolled ESR ingots as compared to cast and hot-rolled AIMFC ingots (Figs 3a and 4a).

The tensile properties at room temperature and 873 K of hot-rolled AIMFC 2 ingots are listed in Table IV. For the purpose of comparison, tensile data from the literature [25] for hot-rolled ESR 2.4, Fe-10.5Al-0.8C-0.2Ce alloy tested under similar condition is included in Table IV. A comparison of tensile properties of AIMFC 2 and ESR 2.4 ingots in the hot-rolled condition

TABLE IV Effect of processing on tensile properties of Fe-10.6Al–0.8C-0.3Ce alloys

		Tensile properties at room temperature			Tensile properties at 873 K		
Expt. no	Processing	UTS (MPa)	YS (MPa)	El (%)	UTS (MPa)	YS (MPa)	El (%)
AIMFC-2	AIMFC + hot-rolling	872	680	3.5	283	278	70
ESR-2.4[a]	AIMFC + ESR + hot-rolling	844	660	6.4	292	275	90

[a] After [25].

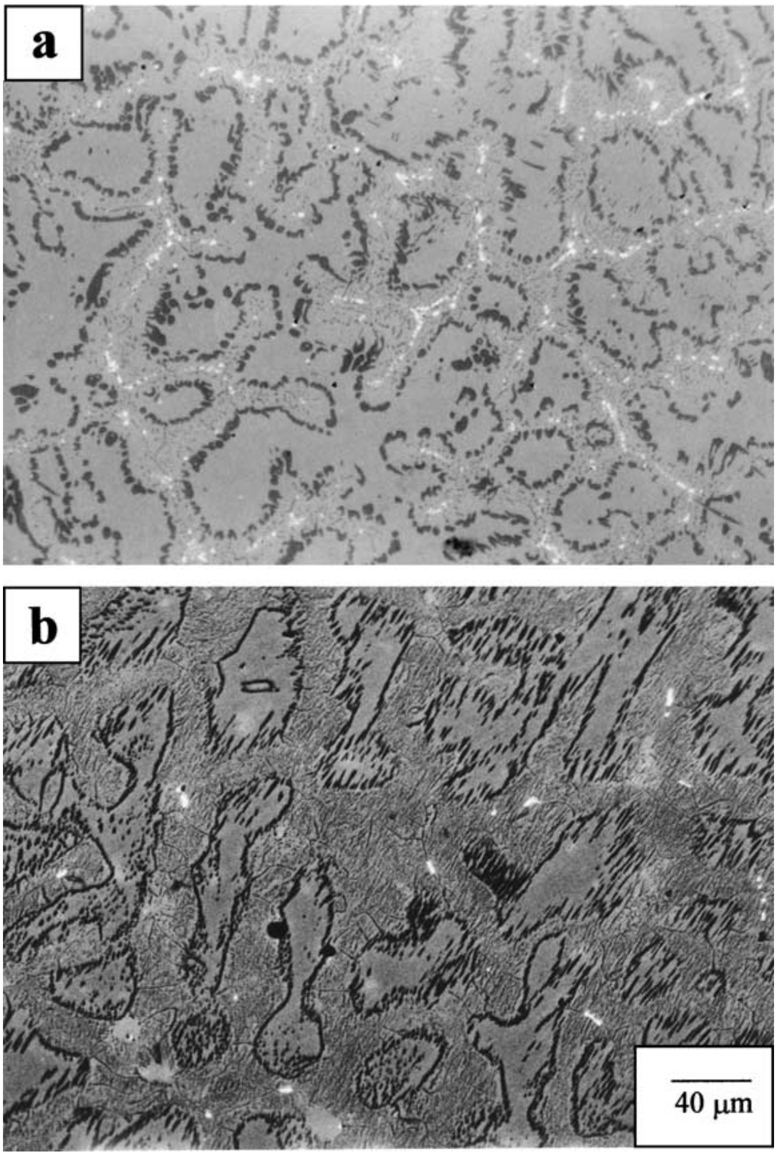

Figure 3 SEM back-scattered electron micrographs showing large volume fraction of dark precipitates and small volume fraction of bright precipitates in the interdendritic region of (a) cast AIMFC 2 ingot and (b) cast ESR 2.4 ingot. The precipitates size was finer in ESR ingots.

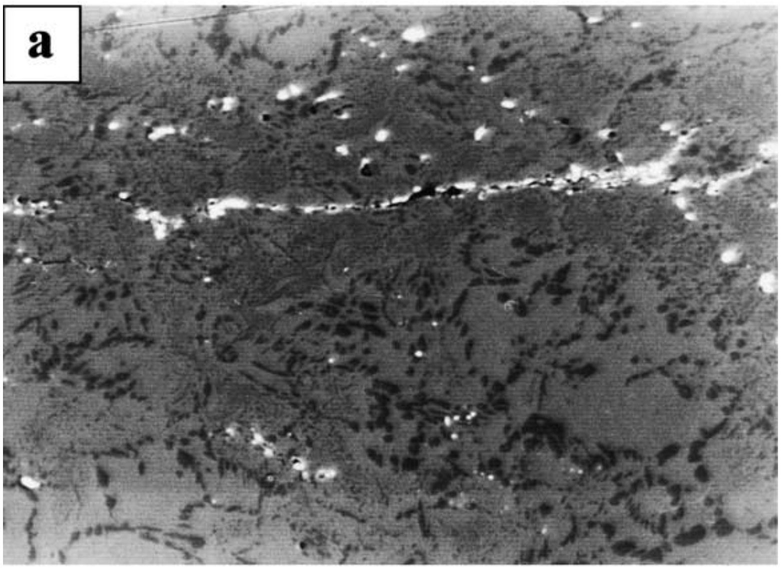

Figure 4 SEM back-scattered electron micrographs showing large volume fraction of dark precipitates in hot-rolled (a) AIMFC 2 ingot and (b) ESR ingot. The precipitates are fine and more uniformly distributed in ESR ingot as compared to hot-rolled AIMFC 2 ingots. (*Continued.*)

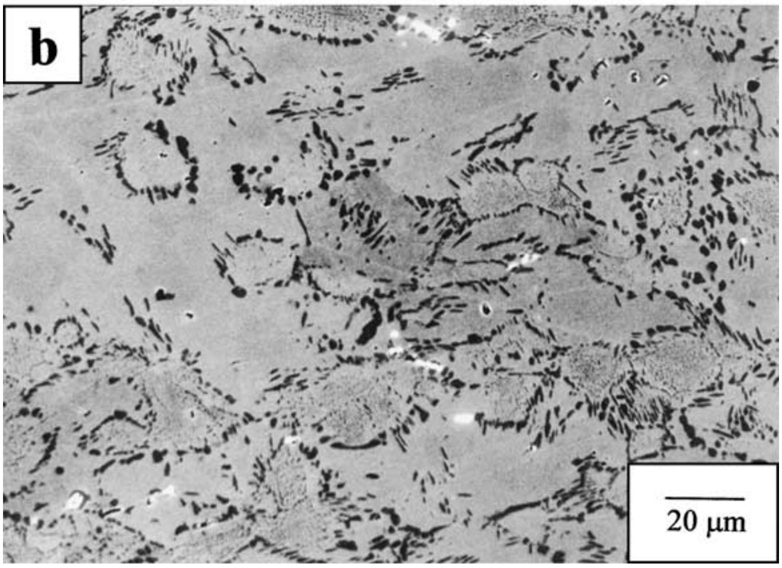

Figure 4 (*Continued*).

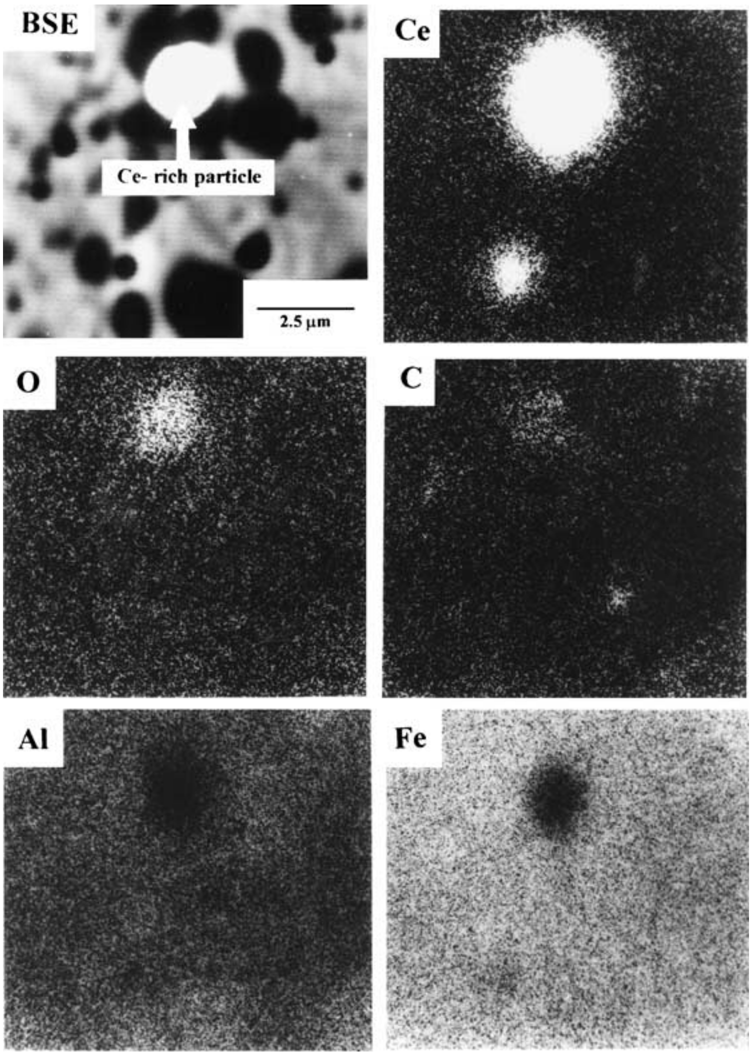

Figure 5 BSE and EDS images observed in EPMA showing cerium oxy-carbide precipitate in ESR 2.4 ingot.

(Table IV) shows that electroslag remelting of AIMFC ingots results in significant improvement in tensile ductility at room temperature and at 873 K. This may be attributed to the comparatively homogenous, sound and clean ingot with very low levels of impurities, refined microstructure and fine uniform distribution of (cerium oxy-carbide) precipitates observed in hot-rolled ESR ingots.

4. Conclusions

The results obtained in the present work serve as a qualitative guide to understand the behavior of Ce in

Fe-Al alloy during AIMFC and ESR. Good recovery of highly reactive element like cerium was obtained by air induction melting with flux cover. While conventional fluxes were found to be unsatisfactory, modified fluxes containing CeO_2 gave better recovery of cerium during ESR. The recovery of Ce was significantly improved by the use of calcium as a deoxident during ESR.

The combination of AIMFC and ESR processing yields a sound ingot of Fe-Al-C-Ce quaternary alloys free from gas and shrinkage porosity with very low oxygen, nitrogen and sulphur content. Processing of AIMFC ingots through ESR has resulted in improved hot workability. This may be attributed to the axially oriented columnar grain structure free from internal defects.

The ESR processed and hot-rolled alloy exhibited superior tensile elongation as compared to hot-rolled AIMFC alloy at room temperature as well as at 873 K. This may be attributed to the comparatively sound, homogeneous and clean ingot with very low levels of impurities, a refined microstructure and fine uniform distribution of $Fe_3AlC_{0.5}$ & cerium oxy-carbide precipitates observed in hot-rolled ESR ingots.

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