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Effect of hydrogen on the mechanical behaviour of carbon-alloyed Fe_3Al-based iron aluminides

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Abstract. The effect of hydrogen on the mechanical behaviour of two carbon-alloyed iron aluminides was studied. Weakening of some carbide–metal interfaces in the presence of hydrogen was indicated. The effect of cathodic hydrogen charging on the microstructure has also been addressed.

Keywords. Iron aluminide; hydrogen embrittlement; mechanical properties; scanning electron microscopy; microstructure.

1. Introduction

Ordered intermetallic alloys based on iron aluminide compositions of Fe_3Al and FeAl are candidate high-temperature structural materials, due to their excellent oxidation and sulphidation resistance. Although the binary alloys exhibit poor room temperature ductility and low fracture toughness, significant improvement in these properties can be achieved by alloying and process control. Most of the earlier studies have been conducted on iron aluminides with very low (0·01 wt%) carbon contents. Baligidad *et al* (1997) reported that addition of carbon in the range of 0·14 to 0·50 wt% significantly increased the room temperature strength of Fe–16 wt% (28 at%) Al alloys. The increase in room temperature yield strength was attributed to solid solution strengthening by the interstitial carbon, as well as precipitation hardening due to the presence of $Fe_3AlC_{0·5}$ precipitates (Baligidad *et al* 1997). These alloys also exhibited some room temperature ductility, which was attributed to irreversible trapping of hydrogen by $Fe_3AlC_{0·5}$ (Baligidad *et al* 1997, 1998). Recently, the mechanical behaviour of carbon-alloyed iron aluminides, containing Ti and V, has been studied (Prakash and Sauthoff 2001). The ductility improvements, which did not depend upon the amount and type of carbide or graphite present, was explained by the blocking of interstitial sites by carbon (Prakash and Sauthoff 2001). The diffusivity of hydrogen was an order of magnitude lower in the two carbon-alloyed intermetallics compared to the base binary intermetallic Fe–25Al (Sen and Balasubramaniam 2001). In the present study, the effect of irreversibly trapped hydrogen on the mechanical behaviour of these two carbon-alloyed iron aluminides would be addressed. It is important to understand the interaction of hydrogen with iron aluminides because hydrogen embrittlement still remains a major cause for concern (Balasubramaniam 1999).

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2. Experimental

The carbon-alloyed iron aluminides were obtained from the Defence Metallurgical Research Laboratory (DMRL), Hyderabad. Their compositions (in at%) were Fe–28·1Al–2·1C and Fe–27·5Al–3·7C. Tensile specimens (25 mm gauge length, 9 mm wide and 3·01 mm thick) were prepared from the rolled strips with the gauge length parallel as well as perpendicular to the rolling direction. One set of experiments was conducted without any hydrogen charging in order to obtain reference properties. Before performing the tensile test, each sample was baked at 250°C for 1 h to remove all reversibly trapped hydrogen from the intermetallic. Tensile specimens were coated with silicone oil prior to testing in order to minimize hydrogen entry. Tensile testing was performed on an Instron testing machine 1195 (screw-driven with a capacity of 100 kN) at a constant strain rate of 10^{-4} sec^{-1}. The second set of experiments was conducted on tensile specimens prepared by the following method. The tensile test sample was used as a cathode in an electrolytic cell. The electrolyte used was 0·05 mol/l H_2SO_4 solution and 100 ppm of sodium arsenate was added to the solution to serve as a hydrogen recombination ‘poison’. A Pt electrode served as the anode. Hydrogen was charged for 12 h at a current density of 10 mA/cm^2. After hydrogen charging, the sample was baked at 250°C for 1 h. This procedure ensured the removal of reversibly trapped hydrogen but not the irreversibly trapped hydrogen (Kasul and Heldt 1991, 1994). The tensile test was performed as described above.

In order to understand the effect of cathodic hydrogen charging on the microstructure, metallographic specimens of the alloys were hydrogen charged for 12 h in 0·05 mol/l H_2SO_4 solution (with 100 ppm sodium arsenate ions) at a current density of 10 mA/cm^2. After hydrogen charging, the samples were slightly polished and then etched to reveal the microstructure. The microstructures as well as the fracture surfaces were observed in a JEOL 840A scanning electron microscope (SEM).

3. Results and discussion

The equiaxed microstructure of the intermetallics contained bulky $Fe_3AlC_{0·5}$ precipitates (figure 1, see also Sen *et al* 2000). The composition and structure of carbide were confirmed by X-ray diffraction.

The tensile test results have been presented in table 1. The intermetallic containing higher amount of carbon generally exhibited higher UTS. A significant difference in UTS was also observed for the same intermetallic for testing performed in the direction parallel and perpendicular to the rolling direction. All the specimens exhibited low ductilities and hence, general trends regarding hydrogen embrittlement could not be discerned. However, analysis of fractographic features provided insights on the role of hydrogen. Fractographic study of fracture surfaces revealed that cracks generally originated from carbide–matrix interfaces. In figure 2, a secondary crack can be seen originating from the carbide particle located in the centre of the micrograph. The sample was not charged with hydrogen in this case and therefore, this observation indicated that carbide–matrix interfaces could act as crack initiation sites. The cracking in the case of iron aluminide intermetallics is cleavage-type and the cleavage fracture surfaces are characterized by the presence of river marks (Agarwal and Balasubramaniam 1996). These river marks lead to the origin of crack initiation (Agarwal *et al* 1996). SEM fractographic observation revealed that the river marks on the fracture surfaces, in several cases, originated from the carbide–matrix interfaces. In the hydrogen charged + baked specimens, the relatively weaker nature of the carbide–matrix interfaces in the presence of hydrogen was indicated. This could result due to irreversible trapping of hydrogen at these locations (Sen and Balasubramaniam 2001). For example, the fracture surface of Fe–28·1Al–2·1C after hydrogen charging + baking treatment (figure 3), revealed delamination at carbide–matrix interfaces. Notice, additionally, in figure 3 that the propagating crack (that resulted in fracture) has cleaved across some carbides and, in the process, has cracked them in the carbides seen in the figure. As delamination of the interfaces consumes energy, this could probably explain the relatively higher strengths and ductilities obtained generally after charging + baking treatment (table 1). The only exception to this general trend was for the Fe–27·5Al–3·7C tested in the rolling direction (table 1).

Table 1. Room temperature mechanical properties of baked and hydrogen charged + baked carbon-alloyed intermetallics. *R* denotes specimens with their gauge lengths parallel to rolling direction while *T* denotes specimens with their gauge lengths perpendicular to the rolling direction.

Specimen	Condition	U.T.S. (MPa)	Elongation (%)
Fe–28·1Al–2·1C	*R*, baked	402	2·12
	R, charged + baked	427	2·93
	T, baked	400	2·23
	T, charged + baked	409	3·03
Fe–27·5Al–3·7C	*R*, baked	573	3·60
	R, charged + baked	531	3·44
	T, baked	400	2·53
	T, charged + baked	488	3·04

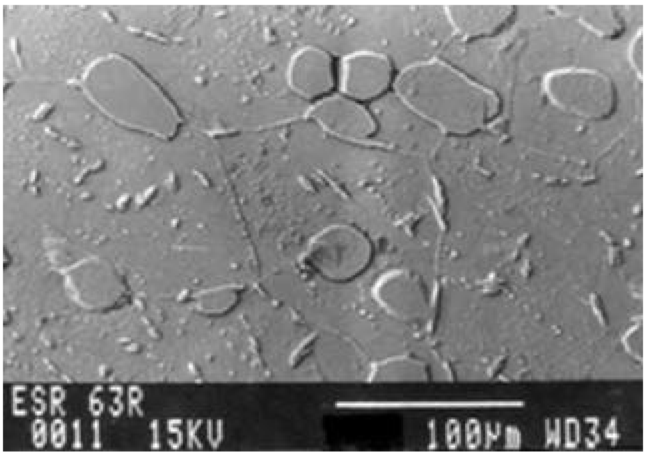

Figure 1. Microstructure of Fe–28·1Al–2·1C showing bulky $Fe_3AlC_{0·5}$ precipitates.

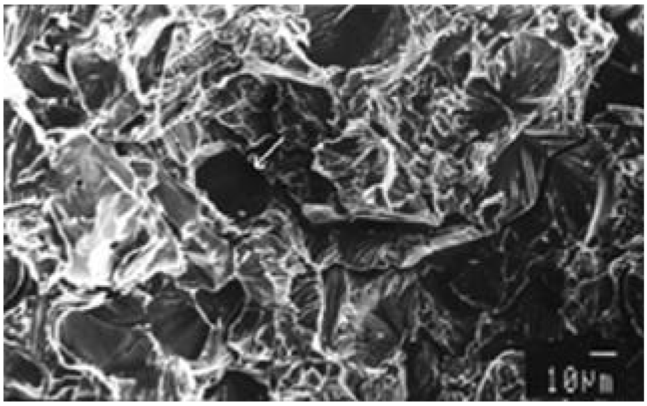

Figure 2. Fracture surface of tensile specimen of Fe–28·1Al–2·1C. Notice the cleavage-type brittle fracture surface with river line markings. A large secondary crack originating from a carbide–matrix interface can be distinguished.

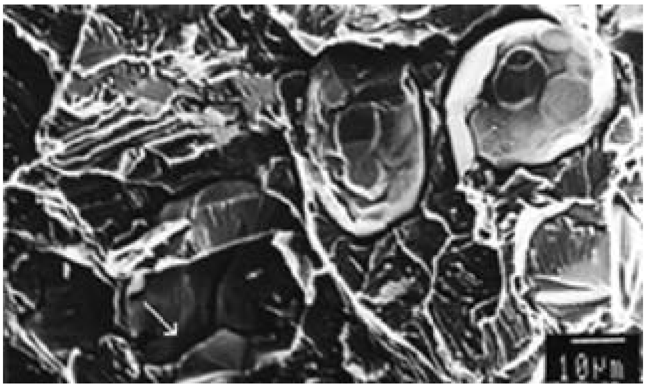

Figure 3. Fracture surface of tensile specimen of Fe–28·1Al–2·1C after hydrogen charging + baking treatment. Notice extensive delamination of carbide–matrix interfaces. The propagating crack, that resulted in the fracture surface, has cleaved across some carbides (arrow).

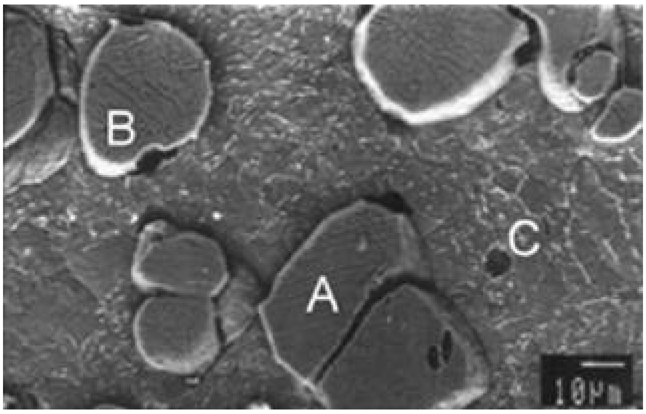

Figure 4. SEM micrograph of surface of Fe–28·1Al–2·1C after cathodic hydrogen charging at the current density of 10 mA/cm^2 for a total period of 12 h in 0·05 mol/l H_2SO_4 solution. Notice some cracked carbide particles (arrowed A), pores at the carbide–matrix interfaces (arrowed B) and pores in the matrix (arrowed C).

The following observations were recorded in the hydrogen-charged metallographic samples. There were several carbide precipitates that were cracked, with the cracks running through them (marked A in figure 4). It is difficult to attribute solely the cracking of the carbides to hydrogen charging because some cracked carbides were also observed in the as-received materials (figure 1, see also Sen *et al* 2000). However, the following observations were unique to the hydrogen charged samples. First, pores could also be identified at some locations in the matrix (marked B in figure 4). Secondly, there were several carbide particles where pores could be identified at the carbide–matrix interfaces (marked C in figure 4). The cracking of the carbides and the formation of pores at carbide–matrix interfaces may be related to the interaction of hydrogen with the carbides. The formation of pores in the matrix locations could be due to local hydrogen accumulation at defects and grain boundaries, leading to hydrogen recombination and the likely formation of hydrogen bubbles, as has been recorded on fracture surfaces of hydrogen embrittled materials (Engel and Klingele 1981). In order to accelerate and understand the interaction of hydrogen with the carbon-alloyed iron aluminides, it is important to investigate the effect of hydrogen at higher (200°C to 1000°C) temperatures.

4. Conclusions

The effect of hydrogen on the mechanical behaviour of two carbon-alloyed iron aluminides has been studied. The alloys exhibited low ductilities both in the baked and hydrogen charged + baked conditions. The alloys exhibited brittle cleavage-type fracture. Weakening of the carbide–matrix interfaces was observed in the presence of hydrogen. The effect of cathodic hydrogen charging on the microstructure has also been studied.

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