Effect of niobium on microstructure and mechanical properties of hot-rolled Fe-8.5 wt% Al-0.1 wt% C alloy

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Fe_3Al-based intermetallic alloys are being developed for elevated temperature structural applications at temperature up to 873 K [1–3]. Although binary Fe_3Al alloys can exhibit brittle behavior at room temperature, poor toughness, poor workability, poor high temperature strength, and creep properties, an improvement in these can be achieved by alloying addition and process control [3, 4]. The additions of Nb, Mo, and W have shown significant improvements in the high temperature strength and creep properties [5–7]. Chromium addition resulted in enhanced room temperature ductility [8], whereas cerium addition resulted in significant improvement in both room temperature ductility and strength [9–11]. Fe_3Al alloys contain 13 to 21 wt% aluminum. The reduction in aluminum content to 8.5 wt% was also found to increase the room temperature ductility [1, 12]. Recently addition of carbon to Fe-8.5 wt% Al alloys was shown to result in improved strength [13]. In this letter, the effect of niobium addition on microstructure and mechanical properties of electroslag refined (ESR) Fe-8.5 wt% Al-0.1wt% C alloy is reported.

Fifty-kilogram melts of Fe-8.5Al-0.1C and Fe-8.5Al-0.1C-1.5Nb (all compositions are in wt% unless otherwise specified) alloys were prepared by a combination of air induction-melting with flux cover (AIMFC) and electroslag remelting (ESR). The melting practice has been discussed in detail elsewhere [14, 15]. The ESR ingots of 80-mm diameter were hot-forged at 1373 K to a reduction of 80%. The forged billets were subsequently hot-rolled at 1373 K to a reduction of 50%. The final thickness of rolled plate was 12-mm. All the investigation were carried out using the 12-mm thick plate product.

Longitudinal sections of the plates were cut using a bi-metallic band saw blade. The cutoff sections were mechanically polished to 0.5 μm grade diamond powder finish for microstructural studies in a scanning electron microscope (SEM). The polished sections were subsequently etched with an etchant composed of 3.3% HNO_3 + 3.3% CH_3COOH + 0.1% HF + 93.3% H_2O by volume for microstructural examination under an optical microscope. The bulk hardness measurements were made on the metallographic samples using a Vickers hardness machine with 30-kg load. Tensile specimens of 4.0-mm gauge diameter and 20-mm gauge length conforming to ASTM–E 8M standard were machined from blanks oriented along the rolling direction. A few specimens cracked during machining and were rejected. Tensile tests were carried out at room temperature and at 873 K on 100 KN Instron 1185 universal testing machine at a strain rate of 0.8×10^{-4} s^{-1}. Fracture surfaces from selected samples were examined by SEM. Stress-rupture and creep tests were carried out using specimens of 5-mm gauge diameter and 25-mm gauge length. All the creep tests were carried out at 873 K and 140 MPa till the specimen failed. Minimum creep rates (MCR) were measured as the slope of the linear portion of the test curves.

Optical micrographs of both the alloys showed the presence of precipitates (Fig. 1a and b). The precipitates in the Nb-free alloy (Fig. 1a) were identified to be $Fe_3AlC_{0.5}$, while those in the Nb-containing alloy (which appeared bright in the back-scattered electron micrograph (Fig. 1c) of SEM) were identified to be niobium carbide by electron probe microanalysis (EPMA) in the pervious study [16]. Both the alloys revealed partially recrystallized grains, indicating that dynamic recrystallization had occurred during rolling at 1373 K (Fig. 1). The addition of niobium resulted in a significant reduction in the as-rolled grain size (Fig. 1a and b), conforming earlier findings of Zhanghu *et al.* [7]. Microcracks were observed in both the alloys. These cracks were probably responsible for the failure of the few tensile testing samples during fabrication and/or failure at very low-tensile stress with no measurable plasticity. Similar types of microcracks were observed in the as-cast condition [16]. The cracks in the as-cast ESR ingots were attributed to an earlier study on the presence of high level of residual hydrogen [16, 17], these cracks might have persisted even after hot working.

Niobium addition to binary Fe-Al alloys has been found to increase the yield strength at temperature up to 923 K [18]. This has been attributed to a solid solution strengthening as well as to the formation of fine niobium carbide precipitates. In the present work the niobium addition to the ternary alloy resulted in a marginal improvement in tensile yield strength, at room temperature and a significant improvement in the tensile yield strength at 873 K (Table I). Similar improvements in hardness and compressive yield strength by the addition of 1.5 and 3.5 wt% Nb have been reported in as-cast ESR ingots [16]. Since Fe–Al alloy is known to have a very low solubility for niobium [7], it is expected that all the Nb would form niobium carbide precipitates in the present alloy containing carbon. Therefore, the improved yield strength of the Nb-containing alloy may be attributed to the higher hardness (2050 HV) [19] of

TABLE I Mechanical properties ESR hot-rolled alloys

			RT tensile properties**			873 K tensile properties**		
Alloy	Alloy composition (wt%)	Hardness* (HV)	UTS (MPa)	YS (MPa)	El (%)	UTS (MPa)	YS (MPa)	El (%)
Alloy 1	Fe-8.5Al-0.1C	$\frac{235}{226\text{–}245}$	$\frac{554}{544\text{–}564}$	$\frac{515}{510\text{–}520}$	$\frac{1}{1\text{–}1}$	$\frac{251}{248\text{–}254}$	$\frac{241}{229\text{–}253}$	$\frac{41}{40\text{–}42}$
Alloy 2	Fe-8.5Al-0.1C-1.5Nb	$\frac{238}{230\text{–}240}$	$\frac{686}{680\text{–}692}$	$\frac{550}{545\text{–}556}$	$\frac{6.3}{6.2\text{–}6.4}$	$\frac{349}{242\text{–}256}$	$\frac{336}{234\text{–}238}$	$\frac{30}{28\text{–}33}$

UTS: Ultimate tensile strength; YS: Yield strength; El: Elongation.

$^{*}\ \frac{\text{Average of five measurements}}{\text{Minimum–Maximum}}$.

$^{**}\ \frac{\text{Average of two measurements}}{\text{Minimum–Maximum}}$.

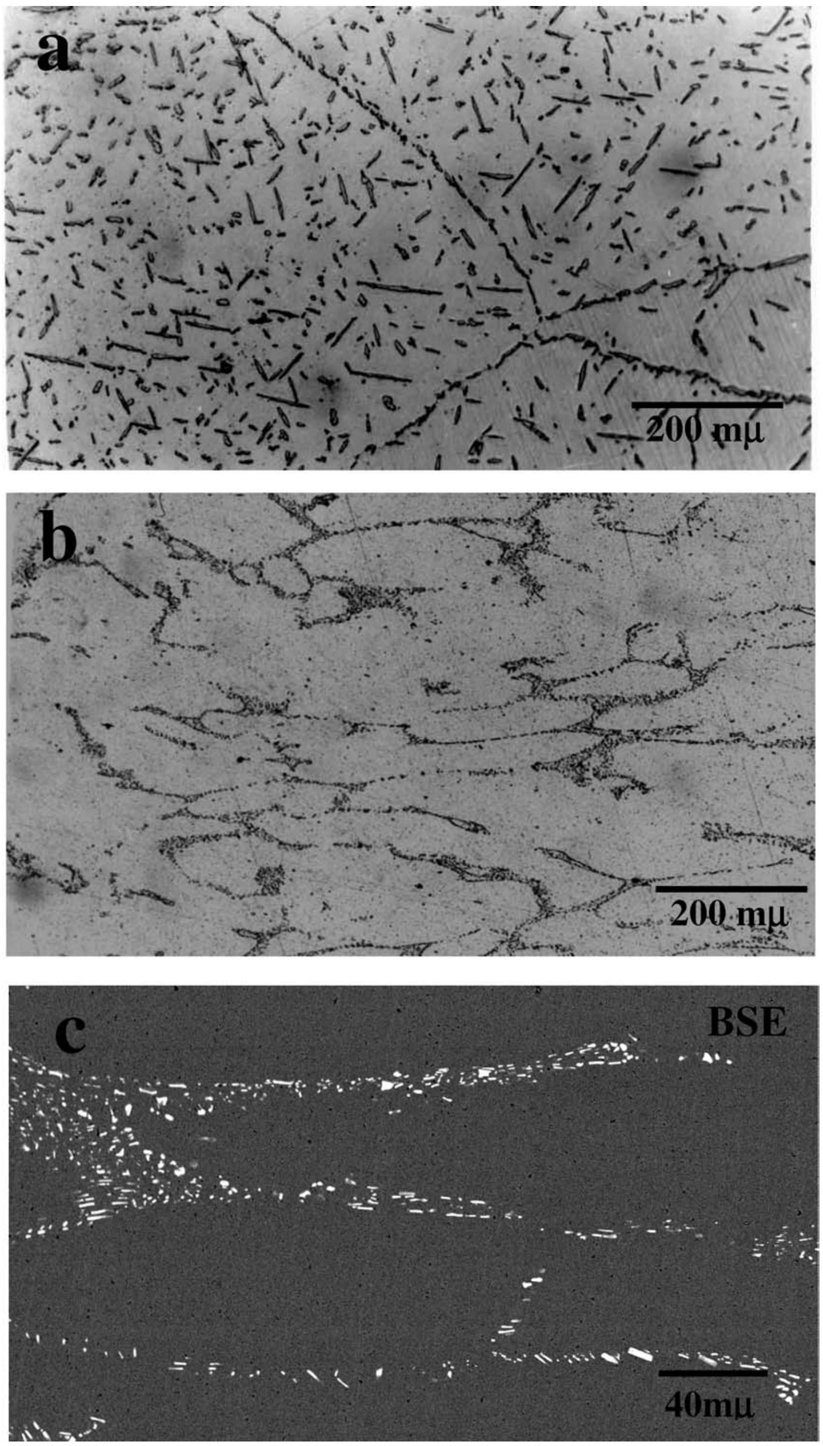

Figure 1 Optical micrographs of hot-rolled ESR ingots of: (a) Fe-8.5Al-0.1C and (b) Fe-8.5Al-0.1C-1.5Nb alloys and SEM back-scattered electron micrographs of hot-rolled ESR ingots of (c) Fe-8.5Al-0.1C-1.5Nb alloy.

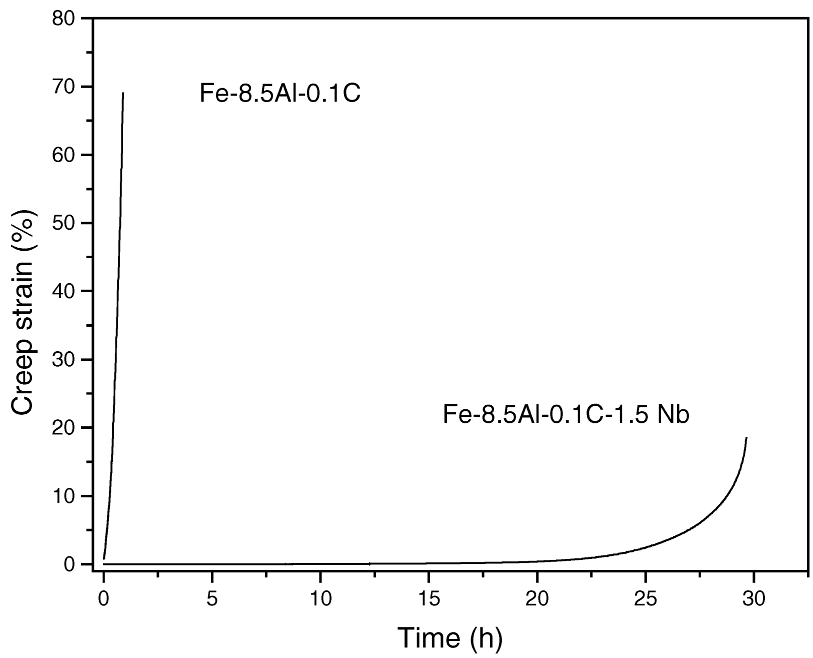

Figure 2 Creep-rupture curves of ESR hot-rolled Fe-8.5Al-0.1C and Fe-8.5Al-0.1C-1.5Nb alloys at 873 K and 140 MPa.

the niobium carbide precipitates, compared to that of $Fe_3AlC_{0.5}$ precipitate (600 HV) [20] in Nb-free alloy.

Table I also shows that the room temperature tensile elongation of Fe-8.5Al-0.1C-1.5Nb alloy is superior (6.3%) to that of the Fe-8.5Al-0.1C alloy (1%). The tensile specimens of both the alloys failed by cleavage at room temperature indicating that there is no significant effect of niobium on the fracture mode. This improvement in room temperature ductility is significant because it has been reported earlier that the presence of niobium in the Fe–Al alloys is not beneficial to the room temperature tensile elongation [7]. The mechanism by which Nb improves the ductility of the Fe-8.5Al-0.1C alloy has not been understood yet, but it may be related to grain refinement during solidification by formation of high melting temperature niobium carbide precipitates. Fig. 2 compares the creep curves of the two alloys. It can be seen from the figure that the addition of niobium resulted in a significant improvement in creep life and reduction in the steady-state creep rate. This is consistent with the higher strength of Nb-containing alloy at 873 K. The creep life increased from approximately 1 to 30 hr, and the minimum creep rate decreased from 30%/h to 25×10^{-2}%/h. This may be attributed to the presence of hard Nb_2C precipitates.

To summarize a small (1.5 wt%) addition of niobium to Fe-8.5 wt% Al-0.1 wt% C alloy results in significant improvement in high temperature strength, creep resistance, and room temperature ductility. The improvement in strength and creep resistance may be attributed to precipitation hardening by niobium carbide, and the improvement in ductility may be attributed to the grain refinement.

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