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Journal of Alloys and Compounds 389 (2005) 198–203

Journal of ALLOYS AND COMPOUNDS

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Hydrogen permeation behavior in a Fe_3Al-based alloy at high temperature

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Received 13 February 2004; received in revised form 29 March 2004; accepted 28 May 2004

Abstract

The hydrogen permeation behavior in a Fe_3Al-based alloy, with three types of heat treatments, was investigated by an ultrahigh vacuum gaseous permeation technique at the temperature range of 240–320 °C. A well-defined second stage is observed in hydrogen permeation curves of the Fe_3Al alloy superimposed on the basic curves. On the basis of optical microstructures, determined permeabilities and diffusivities, corresponding activation energies, it is suggested that the two sigmoidal curves result from the ordered B_2 phase and disordered α-Fe phase in the Fe_3Al alloy; the first stage of permeation reflects the hydrogen transport in matrix disordered α-Fe phase, while the second stage reflects the hydrogen transport in ordered B_2 phase.

Keywords: Iron aluminides; Hydrogen diffusivity; Activation energy; Gaseous permeation technique

1. Introduction

Iron aluminides, based on Fe_3Al, have long been stimulating the interests of material scientists because of their excellent oxidation resistance and relatively low cost and density. However, limited ductility at ambient temperature is the major factor to restrict their commercial applications. Liu et al. [1] declared that Fe_3Al is intrinsically quite ductile. The low-ductility observed in air was attributed to environmental embrittlement involving hydrogen generated from the reaction of aluminum atoms with water vapor in air, entered the metal and embrittled the crack-tip region. Wan et al. [2] found in AES that a saturation value of water is reached after an exposure of 10^{-4} Pa s for Fe_3Al alloy. Therefore, the hydrogen transport may be one of the important factors to control environmental embrittlement.

However, there are relatively few investigations [3–10] to determine the diffusivity of hydrogen in Fe–Al alloys because of a strong tendency to form protective oxides, which impede hydrogen permeation. These investigations mainly indirectly measured the diffusivity of hydrogen in Fe–Al alloy and these data of hydrogen diffusivity were very dispersing. Hosada et al. [4] estimated the diffusivity of hydrogen in two binary iron aluminides based on hardness increase after exposing the alloys to different environments. It was shown that diffusivity in Fe–18Al was 10^{-11} m^2/s while it was 10^{-12} m^2/s in Fe–25Al. Kasul and Heldt [5] showed the reversibility of hydrogen embrittlement by estimating the ductility after baking a hydrogen-charged Fe–35Al polycrystal. The rate of ductility recovery provided the room temperature diffusivity of hydrogen in this intermetallics as 4×10^{-16} m^2/s. Yang and Hanada [6] studied hydrogen absorption in ordered Fe–40Al and estimated the hydrogen diffusivity at room temperature to be 4×10^{-13} m^2/s. The higher diffusivity measured in absorption compared to that in desorption may suggest differences in the diffusion mechanism between absorption and desorption [6]. Based on the three-dimensional distribution of hydrogen reported by Zhu et al. [9] for Fe–36Al, the diffusivity of hydrogen was estimated to be 2.4×10^{-15} m^2/s [7]. In a study of the environment-sensitive cracking of Fe–28Al–5.0Cr–0.5Nb–0.5Mo–0.2C–0.2B–0.1Zr, Chiu et

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doi:10.1016/j.jallcom.2004.05.080

Table 1
Heat treatment of the sheets

No.	Heat treatment	Grain shape
F11	700 °C/1 h, OQ	Elongated grain
F12	900 °C/1 h, FC +700 °C/1 h, OQ	Equiaxed grain
F13	1000 °C/1 h, FC +700 °C/1 h, OQ	Equiaxed grain

al. [8] determined the room temperature diffusivity of hydrogen in the intermetallics by the time-lag technique to be 1.6×10^{-9} m^2/s. They concluded that the reported value of hydrogen diffusivity was reasonable as it was of the same order of magnitude as that for hydrogen diffusivity in bcc metals like α-Fe, V and Nb [10]. In a word, it has been pointed out the fact that hydrogen diffusivity is lowered with increasing Al content in Fe–Al alloys and the hydrogen diffusivity is higher in Fe_3Al-based intermetallics than in FeAl-based intermetallics [11,12]. However, the attribution of retardation effect on hydrogen transport in Fe–Al alloys is still obscure. Especially, the activation energy of hydrogen diffusion in Fe–Al alloy is absent. Therefore, we will use a situ coating technique to rule out the barrier effect of oxide and investigate the hydrogen permeation in a Fe_3Al-based alloy at the temperature range of 240–320 °C using an ultrahigh gaseous permeation technique in this paper.

2. Experimental procedure

A Fe_3Al-based alloy (chemical composition is Fe–27.78Al–4.73Cr, at.%) were produced by vacuum melting in an induction furnace using commercial pure aluminum, iron and chromium, and subsequently cast into 20 mm × 50 mm × 200 mm ingot. After homogenizing at 1150 ° for 5 h, the ingot was then enveloped in stainless steel, hot rolled at 800–1000 °C to 3.4 mm thickness, and warm rolled at 600–650 °C to 1 mm thickness. The 1 mm thickness sheets were heat treated as in Table 1. The microstructure of the heat-treated samples was examined using optical microscopy. 2θ-scans, using Cu Kα radiation, were carried out to determine whether ordering occurred after heat treatment.

Disc specimens with diameter of 19.95 ± 0.05 mm were cut using electro-discharged machine from the heat-treated sheets and sliced up with a thickness of about 0.5 mm. Each surface of the specimens was polished with abrasive paper to No. 5 metallographic emery (the final thickness of each specimen is denoted in Figs. 4–6), rinsed carefully in alcohol and ultrasonically cleaned in acetone. After being dried using flowing air at room temperature, both surfaces of the specimens were coated in ANELVA SPC-350 multi-target magnetron sputtering system. The detailed method is described in previous work [13]. Oxygen was not detected at the interface between the Pd coat and the base alloy by Auger electron spectroscopy (AES) [13]. It is shown that no oxidation film exists at the interface between Pd and Fe_3Al alloy.

After coating, the specimen was mounted in the ultra-high vacuum gaseous hydrogen permeation apparatus. The apparatus and operation procedure have been described in detail elsewhere [14–16]. Measurements were made from temperature 240 to 320 °C with an applied hydrogen pressure of 1.01×10^5 Pa on the entrance side. Specimen temperature during the test was controlled to ±1 °C. Until steady state conditions were achieved, the diffusion rate of hydrogen through the sample (hydrogen flux) was recorded as a function of time using an X–Y recorder. Then, the hydrogen gas on the entrance side was outgassed and the hydrogen flux continued to be recorded. On completion of the degassing procedure, next higher temperature was applied and same procedure was repeated.

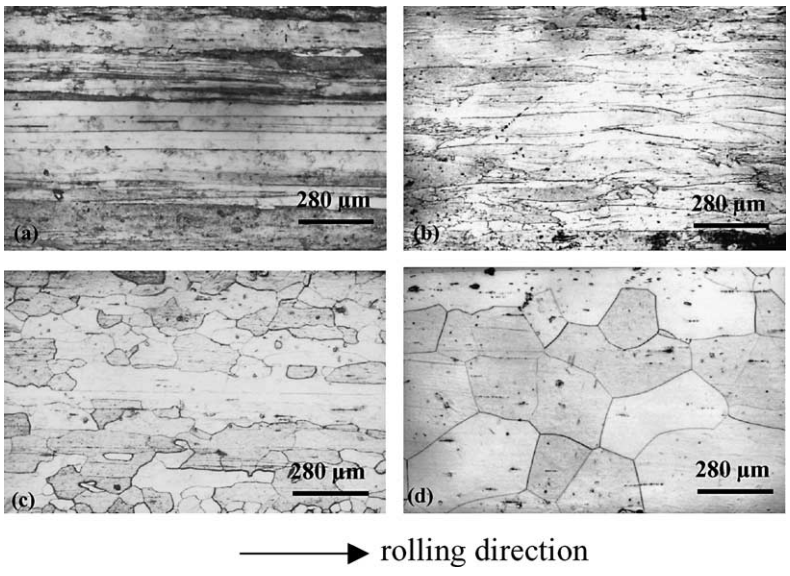

Fig. 1. Optical microstructure (a) before heat treatment; (b) F11; (c) F12; and (d) F13.