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Effect of hydrogen on room-temperature plasticity of B2 iron aluminides

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ABSTRACT

Different sensitivities to hydrogen embrittlement for the disordered and ordered B2 FeAl alloys have been investigated. The plasticity of annealed alloys (before hydrogenating) has been found to decrease with increasing the long-range order parameter. An intrinsic weakness of FeAl phase grain boundaries revealed at a high degree of ordering. The results of bending tests on specimens annealed and cathodically charged with hydrogen have shown that the influence of hydrogen on the plasticity diminishes with the increase in the degree of FeAl phase ordering.

1. Introduction

Iron aluminides, like many other ordered intermetallic alloys containing reactive elements (Al, Si, V, Ti) show severe environmental embrittlement when tested in moist air at ambient temperatures [1]. Aluminium atoms in iron aluminides react with moisture in the air, resulting in the generation of atomic hydrogen. Atomic hydrogen that penetrates into crack tips and causes brittle cleavage crack propagation was suggested as the chemical species responsible for premature failure [2]. Zhu et al. [3] utilized a laser desorption mass spectrometric method to detect the presence of hydrogen after contact of Fe–36 at.% Al alloy with water.

Moisture-induced environmental embrittlement is similar to the hydrogen embrittlement, except that here hydrogen is generated by moisture in the air, rather than by hydrogen gas or cathodically charged hydrogen [4]. The environmental embrittlement is not the only reason for a poor ductility and brittle fracture in FeAl alloys. The intrinsic grain-boundary weakness [5] and a high concentration of vacancies [6,7] are quoted among other reasons for embrittlement.

The amount of hydrogen penetrating into the iron aluminide depends on the state of the protective alumina surface layer, on the time of its exposure to the environmental interaction and on the hydrogen diffusion rate [8–10]. The environmental embrittlement is the problem that needs to be solved for practical application of iron aluminides. Alloy design efforts indicate that the embrittlement can be alleviated or reduced by the control of surface conditions, control of the grain structure, control of the alloy composition and technological conditions [4,11]. However, the mechanism involving the effect of atomic order on the internal and hydrogen embrittlement of B2 FeAl phase is still not well understood.

In the present studies an attempt has been made to determine the influence of hydrogen and degree of atomic order on the plasticity of binary B2 FeAl alloys at room temperature.

2. Experimental procedure

The chemical compositions and average grain size of tested B2 FeAl alloys are listed in Table 1. The ingots 6 mm in diameter and 70 mm long were obtained by induction melting in vacuum. The ingots were homogenized at 1273 K for 72 h in argon atmosphere followed by furnace cooling to room temperature at a cooling rate of 0.005 K s^{-1}. After furnace cooling, the vacancy concentration is relatively low [12]. Microstructure was observed using a light microscope NEOPHOT-2 and the grain sizes were measured by the linear intercept method.

The classical electrochemical method of Devanathan and Stachurski described in Ref. [13] was used to saturate the alloy with hydrogen. In this method, a specimen is placed between two independent electrochemical cells. Hydrogen is introduced into the specimen on the entry side, diffuses through the sample and finally escapes from its exit side, where it is immediately oxidised under potentiostatic conditions. The block diagram of the measuring apparatus connections is presented in Fig. 1. The specimen surface was cleaned by grinding and polishing. The specimen was then mounted in a Teflon fixture between two electrolysers. In the cell

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Table 1
Alloys composition and the average grain size.

Alloy	Elements content (wt%)								D (μm)
	Al	Si	Mn	C	S	P	Fe	O (ppm)	
Fe37Al	21.95	0.11	0.019	0.013	0.008	0.019	77.68	117	430 ± 47
Fe40Al	24.12	0.19	0.015	0.046	0.006	0.055	75.42	70	389 ± 39
Fe43Al	26.57	0.21	0.034	0.037	0.005	0.025	73.13	43	340 ± 28
Fe46Al	29.06	0.15	0.029	0.014	0.003	0.022	70.59	56	320 ± 30
Fe50Al	32.21	0.03	0.026	0.018	0.005	0.016	67.29	37	312 ± 10

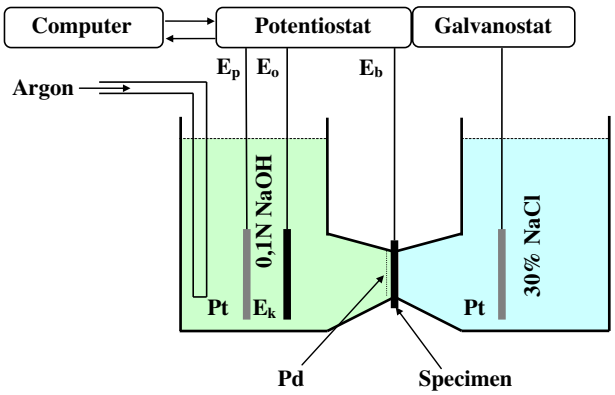

Fig. 1. The diagram of apparatus for hydrogen charging: E_b – investigated electrode (specimen); E_o – reference electrode; E_p – auxiliary electrode; E_k – calomel electrode; Pt – platinum grid; Pd – an output side of specimen covered with palladium.

on the entry side of a specimen there was a NaCl water solution educing hydrogen. During hydrogen charging, the entry side of the specimen was the cathode, while a platinum electrode was the anode. On the exit side, specimen's surface covered with palladium was in constant contact with the 0.1 N NaOH solution and was the anode, while a platinum electrode – the cathode. Saturated calomel electrode was the reference electrode, which through a potentiostat maintained specimen's exit side on a constant potential of +50 mV. As a result, hydrogen penetrating through the specimen was oxidised, $H - e \Rightarrow H^+$. Hydrogen will penetrate into the FeAl alloy when its surface layer of alumina is destroyed. This can be achieved by a proper choice of the electrolyte that will chemically destroy the layer at the input surface of the specimen, but it will not or only weekly interact with the specimen itself. After some experiments [10], the water 30 wt% NaCl solution was chosen. The cathode charging with hydrogen was carried out at room temperature by applying a cathodic current density of 0.013 A/m^2 at the specimen entry side. The hydrogen concentration in specimens was determined on three specimens (for each alloy composition) by means of a STRÖHLEIN INSTRUMENTS H-MAT 2500 hydrogen analyzer of 0.01 wt ppm sensitivity.

Bending specimens were 4 mm in diameter and 26 mm long. Three-point bending tests on non-charged and hydrogen-charged specimens were carried out using an INSTRON 1195 testing machine in high purity argon atmosphere. The cross-head speed was 0.1 mm/min. Three specimens were tested per each alloy composition. All fractured specimens were examined by a scanning electron microscope (SEM).

In order to measure the long-range order parameter, S, X-ray diffraction patterns were obtained using a PHILIPS diffractometer. The spectra were obtained using Cu Kα radiation of a wavelength of 1.54178 Å for the angle range $2\Theta = 20–120°$ with the steps of 0.02°/4 s.

The long-range order parameter S was estimated on the basis of the equations described in paper [14].

Dimensionless vacancy quantity parameter, W, was determined using the Doppler broadening technique applied in paper [15]. In these measurements the momentum component p_2 of the positron–electron pair annihilation in the direction of the semiconductor detector is measured. Positrons get trapped by vacancies because of their negative charge in reference to the bulk lattice. The first approximation of annihilation peak shape is Gaussian and vacancy trapped positrons contribute to the central part of the peak. The W parameter reflects the fractions of positrons annihilating with electrons of low momentum. An increase in the W parameter is an indication of positron trapping in open volume defects, which is a measure of the vacancy amount. The numerical analysis of the Doppler broadening spectra was performed and the statistical error determined $\Delta W = \pm 0.002$, at the ratio of counts 10^6 for every positron annihilation curve.

3. Results and discussion

Measurements of an average grain size revealed a decreasing grain size with the increase in aluminium content in the alloys (Table 1). A decrease in the average grain size with increasing aluminium content in B2 FeAl alloys was observed also in paper [16].

The results of hydrogen content determination depending on the alloy composition, after cathode saturating, are given in Fig. 2. Yan and Weng explain that the real hydrogen concentration measured in pre-charged specimen comprised the difference of the hydrogen content in samples and the quantity determined for the blank sample [17].

Fig. 2 presents, for comparison, the hydrogen concentrations before hydrogen charging. The measured values of hydrogen

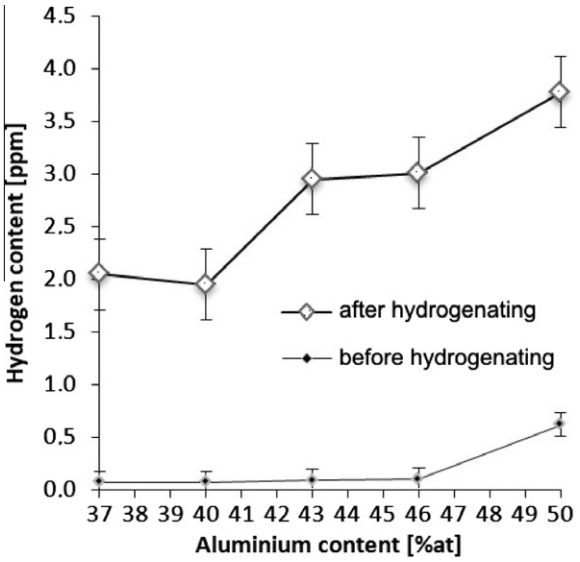

Fig. 2. Hydrogen content in B2 FeAl phase vs. aluminium content for specimens non-charged (annealed) and hydrogen charged for 10 h.