## **Effect of carbon on embrittlement, processing and mechanical properties of Fe-9 wt% Al alloys**

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Intermetallic alloys based on Fe3Al containing 14–21 wt% Al, are being developed for structural applications for temperatures up to 873 K  $[1-3]$ . However, these alloys generally exhibit poor room temperature ductility and low fracture toughness. Recently it has been reported that a reduction in aluminum content to 8.5 wt% resulted in enhanced ductility [4]. The improvement in ductility has been attributed to a reduced susceptibility to environmental embrittlement. Further, the dislocation movement may be easier in the disordered bcc structure exhibited by the alloys with low Al-content than in the ordered  $Fe<sub>3</sub>Al$  based alloys. Recently we have reported that the addition of high concentration (0.5 and 1.0 wt%) of carbon to cast and hot-rolled electoslag refined (ESR) Fe-8.5 wt% Al alloy resulted in improved strength [5–7]. In the present work, we report the effect of low  $(0.05 \text{ and } 0.1 \text{ wt\%})$  and high (0.5 and 1.0 wt%) concentration of carbon addition to hot-rolled ESR Fe-9 wt% Al alloy on hydrogen embrittlement, hot-working, room temperature and 873 K tensile properties as well as creep properties at 873 K and 140 MPa.

Fifty-kilogram melts of Fe-Al alloy containing  $9$  wt% (nominal) aluminum each and 0.05, 0.1, 0.5 and 1.0 wt% carbon respectively were air induction melted with flux cover (AIMFC) and chill cast into 55-mm diameter ingots using split cast iron molds. The AIMFC ingots were subsequently remelted by ESR into 80-mm diameter ingots. The melting practice has been discussed in detail elsewhere [8, 9]. The ESR ingots of 80-mm diameter were preheated in a hearth furnace at 1373 K for 1 hr and hot-forged by 80% in a pneumatic one tonne forge hammer with die platens at room temperature, as well as in a oil hydraulic forge press with dies at 1173 K. The 24-mm thick sections of the forged billets were preheated for 1 hr at 1373 K 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 and short transverse sections of the plates were cut using bi-metallic band saw blade. The cut-off sections were mechanically polished to 0.5  $\mu$ m grade diamond powder finish for microstructural studies by scanning electron microscopy (SEM). The polished sections were subsequently etched with an etchant composed of  $3.3\%$  HNO<sub>3</sub> +  $3.3\%$  CH<sub>3</sub>COOH +  $0.1\%$  $Hf + 93.3\% H_2O$  by volume for microstructural examination by optical microscope.

Bulk hardness measurements were made on polished samples using a Vickers hardness machine with 30-kg load. Longitudinal ASTM-E 8M tensile specimens of 4.0-mm gauge diameter and 20-mm gauge length, for room temperature and high temperature tests, were machined from the plates, and polished using 600-grit abrasive. Tensile tests were carried out at room temperature and at 873 K in a 100 kN Instron 1185 Universal Testing Machine at an initial strain rate of  $0.8 \times 10^{-4}$ s<sup>−1</sup>. Selected tensile fracture surfaces were examined in SEM. Stress-rupture specimens of 5-mm gauge diameter & 25-mm gauge length were machined and polished using 600-grit abrasive. 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 strain vs. time curves.

It has been reported [10] earlier that ESR ingots of Fe-16 wt% Al alloy having low  $(0.013 \text{ to } 0.06 \text{ wt%)}$  carbon content cracked severely during hot-forging, whereas ESR ingots with high (0.5 wt%) carbon content were successfully forged at 1373 K. In the present work, ESR ingots of Fe-9 Al alloys containing high (0.5 and 1.0 wt%) carbon exhibited excellent hot-workability similar to the high carbon Fe-16 wt% Al alloys reported above. The rolled plates were sound and free from macro- and microcracks. The hot-working response of Fe-9 wt% Al alloy ingots, containing low (0.05 and 0.1 wt%) carbon, was poor. Surface as well as internal cracks formed during hot forging and further rolling. The low carbon alloys could be forged without macrocracks when oil hydraulic forge press with heated dies was used. Further hot rolling did not result in any macrocracking, though revealing some microcracks. These cracks were probably responsible for failure of few tensile testing samples during fabrication and/or failure at low tensile stress without exhibiting any measurable plasticity. Similar microcracks were observed in the as-cast alloys having similar composition [11]. These cracks may occur due to the presence of high level of residual hydrogen in the as-cast ingots and they may persist even after hot-working. No attempt was made to confirm the presence of microcracks in hot forged billets. In the present study, the chemical analyses showed 5 ppm hydrogen in the low carbon alloys as against 1.7 ppm in the high carbon alloys. The effect of carbon on the solubility of hydrogen in Fe-Al alloy is not reported in the literature. However, the solubility of hydrogen in pure iron is known to decrease with increase in carbon content [12], and it may be argued that alloys with lower carbon contents are more susceptible to cracking as compared to the alloys with high carbon content due to the higher concentration of dissolved hydrogen. The processing routes followed by earlier investigators [1, 13] used vacuum induction melting, arc melting and drop casting, or powder metallurgy, all using vacuum or inert atmosphere, thus reducing the chances of hydrogen pick-up during melting. Therefore these alloys do not suffer from cracking due to hydrogen embrittlement, in contrast to AIMFC and ESR processed alloys.

In the present work, the plates revealed a variety of grain structures. The low carbon alloys showed coarse pancake-shaped grains indicating absence of recrystallization (Fig. 1a–d). In the high carbon alloys the grain boundaries could not be revealed clearly, owing to high volume fraction of the precipitates. However some grain boundaries could be seen without any preferred



*Figure 1* Optical micrographs of ESR hot-rolled samples of (a and b) Fe-8.7 Al-0.05C, (c and d) Fe-8.8Al-0.1C, (e and f) Fe-8.7Al-0.5 and (g and h) Fe-8.8Al-1C alloys. L, LT and ST refer to the longitudinal, long transverse and short transverse directions w. r. t. rolling direction, respectively.

TABLE I Effect of carbon on mechanical properties ESR hot-rolled Fe-9 wt% Al alloys

Alloy	Alloy composition (wt. $%$ )	Hardness (HV)	RT tensile properties			873 K tensile properties		
			UTS (MPa)	YS (MPa)	El (%)	UTS (MPa)	YS (MPa)	El (%)
	Fe-8.7Al-0.05C	225	535	517	1.5	241	240	53
2	Fe-8.8Al-0.1C	230	550	525	1.6	255	243	55
3	Fe-8.7Al-0.5C	250	697	545	3.2	320	290	75
$\overline{4}$	$Fe-8.8Al-1C$	323	995	650	4.5	345	300	76
5	AMDC wrought Fe-8.5Al-0.01C <sup>a</sup>		480	380	25	$\qquad \qquad$		

UTS, Ultimate tensile strength; YS, Yield strength; El, elongation.

<sup>a</sup>Arc melted and drop cast (AMDC) wrought alloy after [4].

orientation, indicating occurrence of recrystallization. Occurrence of recrystallization in the high carbon alloys can be attributed to the lower volume fraction of the softer  $\alpha$  phase (matrix), leading to higher accumulation of deformation strain, providing a greater degree of driving force for recrystallization. It can also be seen from the figure that carbon addition leads to the formation of uniformly distributed dark colored phase, and the volume fraction of the phase increases with increasing carbon content. This phase was identified to be Fe<sub>3</sub>AlC<sub>0.5</sub> in the previous studies [6]. These results are consistent with the Fe-Al-C phase diagram determined by Palm and Inden [14]. The alloy containing low (0.05 and 0.1 wt%) carbon exhibits the presence of fine needle-shaped  $Fe<sub>3</sub>AlC<sub>0.5</sub>$  precipitates in the matrix and along the grain boundaries. The precipitates appeared to be coarser in the alloy containing 0.5 and 1.0 wt% carbon. However on observation under scanning electron microscope the precipitates were found to be fine and discreet.

Table I lists the bulk hardness, room temperature, and 873 K tensile properties of hot-rolled ESR alloys. Each hardness data point reported here represents an average of 5-measurements and each tensile and creep-rupture data point reported here represents an average of two tests. It can been seen from Table I that the addition of carbon to Fe-9 wt% Al alloy has significant influence on hardness and room temperature tensile properties. The hardness, room temperature ultimate tensile strength (UTS) and yield strength (YS) as well as tensile elongation (El) increase with increasing carbon content. The improvement in hardness, UTS, YS and El with increasing carbon content from 0.05 to 0.1 wt%, was marginal. The increase in the yield strength was significant however when the amount of carbon increased from 0.5 to 1.0 wt%. The ultimate tensile strength and elongation are much higher for the alloy containing  $1.0 \text{ wt\%}$  carbon as compared to the alloy containing 0.05, 0.1 and  $0.5$  wt% C. The large difference in the rupture strength is also indicative of higher tensile elongation for 1.0 wt% C alloy as compared to the alloys containing 0.05 to 0.5 wt% C. Table I show the change of tensile properties at 873 K with increase in carbon concentration. The yield strength increased with increase in carbon content, especially when the carbon content increased from 0.1 to 0.5 wt%.

The improvement in the hardness and strength in the present work may be attributed to the increase in volume fraction of  $Fe<sub>3</sub>AlC<sub>0.5</sub>$  precipitates with the increase in

carbon content, because  $Fe<sub>3</sub>AlC<sub>0.5</sub>$  is hard and in brittle phase and its presence can strengthen the matrix. The increase in room temperature ductility with increase in carbon is significant, because it has been reported earlier that carbon has an adverse effect on room temperature ductility though the reason for this is not clear [15, 16]. The mechanism by which the carbon enhances the ductility of Fe-9 wt% Al alloy has not been clearly understood yet but it may be related (a) to the improved resistance to hydrogen embrittlement because of low level of dissolved hydrogen in the alloy and trapping of hydrogen by the  $Fe<sub>3</sub>AIC<sub>0.5</sub>$  precipitates as well as (b) reduction in grain size due to recrystallization during hot-working.

Previous work by other investigators [4] had shown that Fe-8.5Al alloy processed under inert atmosphere exhibited high (25%) room temperature ductility. Alloys of similar composition with 0.05 and 0.1% carbon, processed by a combination of AIMFC and ESR showed very low (1.5%) ductility, and can be attributed to the presence of microcracks. Though the higher concentration of carbon  $(0.5 \text{ and } 1.0 \text{ wt\%)}$  addition has prevented the formation of cracks and improved the ductility by reducing the hydrogen pick-up, the ductility of these alloys are still much lower (at much higher strength, though) than the Fe-8.5Al alloys produced under inert atmosphere. This is because carbon is expected to lower the inherent ductility of the alloys.

The results of creep and stress-rupture tests of hotrolled ESR alloys are summarized in Table II. Alloys containing high (0.5 and 1.0 wt%) carbon exhibit a higher stress-rupture life and lower minimum creep rate as compared to alloys containing low (0.05 and 1.0 wt.) carbon. This is consistent with the behavior of the strength at 873 K. The improved creep properties may be attributed to the extensive precipitation of

TABLE II Effect of carbon on creep properties of ESR hot-rolled Fe-9 Al alloys at 873 K and 140 MPa

		Creep properties					
Alloy	Alloy composition	Life (h)	Minimum creep rate $(\%$ /hr)	Strain to fracture $(\% )$			
	Fe-8.7Al-0.05C	0.85	3.4	50			
2	Fe-8.8Al-0.1C	0.67	3.2	76			
3	Fe-8.7Al-0.5C	3.67	1.4	72			
4	Fe-8.8Al-1.0C	4.59	1.8	83			

stable  $Fe<sub>3</sub>AIC<sub>0.5</sub>$  phase because of the presence of a high concentration of carbon in the alloys.

To summarise the above, the low  $(0.05 \text{ and } 0.1 \text{ wt\%)}$ carbon ESR ingots showed a higher concentration of hydrogen resulting in the formation of microcracks (hydrogen embrittlement). The pre-existing microcracks in the low carbon alloy ingots were considered responsible for their poor hot-workability and low tensile ductility. On the other hand, alloys with high (0.5 and 1.0 wt%) carbon were free from microcracks, exhibited excellent hot-workability, and possessed superior tensile and creep properties.

## **Acknowledgments**

The author is grateful to the Defence Research and Development Organization, Ministry of Defence, New Delhi for the financial support in carrying out this research work. The author wishes to thank Dr. D. Banerjee, C C R & D DRDO HQrs, Delhi and Dr. A. M. Srirama Murthy, Director DMRL for their interest and encouragement, and Dr. A. Gokhale for his valuable discussions. The author would also like to thank his fellow officers and the staff of varies groups of DMRL such as ERG (melting & casting), ACG (chemical analysis), MEG (sample making), MWG (radiography, forging & rolling), MBG (tensile & creep), SFAG (Metallography and SEM).

## **References**

1. C. G. MCKAMEY, J. H. DEVAN, P. F. TORTORELLI and V. K. SIKKA, *J. Mater. Res.* **6** (1991) 1779.

- 2. V. K. SIKKA, S. VISWANATHAN and C. G. MCKAMEY, in "Structural Intermetallic," edited by R. Darolia, J. J. Lewandowsky, C. T. Liu, P. L. Martin, D. B. Miracle and M. V. Nathal (The Minerals, Metals and Materials Society, TMS Warrendale, PA, 1993) p. 483.
- 3. U. PRAKASH, R. A. BUCKLEY, H. JONES and C. M. SELLARS , *ISIJ Int.* **31** (1991) 1113.
- 4. V. K. SIKKA, S. VISWANATHAN and S. VYAS, in "High-Temperature Ordered Intermetallic Alloys V I," edited by Baker, R. Darolia, J. Wittenberger and M. Yoo, MRS Symp. Proc, ( MRS Pittsburgh, Pennsylvania, 1993) Vol. 288 p. 971.<br>5. R. G.
- 5. R. G. BALIGIDAD, U. PRAKASH and A. RADHAKRISHNA, *Mater. Sci. Eng.* A**269** (1999) 120.
- 6. *Idem.*, *ibid.* **255** (1998) 162.
- 7. *Idem.*, *ibid.* **281** (2000) 143.
- 8. *Idem.*, *Intermetallics* **6** (1998) 765.
- 9. R. G. BALIGIDAD, U. PRAKASH, V. RAMAKRISHNA RAO, P. K. RAO and N. B. BALLAL, *Ironmak & Steelmak* **21** (1994) 324.<br>10. R. G.
- BALIGIDAD, U. PRAKASH, A. RADHAKRISHNA, V. RAMAKRISHNA RAO, P. K. RAO and N. B. BALLAL, *ISIJ Int*. **36** (1996) 1215.
- 11. R. G. BALIGIDAD, A. DUTTA and A. SAMBASIVA RAO, Communicated to *J. Mater. Sci.* (2003).
- 12. R. J. FRUEHAN, "Metals Hnadbook" (ASM. Ohio, USA, 1988) Vol. 15, p. 81.
- 13. V. K. SIKKA, "Processing Properties and Applications of Iron-Aluminides" (The Miner., Met., Soc. Pennsylvania, USA, 1994) p. 3.
- 14. M. PALM and G. INDEN, *Intermetallics* **3** (1995) 443.
- 15. W. R. KERR, *Metall. Trans.* **17A** (1986) 2298.
- 16. W. JUSTUSSON, V. F. ZACKAY and E. MORGAN, Am. *Soc. Met.* **49** (1957) 95.

*Received 12 April and accepted 18 May 2004*